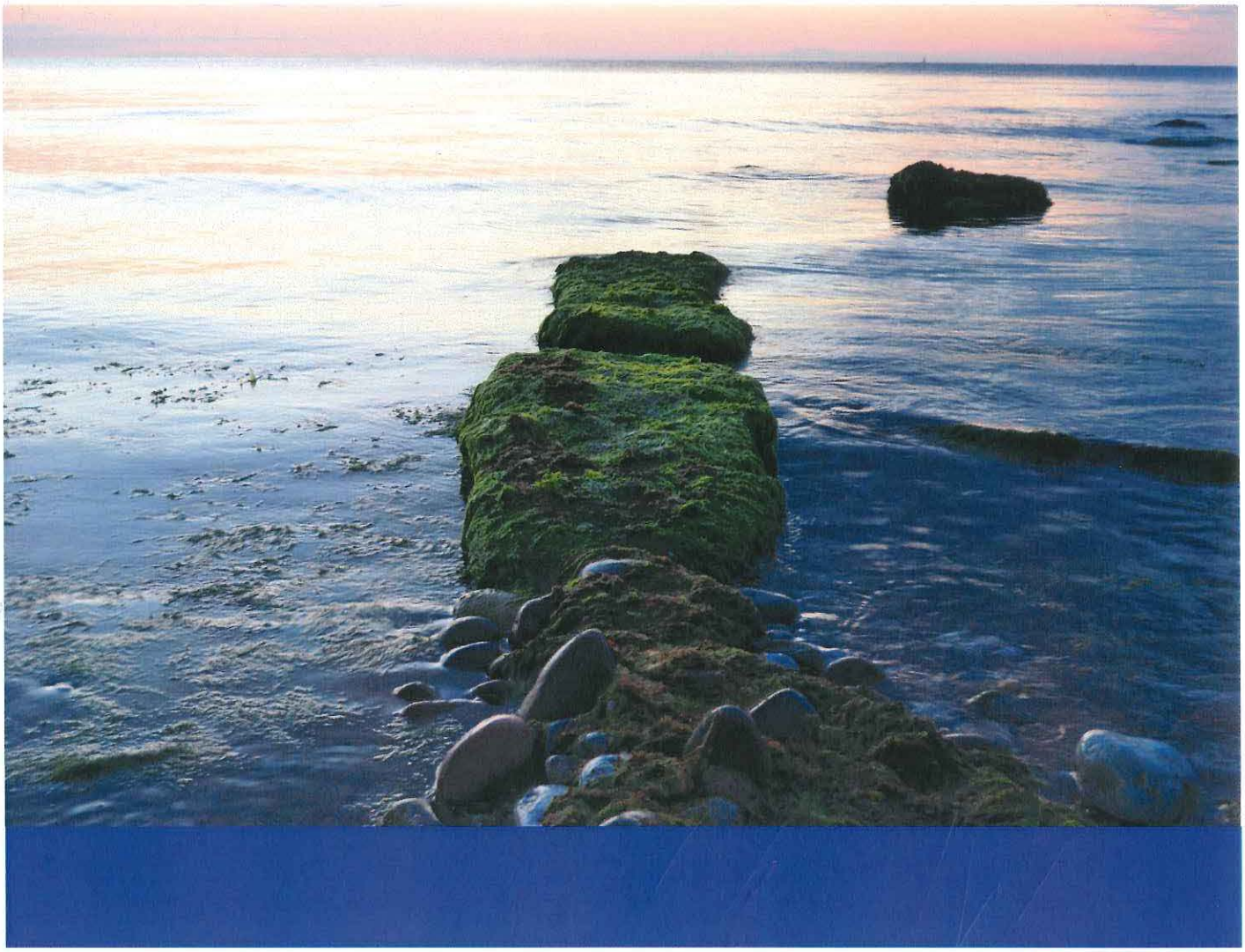

WORKPLAN TO COMPLETE SITE CHARACTERIZATION AND ADDRESS OUTSTANDING DERA ISSUES BINDER 2 OF 3

BINDER 2: QAPP PART 1

Appendix B – Quality Assurance Project Plan

APPENDIX B – QUALITY ASSURANCE PROJECT PLAN (QAPP)

Revision 2

3/28/2014

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1 Title/Signature Page

QUALITY ASSURANCE PROJECT PLAN
FOR THE RCRA PHASE II
QUEMETCO, INC., INDIANAPOLIS, INDIANA

U.S. EPA IDENTIFICATION NUMBER: IND 000 199 653

REVISION 2

March 28, 2014

Prepared by: WSP

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2 Table of Contents

Section# - Page#

1 Title/Signature Page 1-1

2 Table of Contents 2-1

3 Project Description 3-1
3.1 Introduction 3-1
3.1.1 Overall Project Objectives and Decision Statements 3-1
3.1.3 Project Status 3-4
3.1.4 QAPP Preparation Guidelines 3-4
3.2 Facility Description 3-5
3.2.1 Location 3-5
3.2.2 Facility Size and Borders and Surrounding Land Use 3-5
3.2.3 Natural and Manmade Features 3-5
3.2.4 Topography 3-6
3.2.5 Geology and Hydrogeology 3-6
3.2.6 Ecological Communities and Habitats 3-8
3.3 Facility History 3-8
3.3.1 General History 3-8
3.3.2 Past Data Collection Activities 3-9
3.3.3 Current Status 3-11
3.4 Project Objectives and Intended Data Usages 3-12
3.4.1 Decision Rules 3-13
3.4.2 Chemical Analysis Project Target Parameters 3-14
3.4.3 Ecology-Biology-Toxicology Analysis 3-18
3.5 Sampling Locations 3-19
3.5.1 Rationale of Selected Sampling Locations 3-20
3.6 Project Schedule 3-20
3.7 Task Bar Chart and Associated Time Frames 3-20

4 Project Organization and Responsibility 4-1
4.1 Project Organization Chart 4-1
4.2 Management Responsibilities 4-3
4.2.1 U.S. EPA Project Manager 4-3
4.2.2 Quemetco Project Coordinator 4-3
4.2.3 WSP Project Director 4-3
4.3 Quality Assurance Responsibilities 4-3
4.3.1 WSP Project Quality Assurance Officer 4-3
4.3.2 Third-Party Data Validator 4-4
4.3.3 Ecotoxicology Expert 4-4
4.4 Laboratory Responsibilities 4-4
4.4.1 Pace Analytical Services, Inc. 4-7

4.4.2 Northwestern Aquatic Sciences 4-10
4.4.3 RTI Laboratories 4-11
4.4.4 CQM, Inc. Laboratories 4-13
4.5 Field Responsibilities 4-14
4.5.1 WSP Field Leader 4-14
4.5.2 WSP Field Technical Staff 4-14
4.6 Special Training Requirements and Certifications 4-15
4.6.1 Training 4-15
4.6.2 Certifications 4-15

5 Quality Assurance Objectives for Measurement Data 5-1
5.1 QA Objectives for Site Characterization 5-1
5.1.1 Precision 5-1
5.1.2 Accuracy 5-3
5.2 QA Objectives for DERA Sampling 5-4
5.2.1 Precision 5-4
5.2.2 Accuracy 5-6
5.3 Completeness 5-7
5.3.1 Definition 5-7
5.3.2 Field Completeness Objectives 7
5.3.3 Laboratory Completeness Objectives 7
5.4 Representativeness 5-7
5.4.1 Definition 5-7
5.4.2 Measures to Ensure Representativeness of Field Data 5-7
5.4.3 Measures to Ensure Representativeness of Laboratory Data 5-8
5.5 Decision Rules 5-8
5.5.1 Definition 5-8
5.5.2 Decision Rule Objectives 5-8
5.6 Comparability 5-9
5.6.1 Definition 5-9
5.6.2 Measures to Ensure Comparability of Field Data 5-9
5.6.3 Measures to Ensure Comparability of Laboratory Data 5-9
5.7 Level of Quality Control Effort 5-9

6 Sampling Procedures 6-1

7 Custody Procedures 7-1
7.1 Field Custody Procedures 7-1
7.1.1 Field Notebook 7-1
7.1.2 Sample Collection Data 7-2
7.1.3 Custody of Samples 7-2
7.2 Laboratory Custody Procedures 7-3
7.3 Final Evidence Files 7-3

8 Calibration Procedures and Frequency 8-1

8.1 Field Instrument Calibration 8-1
8.2 Laboratory Instrument Calibration 8-1
8.2.1 Pace Analytical 8-1
8.2.2 Northwestern Aquatic Sciences 8-3
8.2.3 RTI 8-4
8.2.4 CQM 8-4

9 Analytical Procedures 9-1
9.1 Field Analytical Procedures 9-1
9.2 Laboratory Analytical Procedures 9-2
9.2.1 Project Target Compounds and Detection Limits 9-2
9.2.2 List of Associated Quality Control Samples 9-2

10 Internal Quality Control Checks 10-1
10.1 Field Quality Control Checks 10-1
10.2 Laboratory Quality Control Checks 10-1

11 Data Reduction, Validation and Reporting 11-1
11.1 Data Reduction 11-1
11.1.1 Field Data Reduction Procedures 11-1
11.1.2 Laboratory Data Reduction Procedures 11-1
11.2 Data Validation 11-1
11.2.1 Procedures Used to Validate Field Data 11-1
11.2.2 Procedures Used to Validate Laboratory Data 11-1
11.3 Data Reporting 11-2
11.3.1 Field Data Reporting 11-2
11.3.2 Laboratory Data Reporting 11-2
11.3.3 Data Acquisition Requirements and Data Quality Management 11-3

12 Performance and Systems Audits and Frequency 12-1
12.1 Field Performance and Systems Audits 12-1
12.1.1 Internal Field Audits 12-1
12.1.2 External Field Audits 12-1
12.2 Laboratory Performance and Systems Audits 12-2
12.2.1 Internal Laboratory Audits 12-2
12.2.2 External Laboratory Audits 12-2

13 Preventative Maintenance 13-1
13.1 Field Instrument Preventative Maintenance 13-1
13.2 Laboratory Instrument Preventative Maintenance 1
13.3 Inspection/Acceptance Requirements of Supplies and Consumables 13-1

14 Specific Routine Procedures Used to Evaluate Data Precision, Accuracy and Completeness 14-1
14.1 Accuracy Assessment 14-1
14.1.1 Accuracy for Chemical Analysis 14-1

14.1.2 Accuracy for Ecological-Biological-Toxicology Analysis 14-1
14.2 Precision Assessment 14-1
14.2.1 Precision for Chemical Analysis 14-1
14.2.2 Precision for Ecological-Biological-Toxicology Analysis 14-2
14.3 Completeness Assessment 14-2
14.3.1 Completeness for Chemical Analysis 14-2
14.3.2 Completeness for Ecological-Biological-Toxicology Analysis 14-2
14.4 Assessment of Data 14-2

15 Corrective Action 15-1
15.1 Field Corrective Action 15-1
15.2 Laboratory Corrective Action 15-2
15.3 Corrective Action During Data Validation and Data Assessment 15-2

16 Quality Assurance Reports to Management 16-1
16.1 Contents of Project Quality Assurance Reports 16-1
16.2 Frequency of Quality Assurance Reports 16-1
16.3 Individuals Receiving/Reviewing Quality Assurance Reports 16-1

17 References 17-1

List of Figures

Figure B1 – Site Location
Figure B2 – Proposed DERA Sample Locations
Figure B3 – Proposed XRF Sampling Grid
Figure B4 – Proposed Vertical Soil Sampling Locations
Figure B5 – Project Organization Chart

List of Tables

Table B1 – Objectives for Field and Laboratory Data Collection ..... Section 3, page 2
Table B2 – Field Parameters ..... Section 3, page 16
Table B3 – Laboratory Parameters ..... Section 3, page 17
Table B4 – Summary of Sample Analysis and Assigned Laboratories .. Section 4, page 5
Table B5 – Site Characterization Precision Control Limits ..... Section 5, page 2
Table B6 – DERA Precision Control Limits ..... Section 5, page 4
Table B7 – Summary of QA/QC Sample Collection and Analysis ..... Section 5, page 11
Table B8 – Project Sampling Procedures ..... Section 6, page 1
Table B9 – Summary of Field Analytical Procedures ..... Section 9, page 1

List of Attachments

Attachment A – Statement of Qualifications of Key Personnel
Attachment B – WSP Standard Operating Procedures
Attachment C – Laboratory Standard Operating Procedures
Attachment D – Laboratory Certifications
Attachment E – Example Chain-of-Custody Form
Attachment F – Manufacturer's Instruction Manuals

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3 Project Description

This Quality Assurance Project Plan (QAPP) presents the organization, objectives, planned activities, and specific quality assurance/quality control (QA/QC) procedures associated with the Phase II Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) for the Quemetco, Inc., facility (Site) located in Indianapolis, Marion County, Indiana, and in response to comments and supplemental information requested by U.S. Environmental Protection Agency (EPA), Region 5 regarding a Human Health Risk Assessment (HHRA) and Ecological Risk Assessment (ERA) for the Site submitted on October 19, 2006. The most recent comments from U.S. EPA are dated July 12, 2013.

Specific rationale for sample collection, protocols for sampling, sample handling and storage, chain-of-custody, and laboratory and field analyses are described in this QAPP. All QA/QC procedures will be structured in accordance with applicable technical standards and U.S. EPA's requirements, regulations, and guidance. This QAPP has been prepared in accordance with the U.S. EPA Region 5 QAPP policy as presented in U.S. EPA RCRA QAPP Instructions, April 1998, and other relevant guidance documents, including previous environmental reports, ASTM standards, individual laboratory information, and operating procedures.

3.1 Introduction

This QAPP has been prepared by WSP on behalf of Quemetco, Inc., a subsidiary of RSR Corporation. This QAPP, a Sampling and Analysis Plan (SAP), and a Health and Safety Plan (HASP) are appended to the Workplan to Complete Soil Characterization and Address Outstanding Detailed Ecological Risk Assessment (DERA) Issues (Workplan), Quemetco, Inc., Indianapolis, Indiana, dated March 28, 2014.

3.1.1 Overall Project Objectives and Decision Statements

The purpose of the Workplan and this QAPP is to satisfy two objectives as requested by the U.S. EPA in their October 8, 2009, comments to WSP's May 19, 2008, Workplan Address Outstanding DERA Issues (WSP Environment & Energy 2008). These conditions include:

1) to satisfactorily address the U.S. EPA's specific comments regarding the Ecological Investigation and Quality Assurance procedures presented in WSP's May 19, 2008, workplan; and

2) to further delineate the nature and extent of lead contamination across the Site.

The X-Ray Fluorescence (XRF) investigation, which followed the strategy presented by U.S. EPA in their October 8, 2009, comment letter, delineated the lateral distribution of lead in surficial and shallow soils across a large portion of the Site surrounding the manufacturing facility, including the property east of the Site belonging to Carrier Corporation.

The objectives of the revised Workplan will be achieved through the collection and analysis of additional samples taken both on and offsite from various environmental media including soil, sediments, surface water, benthic invertebrates, and groundwater. The data will be used to evaluate the lateral and vertical extent of lead contamination and the potential migration pathways for lead and other Site-related constituents identified as potential risks in the 2006 DERA (Cantox Environmental, October 19, 2006, Executive Summary, page vi). These other Site-related constituents include: antimony, arsenic, barium,

cadmium, chromium, nickel, zinc, and mercury. A summary of the proposed sampling and analysis for this investigation is presented in Table B4.

The Decision Statements for this investigation are as follows:

- In order to further delineate lateral extent of lead contamination, additional soil samples will be analyzed using XRF. Results from the XRF analysis will be used to determine if additional step-out samples will be analyzed
- In order to further characterize the vertical extent of lead contamination, soil samples will be collected from specific locations using Geoprobe drilling equipment. Results of the vertical soil sampling will be included with previous investigations and will be used to determine whether additional sampling or corrective actions are required at the Site.
- Soil, surface water, benthic invertebrate, and sediment samples will be collected from specific locations throughout the Site and used for ecological-biological-toxicology testing. The chemical of concern is predominately lead; however, this investigation will also address how other chemicals of concern have impacted the Site. The results of the ecological-biological-toxicology analyses will be used to update the 2006 ERA. Laboratory results will be used to compare potential adverse effects between upstream and downstream locations and within Pond Nos. 1, 2 and 3. The results of the addendum to the DERA will determine the Corrective Actions at the Site.
- At the request of U.S. EPA, groundwater samples will be collected from existing monitoring wells to assess the current groundwater conditions at the Site. Results of the groundwater sampling will be used to determine if additional groundwater monitoring is required.

The specific sampling tasks and objectives to be performed in the generation of field and laboratory data and the specific decision statement are provided as Table B1 below. The target action levels for each parameter are presented in Tables B2 and B3.

Table B1—Objectives for Field and Laboratory Data Collection

Sampling Task	Objective and Decision Statement
XRF soil sampling with XRF machine and hand trowel	Collect soil samples from 0 to 1 in bgs from the north and northeast area of the Site for analysis of lead in the field to further characterize the Site. ▪ If the XRF result is greater than 400 mg/kg than we will step out to the next grid and collect another sample. ▪ If the XRF result is equal to 400 mg/kg than we have defined the lateral extent of lead contamination. ▪ If the XRF result is less than 400 mg/kg than we have defined the lateral extent of lead contamination.

Table B1 (cont.)—Objectives for Field and Laboratory Data Collection	
Sampling Task	**Objective and Decision Statement**
XRF verification soil sampling with hand trowel	Collect soil samples from 1 to 6 in bgs in alternate grid squares from the north and northeast area of the Site for analysis of lead in the laboratory. Correlation between XRF sampling and laboratory data will be accomplished by calculating the relative percent difference (RPD) between the two results (XRF and laboratory data). No pass/fail criteria will be established for this portion of the project. Use of the newly generated XRF data is as a screening tool to broadly delineate the lead contamination.
Vertical soil sampling with Geopropbe drill rig	Collect subsurface soil samples from specific locations at the Site to further characterize vertical extent of lead. Results will be included with previous vertical soil samples collected from the Site. The information will used to determine if additional sampling is required.
Groundwater sampling of existing monitoring wells onsite	Collect groundwater from existing wells to assess current groundwater conditions at the Site. Results will be used to determine if further actions are required.
Composite soil sampling from slag pile area, northeast side of plant and background locations with a hand trowel	Collect composite soil samples to be used for earthworm toxicity testing. Results of toxicity testing will be incorporated into the Effects Assessment of the updated DERA.
Soil sampling from the three onsite ponds and Julia Creek using a hand trowel	Collect soil samples from 0 to 6 in bgs to be used as supporting data for earthworm toxicity testing. Results will be used to supplement the toxicity testing and evaluate statistically significant affects between the Ponds and upstream and downstream of Julia Creek.
Surface water sampling from the Pond #3 and Julia Creek using laboratory-supplied containers	Collect surface water samples for chemistry and toxicology testing using Fathead minnow (*P. promelus*) and water flea (*C. dubia*) Results of toxicity testing will be incorporated into the Effects Assessment of the updated DERA.
Sediment sampling from Julia Creek and the three ponds using hand trowels, Ponar or Eckman	Collect sediment samples for chemistry, physical characterization and 28-day *H. azteca* and 10-day *C. dilutes* toxicity testing. Results will be used for risk analysis associated with the DERA.
Benthic invertebrate sampling in Julia Creek with kick screens	Collect benthic invertebrates for tissue analysis. Results will be used in the food chain model for the Indiana Bat and Spotted Sandpiper.

3.1.3 Project Status

In February 1989, Quemetco entered into a Consent Decree with U.S. EPA Region 5, which required the investigation and remediation of the blast furnace slag pile and adjacent ponds. In accordance with the Consent Decree, Quemetco initiated an RFI and has implemented several of the various investigation requirements.

On October 19, 2006, a revised HHRA and ERA for the referenced Site were submitted by WSP Environmental Strategies LLC (now WSP) to the U.S. EPA Region 5. The U.S. EPA responded to the risk assessments with comments in a May 29, 2007, letter to RSR Corporation. Supplemental information was provided on August 10, and October 11, 2007. The U.S. EPA responded on December 12, 2007, and WSP responded to the December 12, 2007, letter on January 11, 2008. Based on these comments and the letter from U.S. EPA, dated February 13, 2008, a workplan for additional sampling was completed on May 16, 2008 (WSP Environment & Energy 2008). On October 8, 2009, EPA issued comments on the workplan and the QA documents submitted on May 19, 2008.

In April and October 2010, and December 2011, an XRF soil characterization study was conducted as recommended by the U.S. EPA in their October 8, 2009, letter to Quemetco. The XRF investigation was conducted in accordance with the approved XRF Sampling and Analyses Plan, dated February 17, 2010. The final XRF investigation report was submitted to the U.S. EPA on March 23, 2012.

The following field sampling will be conducted at the Site and is further explained in the Workplan. Additionally, the activities below are presented in Table B4.

- Soil samples will be collected from various areas of Site and analyzed for lead and the Site-related constituents presented in Section 3.1.1 and Table B4.
- Soil samples will be collected for earthworm toxicity tests.
- Surface water samples will be collected from Pond No. 3 and Julia Creek for chemistry and toxicity testing using *P. promelas* and *C. dubia*.
- Sediment samples will be collected from all three ponds and Julia Creek for chemistry, physical characterization and toxicity tests. Sediment sample results will be used to assess the risk to the Spotted sandpiper and endangered species Indiana bat.
- Groundwater samples will be collected from 11 existing groundwater monitoring wells and analyzed for lead and Site-related constituents.
- Benthic invertebrates will be collected from Julia Creek and the Ponds for tissue analysis and inclusion in food chain modeling.

Analytical results from the XRF soil sampling will be used to determine whether additional sampling and/or corrective action should be conducted at the Site. Ecological-biological-toxicology data from these investigations will be used in the addendum to the DERA. A RFI Phase II Supplemental report presenting the additional data collected will be prepared and submitted to U.S. EPA Region 5 for review and approval.

3.1.4 QAPP Preparation Guidelines

This QAPP has been prepared in accordance with the U.S. EPA Region 5 RCRA QAPP Instructions (U.S. EPA 1998). Furthermore, WSP incorporated comments from U.S. EPA Region 5, dated October 8, 2009, and July 13, 2013, and telephone conversations in October 2011, April 2012, and February 2014.

3.2 Facility Description

Quemetco manufactures lead and lead alloys, and reclaims polypropylene from a variety of materials. The process materials used at the facility include spent automobile, truck, and industrial lead-acid batteries, factory scrap, coal fines, coke, hard rubber battery cases, pebble lime, sodium carbonate, red phosphorous, pyrite, sulfur, sodium nitrate, sodium hydroxide, tin, antimony, arsenic, and copper. Wastewater treatment materials used onsite include soda ash, ferric sulfate, hydrogen peroxide, sodium carbonate, sodium hydroxide, ferric chloride, and flocculants. The facility operates under the primary standard industrial classification code 3341, which is specific to establishments primarily engaged in secondary smelting and refining of nonferrous metals.

3.2.1 Location

Quemetco, Inc., a subsidiary of RSR Corporation, operates a secondary lead smelting facility located at 900 Quemetco Drive, in Indianapolis, Marion County, Indiana (Figure B1). The study area includes approximately 8.0 acres and encompasses the Northeast area, Slag Pile area, onsite Ponds Nos. 1, 2, and 3, and Julia Creek (Figure B2).

3.2.2 Facility Size and Borders and Surrounding Land Use

The Quemetco facility is 408 acres and is located in an area of mixed industrial, residential, and agriculture land use. The portion of the Site developed for industrial use is approximately 8.0 acres. The immediate area consists of undeveloped woodlands and land zoned for industrial use. Property zoned for residential use is located approximately 0.5 mile northeast, northwest, and southwest of the facility. To prevent future residential development, additional land surrounding the property has been purchased creating an approximately 400-acre buffer zone around the facility.

The facility is bordered to the north by a railroad line and the Power Transmission Company facility; to the northeast by West Park View residential area; to the south by Heritage Environmental, a hazardous waste treatment facility; to the east by farmland, a furnace and air conditioning manufacturing facility, and a freight carrier company; and to the west by the Pennsylvania Railroad, and farmland.

3.2.3 Natural and Manmade Features

Natural features of the facility include an approximately 400-acre undeveloped buffer zone owned by subsidiaries of the RSR Corporation. The buffer zone was created to prevent encroachment and minimize citizen complaints. Julia Creek lies adjacent to the southeast side of the facility's parking lot.

Manmade features of the facility include a concrete containment channel bordering the main processing areas. Two man-made ponds (Ponds 1 and 2) are east of the main parking area, and one man-made pond (Pond No. 3) is located west of the main parking area, adjacent to the blast furnace slag pile. The man-made ponds and their adjacent soil hills were excavated for the storage of slag wastes, but were never used.

3.2.4 Topography

Marion County is located in the southern portion of the Tipton Till Plain physiographic unit of Indiana. This region is characterized by relatively flat surface topography with few abrupt changes in elevation. The till plain is dissected by numerous perennial and seasonal water bodies including creeks, streams, and rivers.

3.2.5 Geology and Hydrogeology

3.2.5.1 Regional Geology

The geology of the Indianapolis area consists of unconsolidated Pleistocene glacial drift and alluvial sediments overlying Middle to Late Paleozoic sedimentary rocks. The following sections summarize the geology of the unconsolidated deposits and underlying bedrock.

The unconsolidated deposits range in thickness from approximately 10 to 400 feet over Marion County. Glacial drift is present over the entire area, whereas the alluvial deposits form a relatively thin veneer over the glacial drift in major stream valleys.

The majority of the glacial cover consists of unstratified drift or till, a poorly sorted aggregation of clay, silt, sand, and gravel sediments that was deposited during the last (Wisconsin) glacial episode. Thin lenses of moderate to well sorted silt, sand, and gravel sediments may be present within the finer grained till deposits. The till lithology, which appears to be fairly uniform over the area, is predominately silt and fine to medium sand with less than 20 percent clay. The stratigraphic boundary between the Wisconsin and older tills in the Indianapolis area is typically marked by a layer of hard, indurated gravelly clay.

Stratified glacial drift is also present in certain areas of Marion County. The most voluminous of these deposits consists of outwash sediments which originated from glacial melt water streams. These deposits consist of relatively well sorted sands and gravels and are typically found along the major stream valleys in the area (e.g., Eagle Creek, White River). Regional studies indicate that some sand and gravel layers within the till interfinger with these outwash deposits. Recent alluvial deposits typically form a thin surface cover over the outwash sediments. These fine- to coarse-grained sediments are the result of the erosion and redeposition of the older glaciogenic materials.

3.2.5.2 Site Geology

The following sections summarize the relevant data on subsurface conditions from previous work. Where Site-specific data are lacking, published information is used. Based on boring logs, the subsurface geology to a depth of approximately 50 feet consists of a silty loam soil underlain by weathered to unweathered, generally fine-grained till. These geologic conditions are consistent with the location of the Quemetco facility in the till plain. Sand and gravel layers representing the confined water-bearing zones of the Pleistocene Aquifer were not encountered, nor were any bedrock encountered during the hydrogeologic investigations.

The soil horizon ranges from 4 to 6 feet in thickness and consists of an upper sandy silt and clay which grades downward to predominantly sand and silt sediments. The contact between the soil and underlying glacial till is distinctive and characterized by moist to very moist conditions.

Subsurface samples indicate the till consists primarily of brown to gray fine grained (silt and clay) sediments with occasional thin sand laminae and lenses. The transition from brown to gray colored sediments marks the base of the weathered (or oxidized) till at the Site. Discontinuous clayey-silty sand to sand to gravelly sand layers greater than 2 feet thick were encountered at depths below 20 feet around the slag pile. However, based upon regional information, these sand and gravel layers do not represent the confined water-bearing sand zones in the Pleistocene Aquifer.

3.2.5.3 Regional Hydrogeology

The principal water-bearing zones in Marion County include the thick sand and gravel deposits within the stratified (outwash) and unstratified (till) drift and deeper Devonian and Silurian carbonate rocks. Previous studies have grouped the highly complex glaciogenic sand and gravel deposits into a single unit, the principal Pleistocene Aquifer (Hartke et al. 1980). Although the aerially extensive sand and gravel deposits are the primary zones for groundwater movement, outwash sands within the till material may provide local pathways for groundwater flow. The permeable bedrock zones behave hydraulically as a single water-bearing unit and thus are combined and referred to informally as the Silurian-Devonian aquifer.

The principal Pleistocene Aquifer includes the coarse-grained deposits within the glacial till and outwash along the major stream valleys. Analysis of information from driller's logs and test borings identified three water-bearing sands and gravels in the till plain (Meyer et al. 1975). These units are designated as the upper, middle, and lower confined zones after Meyer et al. (1975). Each unit is less than 40 feet thick and is typically separated from other units by varying thicknesses of finer grained sand, silt, and clay deposits, which act as leaky confining beds. The upper confined zone does not exist in areas of the till plain west of the White River and Fall Creek. The potentiometric surface in the till plain occurs in the fine grained deposits, which overlie the uppermost sand and gravel unit. The unconfined outwash aquifer consists primarily of sand and gravel with discontinuous clayey layers (Meyer et al. 1975). Using 1974 water-level data, the saturated thickness of this unit ranges from less than 20 feet to over 160 feet, with values less than 100 feet typical for most of the area.

In general, groundwater flow within the principal Pleistocene Aquifer is toward the major streams such as Eagle Creek and White River. However, local variations in the hydrogeologic system often produce a more complex flow regime. In upland areas underlain by glacial till, groundwater recharge may flow through the fine grained materials and discharge to the surface water system at small streams or springs, or may enter the uppermost confined water- bearing zones and circulate downward and laterally toward the major streams (Meyer et al.1975). The flow pattern indicated for the uppermost confined water-bearing zone also extends through the deeper confined zones and the unconfined outwash aquifer.

The primary water-bearing zone in the Paleozoic bedrock occurs within the Silurian and Devonian limestones and dolostones. Groundwater yields from wells in the New Albany Shale and Borden Group lithologies are minimal due to the low permeability of these bedrock units (Hartke et al. 1980). Secondary permeability in the carbonate rocks is greatest in the upper 100 feet of the sequence due to fracturing and development of solution features along paleokarst surfaces. A coupled glacial-bedrock aquifer system occurs in areas where the limestones and dolostones are directly overlain by coarse stratified glacial drift. The direct hydraulic connection between the unconsolidated and consolidated materials allows for significant inter-aquifer flow and is responsible for high yields in bedrock wells over the area. The general flow pattern depicted for the principal Pleistocene Aquifer is also believed to be present to an unknown depth in the underlying bedrock (Meyer et al. 1975). Thus, horizontal groundwater flow is toward the major stream valleys in the area.

3.2.5.4 Site Hydrogeology

The layers of poorly sorted sand and gravel sediments at a depth of greater than 20 feet represent the uppermost water-bearing zones at the Quemetco facility. Thin perched zones were encountered in silt and sand deposits in the upper portions of the till at depths ranging from 3 to 16 feet below ground surface (bgs), which are believed to represent transient saturated intervals whose occurrence is dependent on surficial infiltration from precipitation events. Groundwater within the sand and gravel deposits at depths of greater than 20 feet bgs occurs under confined to semi-confined conditions. The finer grained silt and clay deposits overlying and underlying the sand and gravel units are characterized as leaky aquitards. The characterization of these materials as aquitards is supported by the low vertical hydraulic conductivity of samples from the MW-2 and MW-4 boreholes completed during the preliminary RI. Saturated hydraulic conductivities for samples of the silt and clay sediments range from 10-3 to 10-4 ft/day (Environmental Strategies Corporation [ESC] 1993). Recharge to these uppermost water-bearing zones probably occurs via leakage through the finer grained till deposits overlying the sand and gravel units.

The potentiometric surface maps generated from the data show a consistent spatial hydraulic head distribution for the upper water-bearing unit beneath the Site. Assuming a horizontally isotropic aquifer material, the general direction of groundwater flow is southwestward beneath the plant and associated production areas and northward in the portions of the Site underlying the parking area and Ponds No.2 and No. 3. The groundwater flow paths converge in the area around the water treatment facility and adjacent water storage tanks.

The hydraulic conductivity (K) of the sand and gravel material was estimated from the slug tests and constant-discharge pumping tests conducted during the Phase II RFI. These data vary across five orders of magnitude. The range in hydraulic conductivity values reflects the lithologic variability in the subsurface. An average hydraulic conductivity for the aquifer, computed as the geometric mean was determined to be 5 ft/day. This K value was used to calculate a linear groundwater velocity using a modified version of the Darcy equation: $v=Ki/n$. Assuming a porosity (n) of 30 percent, (based on the effective porosity of fine to coarse grained sand deposits) and using the average, Site-wide hydraulic gradient (i) of 0.01 calculated from potentiometric surface maps, the flow velocity through the aquifer is 0.2 ft/day (ESC 1998).

3.2.6 Ecological Communities and Habitats

The ecological communities and habitats at the Site are addressed in detail in the Final ERA for Quemetco, Inc., prepared by Cantox Environmental, dated October 19, 2006. The Site habitat is described as fragmented with industrial buildings, disturbed terrestrial and aquatic areas. The species in these habitats include generalists that are tolerant of disturbed habitats including, but not limited to, Blue Jay, Robin, Redwing Blackbirds, voles, mice, shrew, raccoons, and skunks.

3.3 Facility History

3.3.1 General History

The Site contains 408 acres and was constructed on farmland in 1972 as a secondary lead smelter. Quemetco purchased the newly constructed facility from St. Joe Minerals in 1972. Up until 1976, the facility operated a blast furnace. Approximately 15,000 cubic feet of lead-bearing slag and refractory brick from the former blast furnace operations is still stored on the west side of Pond No. 3.

In 1996, a slag reduction furnace was installed and as a result, the facility's coal-charge electric arc furnace has not been used since approximately 1998.

In 2001, a hole was discovered in a secondary containment system. As a result, solid lead material impacted onsite soils. Facility personnel removed the lead material and excavated soil to approximately 3 feet bgs. The area was backfilled with clean soil and a new secondary containment steel shell was installed.

Based on the previous releases onsite and results from the RFI conducted onsite in 1996-1997, elevated levels of lead are present in surficial soils near the process areas of the plant, in sediments and surface water in the nearby manmade ponds and Julia Creek, and in unfiltered groundwater samples.

3.3.2 Past Data Collection Activities

The Quemetco facility has been subject to many investigations since 1989. The following summaries are based on a review of reports and supporting documents prepared by WSP and its contracted laboratories and consultants. The results from the referenced investigations have been evaluated in accordance with the Region 5 Memorandum Concerning the Use of Historical Data. None of the investigation results will be used to substitute for data needed for the proposed additional sampling discussed in this Workplan and QAPP. However, the Site-related constituents list, which is based on Site operational history and waste management practices, is consistent with the results of historical data from previous investigations.

Beginning in 1982, Quemetco has contracted with several consulting firms to assess the degree of contamination at the facility, and evaluate potential remedial actions for the identified contamination, which is primarily lead.

- Three investigations were conducted between 1982 and 1987 and are further described below:
  - The first investigation was performed in September 1982 by NFS Services, Inc. and included the installation of one groundwater monitoring well (MW-1) and periodic sampling of the ponded surface waters.
  - The second investigation, conducted in August 1985 by Resource Consultants, Inc., included tests pits excavated from the slag pile and samples were collected from to determine its leaching potential.
  - A third investigation, conducted in April 1987 by the National Enforcement Investigation Center (NEIC) of the U.S. EPA, was conducted to determine the effect of the blast furnace slag pile on the surrounding area and the adjacent ponds. The investigation included sampling of the slag pile, pond sediments, and pond water.

The sampling results from these investigations indicated that the slag pile contains residual amounts of lead at levels exceeding the U.S. EPA's limit for determining whether a waste is hazardous. Elevated levels of lead were also detected in pond sediments. Pond Nos. 3 and 2 had total lead concentrations up to 2,660 mg/kg and 1,830 mg/kg, respectively. Water samples collected from Pond Nos. 3 and 2 had lead concentrations up to 50 µg/l and 900 µg/l, respectively (ESC 2002): NFS Services, Inc. 1982; Resource Consultants, Inc. 1985; and NEIC 1987).

In 1991, Quemetco entered into a consent agreement with the U.S. EPA that provided for the investigation and remediation of the area in the vicinity of the blast furnace slag pile and ponds. Canonie Environmental was retained to implement the RFI, which included the following activities:

- installation of three borings and four monitoring wells to characterize the soil lithology
- collection and analysis of groundwater samples from the existing well (MW-1) and from the newly installed wells (MW-2 through MW-5)
- performance of slug tests on MW-2 through MW-5
- collection and analysis of shallow soil samples in the vicinity of the containment trench and the three soil hills adjacent to the ponds
- collection and analysis of sediment and surface samples from the three ponds and Julia Creek collection and analysis of samples from the slag pile.

Results of the investigation and remediation indicated pond sediments exceeded the hazardous waste criteria for lead by using the toxicity characteristic leaching procedure (TCLP). Additionally, the surface water samples collected from the three ponds and Julia Creek indicated lead was above the maximum contaminant level (MCL; ESC 2002 and incorporated by reference: Canonie Environmental 1991).

In September 1993, ESC conducted limited soil sampling and soil excavation in conjunction with the closure of the hard rubber bins and assessment of the soil quality in the area of the former underground storage tanks. The soils were analyzed for total antimony, cadmium, chromium, copper, lead, nickel, and zinc. Additionally, clean up goals for the soils at the Site were established. Based on the analytical results of soils collected as part of the preliminary investigation, the area underlying the storage bin was excavated to a depth of 4 feet, and the eastern area of the work zone was excavated to a depth of 6.5 feet. The analytical results for the soil samples collected from depths greater than 6 feet in the eastern portion of the bin indicate total metal concentrations below the soil cleanup levels. All soils within the excavation zone were remediated to below the proposed cleanup goals.

RSR received a No Further Action notice from Indiana Department of Environmental Management that certified the closure was complete (ESC 1995).

In May 1994, August Mack Environmental conducted an investigation of Julia Creek to determine whether lead contamination was present in the stream. Sediment, surface water, and soil samples were collected and analyzed for total lead only. The investigation focused on the portion of Julia Creek on the Quemetco property. The surface water results from Julia Creek found that all metals were below their respective MCLs with the exception of lead. The composite sediment sample from Julia Creek contained lead at 354 mg/kg with relatively low concentrations of other metals. TCLP concentrations in the sediment sample did not cause the material to exhibit the characteristic of toxicity (ESC 1995; ESC 2002 and incorporated by reference: August Mack Environmental, Inc. 1994).

In May 1996 through April 1997, ESC performed soil sampling as part of the Phase II RFI to characterize Site-wide conditions. The following activities were conducted as part of the RFI:

- Soil samples were collected at the slag pile, containment trench, along Julia Creek, in the Northeastern area and throughout the property for background data.
- Groundwater samples were collected from six wells and nine piezometers including all of the groundwater monitoring wells required for the quarterly groundwater monitoring program.
- Surface water and sediment samples were collected from all three ponds and the stream and a weir was installed onsite.

The soil, sediment, surface water, and groundwater sampling results were compared to regulatory screening criteria (human health and ecological screening levels) or background levels for the

constituents of concern. These standards are used as screening levels to eliminate samples that clearly do not pose an unacceptable risk to human health or the environment.

Results of the Phase II RFI indicated that metals, and primarily lead, are the primary constituents of concern in the soil, sediment, and surface water at the Site. The Phase II RFI included an ERA and recommended a DERA be conducted onsite (ESC 1999).

In June 1999, a Supplemental Sampling Investigation was conducted and involved the collection and analysis of soil samples from the northeast area and adjacent to a former drainage ditch, the installation of three new groundwater monitoring wells, and the collection and analysis of groundwater samples from all of the monitoring wells and two piezometers at the Site.

Results indicated antimony, arsenic, cadmium, copper, lead, mercury, nickel, and zinc were detected in one or more soil samples at levels exceeding background concentrations in the northeast area. Lead was detected at the highest concentrations, ranging from 76.6 mg/kg to 22,700 mg/kg. Levels of other metals in each sample generally varied according to the relative lead content of the sample.

Much lower metal concentrations were detected in soil samples collected adjacent to the former drainage ditch. Only chromium and zinc were detected at concentrations marginally above background levels in any of the soil samples. None of the primary constituents of concern (lead, arsenic, cadmium, and antimony) were detected in the groundwater sample and no constituents exceeded maximum contaminant levels (ESC 1999).

In April and October 2010, and December 2011, WSP conducted an investigation using an XRF-measuring device along with laboratory analyses to characterize surficial and shallow soil (0 to 6 inches bgs) around the entire facility for impacts by lead. A total of 266 (minus one sample that was not recorded) surface and 128 subsurface soil samples were analyzed for lead using the XRF.

Based on the results of the April 2010, October 2010, and December 2011 XRF sampling events, the highest concentrations of lead (i.e., greater than 10,000 ppm) were detected in four areas of the Site: along the eastern and northern fence lines of the facility; along the east side of Quemetco Drive (north of the scale house); and adjacent to the slag pile that is between the railroad tracks and Pond No. 3. Results indicate that soil concentrations below the action level of 400 ppm were delineated to the west and south of the facility. Lead concentrations remained above 400 ppm to the northern and eastern limits of the investigation. Additionally, based on surface and subsurface XRF data combined, 22.1 percent (87 of 393) of the samples analyzed were below the action level of 400 ppm. These results break down to 22.9 percent (61 of 266) of the surface samples and 20.4 percent (26 of 127) of the subsurface samples were below 400 ppm. Generally, there is a correlation between both surface and subsurface concentrations (collected from the same sample location) in that both surface and subsurface samples are either above or below the action level (WSP Environment & Energy 2012).

3.3.3 Current Status

Based on the results of the Phase II RFI and DERA, the letter from U.S. EPA, dated February 13, 2008, and the U.S. EPA's October 8, 2009, comments to the workplan, dated May 19, 2008, the following areas of the Quemetco Site will be further investigated:

Man-made Ponds. Even though the three onsite ponds were constructed for the storage of slag waste, facility records indicate that no materials were ever placed in these areas. However, the location of these surface water bodies and the presence at the Site since the early 1970s suggests the possibility that Site-related constituents may have accumulated in the areas. The presence of metals in the ponds

may be related to runoff from the slag waste pile (Pond No. 3 only), airborne particulate emissions and general facility runoff.

Julia Creek. Julia Creek is an intermittent stream that flows across the southeastern portion of the Quemetco property. The creek flows west onto the property, and then south parallel to Quemetco Drive. There are two discharge locations identified in the facility's National Pollutant Discharge Elimination System (NPDES) permit: the discharge for NPDES 001 is not used by the facility, and NPDES outfall 002 receives outflow from Pond No. 3. The shallow swale at the southeastern portion of Pond No. 3 flows into a 30-inch sewer line that leads to Julia Creek. Constituents may have entered Julia Creek from Pond No. 3.

Northeast (NE) Area. This area of the Site is wooded and has been identified as having lead concentrations exceeding screening levels. Additional horizontal and vertical delineation will be conducted in this area. Additional information is presented in Section 2.0 of the Workplan to Complete Site Characterization and Address Outstanding DERA Issues, dated July 23, 2012.

Groundwater Monitoring Wells. Groundwater monitoring wells were installed throughout the property and most recently sampled in 1999. One round of monitoring will be conducted to evaluate the current groundwater quality and condition.

3.4 Project Objectives and Intended Data Usages

The scope of the Site Characterization and Additional DERA sampling involves the collection of information to assess potential impacts from facility operations within and around the slag waste pile, man-made ponds, Julia Creek, and the NE Area. As requested by the U.S. EPA, soil, sediment, benthic invertebrate and surface water samples will be collected to further define the nature and extent of contamination at the Site. An addendum to the DERA will be completed to incorporate the new data and assess new receptors (i.e., Indiana bat, Spotted sandpiper). As requested by EPA, the risk characterization will include fish and aquatic invertebrates in Julia Creek and the Ponds; benthic invertebrates in Julia Creek and the Ponds; and the Indiana bat, earthworms and Spotted sandpiper. The risk characterization will utilize a weight of evidence approach for the receptors that have multiple lines of evidence (i.e., earthworms, fish, pelagic and benthic invertebrates).

The DERA will include assessment of the Spotted sandpiper, American robin, and Indiana bat, an endangered species. The American robin was previously included in the 2006 assessment; however, due to the risks noted at that time, a reassessment of risks to the robin, incorporating new soil data, updated exposure pathways and toxicity reference values, will be included in the addendum.

Groundwater samples will be collected to obtain current information on the groundwater condition beneath the Site. A detailed description of the sampling areas is included in the Workplan to Complete Site Characterization and Address Outstanding DERA Issues, dated March 28, 2014.

The data collection activities will specifically address the following concerns:

1. Evaluate the impact of soil contamination on potential human receptors.
2. Evaluate the impacts of sediment, soil, and surface water contamination on potential ecological receptors.
3. Characterize the extent of soil contamination both laterally and vertically at the Site.
4. Assess the current groundwater quality and condition beneath the Site.

Analytical results will be presented as the true results and not the mean, maximum, or range.

3.4.1 Decision Rules

The decision rules associated with Site characterization and groundwater sampling activities are as follows:

- If XRF soil screening in any grid exceeds 400 mg/kg for lead (human health-based residential Regional Screening Level [RSL]), then step-out screening will continue into the next row of grids. Step-out screening will continue until all measurements are below 400 mg/kg for lead.
- If vertical soil samples analyzed for lead exceed 400 mg/kg, than additional information will be required to determine vertical extent of contamination. This may or may not require intrusive investigations.
- If analytical results from the groundwater monitoring well sampling event exceed the federal maximum contaminant levels (MCLs) then the data will be used to review potential additional monitoring or remedial alternatives, if necessary.
- If analytical results are Non-Detect, then the analytical sensitivity of the project will be assessed. If reporting limits are less than the RSL, then it has been determined that this area has not been impacted at a level of unacceptable risk and no further action is required.

The decision rules associated with DERA sampling activities are as follows:

- All ecological-biological-toxicity data collected will be included in the addendum to the DERA.
- Results from the chemical analyses for sediment, soil, and surface water will be used to supplement the ecological-biological-toxicology results. Chemical results for soils will not be compared to ecological screening levels for soils (EcoSSLs). Chemical results will be used to evaluate the statistically significant affects and any potential correlations between ecological-biological-toxicology results from the Ponds and upstream and downstream of Julia Creek. Target parameters (laboratory reporting limits), will be reviewed and all applicable data will be incorporated into the revised DERA, as necessary.

3.4.1.1 Site Characterization Soil Sampling

The objective will be to delineate soil lead concentrations laterally and vertically to below the U.S. EPA RSL for lead in residential soil of 400 mg/kg. The XRF results are not being compared to the 2003 U.S. EPA Region 5 ecological screening levels (ESLs) of 0.05 mg/kg due to the fact that this value is below the background concentrations of lead in soil and approximates the laboratory reporting limit for lead. At the request of and by approval from the U.S. EPA, an ecological-based Action Level has been developed for lead for the site. In a memo from Intinsik Environmental Sciences to the U.S. EPA, dated February 14, 2014, it was calculated that the overall Ecological Action Level for lead is 130 mg/kg (Intrinsik Environmental 2014). Furthermore, it was agreed upon that the Ecological Action Level will not be used to screen the data and that any proposed remediation criteria will be based on the ERA and not this Ecological Action Level.

In addition, five percent of the field XRF samples will be sent to a fixed-laboratory for analysis confirmation. The fixed-laboratory will use SW-846 6020 of total lead using ICP-MS. Correlation between field XRF sampling and fixed-laboratory data will be accomplished by calculating the relative percent difference between the two results (field XRF and laboratory data). No pass/fail criteria will be

established for this portion of the project. Use of the newly generated XRF data is as a screening tool to broadly delineate the lead contamination. A difference in moisture content is anticipated between the soil tested in the field using the XRF compared to the samples being tested in the laboratory recorded in dry weight, WSP does not expect the field readings and the laboratory results to match precisely. Furthermore, the XRF will be calibrated to a 1,345 ppm lead standard. The XRF operates best within the lower range of lead concentrations. It was observed from the previous XRF investigation that at high concentrations (greater than 1,295 ppm) the XRF results varied more greatly from the laboratory generated results.

Vertical soil samples will be collected with Geoprobe drilling equipment from specific locations at the Site to further characterize the vertical extent of lead. The vertical soil sampling results will be compared to the U.S. EPA RSL (400 mg/kg) and included with the data previously collected at the Site. If results for specific locations exceed 400 mg/kg, than further review of the site will be conducted and appropriate actions will be proposed to U.S. EPA.

3.4.1.2 Groundwater Sampling

At the request of U.S. EPA, a one-time sampling event is proposed to assess the current groundwater flow direction and groundwater quality. The work will involve development of each of the wells and piezometers, measurement of water levels in all 20 wells and piezometers, and sampling the 11 monitoring wells (MW-1R, MW-2 through MW-10 and MW-13). Also, each of the wells and piezometers will be re-surveyed for vertical elevation. None of the piezometers at the Site will be sampled. Groundwater samples collected from the existing monitoring wells will be analyzed for metals, phenols and total suspended solids. Results will be compared to federal MCLs presented in Table B3. If groundwater constituents exceed the MCLs, then the data will be reviewed to determine if further actions are required.

3.4.1.3 DERA Sampling

The DERA field sampling will include benthic invertebrates and animal toxicity tests on soil, sediments, and surface water; chemical analysis of metals in soil, sediment, surface water; and evaluation of the risk to the Spotted sandpiper and the endangered Indiana Bat. A summary of the proposed DERA sampling and analyses is provided in Table B4. The proposed sampling locations for the DERA are shown in Figure B2.

Toxicity testing results will be used in the Effects Assessment component of the DERA for the various receptors that have multiple lines of evidence in Julia Creek, the Ponds and surrounding areas (i.e., earthworms, fish, benthic invertebrates). The rationale for using all of the data in the addendum to DERA is to completely assess new receptors (i.e., Indiana bat, sandpiper) and reassess the risks to the American robin by incorporating new soil data. These results will be incorporated into the weight-of-evidence evaluation in the Risk Characterization component of the addendum to the DERA and will include updating exposure parameters and toxicity reference values, as needed.

3.4.2 Chemical Analysis Project Target Parameters

Based on historical data from the Site, the project target parameters for chemical analysis are lead, antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, zinc and phenols. Based on current and historical operations conducted by RSR, other metals found in Appendix IX for RCRA metals analysis have been eliminated (See ERA, October 19, 2006, prepared by Cantox Environmental).

Additionally, the following parameters will be used to supplement analytical data: acid volatile sulfide/ simultaneously extracted metals (AVS/SEM), pore water pH, and pore water ammonia. The following parameters will not be used as a screening tool: total organic carbon (TOC), soil moisture content, water holding capacity, particle size analysis, cation exchange capacity (CEC), water hardness.

3.4.2.1 Field Parameters

XRF soil sampling will be conducted in accordance with WSP's XRF Sampling and Analyses Plan, dated February 17, 2010. These procedures followed the US EPA's approach presented in their letter, dated October 8, 2009. The sampling strategy involves creating a grid composed of 70-foot by 70-foot squares within the target areas of the Site then collecting XRF samples of surface soils (0-1 inch bgs from within each grid square. If an XRF reading exceeds 400 mg/kg lead in a single grid square, then the investigation will extend into the grid square immediately adjacent to each detection. XRF sampling will continue laterally until the lead concentrations have been delineated to below 400 mg/kg or until the Quemetco property line is reached to the north and east.

Surface water samples will be collected with a glass or plastic jar and in accordance with WSP's SOP #4. Surface water will be analyzed in the field for general chemistry parameters including pH, temperature, specific conductivity, oxidation-reduction (redox) potential, and turbidity. These general chemistry parameters will be used to supplement the analytical data for surface water and are not being used for screening purposes. The samples will be analyzed and reported in the field using a YSI 556 handheld multi-parameter instrument. The rationale for conducting surface water sampling is to determine whether contamination on the property has impacted surface waters at the Site.

The sediment samples will be collected after all surface water samples are collected. Sediment sampling will begin with the most downstream location and will progress upstream to minimize the possibility of cross-contamination. At all locations, sediment samples will be collected from the sediment surface at approximately 0 to 0.5-foot of the sediment layer. Depending on the depth of water at the time of sampling, the shallow sediment samples will be collected using a stainless steel scoop, Ponar or Eckman dredge, or hand corer in accordance with WSP's SOPs #4, 5 and 6. Field parameters will be collected for sediments in accordance with SOP #s 4, 5, and 6. Field parameters collected during the sediment sampling activities will include pH and visual observations including color, texture, and presence of oil sheens, macrophites, or animals. The field parameters collected during sediment sampling will be used to supplement the analytical data and not used as for screening purposes.

Groundwater samples will be collected with glass or plastic jars and in accordance with WSP SOP #3. Groundwater indicator parameters will be collected during purging and will be measured by the field technician. The groundwater indicator parameters will include pH, specific conductivity, temperature, redox potential, turbidity, and dissolved oxygen and will be used to determine whether groundwater has stabilized and samples can be collected.

The WSP SOPs are included in Attachment B of this QAPP.

Table B2—Field Parameters				
Constituent	**Regulatory Screening Level**	**Ecological Action Level (not for screening purposes)**	**Stabilization Parameters**	**Matrix**
Lead	400 mg/kg	130 mg/kg	NA	Soil
pH	NA	NA	± 0.1 SU	Surface Water, Purged Groundwater, Sediments
Specific Conductance	NA	NA	± 3%	Surface Water, Purged Groundwater
Temperature	NA	NA	± 3%	Surface Water, Purged Groundwater
Dissolved Oxygen	NA	NA	± 0.2 mg/l or 10% (for flow-through cell)	Purged Groundwater
Turbidity	NA	NA	± 10% for values greater than 1 NTU	Surface Water, Purged Groundwater
Oxidation-Reduction Potential	NA	NA	± 10 millivolts (for flow through cell only)	Surface Water, Purged Groundwater
Note: NA= not applicable for constituent				

3.4.2.2 Laboratory Parameters

Soil, sediments, surface water, and groundwater will be submitted to laboratories for various analyses. The specific analyses and U.S. EPA methods are presented in Table B4.

Soil samples collected from the three ponds and Julia Creek will be analyzed for metals, pH, TOC, soil moisture, water holding capacity, particle size analysis and CEC. Soil samples will be collected from 0 to 6 inches bgs. Analytical results from these soil samples will be used to assess risks to designated wildlife receptors and potential human receptors using exposure and toxicity assumptions.

Fixed-laboratory soil samples collected from the north and northeast grids during XRF sampling will be analyzed for lead. Additionally, vertical soil samples collected from specific locations at the Site will be analyzed for lead. If analytical results for fixed-laboratory and vertical soil samples exceed 400 mg/kg then lead impacted soils have not been delineated and additional actions will be required.

Sediment samples collected from the three ponds and Julia Creek will be analyzed for total metals (including arsenic, cadmium, copper, lead and nickel), phenols, AVS/SEM, interstitial pore water ammonia, interstitial pore water pH, TOC, particle size analysis, and CEC. Analytical results from sediment sampling will be used to assess risks to designated wildlife and potential human receptors using exposure and toxicity assumptions.

Surface water samples collected from the three ponds and Julia Creek will be analyzed for total and dissolved metals, pH, and water hardness. Analytical results from surface water sampling will be used to assess risks to designated wildlife receptors using exposure and toxicity assumptions.

Groundwater will be collected from 11 existing monitoring wells and analyzed for total and dissolved metals, total suspended solids, and total phenols. Analytical results from groundwater will be used to assess current groundwater conditions beneath the Site.

Table B3—Laboratory Target Parameters

Constituent	Drinking Water MCLs (mg/L)	Surface Water Laboratory Reporting Limits (mg/L)	Sediment Laboratory Reporting Limits (mg/kg)	Soil Laboratory Reporting Limits (µg/kg)
Antimony	0.006	0.001	0.10	2000
Arsenic	0.010	0.001	0.10	2000
Barium	2	0.001	0.10	500
Cadmium	0.005	0.001	0.10	500
Chromium	0.1	0.001	0.10	500
Copper	1.3	0.001	0.10	1000
Lead*	0.015	0.001	0.10	1000
Mercury	0.002	0.0002	0.02	6.7
Nickel	N/A	0.001	0.10	1000
Zinc	5	0.01	2	4000
Phenol	N/A	0.05	1.5	NS
Ammonia	N/A	NS	0.500 mg/L pore water	NS
TOC	N/A	NS	400	NS
CEC	N/A	NS	0.1 meq/100g	0.1 meq/100g
AVS	N/A	NS	0.05881 µmol/g	NS
pH	N/A	0.1 standard units	NA	0.1 standard units
Pore water pH	N/A	NS	0.5	NS
Pore water Ammonia	N/A	NS	0.5	NS

SEM-Antimony	N/A	NS	0.0041068 µmol/g	NS
SEM-Arsenic	N/A	NS	0.0066736 µmol/g	NS
SEM-Cadmium	N/A	NS	0.0011121 µmol/g	NS
SEM-Chromium	N/A	NS	0.0024054 µmol/g	NS
SEM-Copper	N/A	NS	0.0039342 µmol/g	NS
SEM-Lead	N/A	NS	0.0009049 µmol/g	NS
SEM-Mercury	N/A	NS	0.0000249 µmol/g	NS
SEM-Nickel	N/A	NS	0.0042589 µmol/g	NS
SEM-Silver	N/A	NS	0.0023176 µmol/g	NS
SEM-Zinc	N/A	NS	0.0152952 µmol/g	NS

3.4.3 Ecology-Biology-Toxicology Analysis

3.4.3.1 Project Target Parameters

Based on historical data collected from the Site, the ecological-biological-toxicology project target parameters will include lead in surface water and lead, arsenic, cadmium and nickel in sediments. Onsite soils will be collected for earthworm toxicity testing with the project target parameters of lead, arsenic, cadmium and nickel. The following ecological-biological-toxicology parameters will be analyzed:

- Soils will be collected for earthworm toxicity tests.
- Surface water samples will be collected from Pond No. 3 and Julia Creek for chemistry and toxicity testing using *P. promelas* and *C. dubia*.
- Sediment samples will be collected from all three ponds and Julia Creek for chemistry, physical characterization and Hyalella toxicity tests.
- Benthic invertebrates will be collected for tissue analysis.
- The Spotted sandpiper and endangered species Indiana bat will be assessed in the addendum to the DERA. The American robin will be re-assessed in the addendum to the DERA.

3.4.3.2 Field Parameters

Surface water samples will be collected from Pond No. 3 and Julia Creek and used for toxicity testing. Field parameters for surface water samples will include the following: pH, dissolved oxygen, temperature, specific conductivity, turbidity, and redox potential. The field parameter results will be used to assess the stabilization of surface water and supplement the laboratory results.

A study of the Spotted sandpiper and the Indiana bat, an endangered species, will be conducted through modeling. The specific details of the modeling parameters are included in Section 3.7, pages 13-14 of the Workplan to Complete Soil Characterization and Address Outstanding DERA Issues (WSP, March 27, 2014).

The rationale for choosing the field parameters include reviewing the results of the 2006 ERA (Cantox Environmental 2006) and previous investigations conducted at the Site.

3.4.3.3 Laboratory Parameters

Soil samples will be collected from around Pond Nos. 1, 2, and 3, Julia Creek, the Slag Pile, and the NE Area and analyzed for soil pH, TOC, soil moisture content, water holding capacity and CEC. Additionally, soils will be collected and composited to be used for earthworm toxicity tests. Analytical results from the earthworm testing will be used in the addendum to the DERA.

Sediment and surface water samples will be collected from the onsite ponds and Julia Creek, since these features may serve as accumulation areas for constituents present from the slag waste pile and other facility production areas transported via wind or surface runoff.

The surface water samples will be analyzed for toxicity by using *P. promelas* and *C. dubia*. Analysis of the sediment samples will be limited to Chironomus and Hyalella toxicity tests as well as other selected parameters including TOC, particle size, water moisture analysis, and CEC.

The analysis of sediment samples for TOC and particle size analysis will provide additional information on the processes that could influence the fate of metals in the onsite ponds. TOC concentrations will be compared with the metal concentrations detected in sediment samples for each pond. The presence of relatively high metal concentrations with elevated organic carbon levels may indicate that organic complexation is a primary mechanism affecting the transport of these constituents.

Benthic invertebrates will be collected from sediments in the Ponds and Julia Creek and tested for metals in the tissue and moisture content. Analytical results from the benthic invertebrate testing will be included in the addendum to the DERA.

These parameters have been selected based on the results of the 2006 ERA (Cantox Environmental 2006), and were not eliminated as a chemical of concern in the ERA. Additionally, other test parameters, such as water hardness and TOC, have been selected to assist in the interpretation of the toxicity testing results.

Laboratory SOPs are included as Attachment C of this QAPP.

3.5 Sampling Locations

Maps showing intended soil, sediment, benthic invertebrate, surface water, XRF, and groundwater sampling locations are included as Figures B2, B3, and B4 of this QAPP. It is possible, however, that depending on the nature of encountered field conditions, sampling locations may be changed. The person responsible for making such decisions will be the Site Field Manager whose responsibilities are described in Section 4.2 of this QAPP.

3.5.1 Rationale of Selected Sampling Locations

The rationale for the selected sampling locations and media is fully described in the Workplan. The rationale for each sampling location is also based on the results of the previous investigations, the 2006 ERA, discussions with U.S. EPA, the SAP, and previous sections of the QAPP. The number of samples have been determined to further delineate onsite contamination, will be used for corrective action measures at the Site, if applicable, and supplement and revise the DERA. Additionally, background soil samples will be collected from the southwest side of the Site and will be included as a composite sample. Background soil samples will be used in the 14-day earthworm toxicity testing and particle size analysis.

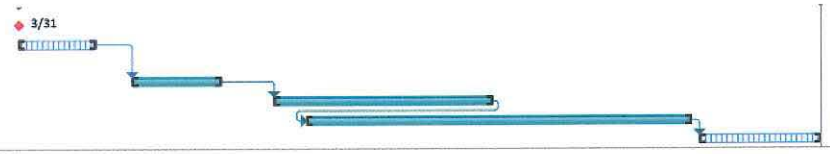
3.6 Project Schedule

The earliest date for which soil, sediment, and surface water sampling will be conducted is 10 days after the Workplan is approved.

3.7 Task Bar Chart and Associated Time Frames

A task bar chart and associated time frames for the project is presented on the next page.

1	Submittal of Final Work Plan, SAP, QAPI		Mon 3/31/14	
2	EPA Approval of Final Work Plan, SAP, QAPP	15 days	Mon 3/31/14	Fri 4/18/14
3	Field Work	16 days	Mon 4/28/14	Mon 5/19/14
4	Receipt of Analytical Data	40 days	Mon 6/2/14	Fri 7/25/14
5	Report Preparation	69 days	Tue 6/10/14	Fri 9/12/14
6	EPA Review/Approval of Report	22 days	Mon 9/15/14	Tue 10/14/14



Project: RSR Indianapolis
Date: Mon 3/17/14

Task	Project Summary	Inactive Milestone	Manual Summary Rollup	Deadline
Split	External Tasks	Inactive Summary	Manual Summary	Progress
Milestone	External Milestone	Manual Task	Start-only	
Summary	Inactive Task	Duration-only	Finish-only	

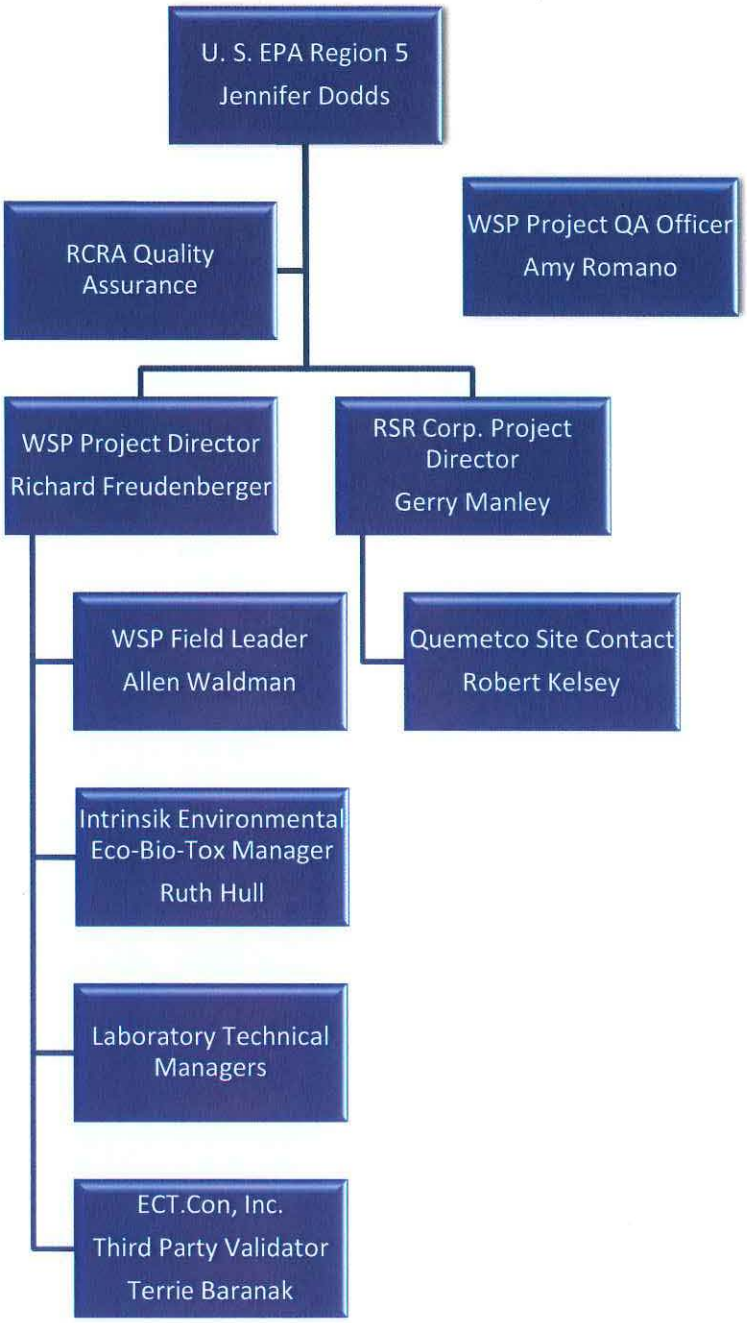
4 Project Organization and Responsibility

At the direction of the Quemetco, WSP has responsibility for all phases of the investigation. WSP is the principal consultant to Quemetco and will be responsible for project management and conducting or managing the performance of all services required to further characterize the Site and address the outstanding DERA issues. These activities include field operations, laboratory testing, data management, data evaluation and reporting. The various QA, field, laboratory and management responsibilities are presented in the Project Organization Chart.

4.1 Project Organization Chart

The lines of authority specific to the investigation are presented on the chart below (see next page). This chart includes all individual discussed below.

Figure B5—Project Organization Chart



4.2 Management Responsibilities

4.2.1 U.S. EPA Project Manager

Jennifer Dodds, the U.S. EPA project manager, has the overall responsibility for all phases of the investigation. Additionally, Ms. Dodds will ensure proper review and distribution of all documents and communicating comments from technical reviewers to the Project Coordinator and project director.

4.2.2 Quemetco Project Coordinator

Gerry Manley, vice president of environmental, health and safety compliance for RSR Corporation is Quemetco's project coordinator. Mr. Manley is responsible for implementing the project and has the authority to commit the resources necessary to meet the project objectives and requirements. Mr. Manley's primary function is to ensure that technical, financial, and schedule objectives are achieved successfully. Mr. Manley is responsible for providing Site information and history, monitor all field leaders, reviewing all results and recommendations, providing management and technical oversight, approving all reports before delivery to U.S. EPA Region 5, represent the project team at all meetings. The Quemetco Project Coordinator will report directly to the U.S. EPA Project Manager.

4.2.2.1 Quemetco Site Contact

Robert Kelsey, environmental, health and safety compliance manager of Quemetco is the Site contact. Mr. Kelsey is responsible for providing Site information, logistical assistance and ensuring that all facility-specific environmental, health and safety procedures during the investigations are followed. Mr. Kelsey will report to Mr. Manley.

4.2.3 WSP Project Director

Richard E. Freudenberger, P.E., senior vice president of WSP, will be WSP's Project Director. He has the authority to commit the firm's resources to accomplish the project objectives. He will have ultimate responsibility for WSP and subcontractor performance. In addition, he will be responsible for the direction and management of all WSP's field, laboratory, and office activities. Mr. Freudenberger has the responsibility and authority to procure the necessary support services and equipment for implementing each phase of the investigative effort. He will also be responsible for staffing and scheduling all of WSP's activities. Mr. Freudenberger will work closely with Mr. Manley to ensure the investigation is conducted according to the Workplan, SAP and QAPP. Additionally, Mr. Manley and Mr. Freudenberger will be responsible for communicating with agency personnel including Ms. Dodds of U.S. EPA Region 5.

4.3 Quality Assurance Responsibilities

4.3.1 WSP Project Quality Assurance Officer

Ms. Amy Romano, senior technical manager of WSP will be WSP's Project Quality Assurance Officer (QAO) and will be responsible for all QA/QC aspects of the work, including data assessment, data validation, and quality reviews. The QAO will remain independent of direct job involvement and day-to-

day operations, and will resolve any QA disputes. Additionally, Ms. Romano will work closely with the contract analytical laboratory QAO to ensure that all QA/QC requirements are being met. Ms. Romano will be responsible for auditing the implementation of the QA program in conformance with the Quemetco policies and U.S. EPA requirements. The Project QAO has sufficient authority to stop work on the investigation as deemed necessary in the event of serious QA/QC issues. The QAO will be responsible for the following activities during the project:

- Performing QA audits on various phases of the field operations;
- Performing QA audits on various laboratory operations
- Reviewing and approving QC plans and procedures;
- Providing QA technical assistance to project staff
- The QAO will be kept informed of all changes, discussions and decisions
- The QAO will review all documents prepared for the project.

4.3.2 Third-Party Data Validator

Ms. Terrie Baranek, technical manager of ECT.CON Inc., will be the Third-Party Data Validator during the project and will be responsible for validation of the fixed laboratory data. The Third-Party Data Validator will remain independent of direct job involvement and day-to-day operations and will report to the WSP Project Manager.

4.3.3 Ecotoxicology Expert

Ruth Hull, senior scientist of Intrinsik Environmental Sciences, Inc., will be the ecotoxicology expert and will be responsible for guidance during the ecological-biological-toxicology sampling. The Ecotoxicology Expert will remain in close contact with field personnel and laboratory personnel and will remain apprised of day-to-day operations. Ms. Hull will report to the WSP Project Director, Mr. Freudenberger, and will resolve any ecotoxicology disputes. Additionally, Ms. Hull will be responsible for evaluating the results of the ecological-biological-toxicology assessments. If necessary, the DERA will be updated by Ms. Hull and she will provide insight on how the data will be used in Site decisions.

4.4 Laboratory Responsibilities

Four laboratories will be used to complete the analytical work for this project. Pace, Northwest Aquatic Services (NAS), RTI Laboratories (RTI), and CQM Inc., Laboratories (CQM) will perform water, soil, sediment and benthic invertebrate analyses. The laboratory directors will report directly to the WSP Project Manager. The laboratory certifications and SOPs are included in Attachment C. Table B5 provides a summary of sample type, analysis, and the associated laboratory performing the analyses.

Table B4—Summary of Sample Analysis and Assigned Laboratories

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method	Laboratory
soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 6020/ 7471	Pace Analytical
soil	pH, total organic carbon , soil moisture, water holding capacity, cation exchange capacity	28	P1-13 through P1-16; P2-10 through P2-13; P3-16 through P3-20; JCS-1 through JCS-9 and JCS-18 through JCS-23	EPA 9045; EPA 9060A; SM 2540G; SM 2540G; EPA 9081;	Pace Analytical; CQM for water holding capacity
soil	14-day earthworm survival and biomass toxicity test; particle size analysis	7	COMP-1 through COMP-3; COMP-JC; COMP-SP; COMP-NE; COMP-BK	EPA 600/3-88-029; ASTMD 422-Atterberg Limit	NAS; CQM for particle size analysis
soil	Lead by XRF	22 grids	Grid #s 267-288. If required, additional step out grids will be sampled	EPA 6020	WSP in field; Pace Analytical for laboratory analyis
soil	Lead	5	VD-1 through VD-5	EPA 6020	Pace Analytical
surface water	Fathead minnow *(P. promelus)* and water flea (*C. dubia*) toxicity test	10	SW-1 through SW-8, P3-SW-1 and P3-SW-2	EPA-821-R-02-013 Method 1000.0	NAS
surface water	pH, water hardness	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8, SW-18 through SW-23	SM 4500 H; SM 2340 B	Pace Analytical
surface water	Total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); Dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	16	P3-SW-1 and P3-SW-2 SW-1 through SW-8, SW-18 through SW-23	EPA 6020/ 7470A	Pace Analytical
sediment	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	23	SD-1 through SD-23	EPA 6020/7471	Pace Analytical
sediment	Total Phenols	23	SD-1 through SD-23	EPA 8270C	Pace Analytical

Sample Matrix	Analysis	Number of Samples	Sample ID (a)	Method	Laboratory
sediment	*H. azteca* (28-day) and *C. dilutus* (10-day) toxicity tests	11	SD-1, SD-3, SD-4, SD-5, SD-10, SD-13, SD-17, SD-18, SD-20, SD-22, SD-23	EPA/600/R-99/064 100.4 and 100.2	NAS
sediment	Interstitial pore water ammonia, Interstitial pore water pH	23	SD-1 through SD-23	EPA 350.1; SM 4500 H-B	Pore water extraction and pH by RTI; ammonia analysis by Pace Analytical
sediment	AVS/SEM	23	SD-1 through SD-23	EPA 1629/6010/ 7471	Pace Analytical
sediment	total organic carbon , particle size analysis, cation exchange capacity	23	SD-1 through SD-23	Walkley-Black; ASTM D 422-Atterberg Limit; EPA 9081	Pace Analytical; particle size by CQM
ground-water	total metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); dissolved metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); total suspended solids; total phenols	11	MW-1R, MW-2 through MW-10 and MW-13	EPA 6020/ 7470A; SM 2540 D; EPA 8270C	Pace Analytical
benthic inverte-brate	Total metals, moisture content	16	BI-1 through BI-5, BI-8, BI-9, BI-12, BI-13, BI-15, BI-17 through BI-20, BI-22 and BI-23	EPA 6020/7471; ASTM D 2974-87	Pace Analytical

4.4.1 Pace Analytical Services, Inc.

Pace Analytical Services, Inc. (Pace Analytical), located in Indianapolis, Indiana, is scheduled to perform the analyses on soil, water, sediments and benthic invertebrate tissue as indicated on Table B4.

The Pace Analytical staff who have responsibilities during the project are outlined below and include the General Manager, Quality Manager, Client Services Manager, Project Manager, Department Manager, Group Leaders and Laboratory Analysts.

4.4.1.1 General Manager

The General Manager provides leadership and support for the QA program and is responsible for overall laboratory efficiency at the local level and reports to the corporate headquarters. The General Manager also provides resources for implementation of the QA program, ensures compliance with all local, state and federal regulations, and reviews and approves the QA Manual for the facility. The General Manager will be responsible for ensuring all the resources of the laboratory will be available on an as-required basis.

4.4.1.2 Quality Assurance Manager

The responsibility of the Quality Assurance Manager (QAM) is to oversee implementation of the quality program and to coordinate QA activities within the laboratory. The QAM works with laboratory production units to establish effective quality control and assessment plans, conducts internal audits, and manages document control.

The QAM reports directly to the General Manager.

4.4.1.3 Client Services Manager

The Client Services Manager is responsible for the Client Services Department (customer services/project chemists, sample receipt and Electronic Data Deliverables). The Client Services Manager provides a complete interface with clients from initial project specification to final deliverables.

The Client Services Manager reports directly to the General Manager.

4.4.1.4 Project Manager

The Project Manager is a senior-level scientist assigned to each client to act as a technical liaison between the client and the laboratory. The project manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements.

The Project Manager reports to the Client Services Manager.

4.4.1.5 Department Manager

The Department Manager has the responsibility to ensure that quality control functions are carried out as planned, and to guarantee the production of high quality data. Department managers have the responsibility to monitor the day-to-day operations to ensure that productivity and data quality objectives are met for each department. Department Managers have the authority to approve and release technical and data management reports.

The Department Managers report directly to the Project Manager.

4.4.1.6 Group Leaders

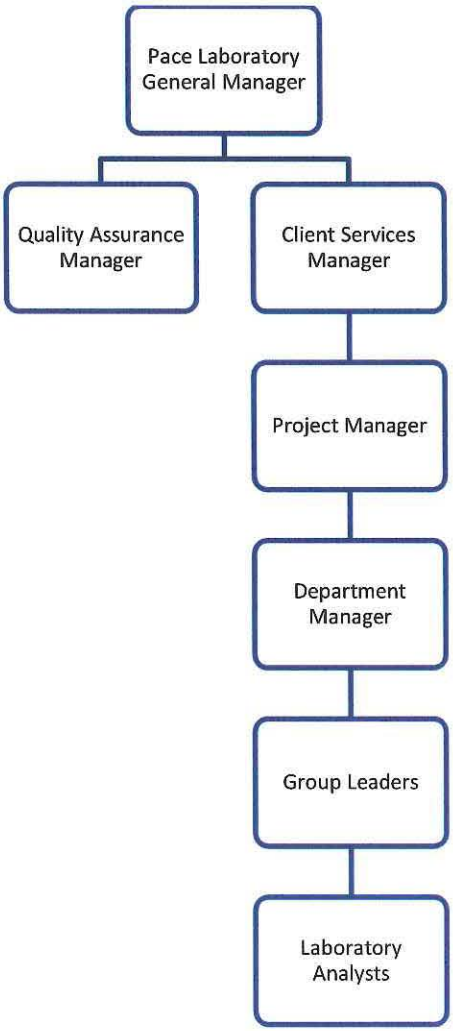
Group Leaders train technical staff in laboratory operations and analytical procedures. Group Leaders organize sample analysis schedules based on holding times, implements data verification procedures, and evaluates instrument performance and schedules instrument calibrations.

Group Leaders report to the Department Manager.

4.4.1.7 Laboratory Analysts

Laboratory Analysts will be responsible for sample preparation and producing data that meets the requirements of published methods and laboratory procedures. Laboratory Analysts reviews and finalizes raw data and monitors quality control measures.

The Laboratory Analysts perform equipment maintenance and will report to their Group Leaders.


Pace Laboratory General Manager
Quality Assurance Manager
Client Services Manager
Project Manager
Department Manager
Group Leaders
Laboratory Analysts

4.4.2 Northwestern Aquatic Sciences

NAS, located in Newport, Oregon, will perform the 14-day earthworm survival and biomass toxicity test for soil; Fathead minnow (P. promelas) and water flea (C. dubia) chronic toxicity test for water; and the 28-day toxicity tests using *H. azteca* and 10-day toxicity tests using *C.dilutus* for sediment.

The NAS staff with responsibilities during this project is presented below and includes the Laboratory Director, QAO, Technical Director, and Technical Staff.

4.4.2.1 Laboratory Director

The Laboratory Director is responsible for ensuring that the responsibility, authority, and interrelationships of all personnel who manage, perform, or verify work affecting the quality of tests are specified and documented, including a clear description of the lines of responsibility in the laboratory to ensure adequate supervision, and job descriptions for all positions.

4.4.2.2 Quality Assurance Officer

The QAO is responsible for evaluating data objectively and performing assessments independent of managerial influence. Additional tasks include coordinating QA/QC procedures and data corrective action process, conducting internal audits, and the maintenance of NAS's Quality Assurance Plan.

The QAO reports directly to the Laboratory Director.

4.4.2.3 Technical Director

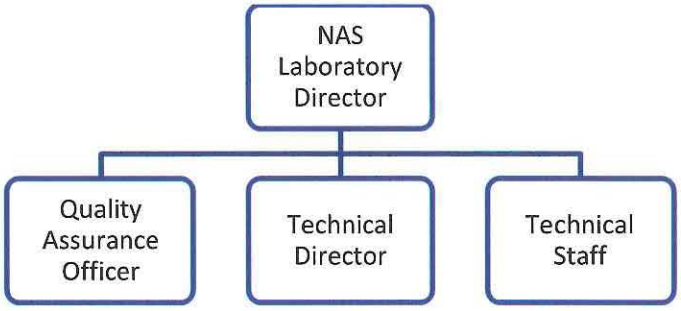
The Technical Director is responsible for monitoring standards of performance in QA/QC, and monitoring the validity of analyses performed and data generated.

The Technical Director reports directly to the Laboratory Director.

4.4.2.4 Technical Staff

The Technical Staff is responsible for sample analysis and identification of corrective actions. All personnel are responsible for complying with all QA/QC requirements that pertain to their organizational/technical function.

The staff report directly to the Laboratory Director.



4.4.3 RTI Laboratories

RTI, located in Livonia, Michigan, will perform the pore water extraction from the sediments and also analyze the pH in the extracted pore water. The pore water analysis for ammonia will be conducted by Pace.

The RTI staff with responsibilities during this project includes the General Manager, Technical Director, Quality Management Director, Laboratory Operations Supervisor, Group Leader/Department Manager, and Chemists/Technical Staff.

4.4.3.1 General Manager

The General Manager is responsible for overall laboratory activities and exercises day-to-day supervision of laboratory operations and reporting of results. Key responsibilities include strategic planning, business development, resource allocation, and profit and loss.

4.4.3.2 Technical Director

The Technical Director is responsible for ensuring that all personnel strictly adhere to policies and procedures enacted to maintain laboratory data integrity, client confidentiality, and quality standards. The Technical Director monitors laboratory performance with regard to QA adherence to ensure data accuracy. The Technical Director maintains oversight of all personnel and ensures that the qualifications and experience are suited to the position of the employees. The Technical Director approves all staff training and authorizes individuals to perform the tasks assigned.

The Technical Director will report to the General Manager.

4.4.3.3 Quality Management Director

The Quality Management Director is responsible for monitoring QA policies and procedures as well as documenting the quality of data reported by the laboratory. Additional tasks include auditing QC functions to ensure compliance with the laboratory protocols, reviewing record keeping and ancillary documents, maintaining quality control materials, and management of proficiency testing samples to ensure that the these sample are analyzed and reported in accordance with the program requirements.

The Quality Management Director reports directly to the General Manager.

4.4.3.4 Laboratory Operations Supervisor

The Laboratory Operations Supervisor is responsible for supervision of laboratory analytical staff, daily scheduling, monitoring work progress and tracking work orders. The Laboratory Operations Supervisor is responsible for ensuring that samples arriving at the laboratory are received in accordance with acceptable sampling programs and for timely logging and storage of samples. The Laboratory Operations Supervisor tracks and schedules work throughout the laboratory in order to ensure compliance with hold time requirements and client expectations. Other responsibilities include approval of laboratory reports.

The Laboratory Operations Supervisor reports directly to the Technical Director.

4.4.3.5 Group Leader/Department Manager

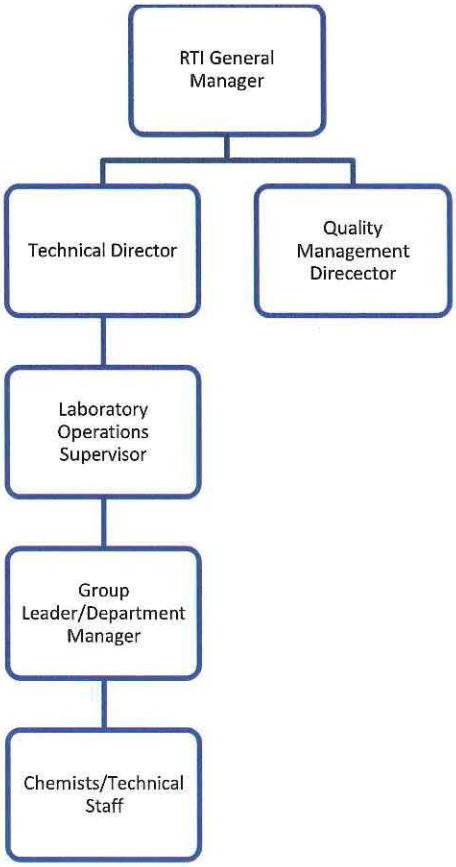
Group Leader/Department Manager is responsible for the status and progress of work in progress and technical aspects of the project. Group Leaders are responsible for overseeing the work performed by the technical staff and with client consultation through the generation of the final report.

The Group Leader/Department Manager reports to the Laboratory Operations Supervisor.

4.4.3.6 Chemists/Technical Staff

Chemists/Technical Staff Members will be responsible for sample preparation and producing data that meets the requirements of RTI's Standard Operating Procedures and Quality Assurance Plan.

These staff members will report to their Group Leader/Department Manager.



4.4.4 CQM, Inc. Laboratories

CQM, located in Green Bay, Wisconsin, will perform the pore water extraction from the sediments and also analyze the pH in the extracted pore water. The pore water analysis for ammonia will be conducted by Pace.

The CQM staff with responsibilities during this project includes the Laboratory Manager, Laboratory Assistant Manager, and Laboratory Technicians.

4.4.4.1 Laboratory Manager

The Laboratory Manager is responsible for overseeing testing procedures, ensures compliance with laboratory quality manuals, provides technical support to assistant manager and technicians, and assigns tasks and procedures to laboratory technicians.

The Laboratory Manager will report to CQM's President.

4.4.4.2 Laboratory Assistant Manager

The Laboratory Assistant Manager is responsible assigning and overseeing physical testing of media sent to laboratory, assists with the interpretation and calculation of test results, and assigns task and procedures to laboratory technicians.

The Laboratory Assistant Manager reports directly to the Laboratory Manager.

4.4.4.3 Laboratory Technicians

Laboratory Technicians will be responsible for sample preparation, testing and producing data that meets the requirements of CQM's Standard Operating Procedures and Quality Assurance Manual.

These staff members will report to the Laboratory Assistant Manager.

4.5 Field Responsibilities

4.5.1 WSP Field Leader

The Quemetco Project Manager will be supported by Mr. Allen Waldman, technical manager of WSP, the WSP Field Leader. Mr. Waldman is responsible for leading and coordinating the day-to-day activities of the various resource specialists under his supervision. Mr. Waldman is a registered geologist and will report directly to the WSP Project Director, Mr. Freudenberger and the Quemetco Project Manager, Mr. Manley. Specific field team leader responsibilities include:

- Provision of day-to-day coordination with the Quemetco Site manager on technical issues in specific areas of expertise
- Developing and implementing of field related work plans, assurance of schedule compliance, and adherence to management-developed study requirements
- coordinating and managing of field staff including sampling, drilling, and supervising field laboratory staff
- implementing of QC for technical data provided by the field staff including field measurement data
- adhering to work schedules authorized by Quemetco
- authoring, writing, and approving of text and graphics required for field team efforts
- coordinating and overseeing of technical efforts of subcontractors assisting the field team
- identifying problems at the field team level, resolving difficulties in consultation with the WSP Project Manager, implementing and documenting corrective action procedures, and provision of communication between team and upper management
- participating in preparation of the final report

4.5.2 WSP Field Technical Staff

The technical staff (team members) for this project will be drawn from WSP's resources. The technical team staff will be utilized to gather and analyze data, and to prepare various task reports and support materials. All of 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. The technical staff will report directly to Mr. Waldman, the WSP Field Leader.

4.5.2.1 Jason Zajdlik – Field Assistant Manager and Health and Safety Officer

The Field Assistant Manager is responsible for assisting the WSP Field Leader with all aspects of the project. Mr. Zadjlik will assist with scheduling, coordinating resources, conducting field work and preparing reports.

As the Health and Safety Officer, Mr. Zajdlik is responsible preparing health and safety plans for the project and ensuring all Site activities are performed in a safe manner.

The Health and Safety Officer will report directly to Mr. Waldman and will be responsible for the following:

- preparation of the Health and Safety Plan and ensuring that it is followed
- ensuring that all Site activities are performed in a safe manner to eliminate danger to personnel performing the field activities
- providing guidance to the injured for immediate medical attention
- filing personnel injury reports to the WSP Project Director

4.6 Special Training Requirements and Certifications

During this investigation, XRF sampling and analysis will be completed and requires training on the proper use of the sampling equipment.

Additionally, field personnel may encounter hazards while conducting sampling. All WSP personnel have received the proper safety and health training.

4.6.1 Training

Training is required for non-routine sampling procedures including XRF. WSP staff received internal training on the proper use of the XRF machine and proper sample collection and analysis.

All WSP personnel have received 40-hour Occupational Safety and Health Administration (OSHA) training with the annual 8-hour refresher training.

4.6.2 Certifications

Certifications maintained by project participants have been attained prior to implementation of the Workplan and in accordance with Quemetco, WSP, and contractor laboratory policies.

5 Quality Assurance Objectives for Measurement Data

The overall QA objective for this project is to implement procedures for field sampling and reporting that will provide results which are high in quality, legally defensible in a court of law, and support the decision rules outlined in Section 3.4.1.

5.1 QA Objectives for Site Characterization

The soil data collected for additional Site delineation should be of sufficient quantity and quality to assess vertical and lateral extent of contamination from facility operations. Lead is considered the target constituent. Field (XRF) and laboratory (verification and vertical samples) results for lead will be compared to the U.S. EPA Region 9 RSL of 400 mg/kg.

The groundwater samples collected from 11 monitoring wells onsite will be analyzed to determine the condition of groundwater beneath the Site and the extent, if any, of contamination from facility operations that have migrated to groundwater. Because an assessment of the overall condition of groundwater is required, the target constituents are total and dissolved metals, total suspended solids and total phenols. The target parameters for groundwater will be the MCLs. Laboratory reporting limits for each media sampled are presented in Tables B3.

5.1.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Precision will be measured for the chemical analyses through the collection and analysis of duplicate and matrix spike/matrix spike duplicate (MS/MSD) samples.

5.1.1.1 Field Precision Objectives

Field precision for the chemical analyses will be assessed through the collection and analysis of duplicate samples during XRF, soils, sediment, surface water and groundwater sampling. Duplicates samples will be collected at the frequency of one duplicate per twenty (20) samples collected for each matrix. The duplicate will then be analyzed for identical metals as the associated sample. Method precision performance will be considered acceptable if the relative percent difference (RPD) between the same and its duplicate is less than 20 percent. The calculation of the RPD used to calculate precision can be found in Section 14.2 of this QAPP.

5.1.1.2 Laboratory Precision Objectives

MS/MSD samples will be collected at the frequency of one per twenty (20) samples collected for each matrix. The MS and MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results, and to indicate precision associated with laboratory procedures. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS and MSD corrected for background concentrations. Precision in the laboratory for the chemical analyses is assessed through the analyses of laboratory control samples/ laboratory control sample duplicates (LCS/LCSD), MS/MSD and analytical duplicates. The RPD will be calculated for three or more replicate samples. The equations used to

calculate precision can be found in Section 14.2 of this QAPP. Precision control limits for the Site Characterization are provided in Table B5.

Five percent of the total XRF measurements will be confirmed via fixed laboratory analysis. The fixed laboratory will analyze lead by SW-846 6020. Correlation between field XRF sampling and fixed-laboratory data will be accomplished by calculating the relative percent difference between the two results (field XRF and laboratory data). However, none of the newly generated XRF data will be discarded and no actual pass/fail criteria will be established. The rationale for using all of the newly generated XRF data is due to the fact that the XRF data is being used as a screening tool to broadly delineate the lead contamination. Additionally, because of the difference in moisture contents between the field readings and the laboratory readings, WSP does not expect the results to be equal; dry weight results provided by the laboratory are expected to be higher than the readings taken by the XRF in natural moisture conditions. The XRF will be calibrated to a 1345 ppm lead standard and operates best within the lower range of lead concentrations. It was observed from the previous XRF investigation that at high concentrations (greater than 1295 ppm) the results varied more greatly from the laboratory generated results.

Laboratory precision for groundwater samples will be assessed through the analysis of analytical duplicates (when applicable), LCS/LCSD and MS/MSD. For these laboratory quality control samples, the entire target parameter list will be used for spiking.

Table B5—Site Characterization Precision Control Limits

Constituent	Media	Method Detection Limits (MDL)	Reporting Limits (PQL)	Relative Percent Difference (RPD)
Lead	Soil	0.008 mg/kg	0.10 mg/kg	20
Total suspended solids (TSS)	Groundwater	0.15 mg/L	1.0 mg/L	20
Total phenols	Groundwater	0.05 mg/L	0.0133 mg/L	20
Antimony	Groundwater	0.115 ug/L	1.0 ug/L	20
Arsenic	Groundwater	0.193 ug/L	1.0 ug/L	20
Barium	Groundwater	0.139 ug/L	1.0 ug/L	20
Cadmium	Groundwater	0.130 ug/L	1.0 ug/L	20
Chromium	Groundwater	0.1156 ug/L	1.0 ug/L	20
Copper	Groundwater	1.00 ug/L	0.381 ug/L	20

Table B5 (cont.)—Site Characterization Precision Control Limits

Constituent	Media	Method Detection Limits (MDL)	Reporting Limits (PQL)	Relative Percent Difference (RPD)
Lead	Groundwater	0.061 ug/L	1.00 ug/L	20
Nickel	Groundwater	0.155 ug/L	1 ug/L	20
Zinc	Groundwater	4.099 ug/L	10 ug/L	20
Mercury	Groundwater	0.1 ug/L	0.200 ug/L	20

5.1.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value.

5.1.2.1 Field Accuracy Objectives

Accuracy in the field for the chemical analyses is assessed through adherence to all sample handling, preservation and holding time requirements. In addition, field, trip, and equipment blanks will be collected and analyzed to monitor possible contamination prior to receipt at the laboratory.

5.1.2.2 Laboratory Accuracy Objectives

Laboratory accuracy for the chemical analyses is assessed through the analysis of MS/MSD samples, LCS/LCSD, internal standards or blank spikes, surrogate standards, and method blanks, where applicable. As previously discussed MS/MSD samples are analyzed at a frequency of 5 percent (1 in 20); LCS/LCSD or blank spikes at a frequency of 5 percent (1 in 20); and surrogate standards, where available, are added to every sample analyzed for organic constituents. Appropriate levels for spike and surrogate standards will be added such that a mid-calibration level is present in the sample at the time of analysis, where applicable.

The results of spiked samples will be expressed as percent recovery and will provide information on positive and negative bias. The equation for determination of percent recoveries can be found in Section 14.1 of this QAPP. The accuracy control limits are included in method spiking solutions for the LCS/LCSD and MS/MSD samples.

5.2 QA Objectives for DERA Sampling

5.2.1 Precision

Precision will be measured for the ecological-biological-toxicology analyses through the comparison of data for a specific toxicant that is collected both upstream and downstream of the project location.

5.2.1.1 Field Precision Objectives

Field precision for the ecological-biological-toxicology portion of the project is not applicable as all of the data collected will be analyzed and no duplicate samples will be analyzed.

5.2.1.2 Laboratory Precision Objectives

Laboratory precision for the ecological-biological-toxicology analyses will be conducted by comparing analytical results to a referenced toxicant.

Chemical analysis of soil, surface water and sediments will be used to support the ecological-biological-toxicology results. Precision in the laboratory for the chemical analyses is assessed through the calculation of RPD for three or more replicate samples. The equations used to calculate precision can be found in Section 14.2 of this QAPP. Precision control limits for the DERA sampling are provided in Table B6.

Table B6—DERA Sampling Precision Control Limits

Constituent	Media	Method Detection Limits (MDL)	Reporting Limits (PQL)	Relative Percent Difference (RPD)
pH	Soil	0.1 Std units	0.1 Std units	N/A
TOC	Soil	600 mg/kg	73.37 mg/kg	N/A
Soil Moisture	Soil	0.10%	0.10%	N/A
Water Holding Capacity	Soil	0.10%	0.10%	N/A
Particle Size Analysis	Soil	N/A	N/A	N/A
CEC	Soil	0.05 meq/100g	0.1 meq/100g	47
Antimony	Soil	0.15 mg/kg	0.10 mg/kg	20
Arsenic	Soil	0.013 mg/kg	0.10 mg/kg	20
Barium	Soil	0.009 mg/kg	0.10 mg/kg	20
Cadmium	Soil	0.011 mg/kg	0.10 mg/kg	20

Table B6 (cont.)—DERA Sampling Precision Control Limits

Constituent	Media	Method Detection Limits (MDL)	Reporting Limits (PQL)	Relative Percent Difference (RPD)
Chromium	Soil	0.0166 mg/kg	0.10 mg/kg	20
Copper	Soil	0.047 mg/kg	0.10 mg/kg	20
Lead	Soil	0.008 mg/kg	0.10 mg/kg	20
Nickel	Soil	0.046 mg/kg	0.10 mg/kg	20
Zinc	Soil	0.856 mg/kg	2.0 mg/kg	20
Mercury	Soil	.0330 mg/kg	0.007 mg/kg	20
Antimony	Surface Water	0.115 ug/L	2.0 ug/L	20
Arsenic	Surface Water	0.193 ug/L	1.0 ug/L	20
Barium	Surface Water	0.139 ug/L	1.0 ug/L	20
Cadmium	Surface Water	0.130 ug/L	1.0 ug/L	20
Chromium	Surface Water	0.1156 ug/L	1.0 ug/L	20
Copper	Surface Water	0.381 ug/L	1.00 ug/L	20
Lead	Surface Water	0.061 ug/L	1.00 ug/L	20
Nickel	Surface Water	0.155 ug/L	1 ug/L	20
Zinc	Surface Water	4.099 ug/L	10 ug/L	20
Mercury	Surface Water	0.1 ug/L	0.200 ug/L	20
Antimony	Sediments[2]	0.15 mg/kg	0.10 mg/kg	20
Arsenic	Sediments	0.013 mg/kg	0.10 mg/kg	20
Barium	Sediments	0.009 mg/kg	0.10 mg/kg	20
Cadmium	Sediments	0.011 mg/kg	0.10 mg/kg	20
Chromium	Sediments	0.0166 mg/kg	0.10 mg/kg	20
Copper	Sediments	0.047 mg/kg	0.10 mg/kg	20

Table B6 (cont.)-DERA Sampling Precision Control Limits

Constituent	Media	Method Detection Limits (MDL)	Reporting Limits (PQL)	Relative Percent Difference (RPD)
Lead	Sediments	0.008 mg/kg	0.10 mg/kg	20
Nickel	Sediments	0.046 mg/kg	0.10 mg/kg	20
Zinc	Sediments	0.856 mg/kg	2.0 mg/kg	20
Mercury	Sediments	.0330 mg/kg	0.007 mg/kg	20
Phenols	Sediments	52.5 ug/kg	167 ug/kg	20
Interstitial pore water-Ammonia	Sediments	0.250 mg/l	0.500 mg/l	20
Interstitial pore water-pH	Sediments	0.250 mg/l	0.500 mg/l	5
AVS	Sediments	0.00784605 umoles/g	0.05881 umoles/g	30
TOC	Sediments	113.1 mg/kg	400 mg/kg	20
Particle Size Analysis	Sediments	N/A	N/A	N/A
CEC	Sediments	0.5 meq/100g	0.1 meq/100g	47

5.2.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference value.

5.2.2.1 Field Accuracy Objectives

Accuracy in the field for the ecological-biological-toxicology analyses is not applicable.

5.2.2.2 Laboratory Accuracy Objectives

Laboratory accuracy in the ecological-biological-toxicology analyses will be conducted by comparing results to a referenced toxicant. Laboratory accuracy for the chemical analyses that will be conducted to support the DERA sampling is assessed through the analysis of MS/MSD samples, LCS/LCSD, internal standards and/or blank spikes, surrogate standards, and method blanks. As previously discussed duplicates and MS/MSD samples are analyzed at a frequency of 5 percent (1 in 20); LCS/LCSD or blank spikes at a frequency of 5 percent (1 in 20); and surrogate standards, where applicable, are added to every sample analyzed for organic constituents. Appropriate levels for spike and surrogate standards will be added such that a mid-calibration level is present in the sample at the time of analysis. The results of spiked samples will be expressed as percent recovery and will provide information on positive and negative bias. The equation for determination of percent recoveries can be found in Section 14.1 of this QAPP. Accuracy control limits for the DERA sampling will include all parameters of concern

identified in Table B3 of this QAPP. The accuracy control limits are included in method spiking solutions for the LCS/LCSD and MS/MSD samples.

5.3 Completeness

5.3.1 Definition

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

5.3.2 Field Completeness Objectives

Field completeness is a measure of the number of valid field measurements obtained from all the field measurements taken in the field during the project for each parameter in a given matrix. The equation for completeness is presented in Section 14.3 of this QAPP. The field completeness goal for this project will be greater than 90 percent.

Field completeness for the ecological-biological-toxicology analysis is 100 percent.

5.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the number of useable laboratory analytical results obtained from all the laboratory analytical results requested for each matrix. The equation for completeness is presented in Section 14.3 of this QAPP. The laboratory completeness goal for this project, as it applies to the parameters outlined in Table B3, will be greater than 95 percent.

Laboratory completeness for the ecological-biological-toxicology analysis is 100 percent for any set of samples that are requested to be tested.

5.4 Representativeness

5.4.1 Definition

Representativeness expresses the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

5.4.2 Measures to Ensure Representativeness of Field Data

All proposed field testing and measurement procedures are designed to maximize the goal that the field data will represent the conditions found at the Site. All sampling efforts will be conducted using procedures designed to maximize the goal that the sample be representative of the matrix from which it was taken. The proper execution of field activities will ensure data representativeness. During development of these protocols, consideration was given to previous investigation areas, previous analytical results, operations conducted onsite and physical Site settings. The rationale of the sampling network is discussed in detail in the Workplan.

5.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory will be satisfied by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicate samples.

5.5 Decision Rules

5.5.1 Definition

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

5.5.2 Decision Rule Objectives

The decision rules for the Site Characterization portion of the project are outlined below:

- If XRF soil screening in any grid exceeds 400 mg/kg for lead (human health-based RSL), than step-out screening will continue into the next row of grids. Step-out screening will continue until all measurements are below 400 mg/kg for lead. Field accuracy and precision must adhere to the data quality objectives. Field data completeness will be greater than 90 percent.
- If fixed laboratory soil samples analyzed for lead exceed 400 mg/kg then the extent of contamination has not been delineated and additional investigations may be required. Laboratory accuracy and precision will be within the above outlined criteria (Table B5). Laboratory completeness will be greater than 95 percent.
- If analytical results are Non Detect, then the analytical sensitivity of the project will be assessed. If reporting limits are less than the RSL, then it will be concluded that this area has not been impacted at a level of unacceptable risk and no further action is required.
- The ecological action level for lead in soil has been calculated at 130 mg/kg (Intrinsik Memo to EPA, February, 2014). However, as agreed upon by EPA, this ecological action level will not be used to screen data and no further Site characterization activities will result from an exceedance of the ecological action level.

The decision rules for the DERA portion of the project are outlined below:

- Results from the ecological-biological-toxicology analyses will be included in the addendum to the DERA. Laboratory completeness for the ecological-biological-toxicology analysis is 100 percent for any set of samples that are requested to be tested.
- Results from the chemical analyses for sediment, soil, and surface water will be used to supplement the ecological-biological-toxicology results. Chemical results for soils will not be compared to EcoSSLs. Chemical results will be used to evaluate the statistically significant affects and any potential correlations between ecological-biological-toxicology results from the Ponds and upstream and downstream of Julia Creek.

For this project, samples shall be collected discretely to provide greater indication of contaminated areas and delineate boundaries of contamination.

Decision levels, expressed as RSL and target parameters, for each critical measurement parameter are specified in Table B3.

5.6 Comparability

5.6.1 Definition

Data comparability expresses the confidence with which one data set can be compared to another. All data collection mechanisms are designed to produce comparable data.

5.6.2 Measures to Ensure Comparability of Field Data

Procedures for field measurements are provided in Section 6 of this QAPP and in the SAP included as Appendix B of the Workplan. These procedures will be implemented to ensure that tests performed at the designated locations across the Site are conducted using accepted procedures for the media that will be sampled. In order to ensure comparability of field data for the chemical and ecological-biological-toxicology analyses, all data will be collected in a consistent manner between locations, over time, and will include appropriate QA/QC procedures (i.e., instrument calibration). The previous data was collected and analyzed in a manner that is consistent with the new data to be collected during this investigation. The new data will be compared with previous data collected from the Site. Any limitations on the comparability of test data will be noted and test results will be evaluated on that basis.

5.6.3 Measures to Ensure Comparability of Laboratory Data

Any soil, sediment, surface water, and groundwater samples collected will be analyzed by a laboratory using consistent protocols for holding times, sample preparation, analytical methods, and QC as described in the approved U.S. EPA analytical procedures.

The data will be reduced, reported, and documented consistently throughout the study and consistent with previous data collected for the Site. For example, surface water and sediment quality data will be reported using a consistent set of units throughout the study. Any deviations from established protocols will be noted so that data comparability can be maintained.

5.7 Level of Quality Control Effort

Field blank, trip blank, equipment blank, field duplicate, laboratory duplicate, method blank, laboratory control standard reference materials, MS/MSD, and LCS/LCSD samples will be analyzed to assess the quality of data resulting from the field sampling and analytical programs. Chemical analyses for samples collected during the investigation will be performed using U.S. EPA-approved methods and protocols.

- Field blanks consisting of distilled water will be prepared and analyzed by the laboratory to provide the means to assess the quality of data resulting from the field sampling program.
- Field blanks will only be included with coolers containing aqueous samples. One field blank sample for every 20 samples for each matrix with no more than 2 per day will be collected during the project. Field blanks will be analyzed to detect sample preparation or system contamination during field work activities.

- Trip blanks consisting of distilled water will be prepared and analyzed by the laboratory to provide the means to assess cross-contamination during the transit of samples. Trip blanks will be included in each cooler where soil, sediment and surface water samples will be analyzed for AVS/SEM, total phenols and for toxicity testing. Trip blanks will not be included in coolers where soil, sediment and water will be analyzed for metals and the remaining constituents as indicated on Table B4. Trip blanks will be analyzed to detect sample contamination during transport to the laboratory.
- Equipment blanks will be prepared and analyzed by the laboratory at the rate of one sample per equipment type per day (e.g. one sample for all the trowels, one sample for the groundwater pump, etc.) Laboratory blanks will be analyzed with each run to detect sample preparation, reagent, or system contamination.
- For purposes of quality control, a minimum of one duplicate sample for every 20 samples collected for each matrix. The duplicate will then be analyzed for the same parameters as the associated sample. These replicates will be submitted to the laboratory. Internal laboratory duplicates will be analyzed at the rate of 1 for every 20 samples analyzed.
- Method blank samples are generated within the laboratory and used to assess contamination resulting from laboratory procedures.
- Laboratory QC reference samples will be integrated into the analytical scheme to assess accuracy. All equipment and laboratory QC samples will be analyzed using the same method protocols as regular samples, including all spikes, dilutions, and processing.
- MS samples will be prepared and analyzed by the laboratory at the rate of 1 per 20 samples of the same matrix. Sample matrices will thus be examined to evaluate their effects on the analytical protocols.

The different types of samples that will be collected will be analyzed for the parameters indicated in Table B4. The number of QC samples scheduled for collection is shown on Table B7 below. Coordination between WSP and the laboratories will be emphasized. When the laboratory encounters problems, such as difficult sample matrices, WSP (with U.S. EPA assistance, if required) will provide professional guidance on how to continue without excessive cost increases or compromise of the data quality.

All QC samples will be evaluated based on the CLP National Functional Guidelines for Organic Data Review, CLP National Functional Guidelines for Inorganic Data Review, or method criteria specified in the associated U.S. U.S. EPA-approved methods.

Table B7—Summary of QA/QC Sample Collection and Analysis

Sample type	Analysis	# of Total Samples in Field	QA/QC Sample Type and #	Method
soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	28	Duplicate (2); MS/MSD (2)	EPA 6020/7471
soil	Cation exchange capacity	28	Duplicate (2); MS/MSD (2)	EPA 9081
soil	pH, TOC, water holding capacity,	28	Duplicate (2); MS/MSD (2)	EPA 9045; EPA 9060A; SM 2540 G;
soil	14-day earthworm survival and biomass toxicity test, particle size analysis	7	Quality Control checks will be conducted by the lab. Trip blank (1 per cooler)	EPA 600/3-88-029; ASTMD 422
soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	1 per day per equipment piece	Equipment Blanks for trowels, spoons and trays	EPA 6020
soil	Lead	1 per day	Equipment Blank for Geoprobe drilling rig	EPA 6020
surface water	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	16	Duplicate (2); MS/MSD (2) Field blank (1 per 20; no more than 2 per day)	EPA 6020/7470A
surface water	Chronic toxicity tests using P. promelas (Fathead minnow) & C. dubia (water flea)	10	Quality Control checks will be conducted by the lab. Trip blank (1 per cooler)	EPA-821-R-02-013 Method 1000.0
sediment	Interstitial pore water ammonia; Interstital pore water pH	23	Duplicate (2); MS/MSD (2)	EPA 350.1; EPA 4500-H-B
sediment	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	23	Duplicate (2); MS/MSD (2)	EPA 6020/7471
sediment	10-day toxicity test using H.azteca & C. dilutus	11	Quality Control checks will be conducted by the lab. Trip blank (1 per cooler)	EPA 600/R-99/064/100.1/100.2

Sample type	Analysis	# of Total Samples in Field	QA/QC Sample Type and #	Method
sediment	AV/SEM	23	Duplicate (2); MS/MSD (2) Trip blank (1 per cooler)	EPA 1629/6010/7471
sediment	TOC, particle size, CEC	23	Duplicate (2); MS/MSD (2)	Walkley-Black; ASTM D 422-63; EPA 9081
sediment	Total Phenols	23	Duplicate (2); MS/MSD (2) Trip blank (1 per cooler)	EPA 8270C
sediment	Metals, Total Phenols, AV/SEM	1 per day per piece of equipment	Equipment Blank for hand trowel or dredge and tray	EPA 6020; 8270C; 6010/7471
groundwater	Total and Dissolved Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn); total suspended solids; total phenols	11	Duplicate (1); MS/MSD (1) Field blank (1 per 20; no more than 2 per day)	EPA 6020/7470;EPA 160.2; EPA 8270C
groundwater	Total Metals; total suspended solids, total phenols	1 per day	Equipment Blank for submersible pump	EPA 6020/7470;EPA 160.2; EPA 420.2
Benthic invertebrates	Total metals	If enough organisms available, 1 in 10 samples	Duplicate (2 max)	EPA 6020/7471
Benthic invertebrates	Total metals	1 per day	Field Blank	EPA 6020/7471

6 Sampling Procedures

The sampling procedures to be used during this Site investigation will be consistent for the objectives and decision rules of this project. The sampling locations are presented in Figures B2, B3 and B4. Additionally, Table B4 presents a summary of sampling and analysis for the project and includes the matrices, field and laboratory parameters, sample identification numbers and assigned laboratories.

The sampling procedures for XRF soil sampling are included in the SAP presented in Appendix A of WSP's Workplan, dated March 28, 2014, and WSP SOP # 31 presented in Attachment B of this QAPP.

The procedures for soil, sediment, groundwater and surface water sampling are included in WSP's Workplan, dated March 28, 2014. WSP's SOPs for soil, sediment, groundwater, surface water and benthic invertebrate sampling are also presented in Table B8 and Attachment B of this QAPP. The laboratory SOPs for analysis of the various parameters are included in Attachment C of this QAPP. Table 1, found in WSP SOP #2, presents the required type, size, number of containers, preservative/temperature, holding time and sample collection container for each parameter/analysis.

Table B8—Project Sampling Procedures

EPA Method Number; WSP SOP Number	Matrix	Field Activity/Parameter	Referenced Document
SW-846; WSP SOP # 31	Soil	XRF Sampling	XRF Sampling and Analysis Plan, prepared by WSP, dated November 16, 2009
EPA Method 6020/7471; WSP SOP #11	Soil	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	Sections 3.2 and 3.3.1 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 9045; EPA 9060A; SM 2540G; EPA 9081 WSP SOP #11	Soil	pH, TOC, soil moisture, water holding capacity, CEC	Section 3.2 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA Method 600/3-88-029; ASTM D 422 Atterberg Limit; WSP SOP #11 and 32	Soil	14-day earthworm survival and biomass toxicity test; particle size analysis	Section 3.3.1 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 6020; WSP SOP #24	Soil	Lead	Section 2.3 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA-821-R-02-013 Method 1000.0; WSP SOP #4	Surface Water	Chronic toxicity tests using *P. promelas & C. dubia*	Section 3.5 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
SM 4500 H; SM 2340 B; WSP SOP # 4	Surface Water	pH; wáter hardness	Section 3.5 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA Method 6020/7470; WSP SOP #4	Surface Water	Total and Dissolved Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	Section 3.5 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014

Table B8 (cont.)—Project Sampling Procedures

EPA Method Number; WSP SOP Number	Matrix	Field Activity/Parameter	Referenced Document
EPA Method 6020/7471; WSP SOP #4, 5 and 6	Sediment	Metals (Sb, As, Ba, Cd, Cr, Cu, Pb, Hg, Ni, Zn)	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 1629/6010/7471; WSP SOP #4, 5 and 6	Sediment	AVS/SEM	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
Walkley-Black; ASTM D 422-63; EPA 9081; WSP SOP #4, 5 and 6	Sediment	Total organic carbon , particle size analysis, cation exchange capacity	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 8270C; WSP SOP #4, 5 and 6	Sediment	Total Phenols	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 350.1, SM 4500 H-B; WSP SOP #4, 5, 6, 33	Sediment	Interstitial Pore Water-ammonia; Interstitial Pore Water-pH	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA /600/R-99/064 100.4 and 100.2; WSP SOP #4, 5 and 6	Sediment	H.azteca (28-day survival and growth) C. dilutus (10-day) toxicity tests	Section 3.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 6020/ 7470; SM 2540D; EPA 8270C; WSP SOP #3	Groundwater	Total and dissolved metals; TSS; total phenols	Section 2.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
EPA 6020/7471; ASTM D 2974-87; WSP SOP #34	Benthic invertebrates	Total metals, moisture content	Section 3.6 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
WSP SOP #21	Various	Collection of QA/QC Samples	Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
WSP SOP #19	Various	Equipment Decontamination	Section 4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
WSP SOP #1	Various	Detailed System for Labeling and Numbering all Samples	Section 4.2 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
WSP SOP #20	Various	Sample Packaging and Shipping	Section 4.1 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014
WSP SOP #26	Various	Investigation-Derived Waste	Section 4.4 of the Sampling and Analysis Plan, Appendix B of Workplan dated March 28, 2014

7 Custody Procedures

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

A sample is considered to be in a person's custody if it is in the person's possession, it is in the person's view after being in his or her possession, it was in that person's possession and that person has locked it in the sampling vehicle or office to prevent tampering, or in a designated secure area.

Sample custody is controlled and maintained through the chain-of-custody procedures. Chain-of-custody is the means by which the possession and handling of samples will be tracked from the source (field) to their final disposition, the laboratory.

Sample containers will be preserved at the laboratory before shipment to the Site. Other chain-of-custody supplies, such as shipping containers (coolers), sample labels, custody seals, and ice packs, will also be provided by the laboratory. Sample containers will be new, and certification that they are contaminant-free will be provided by the manufacturer for each lot number.

7.1 Field Custody Procedures

7.1.1 Field Notebook

WSP personnel will maintain a Field Notebook which will be used to record data collection activities including conditions of the Site, issues encountered during sampling activities and any decisions made in the field during the investigation. Entries in the Field Notebook will include as many details as possible so that persons returning to the facility could reconstruct a particular situation without reliance on memory.

Field Notebooks will be bound field survey books and will be stored in the document control center when not in use. Each logbook will be identified by the project-specific document number and will contain, at a minimum, the following information:

- Person to whom the logbook is assigned.
- Logbook number
- Date, arrival time onsite, weather/conditions
- Personnel onsite
- Time of departure
- Page numbers and signature on each page

7.1.2 Sample Collection Data

Samples will be collected in accordance with sampling procedures outlined in the SAP (Appendix B of the Workplan). The equipment used to collect the sample will be noted in the Field Logbook along with the following information:

- Sample identification
- Time of collection
- Location information or GPS coordinates
- Depth of sample
- Number of containers per sample

All sample containers will be identified with sample tags. The sample tags will be completed with waterproof markers and include the following information:

- Sample number(s)
- Sample location
- Date and time of collection
- Type of analysis (method number)

The protocol for specific sample number and other sample designations are presented in WSP SOP #20.

7.1.3 Custody of Samples

WSP personnel will be responsible for the custody of samples from the time they are collected until they are transferred to the sample shipper for delivery to the laboratory. An example chain-of-custody form is included as Attachment E. The chain-of-custody procedures for the sampling activities will include the following:

1. The sample will be placed in a thermal shipping container with ice and otherwise be preserved as required. The container will remain within the sampler's view or locked in the sampling vehicle for temporary storage and transport to the sample staging area. On arrival at the sample staging area, the sampler will fill out chain-of-custody form(s) to account for each sample. Blanks and QC samples may be sent with each group of samples. Field duplicate samples will be sent as "blind duplicates" (e.g., the laboratory will be unable to distinguish a field duplicate from any other sample). The identity of the field duplicate sample will be recorded in the WSP Field Notebook. The WSP copy of the chain-of-custody form will be retained as a permanent record in the project files.
2. The location of sampling points in the field will be documented in accordance with the procedures described in Sections 2 and 3 and Table 1 of the Work Plan to Address Outstanding DERA Issues (March 28, 2014). These procedures are designed to ensure that sampling points for the investigations can be relocated in the future and are accurately represented in subsequent reports.
3. Following sample collection, the sampler will record the date and time and sign in the appropriate block of the form to relinquish custody. The sample carrier will sign and record the date and time in the appropriate box to accept custody. The original chain-of-custody record will be placed inside the shipping package. The shipping container will then be sealed with custody seals and

secured with strapping tape. The seals will indicate if the samples have been tampered with during transport to the laboratory. The sample shipping receipt will be retained as part of the permanent chain-of-custody documentation. A sample tag will be attached to each sample container.

4. Samples will be transported to the laboratory on the same day as field collection or shipped via overnight carrier.
5. The laboratory will assume custody of the samples on receipt. A designated laboratory sample coordinator will record the date and time and sign the chain-of-custody form on receipt.
6. The sample coordinator will immediately inspect the shipment for damage and completeness and report any problems to the WSP Project QAOs. The laboratory sample coordinator will then complete the appropriate lab tracking forms and logs.

7.2 Laboratory Custody Procedures

The laboratory sample coordinator is responsible for custody of the samples from the time of sample receipt to the time of discard. Each laboratory has established procedures for sample handling and tracking including transfer within the laboratory, sample storage, extract or digestate disposal and analytical data generation and maintenance.

Many of the parameters that will be analyzed for this project require special chemical preservatives and holding samples at cool temperatures (4°C). Each laboratory will be responsible for providing pre-preserved containers. All containers will be brand new from the manufacturer and documentation for each lot number will ensure that they are free of contamination. Holding conditions of the samples will be checked and recorded. Preservation of the samples is verified by the laboratory sample coordinator when the samples are received.

7.3 Final Evidence Files

Chain-of-custody protocols also involve maintaining a project evidence file. The final evidence file will be the central repository for all documents which constitute evidence relevant to sampling and analysis activities as described in this QAPP. WSP's administrative staff will coordinate with the WSP Project Director, Richard E. Freudenberger, to act as custodians of the evidence file and will maintain the contents of evidence files for the RFI, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports and data reviews in a secured, limited access area and under custody of the WSP Project Director.

The final evidence file will include at a minimum:

- field logbooks
- field data and data deliverables
- photographs
- drawings
- laboratory data deliverables
- data validation reports

- data assessment reports
- progress reports, QA reports, interim project reports, etc.
- all custody documentation (tags, forms, airbills, etc.)

The project evidence file will be maintained at WSP's San Jose, California, office in a secure, limited access area for 2 years after the completion of the project. After 2 years, the entire project evidence file, consisting of the items listed above, will be placed in cardboard boxes that are each assigned a WSP document control number. The contents of each box and its assigned document control number are recorded by WSP personnel and this information is maintained at the San Jose office. The boxes containing the project file are then transported to an offsite storage area located in San Jose, California, where they will be maintained for an additional 3 years. The offsite storage area is a high-security, fireproof, climate-controlled records center. Using the WSP document control number, WSP can retrieve the project file from the storage center within 48 hours of a request. The file will be offered to U.S. EPA prior to disposal by WSP.

8 Calibration Procedures and Frequency

The calibration procedures and schedules established for the measuring instruments apply to all field and laboratory equipment. Specific calibration procedures for each instrument to be used are detailed in the following sections.

8.1 Field Instrument Calibration

Field parameters that will be measured include: XRF, pH, temperature, turbidity, dissolved oxygen, specific conductivity, and redox potential. WSP will use the S1 Titan XRF device and a YSI 556 Water Quality Checker for all field measurements except turbidity. Turbidity will be analyzed using a LaMotte 2020e Turbidity Meter.

Field instruments will be calibrated in accordance with the manufacturer's instruction manuals which are included as Attachment B of the SAP. At a minimum, field instruments will be calibrated before initial use and continuing through the project on a daily basis. All the calibration procedures performed will be documented in the field logbook and will include the date/time of calibration, name of person performing the calibration, reference standard used, temperature at which readings were taken and the readings. Multiple readings on one sample or standard, as well as readings on replicate samples, will likewise be documented.

8.2 Laboratory Instrument Calibration

Calibration procedures for each laboratory will be specific to the instruments used and the analyses performed by each laboratory. Pace Analytical, NAS, RTI and CQM's QA/QC manuals are presented in Attachment C. The laboratory SOPs describe calibration procedures, frequency, acceptance criteria and conditions that require recalibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution.

The specific calibration methods used by each laboratory for this project are described below.

8.2.1 Pace Analytical

Pace Analytical will be performing the following analyses: soil analyses include metals (including antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, and zinc); pH; organic matter; soil moisture; water holding capacity; CEC. The following water analyses will be conducted by Pace Analytical: pH; water hardness; interstitial pore water ammonia and interstitial pore water pH; total and dissolved metals in water including antimony, arsenic, barium, cadmium, chromium, copper, lead, mercury, nickel, and zinc. Pace Analytical will analyze sediment samples for total metals; phenols; AVS/SEM; TOC, particle size, and CEC. Pace Analytical will also conduct the benthic invertebrate tissue sampling for metals and moisture content.

All equipment and instruments used at Pace Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in

appropriate reference files. Records are maintained to provide traceability of reference materials. A brief description of the calibration procedures for the major laboratory equipment and instruments are described below:

- ICP-MS

Each element of interest is calibrated by typically using one blank to three standards. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard is analyzed with every ten samples. Tune measurement procedures are provided in Pace Analytical's Quality Assurance Manual.

- Gas Chromatograph/Mass Spectrometry (GC/MS) Systems

All GC/MS instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. Compounds selected as system performance check compounds must show a method-specified response factor in order for the calibration to be considered valid. Calibration check compounds must also meet method specifications for percent difference from the multipoint calibration. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene for volatile organic chemical analysis, or decafluorotriphenylphosphine for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed.

- Atomic Absorption Spectrophotometers

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be "dropped" from the resulting calibration curve.

- Gas Chromatographs (GC) and High Performance Liquid Chromatographs (HPLC)

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).

- UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods include total phenolics, phosphates, surfactants and tannin-lignin.

- Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the SOP for Checking Pipet Calibration. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

- Other Instruments

Calibration for the TOC, total organic halogen, and other instruments is performed following manufacturer's recommendations and applicable SOPs.

An evaluation of the effect of this defect on previous calibrations or tests is made and documented appropriately.

Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. For NELAP accredited methods, the concentration of calibration verification standards are varied within the calibration range periodically.

8.2.2 Northwestern Aquatic Sciences

NAS will be conducting the 14-day earthworm survival and biomass toxicity test in soil; Fathead minnow (P. promelas) and water flea (C. dubia) chronic toxicity test in water; and the 28-day toxicity tests using H.azteca and the 10-day toxicity test using C.dilutus in sediment. Instruments used by NAS during all testing procedures include pH/temperature meters, dessicator, microbalance, light box, drying oven, autopipetter, light meter, dissecting microscope and a water purification system. All equipment is properly and frequently calibrated to ensure accurate and reliable measurements. All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before putting into use and on a continuing basis. Calibration frequency and procedures for balances, pH meters, thermometers, and other support equipment are included as part of the procedure manual and/or in standard operating procedures.

Wherever applicable, calibration of analytical support equipment and instruments is traceable to national standards of measurement. Reference standards of measurement (such as Class S or equivalent weights or traceable thermometers) are used for calibration only. Reference standards are subjected to in-service checks between calibrations and verifications. NAS maintains a contract with Quality Control Services, Portland, Oregon for regular calibration of reference thermometers (every three years), Class S weights, and laboratory balances (annually). Standards and other reagents are labeled when received. The label includes receipt date, date opened, and expiration date. Normally reagents expire one year after opening unless otherwise indicated.

Reference toxicants are weighed using an analytical balance accurate to the nearest 0.1 mg. Balances are checked before and after each use using weights traceable to NIST standards. Balances are serviced annually by QC Services, Portland, Oregon. Effluent and reference toxicant dilutions are made using Class A volumetric flasks and pipettes and graduated cylinders.

8.2.3 RTI

Instruments and support equipment used by RTI are calibrated in accordance with the referenced analytical methods. Details of the calibration criteria are contained in the QC Table for metals included in the laboratory SOPs (Appendix C). Where available, the calibration standards and instruments are checked through the use of second source standards. Certified titrants do not require verification provided they are used within the manufacturer specified expiration date. Unless stated otherwise in the appropriate SOP, the lowest standard in the calibration curve must be at or below the PQL for that analyte.

8.2.4 CQM

CQM will be particle size analysis for soils. All equipment and instruments used at CQM are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials. A brief description of the calibration procedures for the main laboratory equipment used for the particle size analysis procedure (scales, sieve, hygrometer and air dispersion instruments) is included in CQM's SOP included in Attachment C.

9 Analytical Procedures

Soil, surface water, groundwater and sediment samples will be collected from the Quemetco facility and sent to four laboratories for analysis. The laboratories used for this project include Pace, NAS, RTI and CQM. Specific laboratory responsibilities are described in Section 4.4 of this QAPP. Specific QA criteria for all analytical procedures are included in Table B7.

9.1 Field Analytical Procedures

Field analytical procedures for this project are presented in Table B9. Field measurements will be obtained in accordance with the manufacturer's standard operating procedures for the respective instruments. XRF sampling will be conducted in the Surficial Zone and Subsurface Zone soils at the proposed locations. Specific field sampling procedures are included in the XRF Sampling and Analysis Plan, dated March 28, 2014 (Appendix B of the Workplan) and referenced in Attachment B of this QAPP.

Surface and groundwater field measurements will include pH, dissolved oxygen, temperature, specific conductivity and redox potential. Water samples will be collected in a polyethylene cup and the probe of one instrument at a time will be inserted in the cup. Readings will be taken after the instrument stabilizes and recorded in the Field Notebook.

Sediment field measurements will include pH and visual observations including color, texture, and presence of oil sheens, macrophites or animals. Sediment samples will be collected with a corer, Ponar or Eckman dredge and placed in a polyethylene cup for field observations. Readings and observations will be recorded in the Field Notebook.

Field sampling equipment will be decontaminated between each sample point. Specific field sampling procedures for groundwater, sediments and surface water are included in Sections 2.4.4, 3.4.1 and 3.5.1 of the SAP presented as Appendix B of the Workplan (March 28, 2014). Field sampling procedures are also provided in Table B9 and Attachment B of the QAPP.

Table B9—Summary of Field Analytical Procedures

Matrix	Field Analytical Procedure	Parameters Analyzed	SOP
Soil	XRF	Lead	SW-846; WSP #31
Sediment	Hand trowel, Ponar or Eckman Dredge	pH and visual observations of color, texture, presence of oil sheens, macrophites or animals	WSP SOP #4, 5, 6
Surface Water	Polyethylene Cup	pH, turbidity, dissolved oxygen, temperature, specific conductivity, redox potential	WSP #4
Groundwater	Polyethylene Cup	pH, turbidity, dissolved oxygen, temperature, specific conductivity, redox potential	WSP #25

9.2 Laboratory Analytical Procedures

The laboratories named above will implement the project required analyses identified in Table B4 and in accordance with the SOPs included in Attachment C of this QAPP. Laboratory analytical procedures for this project will include soil, sediments, groundwater, surface water and benthic invertebrates. Each matrix will be analyzed for both chemical and ecological-biological-toxicology parameters as described in Table B4. Specific SOPs for collecting each sample is presented in Table B8 and Attachment B of this QAPP. The SOPs for each laboratory are presented in Attachment C of this QAPP.

9.2.1 Project Target Compounds and Detection Limits

A complete listing of project target compounds and project action levels for each analyte group is listed in Tables B2 and B3 and can be found in Section 3.4.1 of this QAPP.

9.2.2 List of Associated Quality Control Samples

Section 5.7 and Table B7 of this QAPP contains a complete listing of the associated QC samples for each parameter and matrix. Sample preparation methods are outlined in the laboratory SOPs included in Attachment C of this QAPP.

10 Internal Quality Control Checks

10.1 Field Quality Control Checks

QC procedures for XRF, chemical, and ecological-biological-toxicology analysis for the project specific parameters include calibrations as described in Section 6.1 of the QAPP. Measuring duplicate samples and checking the reproducibility of the measurements will be completed by taking multiple readings on a single sample or reference standard. The QC criteria for each field measurement are provided in WSP's SOPs presented in Attachment B of this QAPP. Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection of the QC samples will be in accordance with the applicable SOPs discussed in Section 5.7 and at the frequency indicated in Table B7 of this QAPP.

10.2 Laboratory Quality Control Checks

Each laboratory for this project has a QC program it uses to ensure the reliability and validity of the analysis performed at the laboratory. All analytical procedures are documented in writing as laboratory SOPs or in respective EPA-approved methods. Each laboratory's QC SOPs are included in Attachment C of this QAPP. The internal quality control checks might differ slightly for each individual procedure but in general the QC requirements include the following:

- Method blanks
- Reagent/preparation/calibration blanks (applicable to inorganic analysis)
- Instrument blanks
- MS/MSDs
- Surrogate spikes
- Analytical spikes
- Field duplicates
- Laboratory duplicates
- Laboratory control standards
- Internal standard for ICP/MS and gas chromatography/mass spectrometry(GC/MS) analysis; control limits
- Mass tuning for GC/MS analysis

All data obtained will be properly recorded. The data package will include a full deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. Any samples analyzed in non-conformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume and appropriate hold times are met.

For XRF verification and vertical soil samples analyzed in the laboratory, a Level IV data package will be obtained. This is due to the fact that the soil samples will be analyzed to verify the extent of lead contamination and verify field measurements and will only be used as a screening tool.

All other chemical analysis, including the phenol parameter isolated in EPA Method 8270C, will include a full Contract Laboratory Program (CLP)-type deliverable package capable of allowing the recipient to reconstruct QC information and compare it to QC criteria. The data package for the chemical analysis is sometimes referred to as a Level IV package and includes all raw data applicable to the samples analyzed. The raw data includes chromatograms and quantitation reports for tuning (for GC/MS analyses), calibration standards, field samples and quality control samples including blanks, spikes, duplicate analyses, and laboratory control samples.

In a similar fashion, ecological-biological-toxicology standard reports include copies of all raw data for the toxicity tests including endpoint data and water quality data, as well as all raw data for reference toxicant tests conducted with the toxicity test, including control charts to evaluate the reference toxicant test results. An outside reviewer will be able to review and validate the data with the information included in each report. The ecological-biological-toxicology reports could therefore be considered "Level IV-type deliverable packages"; however, CLP does not apply to ecological-biological-toxicology testing.

Any samples analyzed in nonconformance with the QC criteria will be reanalyzed by the laboratory, if sufficient volume is available. It is expected that sufficient volumes/weights of samples will be collected to allow for reanalysis when necessary.

11 Data Reduction, Validation and Reporting

Samples will be collected by WSP personnel and sent by overnight carrier with the proper chain-of-custody documentation to the specified laboratory indicated in Table B4.

11.1 Data Reduction

11.1.1 Field Data Reduction Procedures

All field data will be written into field log books immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, it can be determined whether any errors have been made by the field crew.

11.1.2 Laboratory Data Reduction Procedures

WSP and each laboratory will attempt to minimize manual data entry through the use of personal computers and software packages. Each laboratory will supply "CLP-like" level IV data packages, where specified, that include all raw data to WSP. Also, analytical data results will be supplied electronically. Laboratory data will be reduced by each laboratory by direct output of data, as necessary. It is not expected that data from the direct reading instruments will require data reduction.

11.2 Data Validation

11.2.1 Procedures Used to Validate Field Data

Validation of the analytical data obtained in the field is the responsibility of the Field Manager. One hundred percent of the field data will be validated. The procedures to evaluate field data for this project include checking for transcription errors, ensuring all field equipment has been calibrated and decontaminated and review of field log books.

11.2.2 Procedures Used to Validate Laboratory Data

The fixed laboratory data will be validated in accordance with the U.S. EPA National Functional Guidelines, the analytical methods, and data quality objectives specified in this QAPP. Validation will be conducted on a "CLP-like" Level IV data package.

The laboratories will produce data reports for all chemical and ecological-biological-toxicology analytical results that allow for validation per the analytical methods indicated on Table B4.

For the ecological-biological-toxicology analytical results, all data entry from bench sheets will be compared against printouts from data analyses software and from all software printouts against the report data. All entries are checked for transcription errors and corrected. Ecological-biological-toxicology data is checked to verify that controls passed acceptability criteria and that reference toxicant

test results were within laboratory control chart limits. Ecological-biological-toxicology data will be validated by the laboratory.

Based on the review of the chemical analytical data, a validation report will be prepared. The report will consist of a general introduction section, followed by qualifying statements that should be taken into consideration for the analytical results to be best used. Based on the validation, qualifier codes will be placed next to specific sample results on the data summary table(s). These qualifier codes will serve as an indication of the qualitative and quantitative reliability of the reported analytical results. During the course of the validation, a support documentation package will be prepared that will include backup information to support all qualifying statements presented. The data reporting packages will be reviewed thoroughly by the Third-Party Data Validator.

If field or laboratory data is determined to be unusable, corrective action will be implemented as outlined in Section 15.0 of this document.

11.3 Data Reporting

11.3.1 Field Data Reporting

Field data reporting shall be conducted principally through the transmission of report sheets containing tabulated results of all measurements made in the field and documentation of all field calibration activities.

Additionally, the documentation of sample collection will include the use of bound field logbooks in which all information on sample collection will be entered in indelible ink. Appropriate information will be entered to reconstruct the sampling event, including Site name (top of each page), sample identification, brief description of sample, date and time of collection, sampling method, field measurements and observations, and sampler's initials and date on the bottom of each page.

11.3.2 Laboratory Data Reporting

Analytical data for the chemical analyses will be summarized in tabular form with sample number, sample matrix description, parameters analyzed and their corresponding detected concentrations, analytical qualifiers where appropriate, and the method detection limit. Each fixed laboratory will be required to provide WSP with "CLP-type" Level IV data packages for all chemical analyses. The laboratories will provide all the necessary information for validation as detailed in Section 8.2. CLP Standard Reporting Forms for all data reporting will be used.

As specified in Section 8.2, CLP reporting is not generated for the ecological-biological-toxicology analyses.

The results from the sampling activities will be incorporated into reports as data tables and maps showing sampling locations and analytical concentrations.

WSP will implement a rigorous data control program that will ensure that all documents are accounted for following completion of the investigation. Accountable documents include items such as logbooks, field data records, laboratory data packages, photographs, computer disks, and reports. The Project Director will be responsible for maintaining a central file in which all documents will be inventoried.

11.3.3 Data Acquisition Requirements and Data Quality Management

Data obtained from non-measurement sources will include reference documents and review of previous data collected for the Site.

12 Performance and Systems Audits and Frequency

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the Workplan and this QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

12.1 Field Performance and Systems Audits

12.1.1 Internal Field Audits

Internal audits of field activities including sampling and field measurements will be conducted by the WSP Field Manager and the WSP Project QAO. These audits will verify that all established procedures are being followed.

12.1.1.1 Internal Field Audit Frequency

Internal field audits will be conducted at least once at the beginning of a sampling event. If equipment or sampling protocols have been significantly modified, an additional internal audit will occur before samples are collected for analysis.

12.1.1.2 Internal Field Audit Procedures

The audits will include examination of field sampling records, field instrument operating records, sample collection procedures, handling and packaging in compliance with the established procedures, maintenance of QA procedures, chain-of-custody records, and field notebook entries. The audits will involve review of field measurement records, instrumentation calibration records, and sample documentation. Follow-up audits will be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the investigation.

12.1.2 External Field Audits

External field audits may be conducted by the U.S. EPA Project Manager.

12.1.2.1 External Field Audit Frequency

External field audits may be conducted any time during the investigation. The U.S EPA Project Manager shall contact the Quemetco Project Manager, Mr. Gerry Manley, to schedule the external audit and approve access to the site. These audits are at the discretion of U.S. EPA.

12.1.2.2 External Field Audit Process

External field audits will be conducted according to the planned sampling activities presented in this QAPP. The external field audit process can include (but not be limited to): sampling equipment

decontamination procedures, sample bottle preparation procedures, sampling procedures, examination of field sampling, review of Site safety plans, sample vessel cleanliness, QA procedures, procedures for verification of field duplicates, sample preservation, and sample preparation for shipment. At a minimum, the WSP Field Manager will be present during the external audit.

12.2 Laboratory Performance and Systems Audits

12.2.1 Internal Laboratory Audits

The internal laboratory audits will be conducted by the Project QAO or a designated representative of the Project QAO.

12.2.1.1 Internal Laboratory Audit Frequency

The internal system audits will be done on an annual basis.

12.2.1.2 Internal Laboratory Audit Procedures

The internal laboratory audits will include an examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, instrument operating records, and mandatory equipment maintenance.

The internal laboratory audits will involve preparing blind QC samples and submitting them along with project samples to each laboratory for analysis throughout the project. The Project QAO or Third-Party Validator will evaluate the analytical results of these blind performance samples to ensure that the laboratories maintain acceptable QC performance.

12.2.2 External Laboratory Audits

An external audit will be conducted as required, by appropriate QA staff of the Waste, Pesticides and Toxics Division, U.S. EPA Region 5.

12.2.2.1 External Laboratory Audit Frequency

An external audit may be conducted at least once prior to the initiation of the sampling event and analysis activities. These audits shall be scheduled with the Laboratory Manager and will be at the discretion of the U.S. EPA. At the request of the Laboratory Mangers and U.S. EPA, the WSP Project QAO may attend these audits.

12.2.2.2 Overview of the External Laboratory Audit Process

External audits may include any or all of the following: review of laboratory analytical procedures, laboratory onsite visits, and/or submission of performance evaluation samples to the laboratory for analysis.

Failure of any or all audit procedures chosen can lead to laboratory disqualification, and the requirement that another suitable laboratory be chosen.

An external onsite review can consist of: sample receipt procedures, custody and sample security and log in procedures, sample through-put tracking procedure, review of instrument calibration records, instrument logs and statistics (number and type), review of QA procedures, log books, sample prep procedures, sample analytical SOP review, instrument (normal or extended quantitation report) reviews, personnel interviews, review of deadlines and glassware prep, and a close-out to offer potential corrective action.

It is common practice when conducting an external laboratory audit to review one or more data packages from sample lots recently analyzed by the laboratory.

This review will most likely include, but not be limited to;

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

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

13 Preventative Maintenance

13.1 Field Instrument Preventative Maintenance

The field equipment for this project includes an XRF field screening system, Geoprobe equipment, Ponar and/or Eckman Dredge, sediment testing equipment, water chemistry testing equipment, benthic invertebrate nets, and ecological-biological-toxicology sampling equipment.

Specific preventative maintenance procedures to be followed for the XRF, Geoprobe and Ponar and/or Eckman Dredge, and the benthic invertebrate, sediment and water testing equipment are based on those recommended by the manufacturer. Field instruments will be checked and calibrated daily before use. Calibration checks will be documented in the Field Notebooks.

The maintenance schedule and trouble-shooting procedures for field instruments will be conducted according to manufacturer specifications. Critical spare parts, such as batteries, will be kept onsite to reduce potential downtime. Backup instruments and equipment will be available onsite or within 1-day shipment to avoid delays in the field schedule.

13.2 Laboratory Instrument Preventative Maintenance

A routine preventative maintenance program is conducted by each laboratory to minimize the occurrence of instrument failure and other system malfunctions. Designated laboratory employees regularly perform routine scheduled maintenance and schedule necessary repair of all instruments. All maintenance that is performed is documented in the laboratory's operating record. All laboratory instruments are maintained in accordance with manufacturer's specifications.

13.3 Inspection/Acceptance Requirements of Supplies and Consumables

For this project, all supplies and consumables will be inspected upon receipt from the vendor and any discrepancies will be noted in the Field Notebook. If necessary, contaminated or incorrectly shipped supplies will be returned to the vendor or sent offsite for disposal, if deemed inadequate for the planned sampling activities.

14 Specific Routine Procedures Used to Evaluate Data Precision, Accuracy and Completeness

The purpose of this section is to indicate the methods by which it will be ensured that the data collected for this investigation is in line with the data quality objectives for the Site. Factors considered in this assessment include, but are not limited to:

- Expansion of chemical data to further delineate contamination at the Site. The parameters evaluated are known contaminants identified in the 2006 ERA.
- Sensitivity of analytical program so that conclusions can be drawn from the analytical results.

Analytical data quality will be assessed to determine if the objectives of the investigation have been met. In addition, the data will be reviewed for indications of interferences to results caused by sample matrices, cross contamination during sampling, cross contamination in the laboratory, and sample preservation and storage anomalies (i.e. samples holding time or analytical instrument problems).

14.1 Accuracy Assessment

14.1.1 Accuracy for Chemical Analysis

Accuracy for the chemical analysis (metals and phenols) will be evaluated based on the results of the spike samples. The percent recovery (%R) for a spiked sample is calculated using the following equation:

$$\%R = \frac{\text{Amount in Spiked Sample - Amount in Sample X 100}}{\text{Known Amount Added}}$$

14.1.2 Accuracy for Ecological-Biological-Toxicology Analysis

Accuracy criteria are not applicable to toxicity testing endpoints because there are no standard organism responses against which to compare test results. One method of testing accuracy in toxicity testing will be to compare results to a referenced toxicant.

14.2 Precision Assessment

14.2.1 Precision for Chemical Analysis

Precision in the chemical analysis can be determined by the measurement of duplicate samples. Duplicate samples may consist of MS/MSD, LCS/LCSD and analytical duplicates. Evaluation of the duplicate results allows the analyst to determine the precision of the preparation and analytical techniques associated with the sample. The RPD between the samples is calculated using the following equation:

$$RPD = \frac{\text{Amount in Sample 1 - Amount in Sample 2 X 100}}{\text{0.5(Amount in Sample 1 + Amount in Sample 2)}}$$

14.2.2 Precision for Ecological-Biological-Toxicology Analysis

A standard toxicity test automatically implies an acceptable degree of precision at both the intra- and inter-laboratory level. Intra-laboratory precision can be determined on an on-going basis through the use of control charts to evaluate reference toxicant tests.

14.3 Completeness Assessment

14.3.1 Completeness for Chemical Analysis

Completeness for the chemical analysis is the ratio of the number of valid sample results to the total number of samples analyzed for a specific matrix and analytical method. The percent completeness is calculated by the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements) X 100}}{\text{(number of measurements planned)}}$$

14.3.2 Completeness for Ecological-Biological-Toxicology Analysis

The goal is for 100 percent completeness for any set of samples that are requested to be tested.

14.4 Assessment of Data

The field and laboratory data collected during this investigation will be used to further delineate the extent of contamination at the Site. The QC results associated with each analytical parameter for each matrix will be compared to the objectives presented in Section 3 and Tables B3 and B4 of this QAPP.

In addition, the data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific basis. This assessment will be performed by the WSP Project Manager or his designee. The results will be presented and discussed in detail in the investigation report. Factors to be considered in the assessment of field and laboratory data will include the following:

- Were all samples obtained using the methodologies and SOPs proposes in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were all samples obtained from all proposed sampling locations and depths?
- Are there any analytical results not expected to be present at the facility?

- Were all field and laboratory data validated in accordance with the validation protocols, including project-specific QC objectives, outlined in the QAPP?
- Were any data sets found unuseable?
- Were any data sets found to be useable for limited purposes ("J" flag)?
- What affect do qualifiers applied to the data affect the project decision rules?
- Has sufficient data been generated to support the project decision rules?
- Can valid conclusions be drawn for all matrices under investigation?
- Were all issues requiring corrective actions fully resolved?
- Were the project-specific decision rules used as proposed during the investigation?
- If proposed procedures were not used, has the affect been fully discussed?
- Any remaining data gaps?
- Based on findings of investigation, were all original project objectives met?

15 Corrective Action

Corrective action is the process of identifying, recommending, approving and implementing measures to counter unacceptable procedures or out of quality control performance which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation and data assessment. All corrective action proposed and implemented should be documented in the regular QA reports to management. Corrective action should only be implemented after approval by Richard Freudenberger, WSP's project director.

For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying Mr. Freudenberger, who in turn will notify Jennifer Dodds, U.S. EPA's project manager. If the problem is analytical in nature, information on these problems will be promptly communicated to the U.S. EPA, Quality Assurance Section. Implementation of corrective action will be confirmed in writing through the same channels.

Any nonconformance with the established quality control procedures in the QAPP or Workplan will be identified and corrected in accordance with the QAPP. Mr. Freudenberger, or his designee, will issue a nonconformance report for each nonconformance condition.

15.1 Field Corrective Action

Corrective action in the field can be needed when the sample network is changed (i.e., more/less samples, sampling locations other than those specified in the QAPP, etc.), sampling procedures and/or field analytical procedures require modification, etc. due to unexpected conditions. In general, WSP's field team leader, project director, health and safety officer or QAO may identify the need for corrective action. The field staff, in consultation with the field team leader, will recommend a corrective action. WSP's project director will approve the corrective measure which will be implemented by the field team. It will be the responsibility of the WSP project director to ensure the corrective measure has been implemented.

If the corrective action will supplement the existing sampling plan (i.e., additional soil borings) using existing and approved procedures in the QAPP, the corrective action approved by the field team leader will be documented. If corrective actions resulting in less samples (or analytical fractions), alternate locations, etc. which may cause project QA objectives not to be achieved, it will be necessary that all levels of project management, including Gerry Manley from RSR, and Jennifer Dodds, the U.S. EPA project manager, concur with the proposed action.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The QAO, Amy Romano, will identify deficiencies and recommend corrective actions to the Quemetco project manager, Gerry Manley. Implementation of corrective actions will be performed by the field manager, Allen Waldman. Corrective action will be documented in QA reports to the entire project management.

Corrective actions will be implemented and documented in the field record book. No staff member will initiate corrective action without prior communication of findings through the proper channels. If corrective actions are insufficient, work may be stopped by the U.S. EPA project manager.

If at any time a corrective action issue is identified which directly impacts the data quality objectives, the U.S. EPA project manager will be notified immediately.

15.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the laboratory Quality Control Coordinator to approve the implementation of corrective action. Conditions that may warrant corrective action include dilution of samples, additional sample extract cleanup, and automatic re-injection/reanalysis when certain quality control criteria are not met, etc.

The bench chemist will identify the need for corrective action. The laboratory manager, will approve the required corrective action to be implemented by the laboratory staff. The laboratory QA manager will ensure implementation and documentation of the corrective action. If the nonconformance causes project objectives not to be achieved, it will be necessary to inform all levels of project management including the U.S. EPA Project Manager to concur with the corrective action.

These corrective actions are performed prior to release of the data from the laboratory. The corrective action will be documented in both the laboratory's corrective action log (signed by analyst, section leader and quality control coordinator), and the narrative data report sent from the laboratory to the WSP data validator. If corrective action does not rectify the situation, the laboratory will contact WSP's project director.

15.3 Corrective Action During Data Validation and Data Assessment

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective action may include re-sampling by the field team or reinjection/reanalysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.) When the Third-Party Data Validator identifies a corrective action situation, it is WSP's project manager who will be responsible for approving the implementation of corrective action, including re-sampling, during data assessment. All corrective actions of this type will be documented by WSP's QAO and project director.

16 Quality Assurance Reports to Management

The deliverable associated with the tasks identified in the Workplan will contain a separate QA section in which data quality information collected during the task is summarized. This report will be the responsibility of the WSP Project Manager, or his designee and will include a section by the Project QAO, on the accuracy, precision, and completeness of the data and any corrective action needed or taken during the project.

16.1 Contents of Project Quality Assurance Reports

The QA report will contain, at a minimum and on a routine basis the following elements:

- Summary of corrective actions that were implemented and immediate results on the project.
- If applicable, updates on training provided, changes in key personnel, anticipated problems in the field or laboratory will be reported.
- Detailed reference to QAPP modifications, if necessary.

The final QA report will be prepared in written, final format by WSP project manager or his designees. In the event of an emergency, or in the case it is essential to implement corrective actions immediately, QA reports can be made by telephone to the appropriate individuals identified on the Project Organization Chart.

16.2 Frequency of Quality Assurance Reports

The QA Reports will be prepared on a monthly basis and will be delivered to all recipients by the end of the first full week of the month. The reports will continue without interruption, until this investigation has been completed.

16.3 Individuals Receiving/Reviewing Quality Assurance Reports

The U.S. EPA Project Manager and Quemetco Project Manager will receive copies of the final QA report.

17 References

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U.S. EPA. November 1986. Test Methods for Evaluating Solid Waste, Third Edition SW-846. Prepared by the Office of Solid Waste and Emergency Response.

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Figures

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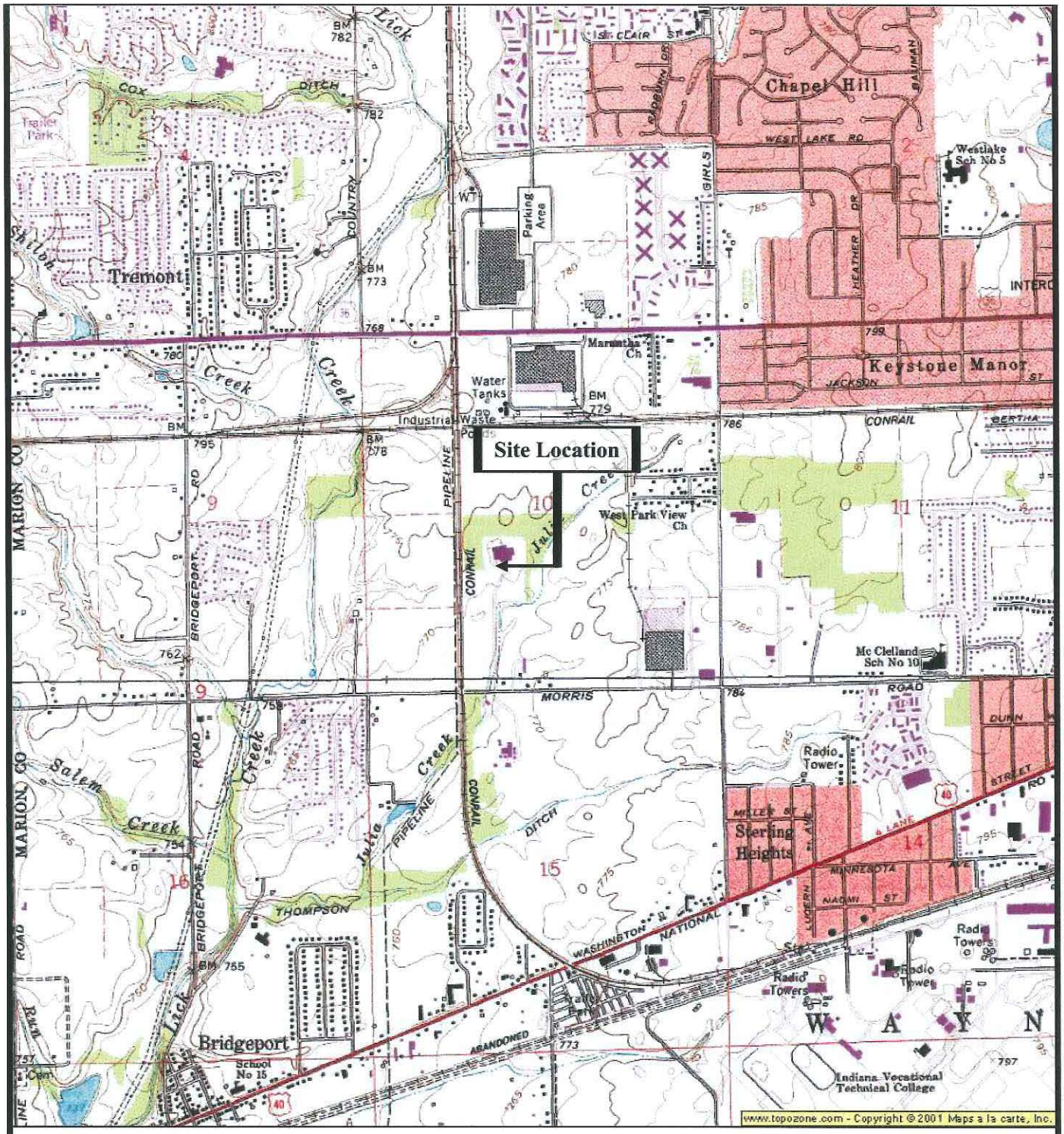
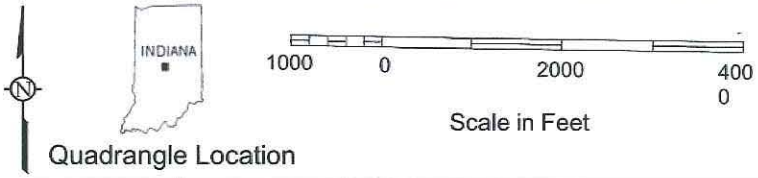


Reference
7.5 Minute Series Topographic Quadrangle
Clermont, Indiana
Photorevised 1984 Scale 1:25,000



Quadrangle Location

WSP

2025 Gateway Place, Suite 435
San Jose, California
(408) 453-6100

Figure B1
Site Location
Location of Quemetco Facility
Indianapolis, Indiana

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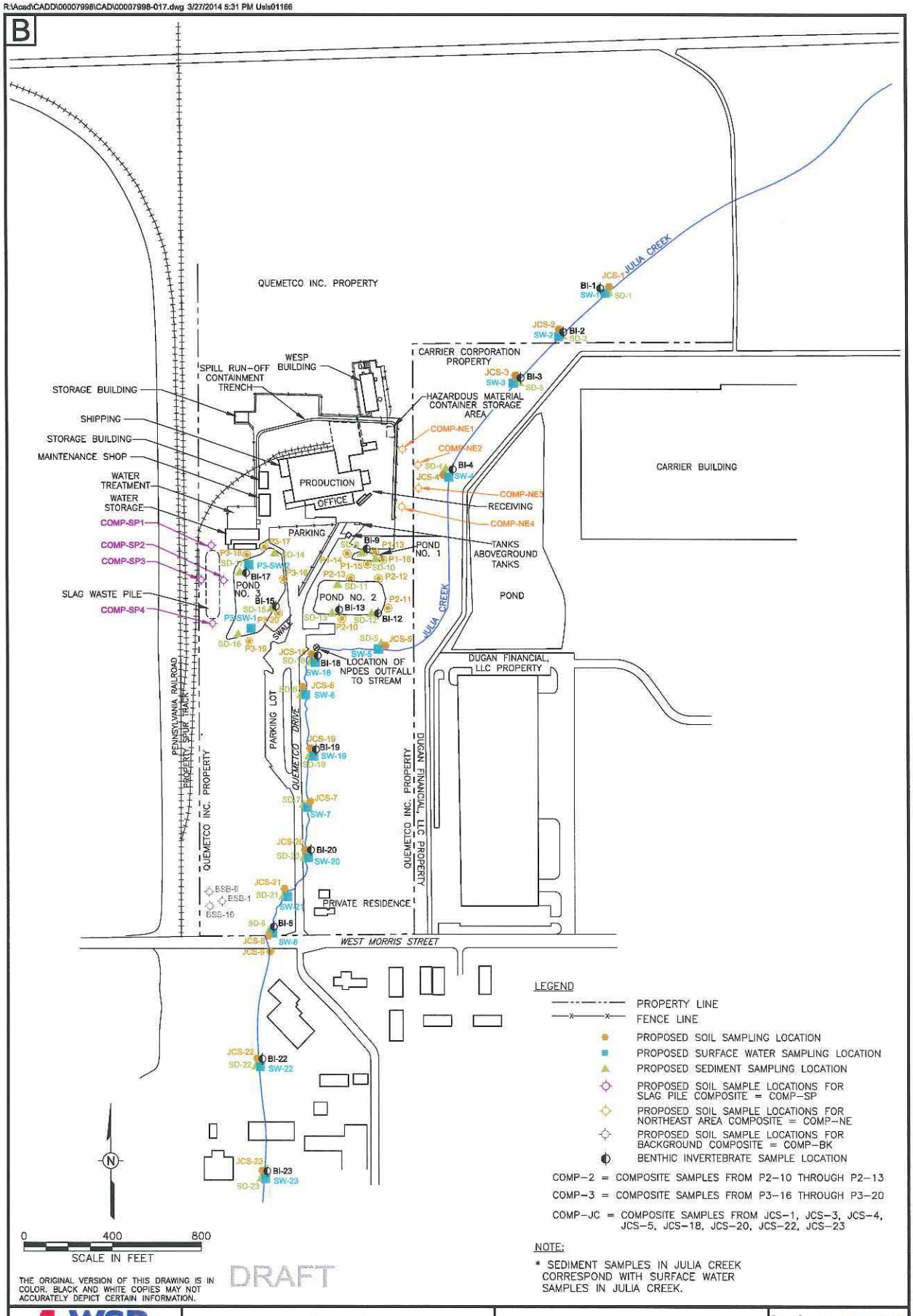

LEGEND

- PROPERTY LINE
- FENCE LINE
- PROPOSED SOIL SAMPLING LOCATION
- PROPOSED SURFACE WATER SAMPLING LOCATION
- PROPOSED SEDIMENT SAMPLING LOCATION
- PROPOSED SOIL SAMPLE LOCATIONS FOR SLAG PILE COMPOSITE = COMP-SP
- PROPOSED SOIL SAMPLE LOCATIONS FOR NORTHEAST AREA COMPOSITE = COMP-NE
- PROPOSED SOIL SAMPLE LOCATIONS FOR BACKGROUND COMPOSITE = COMP-BK
- BENTHIC INVERTEBRATE SAMPLE LOCATION

COMP-2 = COMPOSITE SAMPLES FROM P2-10 THROUGH P2-13

COMP-3 = COMPOSITE SAMPLES FROM P3-16 THROUGH P3-20

COMP-JC = COMPOSITE SAMPLES FROM JCS-1, JCS-3, JCS-4, JCS-5, JCS-18, JCS-20, JCS-22, JCS-23

NOTE:

* SEDIMENT SAMPLES IN JULIA CREEK CORRESPOND WITH SURFACE WATER SAMPLES IN JULIA CREEK.

THE ORIGINAL VERSION OF THIS DRAWING IS IN COLOR. BLACK AND WHITE COPIES MAY NOT ACCURATELY DEPICT CERTAIN INFORMATION.

DRAFT

WSP 2025 Gateway Place, Suite 435 San Jose, California 95110 (408) 453-6100 www.wspgroup.com/usa	Figure B2 PROPOSED DERA SAMPLING LOCATIONS	900 QUEMETCO DRIVE INDIANAPOLIS, INDIANA PREPARED FOR QUEMETCO, INC. INDIANAPOLIS, INDIANA	Drawn By: LS 3/26/2014 Checked: Approved: DWG Name: 00007998-017

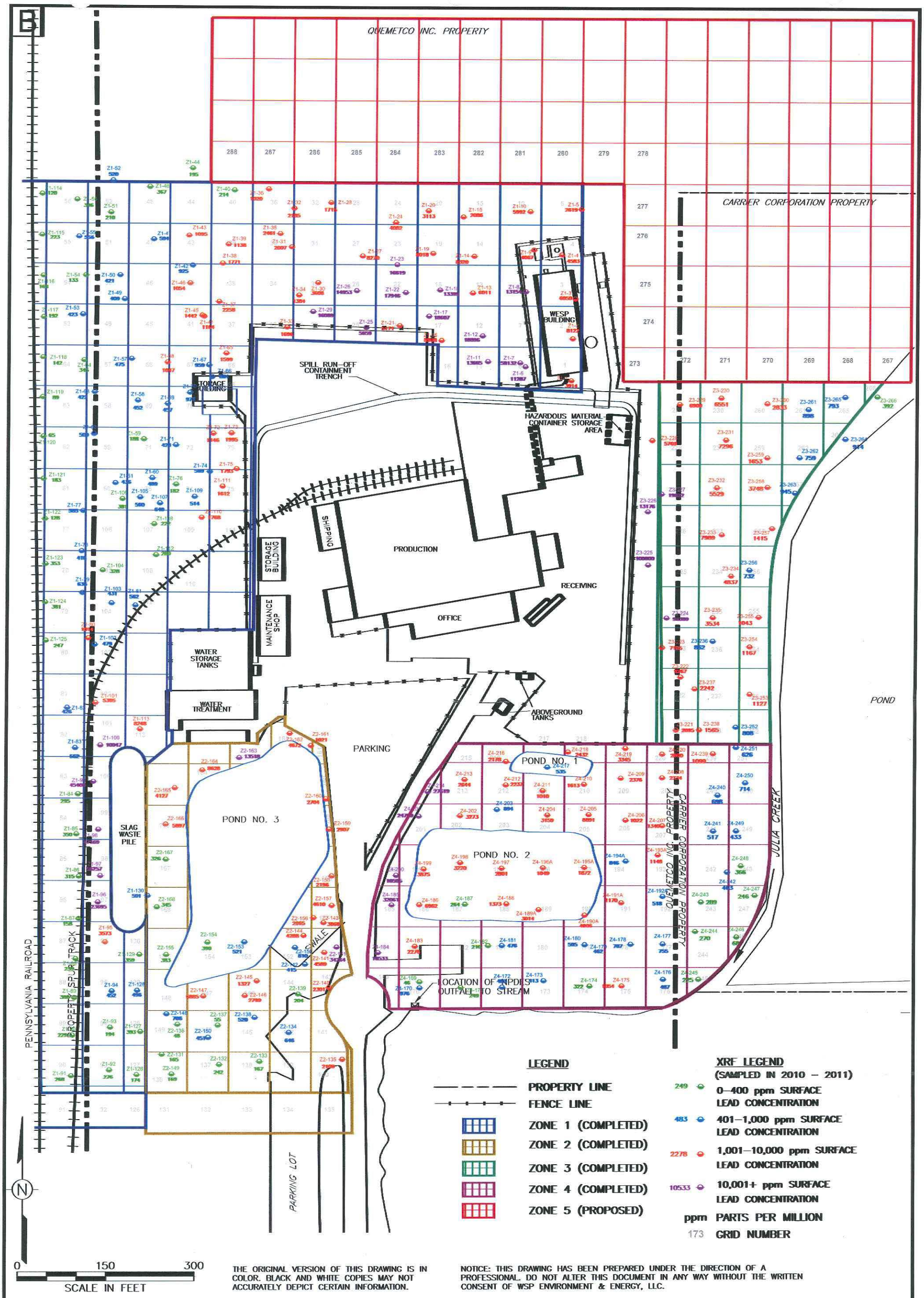

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Figure B3

PROPOSED XRF SAMPLING GRID

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	DWC	7/06/2012
Checked:	AW	7/13/2012
Approved:	BJM	7/13/2012
DWG Name:	00007998–014	

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B

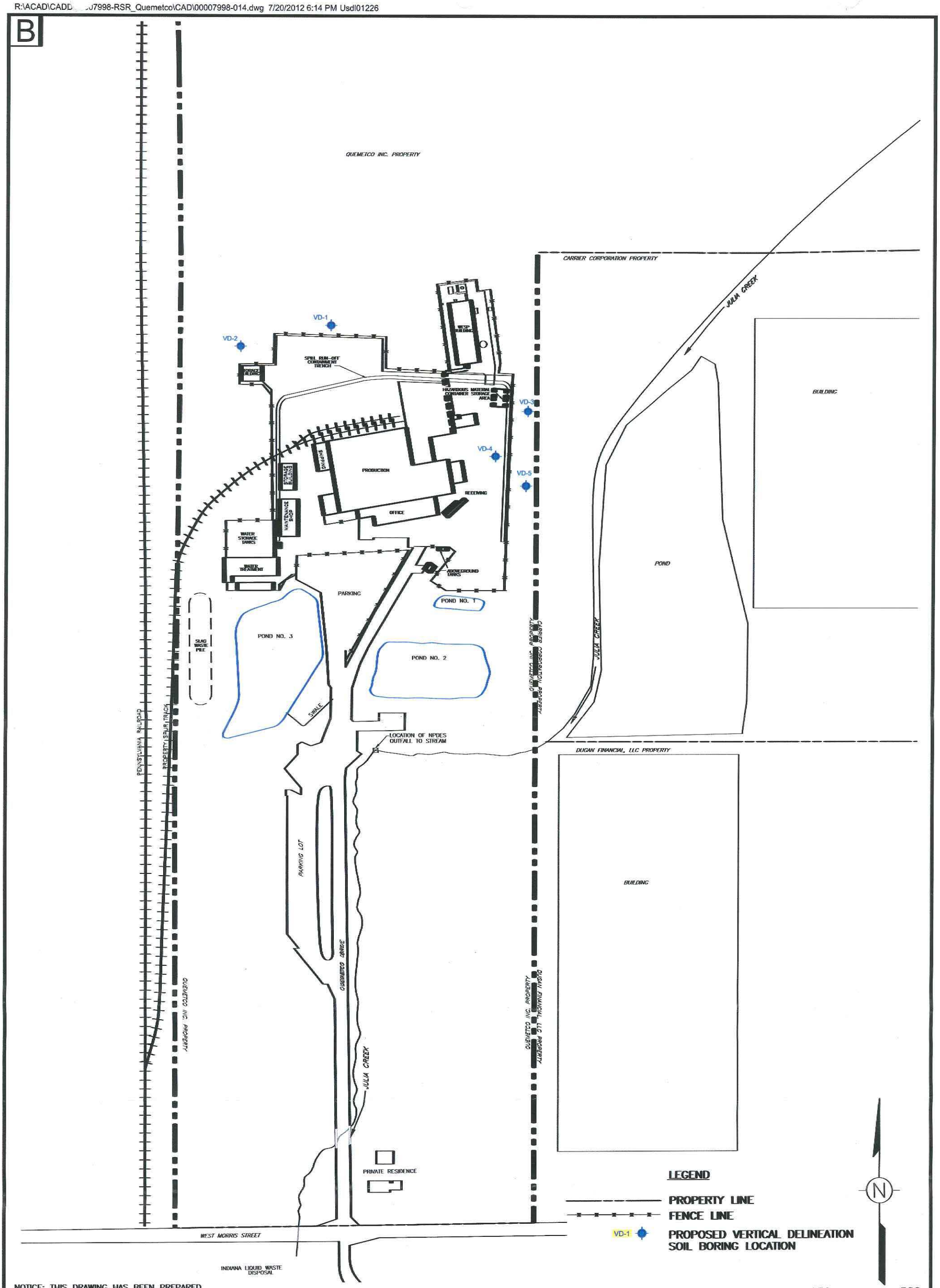

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Figure B4

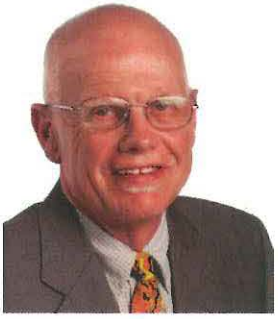
PROPOSED SOIL BORINGS FOR VERTICAL DELINEATION OF LEAD

900 QUEMETCO DRIVE
INDIANAPOLIS, INDIANA

PREPARED FOR
QUEMETCO, INC.
INDIANAPOLIS, INDIANA

Drawn By:	DJL	7/06/2012
Checked:	AW	7/13/2012
Approved:	BJM	7/13/2012
DWG Name:	00007998-014	

Attachment A – Statement of Qualifications of Key Personnel



Rick Freudenberger, PE

Senior Vice President
WSP

Career Summary

Mr. Freudenberger has over 30 years of experience in performing environmental assessments and remedial investigations, in waste treatment process design, and in technical management programs. He directs the technical staff in a wide range of projects and is currently serving as General Manager in the WSP Environment & Energy's San Jose office.

Professional Qualifications

- Registered Professional Engineer: Maryland, New York, Virginia, Georgia, Kentucky, Indiana, Minnesota, and Ohio
- Maryland Certified Wastewater Treatment Plant Operator
- Diplomat of the American Academy of Environmental Engineers
- California Registered Environmental Assessor

Education and Training

- B.S. - Civil Engineering, University of Maryland
- M.S. - Environmental Engineering, University of Maryland

Selected Relevant Experience

Project Experience

- Expert Witness Testimony: Mr. Freudenberger has served as an expert witness in various environmental litigation matters. He has extensive experience in insurance related matters and environmental claims, having worked both with the insured and insurers. This insurance work has included issues involving trigger of coverage, fate and transport modeling, and Superfund PRP cases.
- Environmental Investigations: Mr. Freudenberger has been most heavily involved in the design of groundwater cleanup and Superfund remedial investigation programs, assessments for mergers and acquisitions, environmental audits, and environmental assessments for major corporate clients. His previous service with local governments qualifies him to represent clients effectively in negotiations with regulatory agencies. One of

Mr. Freudenberger's major clients is an international lead company with smelting operations in the United States and Europe. Mr. Freudenberger has directed extensive RCRA Facility Investigations and Corrective Actions at the client's three U.S. smelters.

- RCRA and CERCLA Investigations: Mr. Freudenberger has worked with Environmental Strategies clients in various industries in designing and planning extensive investigations and cleanups under CERCLA (Superfund), RCRA, and ECRA regulations. These industries have included secondary lead smelters, wood preserving, petroleum refineries, telecommunications, electric utilities, automotive parts manufacturing, and industrial chemicals. Specifically, in two Superfund projects, he directed the preparation of remedial investigations and feasibility studies to address the characterization and remediation of groundwater contamination by chlorinated organics.
- Health and Safety: He has designed detailed health and safety and soil sampling plans for dioxin, performed field sampling programs, and directed the remedial activities in the field for the cleanup and secure storage of the dioxin-contaminated soils.
- Remedial Investigations: Mr. Freudenberger has designed sampling and remedial action programs for groundwater and soil contamination at numerous industrial sites and has performed more than 500 industrial site surveys and risk assessments for a variety of industrial clients. He also oversaw a wastewater facility construction management contract for the city of Baltimore from 1979 to 1981.
- Environmental Management: Mr. Freudenberger was Director of Public Works for Howard County, Maryland, with management responsibility for solid waste collection and disposal, environmental engineering, wastewater treatment, road maintenance, engineering, and capital projects. He also was Project Engineer for a $50-million expansion of the county's wastewater treatment plant and successfully directed the permitting and siting process for a new 600-acre sanitary landfill. He spent 3 years as Superintendent of Operations and Maintenance for a 10-million-gallon per day secondary wastewater treatment plant.

Allen J. Waldman, P.G.

Technical Manager
WSP

Career Summary

Mr. Waldman is a licensed geologist with over 17 years of experience in conducting environmental field work in support of site assessments, feasibility studies and landfill monitoring programs. He has hands-on experience with many of the standard field practices used in the environmental industry for assessing indoor air, soil vapor, soil, and groundwater conditions. He has been involved in generating hundreds of reports for site assessments and monitoring programs.

Mr. Waldman works closely with regulatory agencies to streamline the process of developing work plans so all parties are aligned on project objectives from the start and fewer revisions are required.

Mr. Waldman performs and oversees all phases of investigative work designed to safely and expediently characterize subsurface conditions and delineate contamination. His expertise in data collection and interpretation assure that field procedures will yield data of high integrity and scientific value.

Professional Qualifications

- Licensed Professional Geologist - California (#6323), Washington (#2753) and Idaho(#1455)

Education and Training

- B.S.- Geology, San Jose State University
- A.A. - Liberal Arts, Evergreen Valley College
- OSHA Hazardous Waste Operations training
- OSHA Hazardous Waste Supervisor training

Selected Relevant Experience

Project Experience

- Leaking Underground Storage Tank (LUFT) investigations: Mr. Waldman has performed tank removals and site characterizations at numerous gas station sites in Northern and Southern California. He has coordinated site investigations with state and local regulatory agencies designed to characterize the extent of both dissolved phase and free product. In addition to delineating the extent of soil and groundwater contamination,

Mr. Waldman has successfully remediated sites via excavation that have resulted in site closures. He has also coordinated and implemented a free-product recovery program that utilized vacuum trucks to remove floating product from groundwater extraction wells and maintained groundwater monitoring programs for many clients.

- Dense Non-Aqueous Phase Liquid (DNAPL) Investigations: Mr. Waldman has been involved in numerous site investigations involving the release of DNAPLs including organic solvents and polychlorinated biphenyl (PCBs). The characterization of DNAPL sites requires an acute understanding of the geologic strata beneath the site in order to delineate potential paths of migration or areas where DNAPL pooling may occur. Mr. Waldman has made use of high resolution data collection techniques including direct push membrane interface probes and electrical conductance, cone penetrometer testing and continuous core drilling to create geologic cross sections and elevation contour maps of specific geologic units. In one specific case he made use of such data to successfully design and install extraction wells specifically for recovering free phase PCBs. He has characterized other sites where DNAPLs have been released using soil vapor testing and data contouring in conjunction with depth-discrete soil and groundwater sampling to delineate the source and extent of contamination. In one such case where PCE was the primary contaminant, Mr. Waldman was able to show that the soil and groundwater contamination was confined within the site boundaries and then implemented an aggressive remediation plan to reduce the contamination levels. For this site the cost of off-hauling contaminated soil was eliminated by taking advantage of the acreage available on site. Through Mr. Waldman's oversight and coordination with the regulatory agency the excavated soil was spread out in a thin layer and treated through aeration for reuse in backfilling the excavations. The results of the remediation effort along with risk analysis led to site closure. At another site where a release of TCE occurred in a highly-stratified, weathered bedrock setting, Mr. Waldman successfully characterized the plume then designed an in situ remediation program where permanganate solution was introduced via precisely placed injection wells to oxidize the contaminant. The injection program proceeded as planned and the TCE plume was significantly reduced.
- Hydrogeologic Conceptual Site Model (HCSM): Mr. Waldman evaluated regional and site investigation data to prepare a HCSM that shed new insight on subsurface conditions. Based on the HCSM Mr. Waldman evaluated the existing groundwater monitoring network and provided recommendations to improve the groundwater monitoring program.
- Landfill Monitoring: Mr. Waldman has been involved in many phases of landfill monitoring including landfill gas monitoring, stormwater runoff sampling and leachate/groundwater monitoring. He helped assure that the landfill operation complied with the requirements of the General Permit by collecting the required monitoring data as stipulated in the permit, assuring data integrity by reviewing data collection processes and data evaluation, and submitting the reports to the regulatory agency on schedule. He also performed quarterly and monthly landfill observations also as required in the General Permit and addressed any failing conditions as needed.
- Landfill Expansion Projects: Mr. Waldman has logged exploratory borings, performed trench logging, installed monitoring wells, conducted packer tests, slug tests, and pumping tests to evaluate a proposed landfill expansion area. He has also performed exploratory drilling and well installation up to 1,500 feet deep in a proposed hazardous waste repository in the Mojave Desert using hollow-stem auger, air-rotary and mud-rotary drilling methods.
- Vapor Intrusion Investigation: Mr. Waldman worked closely with a risk assessor and the lead regulatory agency to develop and implement an indoor air investigation for a former industrial facility in Southern California. The investigation involved installing and sampling subslab vapor probes and collecting air samples from within the occupied structures then comparing the data to determine whether constituents in the subsurface are impacting indoor air quality. The data were then used in the Human Health Risk Assessment for the Site

Awards and Publications

- Advanced Diagnostic Tools for Natural Attenuation and Bioremediation Applications, April 2010 Groundwater Resource Association Conference, Santa Clara, CA
- Case Study: Sodium Permanganate Injection to Treat TCE Plume, January 2012 ISCO Design and Implementation Boot Camp, Vironex.

Amy M. Romano

Senior Technical Manager
WSP

Career Summary

Amy Romano has extensive experience providing environmental and Department of Transportation (DOT) regulatory support and conducting environmental site assessments, regulatory compliance audits, and human health risk assessments.

Professional Qualifications

Education and Training

- MEM, Environmental Management – Duke University – Durham, North Carolina
- BS, Environmental Studies and Biology, summa cum laude – Saint Lawrence University – Canton, New York
- OSHA Hazardous Waste Operations Training
- U.S. DOT Hazardous Materials Training

Selected Relevant Experience

Project Experience

- Environmental and DOT Compliance Audits: Performed and managed environmental and DOT audits for clients with multiple manufacturing facilities. The audits have included evaluation of compliance with Resource Conservation and Recovery Act (RCRA), Emergency Response and Community Right-to-Know Act (EPCRA), Clean Air Act, Toxic Substances Control Act, Clean Water Act, Safe Drinking Water Act, Federal Insecticide, Fungicide, and Rodenticide Act, and DOT hazardous materials regulations. Ms. Romano has performed audits for chemical and steel manufacturing facilities, computer and electronic assembly plants, aircraft engine manufacturing and repair facilities, metal working facilities, secondary lead smelters, machine and equipment assembly plants, municipal landfills, and hazardous waste treatment, storage, and disposal facilities.
- Environmental Site Assessments: Performed numerous Phase I environmental site assessments projects for clients throughout the United States. The site assessments included an evaluation of historic property uses, current raw material and waste handling and storage practices, wastewater and storm water management, air emissions, and the presence of polychlorinated biphenyls, asbestos, radon, and lead-based paint.

- Regulatory Compliance: Met with state and U.S. Environmental Protection Agency (EPA) inspectors on behalf of clients and has developed policy and procedures manuals for industrial clients to maintain compliance with environmental and DOT regulations. Ms. Romano has prepared RCRA contingency plans and annual/biennial hazardous waste reports; EPCRA Section 302 notifications, Section 312 Tier I/II reports, Section 313 Form A and R reports, and supplier notification letters; wastewater discharge applications, wastewater slug/spill control plans, and discharge monitoring reports; spill prevention, control, and countermeasure (SPCC) plans; storm water permit applications, storm water no exposure certification forms, and storm water pollution prevention plans; air emission inventories and air permit applications; and DOT registrations and security plans.
- Regulatory Compliance Training: Prepared multiple training materials for industrial clients regarding environmental regulations, including requirements for RCRA generators, the Clean Air Act, wastewater, storm water, SPCC plans, and EPCRA requirements. Ms. Romano has performed training on DOT requirements, including hazardous materials classifications, packaging, labeling and marking, shipping papers, emergency response information, materials of trade, registration, and security issues related to hazardous material shipments.
- Risk Assessments: Prepared human health evaluations for Superfund and RCRA sites, coal gasification facilities, petroleum refineries, wood preserving facilities, and various manufacturing facilities. Ms. Romano has characterized the chemicals of concern, modeled exposure doses, and quantified risks to human health and the environment. She has derived risk based cleanup goals for contaminants in soil, groundwater, and sediments for sites regulated under Superfund, RCRA, and state voluntary cleanup programs. She has modeled and evaluated risks associated with indoor air concentrations from subsurface vapor intrusion into industrial and residential buildings.

Awards and Publications

- Silfer, B. and Romano, A. 2004. Vapor Intrusion - Just When You Thought Your Site was Cleaned Up. Environmental Claims Journal.
- Romano, A. 2003. Recent Amendments to EPA's TSCA Inventory Update Rule. Environmental Quality Management.
- Romano, A. and McDermott, E. 2001. Current Technical Developments. TRI Lead Rule. Environmental Claims Journal.
- Romano, A. and Silfer, B. 2000. New Regulatory Requirements for Class V Underground Injection Control Wells. Environmental Quality Management.
- Romano, A. and Silfer, B. 2000. Recent Technological Developments and EPA Initiatives. Environmental Claims Journal.
- Silfer, B., Moore, G., and Romano, A. 1997. Innovative Remedial Technologies and Cost-Saving Regulations. Environmental Claims Journal.
- Romano, A. 1996. The Ins and Outs of EPA's Accidental Release Prevention Program. Environmental Regulation and Permitting.
- Romano, A., and Murphy, M. 1993. Environmental Compliance Auditing. In: D. Machlowitz, ed. Legal Guide to Working with Environmental Consultants. pp. 27-49. Wiley Law Publications.

Intrinsik Environmental Sciences Inc.

RUTH N. HULL, MSc, QP_{RA}

Senior Scientist

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Mississauga, ON, L5T 0A3
Ph: 905-364-7800 Fax: 905-364-7816
rhull@intrinsikscience.com
www.intrinsikscience.com

EDUCATION

1994	M.Sc., Ecotoxicology, Concordia University
1989	B.Sc., Honours, Biology with Chemistry Minor, University of Waterloo
QP_{RA}	Qualified Person for Risk Assessment as defined by the Environmental Protection Act of Ontario (Brownfields Legislation; O.Reg. 153/04)

AREAS OF SCIENTIFIC INTEREST

- Ecological risk assessment
- Human health risk assessment
- Contaminated sites
- Ecological impacts of climate change
- Aquatic and terrestrial toxicology
- Criteria development
- Risk assessment of smelter sites
- Landscape-scale risk assessment

EMPLOYMENT HISTORY

1999-Present **INTRINSIK ENVIRONMENTAL SCIENCES INC.**
(formerly operating as Cantox Environmental Inc.)
Mississauga, Ontario
Senior Scientist

Management, public consultation and completion of projects involving risk assessment for predicting/evaluating the potential impacts of environmental contamination on human health and aquatic and terrestrial ecosystems. Projects include Superfund and RCRA sites in the U.S., risk assessments in Canada, and landscape-scale risk assessments in Canada and Latin America. Technical lead for two of the largest ERAs conducted in Canada, at the Teck Metals lead/zinc smelter in Trail, BC and for the smelters in the City of Greater Sudbury in Ontario. Specialist in risk assessment, including problem formulation (conceptual model development), review of environmental site assessment data, ecological exposure assessment, review of toxicological data, development of ecological TRVs, and risk characterization. Provides expert advice and peer review on toxicology, contaminated sites and risk assessment to government and industry.

1995-1999 Beak International Inc., Brampton, ON
Risk Assessment Specialist, QA Manager for GLP Ecotoxicity Laboratory

1992-1994 Oak Ridge National Laboratory, Oak Ridge, Tennessee
Ecological Risk Assessment Team Leader

1992 State Of Minnesota., St. Paul, Minnesota
Toxicologist

1989 Rijkswaterstaat, Government of the Netherlands, Haren, Netherlands
Toxicologist

PROFESSIONAL AFFILIATIONS

- Society of Environmental Toxicology and Chemistry (SETAC) - member since 1990
  - Director – SETAC North America Board of Directors (Nov. 2011-2014)
  - Co-Chair of the SETAC North America Regional Chapters Committee (2010-11)
  - Co-Chair – SETAC North America Endowment Fund Board of Trustees (Nov. 2013-present)
  - Chair of the SETAC North America Membership Committee (2006-2009)
  - Member of the SETAC World Council Scientific Committee (2006-2008)
  - Member of the SETAC Ecological Risk Assessment Advisory Group
  - Member of the SETAC Human Health Risk Assessment Advisory Group
  - Member of the SETAC Metals Advisory Group
  - Member of the Laurentian Chapter of SETAC
  - Board Member – Laurentian Chapter of SETAC (2003 to 2005)

- American Society of Testing and Materials, Environmental Toxicology and Risk Assessment Committee - member since 1992
  - Subcommittee E47.05 (Risk Assessment and Risk Management)
    Lead author for ASTM Standard Guide to Ecological Risk Assessment Data and Information Needs. Contributing author for ASTM E2348-06 Standard Guide for a Framework for a Consensus-based Environmental Decision-making Process and ASTM E2205 Standard Guide for Risk-based Corrective Action for Protection of Ecological Resources.

PUBLICATIONS

Hull, R.N. and K. Bresee. 2012. Ecological Risk Assessment: Evaluating Risks to Terrestrial Wildlife. Chapter 11 in: Risk Assessment and Environmental Management: A Case Study in Sudbury, Ontario, Canada. C. Wren (Ed.). Maralte Publishing. 450 pp.

Wren, C. and **R. N. Hull**. 2012. Ecological Risk Assessment Problem Formulation. Chapter 9 in: Risk Assessment and Environmental Management: A Case Study in Sudbury, Ontario, Canada. C. Wren (Ed.). Maralte Publishing. 450 pp.

Allard, P., Fairbrother, A., Hope, B.K., **Hull, R.N.**, Johnson, M.S., Kapustka, L., Mann, G., McDonald, B. and B.E. Sample. 2010. Recommendations for the Development and Application of Wildlife Toxicity Reference Values. Integrated Environmental Assessment and Management 6(1): 28-37.

Archbold, J.A., **Hull. R.N.** and M. Diamond. 2007. Potential Importance of Inhalation Exposures for Wildlife using Screening-level Ecological Risk Assessment. Human and Ecological Risk Assessment 13(4): 870-883.

Hull, R.N., Barbu, C. H., and Goncharova, N. (Eds). 2007. Strategies to Enhance Environmental Security in Transition Countries. Springer Publishing. Dordrecht, The Netherlands. ISBN 978-1-4020-5995-7.

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Hull, R.N., and Swanson, S.M. 2006. Sequential Analysis of Lines of Evidence – An Advanced Weight-of-Evidence Approach for Ecological Risk Assessment. Integrated Environmental Assessment and Management. Vol 2(4): 302-311.

Hull, R.N. 2006. Scientific Basis for Ecotoxicology, Ecological Risk Assessment and Multiple Stressors: Canadian Experience in Defining Acceptable Risk Levels for Infrastructure. Ecotoxicology, Ecological Risk Assessment and Multiple Stressors. In: Arapis, G., Goncharova, N. eds. Kluwer Academic Publishers.

Hull, R.N., Belluck, D.A., and Lipchin, C. 2006. A Framework for Multi-criteria Decision-Making with Special Reference to Critical Infrastructure: Policy and Risk Management Working Group Summary and Recommendations. In: Arapis, G., Goncharova, N. eds. Ecotoxicology, Ecological Risk Assessment and Multiple Stressors. Kluwer Academic.

Belluck, D.A., **Hull, R.N**, Benjamin, S.L., Alcorn, J., and Linkov, I. 2006. Environmental Security, Critical Infrastructure and Risk Assessment: Definitions and Current Trends. In: Morel, B., Linkov, I., eds. Environmental Security and Environmental Management: The Role of Risk Assessment. Springer Publ. The Netherlands. pp. 3-16.

Belluck, D.A., **Hull, R.N.**, Benjamin, S.L., Alcorn, J., and Linkov, I. 2006. Are Standard Risk Acceptability Criteria Applicable to Critical Infrastructure Based on Environmental Security Needs? In: Morel, B., Linkov, I., eds. Environmental Security and Environmental Management: The Role of Risk Assessment. Springer Publ. The Netherlands. pp. 93-102.

Willis, R.D., **Hull, R.N.**, and Marshall, L.J. 2003. Considerations regarding the use of reference area and baseline information in ecological risk assessment. HERA 9(7): 1645-1654.

Stenner, R.D., **Hull, R.N.**, and Willes, R.F. 2002. Involving stakeholders to achieve successful development of brownfield sites. Proceedings of the First International Conference on Prevention, Assessment, Rehabitation and Development of Brownfield Sites. Sept. 2-4, 2002, Cadiz, Spain. The Wessex Institute of Technology Press. Ashurst, UK.

Hull, R.N., Ferguson, G.M., Glaser, J.D., and Willis, R. 2002. Risk Assessment Resources on the World-Wide Web (WWW). Human and Ecological Risk Assessment 8(2):443-457.

Greenberg, B.M., **Hull, R.N.**, Roberts, M.H., Jr., and Gensemer, R.W., Eds. 2001. Environmental Toxicology and Risk Assessment: Science, Policy and Standardization - Implications for Environmental Decisions: Tenth Volume, ASTM STP 1403, American Society for Testing and Materials, West Conshohocken, PA.

Hull, R.N., and Sample, B.E. 2001. Ecological Risk Assessment Primer. In: AA Practical Guide to Managing Contractor Produced Risk Assessment Reports. S.A. Benjamin (ed). CRC Lewis Press.

Gilron, G., and **Hull, R.N.** 2001. Toxicity Testing in Risk Assessment. In: AA Practical Guide to Managing Contractor Produced Risk Assessment Reports. S.A. Benjamin (ed). CRC Lewis Press.

Hull, R.N., and Benson, D.C. 2000. Better Communication enhances Risk Assessment Process. EcoLog 28(15):3-4.

Hull, R.N., Klee, U., Bryant, D., and Copeland, T. 1999. Soil Microbial Communities and Ecological Risk Assessment: Risk Assessors' Perspective. Human and Ecological Risk Assessment 5(4):707-714.

Hull, R.N., and Benson, D.C. 1998. Unburied Past: Applying Site-specific Risk Assessment to a TCE-contaminated Site. Hazardous Materials Management: Oct/Nov: 12.

Jones, D.S., Suter II, G.W., and **Hull, R.N.** 1997. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-associated Biota: 1997 Revision. ES/ER/TM-95/R4. Oak Ridge National Laboratory, Oak Ridge, TN.

Hull, R.N., Suter II, G.W., and Barnthouse, L.W. 1995. Comparability of Human and Ecological Risk Assessments: Ecological Risk Assessments Have Been Less Influential Than Human Health Risk Assessments. Human and Ecological Risk Assessment 1(5):482-484.

Suter II, G.W., Cornaby, B.W., Hadden, C.T., **Hull, R.N.**, Stack, M.E., Zafran, F.A. 1995. An Approach for Balancing Health and Ecological Risks at Hazardous Waste Sites. Risk Analysis 15(2): 221-231.

Suter, G.W. II, **Hull, R.N.**, and Sample, B.E. 1994. Ecological Risk Assessment for Comprehensive Environmental Response, Compensation and Liability Act Sites on Federal Facilities: An Introductory Guide for Facility Managers. U.S. Department of Energy, Oak Ridge, Tennessee. DOE/HWP-152.

Hull, R.N., and Suter, G.W.II. 1994. Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Sediment-associated Biota: 1994 Revision. ES/ER/TM-95/R1. Oak Ridge National Laboratory, Oak Ridge, TN.

Belluck, D.A., **Hull, R.N.**, Benjamin, S.L., French, R.D., and O'Connell, R.M. 1993. Defining Scientific Procedural Standards for Ecological Risk Assessment. In: Environmental Toxicology and Risk Assessment: 2nd Volume, ASTM STP 1173, Gorsuch, J.W., Dwyer, F.J., Ingersoll, C.G., and LaPoint, T.W., Eds., American Society for Testing and Materials, Philadelphia.

Ruby, S.M., Jaroslawski, P., and **Hull, R.N.** 1993. Lead and Cyanide Toxicity in Sexually Maturing Rainbow Trout, *Oncorhynchus mykiss* during Spermatogenesis. Aquatic Toxicology 26: 225-238.

Klamer, J.C., **Hull, R.N.**, Laana, R.W.P.M., and Eisma, D. 1990. The Distribution of Heavy Metals and Polycyclic Aromatic Hydrocarbons in the Sediments of the Oyster Grounds (North Sea). Netherlands Journal of Sea Research 26(1): 83-87.

CONFERENCE PRESENTATIONS AND RELATED ACTIVITIES

Hull, R.N. and M. Machmer. Linking a Weight of Evidence Analysis to Restoration in a Landscape-scale Terrestrial Ecological Risk Assessment. Poster Presentation. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2013. Nashville, TN.

McCarthy, C., **Hull, R.N.** and G.W. Suter II. Co-chairs of the Platform session "Implementing ERA Weight-of-evidence Approaches that Contribute to Decision-making". Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2013. Nashville, TN.

Hull, R.N., M. Machmer, S.R. Hilts, D. DeRosa. 2013. The Canadian Lower Columbia Ecosystem Management Program: A Collaborative Approach to Assess, Restore, Conserve and Enhance Terrestrial Ecosystems around a Smelter Site. Invited Platform Presentation. Annual Meeting of the Society for Ecological Restoration. October 2013. Madison, WI.

Hull, R.N., M. Whitfield Aslund, L. Marshall, C. Moore, K. Rankin. 2012. Ecotoxicity and Accumulation of Rare Earth Elements. Poster Presentation. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2012. Long Beach, CA.

Hull, R.N. Sustainability: Innovation through Creative Use of Existing Tools. Panel presentation. Commission for Environmental Cooperation, Sound Management of Chemicals Workshop, May 2012. San Antonio, TX.

Hull, R.N. and J. Archbold. Transparency, Risk Communication and Stakeholder Engagement for a Diverse Audience in the 21st Century. Panel presentation. Commission for Environmental Cooperation, Sound Management of Chemicals Workshop, May 2012. San Antonio, TX.

Hull, R.N., S. Kleywegt, T. Fletcher and J. Schroeder. 2012. Hazard Screening of Substances of Emerging Concern in the Great Lakes Basin. Platform Presentation. Canadian Association on Water Quality. February, 2012. Burlington, ON.

Hull, R.N., D.W. Bryant and E.A. Sigal. 2011. Use of Risk Assessment to Demonstrate Benefits to Local Stakeholders of Regional Action to Decrease Greenhouse Gas Emissions. Platform Presentation. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2011. Boston, MA.

Hull, R.N. and E.A. Sigal. 2011. Human Health and Ecological Risk Assessments Around Mining and Smelting Sites. Platform Presentation. SETAC Focused Topic Meeting "Pollutants in the Environment: Fate and Toxicity", Merida, Mexico. August 2011.

Hull, R.N., R.S. Teed, H. Yee, C. Meays. 2010. Potential influences of climate change on water quality guideline development. Poster presentation. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2010. Portland, OR.

Schroeder, J., **R.N. Hull**, N. Feisthauer, A-M. Jesupillai, R.S. Teed, T. Fletcher. 2010. Challenges associated with evaluating substances of emerging concern. Platform presentation at the Aquatic Toxicity Workshop. Toronto, ON. October 2010.

Hull, R.N. Using risk assessment to evaluate and reduce stress(ors). Plenary presentation at the Annual General Meeting of the Laurentian Chapter of SETAC. Niagara-on-the-Lake, ON. June 2010.

Hull, R.N. and J. Margolin. Co-chairs of the ERA Debates: 20 Years after the First ERA Guidance: The State of Affairs. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2009. New Orleans, LA.

Wren, C., M-K Gilbertson and **R. Hull.** 2009. The Sudbury Soils Study Ecological Risk Assessment. Platform Presentation at the Sudbury Restoration Workshop, October 2009. Sudbury ON.

Sigal, E.A., Moore, C.E., Ferguson, G.F., **Hull, R.N.**, Marshall, L. and R. Willis. 2009. Lessons learned from wide area risk assessment studies of mining and smelting sites. Platform presentation. Canadian Institute of Mining Conference. May 2009.Toronto, ON.

Enns, K.A. and **R.N. Hull.** 2008. Use of biophysical habitat mapping in landscape-scale ecological risk assessment. Platform presentation. Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2008. Tampa, Florida.

Hull, R.N. and P. Allard. 2007. Co-chairs of the Interactive Platform session "Development and Use of Wildlife TRVs in ERA". 28th Annual Meeting of the Society of Environmental Toxicology and Chemistry. November 2007. Milwaukee, Wisconsin.

Hull, R.N., P. Allard, A. Fairbrother, B. Hope, M.S. Johnson, L.A. Kapustka, B. McDonald and B.E. Sample. 2007. Summary of Recommendations for Wildlife TRV Development and Use. Annual Meeting of the Society of Environmental Toxicology and Chemistry. Interactive Platform. November 2007. Milwaukee, Wisconsin.

McDowell, S., K.A. Enns and **Hull, R.N.** 2007. Wildlife Species at Risk Assessment: Challenges and Approaches for Landscape Scale Analysis. Annual Meeting of SETAC. Poster Presentation. November 2007. Milwaukee, Wisconsin.

Hull, R. N. and M-K Gilbertson. 2007. Lessons learned from two smelter ERAs in Canada: You must risk bears and bugs! Sudbury 2007 Mining and Environment Conference. Platform presentation. October 2007. Sudbury, Ontario.

NATO Country Co-Director for a NATO-sponsored Advanced Research Workshop on "Strategies to Enhance Environmental Security in Transition Countries", held September 6-9, 2006 in Sibiu, Romania.

Hull, R.N., and S. Swanson. 2006. Sequential Analysis of Lines of Evidence (SALE) to Evaluate Risks to Avian Populations around a Smelter. 27th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2006. Montreal, Quebec.

Hull, R.N. 2006. Which human and terrestrial ecological receptors are most at risk around smelters? Platform presentation at the NATO-sponsored Workshop "Strategies to Enhance Environmental Security in Transition Countries", Sept. 6-9, 2006. Sibiu, Romania.

Hull, R.N. 2006. It ain't easy being green – who is most at risk around smelters? Platform presentation at the Laurentian SETAC Annual Meeting. May 2006. Sudbury, Ontario.

Hull, R.N. 2006. Sudbury Terrestrial Ecological Risk Assessment: Seeing the forest and the trees. Platform presentation at the Restoration Workshop. February 2006. Sudbury, ON.

Hull, R.N., and S. Swanson. 2005. Sequential Analysis of Lines of Evidence: An Advanced Weight-of-Evidence Approach. 26th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2005. Baltimore, MD.

Hull, R.N. 2005. Thinking Outside the Tox: Indirect Effects in Ecological Risk Assessment. Invited Speaker. 32nd Aquatic Toxicity Workshop. October, 2005. Waterloo, Ontario.

Hull, R.N. 2004. Scientific Basis for Ecotoxicology, Ecological Risk Assessment and Multiple Stressors: Canadian Experience in Defining Acceptable Risk Levels for Infrastructure. NATO Advanced Research Workshop on Ecotoxicology and Ecological Risk Assessment. Invited Talk. Poros, Greece. October, 2004.

Hull, R.N., and Sigal, E.A. 2004. Benefits of Concurrent Human Health and Ecological Risk Assessments for Supporting Remediation Planning around Three Smelters in Sudbury, Ontario. 25th Annual Meeting of the Society of Environmental Toxicology and Chemistry. 4th World Congress. Interactive Platform presentation. November, 2004. Portland, OR.

Hull, R.N., and Swanson, S. 2004. Ecological risk assessment and management at smelters. Co-chair of Interactive Platform session. 25th Annual Meeting of SETAC. 4th World Congress. November, 2004. Portland, Oregon.

Enns, K., Enns, B., Hilts, S., Duncan, W.F.A., **Hull, R.N.**, and Swanson, S. 2004. Landscape-scale Ecological Risk Assessment in Support of Remediation Planning for Teck Cominco's Trail Smelter. 25th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Interactive Platform presentation. Nov. 2004. Portland OR.

Hull, R.N., and E.A. Sigal. 2004. Ecological and Human Health Risk Assessment for the Sudbury Area. Platform presentation at the Restoration Workshop. February 2004. Sudbury, Ontario.

Hull, R.N., Scozzaro, H., Archbold, J., Bacigalupo, C., and Sigal, E.A. 2003. How do we assess risks to pets? A preliminary evaluation. 24th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2003. Austin, Texas.

Hull, R.N., Moore, R.L., Bresee, K.E., Willis, R.D., and Marshall, L.J. 2002. Screening Chemicals of Concern for Ecological Risk Assessment at Combustion Facilities. 23rd Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2002. Salt Lake City, Utah.

Hull, R.N., and Hilts, S.R. 2001. Approaches to Human and Ecological Risk Communication in an Active Smelter Community. 22nd Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2001. Baltimore, MD.

Hull, R.N., and Moore, R.L. 2001. Review of Ecological Studies Conducted at Smelter Sites. 22nd Annual Meeting of the Society of Environmental Toxicology and Chemistry. Oral presentation. November, 2001. Baltimore, Maryland.

Brown, G., O'Brien, G., **Hull, R.N.**, Swanson, M.S., and Duncan, W. 2001. Problem formulation for a large, complex ecological risk assessment. 28th Annual Aquatic Toxicity Workshop. Oral Presentation. Winnipeg, Manitoba.

Moore, R.L., and **Hull, R.N.** 2001. Are the Canadian water quality guidelines protective for metal mixtures? Annual Meeting of the Laurentian Chapter of the Society of Environmental Toxicology and Chemistry. Poster presentation. Hamilton, ON.

Hull, R.N., Brown, G., Duncan, B., and Sentis, R. 2000. Unique considerations when conducting an ecological risk assessment for a wide-area site. 21st Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 2000. Nashville, Tennessee.

Hull, R.N. 2000. Member of Technical Organization Committee. Third Society of Environmental Toxicology and Chemistry World Congress, Brighton, England. May, 2000.

Hull, R.N. 1999. Risk assessment resources on the world wide web (WWW). 20th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1999. Philadelphia, Pennsylvania.

Stecko, P., Russel, C., Schroeder, J., Garisto, F., **Hull, R**., Rinker, M., and van Arkel, G. 1999. Using multiple lines of evidence to determine the cause of ecological impacts downstream of a mine discharge. 20th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1999. Philadelphia, PA.

Hull, R.N., McKee, P.M., Fahey, J.J., and Benson, D.C. 1998. Risk communication during the risk assessment process: An Ontario case study. 19th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1998. Charlotte, North Carolina.

Costello-Walker, C., Menzie, C., Kangas, M., Luke, N., Suedel, B., Swindoll, M., Meyer, A., Montgomery, C., **Hull, R**. 1998. Comparative analysis of ecological risk assessment guidance as part of the ASTM risk based corrective action ecological risk assessment standard. 19th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Interactive Poster presentation. November, 1998. Charlotte, NC.

Swindoll, M., **Hull, R.N**., Saterbak, A., Presiosi, D.V., Sample, B., Mazur, D., Suedel, B. 1998. Selection and use of screening-level benchmarks for risk-based corrective action ecological risk assessments. 19th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Interactive Poster presentation. November, 1998. Charlotte, North Carolina.

Hull, R.N., and Dixon, D.G. 1998. Ecological Risk Assessment. Co-instructor of short-course. Environmental risk assessment and management: Principles, practice and progress. Institute for Risk Research. Hamilton, Ontario.

Hull, R.N., and Kangas, M. 1997. ASTM data and information needs checklist for conducting an ecological risk assessment. 18th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1997. San Francisco, California.

Hull, R.N., and Robertson, J.L. 1996. What do you need to make a decision? The role of analytical chemistry data in the risk assessment/risk management process. ECOTOX Workshop. Platform presentation. June1996. Winnipeg, MB.

Hull, R.N., and Robertson, J.L. 1996. The role of analytical chemistry data in the risk assessment/risk management process. 17th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November,1996. Washington, DC.

Ruby, S.M., **Hull, R.N**., and Anderson, P.D. 1996. Pituitary-gonad interactions during vitellogenesis in female rainbow trout following exposure to sublethal lead. 17th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1996. Washington, DC.

Hull, R.N., and Hart, D. 1996. Ecological Risk Assessment. Co-instructors of Short-course session. Environmental Risk Management. Institute for Risk Research. Waterloo, Ontario.

Suter, G.W., Sample, B.E., and **Hull, R.N.** 1996. Ecological risk assessment at contaminated sites. Co-instructors of short-course. Laurentian Chapter of the Society of Environmental Toxicology and Chemistry. Ottawa, Ontario.

Hull, R.N., and Gilron, G.L. 1995. How much weight should be assigned to toxicity test results in ecological risk assessment? 16th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1995. Vancouver, British Columbia.

Hart, D., Miesenheimer, P., and **Hull, R.** 1995. Assessing risks to fish populations near a proposed disposal facility for used nuclear fuel. 16th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1995. Vancouver, British Columbia.

Hull, R.N., and Pastorok, R. 1995. Ecological risk assessment: Science or magic? Co-chairs of platform session. 16th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Vancouver, British Columbia.

Suter, G.W., Barnthouse L. Sample B.E., and **Hull, R.N.** 1995.Ecological risk assessment at contaminated sites. Co-instructors of short-course. 16th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Vancouver, British Columbia.

Hull, R.N., and Suter, G.W. II. 1994. Using the Weight-of-evidence approach for ecological risk assessment at a DOE facility. 15th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November 1994. Denver, Colorado.

Jones, D.S., **Hull, R.N.**, and Suter, G.W. II. 1994. Issues and implications of coordinated ecological and human health risk assessments. 15th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1994. Denver, Colorado.

Suter G.W., Barnthouse L., Sample B.E., and **Hull, R.N.** 1994. Ecological risk assessment at contaminated sites. Co-instructor of short-course. 15th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Denver, Colorado.

Hull, R.N., Belluck D.A., and S.L.Benjamin. 1993. Shifting from baseline to life cycle risk assessments to estimate ecological risks and impacts at hazardous waste sites. 1st Society of Environmental Toxicology and Chemistry World Congress. Poster presentation.

Belluck, D.A., **Hull, R.N.**, and S.L. Benjamin. 1993. Development of procedural standards for conducting acceptable ecological risk assessments. 1st Society of Environmental Toxicology and Chemistry World Congress. Poster presentation.

Benjamin, S.L., Belluck, D.A., and **Hull, R.N.** 1993. Functional performance standards for determining acceptable ecological risks. 1st Society of Environmental Toxicology and Chemistry World Congress. Poster presentation.

Hull, R.N. *et al.* 1993. Decreasing the risk of an unacceptable ecological risk assessment. 14th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November, 1993. Houston, Texas.

Suter, G.W., Stack, M.E., **Hull, R.N.**, McGinn, C.W., Cornaby, B.W, Hadden, C.T., and F,A. Zafran. 1993. A logic for balancing human health risk-based and ecological risk-based remedial goals and assessments. Society for Risk Analysis. Oral presentation. December, 1993. Savannah, Georgia.

Hull, R.N, Suter, G.W., and Bonczek, R.R. 1993. Analysis of results of a preliminary ecological risk assessment to guide baseline ecological risk assessment efforts at a large, complex DOE site. Society for Risk Analysis. Poster presentation. December 1993. Savannah, Georgia.

Bonczek, R.R., **Hull, R.N.**, and Suter, G.W. 1993. A strategy for ecological risk assessment at a large, complex DOE site. Society for Risk Analysis. Poster presentation. December, 1993. Savannah, Georgia.

Hull, R.N., and Belluck, D.A. 1992. Shifting from baseline to life cycle risk assessments to estimate ecological risks and impacts at hazardous waste sites. 13th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Poster presentation. November 1992. Cincinnati, Ohio.

Hull, R.N., and Ruby, S.M. 1991. Histological effects of low levels of inorganic lead to the pituitary and ovary of rainbow trout (*Oncorhynchus mykiss*). 12th Annual Meeting of the Society of Environmental Toxicology and Chemistry. Oral presentation. November, 1991. Seattle, Washington.

REPRESENTATIVE PROJECT EXPERIENCE

- Project manager and senior scientist for a Review of the Status of Tier 1 and Tier 2 Substances in the Great Lakes Basin under the Canada-Ontario Agreement, for Ontario Ministry of the Environment (2013-2014);
- Project manager and senior advisor for a Comprehensive Ecological Hazard Evaluation of Four Organic Flame Retardants Subject to Chemical Management Plan Assessment, for Environment Canada (2013);
- Project manager and senior advisor for a Comprehensive Literature Search, Data Evaluation and Summary of the Environmental Fate, Concentrations, Persistence, Bioaccumulation and Ecotoxicity of Phthalates Subject to CMP Assessment, for Environment Canada (2013);
- Project Manager and Senior Reviewer for a risk assessment for wildlife with aquatic-based diets (Confidential Client, 2013);
- Project Manager and Senior Ecological Risk Assessor for an ERA for a municipal waste water treatment plant in Ontario (Confidential Client, 2013);
- Project Manager and Senior Reviewer for a risk assessment of an industrial site containing arsenic in Northern Ontario (Confidential Client, 2013);
- Program Manager for the Steering Committee for Canada's Lower Columbia Ecosystem Management Program, on behalf of Teck (2012-ongoing);
- Advisor to EBA Consulting (BC) regarding the ecological risk assessments for five mine sites in NWT (2011-2013);
- Ecological Screening Evaluation for a Preliminary Endangerment Assessment (PEA) report for an industrial recycling facility (metals, PCBs) in California (2012);
- Senior review of aquatic classifications under the Global Harmonization System (GHS) for numerous chemical products (Confidential Client, 2012);

- Project Manager and Senior Advisor/Reviewer for a white paper developed on the Use of Toxicity Testing as an Alternative to Benthic Community Monitoring related to Zequanox™ Use at Decew II Generating Station (Marrone BioInnovations, 2012);
- Hazard screening of selected contaminants in the Great Lakes Basin (water, sediment, biota) for Ontario Ministry of the Environment (2011-2012);
- Ecological risk assessment for PCBs in the Former Spill Pond, Georgia-Pacific Canada, Thorold, ON (2011-2012);
- Human health and ecological screening-level risk categorization of herbicides for Conoco Phillips Canada (2011-2012);
- Project manager and senior ecological risk assessor for a commercial site under development under O. Reg. 153/04 in Markham ON contaminated with VOCs (2011/2012);
- Senior advisor/reviewer for EBA's project "Calculation of Lead Site-specific Soil and Food Ingestion Guideline for Livestock Receptors" (2011);
- Senior ecological risk assessor for a commercial site in St. Thomas contaminated with VOCs, planned to undergo redevelopment under O.Reg. 153/04 (2011/2012);
- The Assessment of Climate Change Impact on Water Quality Guideline Development for CCME (2010);
- Hazard screening of contaminants of emerging concern in the Great Lakes Basin for Ontario Ministry of the Environment (2010);
- Ecological risk assessment for PCBs and dioxin-like PCBs in Beaverdams Channel, near Thorold, ON (2010);
- Review of aquatic toxicity of disinfection by-products for a large municipality in Ontario (2009);
- Senior advisor for an ecological risk assessment of a large former industrial site in eastern Ontario (O. Reg. 15/304);
- Project manager for the human health and ecological risk assessment for a commercial site in Kingston contaminated with VOCs, planned to undergo redevelopment to residential and parkland, evaluated under O.Reg. 153/04 (2008/2009);
- Project manager and senior ecological risk assessor for the human health and ecological risk assessment for La Oroya, Peru (2008-2009);
- Project manager and senior ecological risk assessor for the human health and ecological risk assessment for the Port of Callao, Peru (2008-2009);
- Project manager and senior advisor/reviewer for the literature search, review, and development of Provincial Water Quality Objectives for Ontario Ministry of the Environment for boron, chloride, and 1,4-dioxane (2007);
- Project manager and senior advisor/reviewer for the literature search and review for Ontario MOE of aquatic toxicological data for inorganic fluorides, numerous PAHs, and didecyl dimethyl ammonium chloride, to determine whether recent toxicological data were consistent with older data used to develop the CCME water quality guideline (2006);
- Provided an opinion to a law firm preparing the defence of their mining client related to ecological risk issues (2006);
- Project manager and senior advisor/reviewer for a review for Ontario MOE of previously established aquatic screening benchmark values and aquatic toxicity literature for a number of compounds to review and develop environmental benchmarks for chemicals in municipal effluents (2006);
- Project manager and senior ERA specialist for the Detailed ERA for a secondary lead smelter (RCRA site) in Indiana (2006);
- Review of nickel ecotoxicity in the marine environment for Sherritt International Corporation (2006);
- Lead ecological risk assessor for the off-site area of the Teck lead/zinc smelter in Trail, BC (the largest ERA to be conducted in BC). Included a review of the ecotoxicological literature for metals, de novo development of TRVs for wildlife, and extensive exposure modeling. Participation in public and Technical Advisory Committee meetings (2000 to 2012);

- Planning for the Wide Area Remediation Plan for the off-site area of the Teck Cominco lead/zinc smelter in Trail, BC (2006-2012);
- Lead author for review of terrestrial exposure and effects associated with lead, as part of the update to the U.S. EPA air quality criteria document (2005).
- Project manager and lead ecological risk assessor for the human health and ecological risk assessment for the Arab Abu Said area south of Cairo, Egypt. This area contains over 200 brick factories which burn heavy oil. This work was done to evaluate the health and environmental benefits of converting the brick factories to natural gas. We were a subcontractor to Roche Ltd. (2001-2005);
- Provision of expert advice and review of SSRAs on behalf of the Ontario Ministry of the Environment, in the area of ecological risk assessment (2000 to present).
- Ecological risk assessment technical lead for the City of Greater Sudbury and surrounding area (2003-present);
- Development of the Scope of Work for a human health and ecological risk assessment for the mangrove near the Petrobras Pedra Branca station, near Salvador, Bahia (Brazil). Reviewed available site characterization data and ecological survey and effects data, and worked with local Petrobras staff and biologists to ensure issues relevant to this sensitive ecosystem and the local inhabitants were identified. (October-December 2002);
- Terrestrial risk assessment for a former commercial brownfield site redeveloped into a park. The primary chemicals of concern were lead and cadmium;
- Review of toxic interactions of metals in the aquatic environment, as well as potential approaches for the incorporation of metal interactions in the development of water quality criteria. This work is in support of the CCME re-evaluation of the protocol for development of water quality guidelines for the protection of aquatic life;
- Development of ecological exposure models in support of Ontario Ministry of the Environment's brownfield regulation (O.Reg. 153/04);
- Environmental evaluation of alternatives to sodium chloride (NaCl) road salts, on behalf of Environment Canada. This document was used by Environment Canada in stakeholder consultation meetings;
- Predictive aquatic and terrestrial ecological risk assessment for a proposed thermal treatment facility in Ontario. A novel chemical-screening process was used to identify chemicals of concern, and food chain modeling was used to assess risks associated with chemicals that bioaccumulate. Participated in public meetings;
- Site-specific terrestrial ecological risk assessment for a commercial property in Ottawa. COCs included metals and PAHs. Predictive modeling of chemical uptake into earthworms suggested potential ecological risks to vermivores; thus, earthworm samples were taken to allow site-specific exposure modeling of chemicals in the food chain;
- Human health and ecological risk assessment for a former industrial property which had soil and groundwater contamination on-site, and surface water and sediment contamination off-site. Risks to aquatic organisms, and avian and mammalian wildlife were evaluated. COCs were metals (Pb, Cd, Zn) in the aquatic environment and VOCs in groundwater;
- Aquatic risk assessment for a proposed nickel smelter. Included characterization of current ecosystem properties and health as a reference for future studies conducted during the smelter operation;
- Aquatic and terrestrial ecological risk assessment for a wood preserving site on the Fraser River contaminated with chlorophenols, metals and PAHs in British Columbia. Integration of human health risk assessment with ecological risk assessment results;
- Ecological risk assessment for a river adjacent to a landfill, contaminated with metals, hydrocarbons;
- Human and ecological risk assessment for a site contaminated with PAHs. The change in land use to a park necessitated evaluation of particular human and ecological receptors.
- Terrestrial ecological risk assessment for an Ontario Hydro transformer site contaminated with arsenic. Risks to wildlife in the adjacent wetland were critical to decision-making.

- Aquatic risk assessment for a harbour contaminated with metals and PAHs;
- Project manager and lead risk assessor - Aquatic risk assessment for a RCRA site in the southern U.S. contaminated with chromium;
- Screening human health review of beryllium toxicity for the Hydro One Hamilton Beach Transformer Station, Hamilton, Ontario;
- Ecological risk assessment for a river contaminated with metals and dioxins/furans from a pulp and paper mill. Receptors included the fish community, benthic macroinvertebrate community and piscivorous wildlife;
- Human health risk assessment for a site with VOC contamination in groundwater in the US;
- Ecological risk assessment team leader (while at ORNL) for several screening-level and baseline ecological risk assessments in Ohio and Tennessee. Contaminants present included metals, PAHs, PCBs, metals, dioxins and radionuclides. ERA receptors included the fish communities, benthic communities, threatened and endangered species, small mammals, piscivorous wildlife, terrestrial plants, soil invertebrates, and wetlands.

PEER REVIEWS

- Peer review of the ecological portion of a risk assessment completed by Golder Associates which was conducted in the spirit of Ontario Regulation 153/04 (as amended) for a commercial/industrial site in Cornwall, ON (2012);
- Peer review of the ecological portion of a risk assessment completed by MTE-GlobalTox which was conducted in the spirit of Ontario Regulation 153/04 (as amended) for a commercial site in Kitchener, ON (2012);
- Peer review of the ecological portion of a risk assessment completed by exp which followed the Ontario Regulation 153/04 format, for a Celenese Facility in Edmonton, Alberta (2012);
- Senior advisor and peer reviewer for three ecological risk assessments prepared by EBA for mine sites in NWT assessed using federal guidance (2010-2012);
- Peer review of the ecological portions of the Durham-York generic risk assessment feasibility study for the proposed energy-from-waste facility under consideration by Durham and York regions (2009);
- Scientific expert review panellist for the *Review of Formative Evaluation of the Federal Contaminated Sites Action Plan* completed for Goss Gilroy, on behalf of FCSAP (2008);
- Peer review of the ERA for a proposed new large-scale petroleum refinery facility in New Brunswick (2008);
- Peer review of the ERA for a refinery expansion project in Ontario (2008);
- Peer review of the ERA of a due diligence risk assessment for a confidential client in Ontario;
- Peer reviews of numerous Site-specific Risk Assessments (SSRAs) and Due Diligence Risk Assessments for numerous engineering/consulting firms under the Ontario *Guideline for Use at Contaminated Sites in Ontario* and Ontario Regulation 153/04.

Attachment B – WSP Standard Operating Procedures

WSP Standard Operating Procedures

Table of Contents

SOP Number and Title

1 Note Taking and Log Book Entries
2 Sample Container, Preservatives, and Holding Times (Table 1)
3 Groundwater Sampling (revised July 2012)
4 Surface Water and Sediment Sampling
5 Sediment/Sludge Sampling with Ekman or Ponar Dredge
6 Sediment/Sludge Sampling Using Hand Corer
7 Sludge Sampling with Sludge Judge
8 Sludge Sampling with Coring Tube
9 Soil Sampling Using Bucket Auger
10 Split Spoon Soil Sampling (Figure 2)
11 Soil Sampling Using Hand Trowel
12 Chip, Wipe, and Sweep Sampling
13 Concrete Core Sampling
14 Waste Pile Sampling
15 Decontamination of Drilling Equipment
16 Decontamination of Submersible Pumps
17 Decontamination of Water Level Indicators
18 Decontamination of Interface Probe
19 Decontamination of Sampling Equipment
20 Sample Shipping Procedures
21 Field QA/QC Samples
22 Soil Head Space Screening (Field Procedure)
23 Underground Utility Locating
24 Soil Sampling Using GeoProbe® System or Equivalent
25 Groundwater Sampling Using GeoProbe® System or Equivalent
26 Managing Investigation Derived Waste
27 Soil Sampling Using a Rotosonic Sampler
28 Sampling of Private Water Supply Wells
29 Aquifer Pumping Tests
30 In Situ Single Well Hydraulic Conductivity Test (Slug Test)

31 Screening of Soils for Metals Using a Delta Handheld X-Ray Fluorescence Analyzer
32 Composite Soil Sampling
33 Sediment Pore Water Sample Collection Performed by Laboratory
34 Multi-Habitat Macroinvertebrate Collection Procedure

Standard Operating Procedure – 1

Note Taking and Log Book Entries

Materials:

Permanently bound log book (no spiral-bound log books)

Black or blue ballpoint pen (waterproof ink)

Procedure:

1. Use black or blue ballpoint pen with waterproof ink. Felt-tip pens should not be used.
2. Reserve the inside front cover for business cards from key personnel who visit the site (including the person in charge of the log book).
3. On the first page of the log book, place a return for reward notice, WSP's phone number, and the project manager's name.
4. Enter the following on the second page of the log book: project name, project number, project manager's name, onsite contacts, onsite telephone number and address, telephone numbers for all key personnel, and emergency fire and medical telephone numbers.
5. Number each page, initial each page, and put the date at the top of each page. Start a new page for each day. At the end of a day, summarize the day's activities, sign the page, and put a slash through the rest of the blank lines. Start the next day on a new page.
6. Enter the time (in military time, e.g., 0830) in the left column of each page when an entry is recorded in the field notebook.
7. If a mistake is made in an entry, cross out the mistake with one line and initial the end of the line.
8. At all times, maintain the chain of custody on the field log book.

Content:

1. Be sure that log book entries are LEGIBLE and contain accurate and inclusive documentation of project field activities.
2. Provide sufficient detail to enable others to reconstruct the activities observed.
3. Thoroughly describe all field activities while onsite. Be objective, factual, and thorough. Language should be free of personal feelings or other terminology that might prove inappropriate.
4. Describe problems, delays, and any unusual occurrences such as wrong equipment or breakdowns along with the resolutions and recommendations that resulted.
5. Fully document any deviations from or changes in the work plan.
6. Describe the weather and changes in the weather, particularly during sampling events.

7. Sketch a map of the facility or areas onsite where activities are occurring, especially the location of sampling points.
8. During sampling activities, record all information pertaining to the sampling event. Include descriptive locations and diagrams of the sample locations, time, sample media, analysis, sampling procedure, equipment used, sizes and types of containers, preservation and any resulting reactions, sampling identification (especially for duplicate samples), shipping procedures (record airbill numbers), and addresses.
9. Note decontamination or disposal procedures for all equipment, samples, and protective clothing and how effectively each is performed.
10. If possible, photograph all sample locations and areas of interest. Maintain a photographic log in the field log book and include:

    Date, time, photographer, name of site, general direction faced, description of the subject taken, and sequential number of the photograph and the roll number.
11. Record the names and affiliations of key personnel onsite each day.
12. List all field equipment used and record field measurements, including distances, monitoring and testing instrument readings (e.g., photoionization detector (PID), organic vapor analyzer (OVA), pH, conductivity, model numbers, etc.), and calibration activities.
13. Record proposed work schedules and changes in current schedules in the log book.
14. Describe site security measures.
15. Include drum inventory for all investigation-derived waste (IDW) materials generated during site activities. Provide information on how IDW material was labeled.

Standard Operating Procedure - 2

Sample Container, Preservatives, & Holding Times

Scope:

This operating procedure describes the ways and means of selecting the appropriate sampling containers for environmental sampling.

Application:

The purpose of this procedure is to assure that sample volumes and preservatives are sufficient for analytical services required under EPA-approved protocols.

Materials:

Sample containers

Sample container labels

Indelible (waterproof) markers or pens

Clear tape

Procedures:

1. Refer to Table 1 for minimum sample volume and glassware types required for sampling a particular matrix and compound class.
2. Select the appropriate glassware (i.e., bottles or jars) from those provided by the analytical laboratory. Verify that the analytical laboratory has provided the correct number of sample containers and the correct preservatives for the project per the sampling plan requirements.
3. The analytical laboratory should always provide extra sample containers for all analytical parameters in case of breakage or other problems encountered in the field. This is particularly true for VOC sample containers (i.e., 40-ml vials).
4. Report any discrepancies or non-receipt of specific types of sample containers to the Quality Assurance Officer immediately. Arrangements should be made with the laboratory to immediately ship the missing or additional sampling containers to the project site.
5. Apply WSP sample labels to the sample containers.
6. Information on the sample labels should contain the following data:

    Site/Project name

    Project/Task number

    Unique sample identification number

    Sample date

    Time of sample collection (military system, e.g., 0000 to 2400 hours)

Analytical parameters

Preservative

Sampling personnel

7. Once sample containers are properly labeled, the sample labels should be wrapped with clear tape to prevent deterioration of sample label.
8. Proceed with the sample collection per the sampling plan requirements.
9. Collected samples should be immediately placed in an iced cooler to maintain as close as possible a 4*C atmosphere for shipment to the analytical laboratory. Follow sample shipping procedures detailed in Sample Shipping Standard Operating Procedures.
10. Recommended order of sample collection:

    In-situ measurements (e.g., temperature, pH, specific conductance)

    Volatile organic analytes (VOA)

    Purgeable organic carbon (POC)

    Purgeable organic halogens (POX)

    Total organic halogens (TOX)

    Total organic carbon (TOC)

    Extractable organics

    Total petroleum hydrocarbons (TPH)

    Total metals

    Dissolved metals

    Microbiologicals

    Phenols

    Cyanide

    Sulfate and chloride

    Turbidity

    Nitrate and ammonia

    Radionuclides

Table 1 – Sample Containers, Preservatives, and Holding Times

Parameter	Matrix	Sample Container Size and Type	Preservatives	Holding Time
Metals, except mercury and hexavalent chromium	Solid	4-oz. glass jar	Cool to <6° C	180 days
Mercury	Solid	4-oz. glass jar	Cool to <6° C	28 days
Hexavalent chromium	Solid	4-oz. glass jar	Cool to <6° C	24 hours
Metals, except mercury and hexavalent chromium	Aqueous	500-ml plastic container with Teflon-lined plastic cap	HNO3, pH<2 Cool to <6° C	180 days
Mercury	Aqueous	500-ml plastic container with Teflon-lined plastic cap	HNO3, pH<2 Cool to <6° C	28 days
pH	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	ASAP
Total Organic Carbon	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	28 days
Water Holding Capacity	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	N/A
Cation Exchange Capacity	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	180 days

Parameter	Matrix	Sample Container Size and Type	Preservatives	Holding Time
Particle Size Analysis	Solid	Three 4oz glass jar with Teflon-lined cap; 1 Gallon zip lock bags	Cool to <6°C	N/A
14-Day Earthworm survival & Biomass Toxicity Test	Solid	plastic of PTFE containers 2 L needed	Cool to 4°C; store in dark until testing	72 hours
Lead	Solid	Three 4oz glass jar with Teflon-lined cap	Cool to <6°C	N/A
P. promelas and C.dubia chronic toxicity test	Aqueous	5 gal plastic container; no headspace	Cool to 4°C; store in dark until testing	N/A
pH	Aqueous	Two 250 mL plastic	Cool to <6°C	1 none; 1 HNO3<2 pH
Hardness	Aqueous	Two 250 mL plastic	Cool to <6°C	1 none; 1 HNO3<2 pH
Total Recoverable Phenols	Solid	Three 4oz glass jar with Teflon-lined cap	Cool to <6°C	N/A
H. Azteca 28-Day Survival and Growth C. dilates 10-Day toxicity test	Solid	plastic or PTFE containers; can use 1L or gal; need 5L of sample	Cool to 4°C; no headspace; store in dark until testing	2 weeks; up to 8 weeks under nitrogen
Interstitial pore water Ammonia	Solid	3 gallon covered plastic bucket	Cool to 4°C	14 days
AVS/SEM	Aqueous	4oz glass jar; no headspace	None	14 days
Total Suspended Solids	Aqueous	1L plastic bottle	none	7 days
Total phenols	Aqueous	1L plastic bottle	H2SO4 <2 pH	28 days
Metals	Benthic	5 grams in plastic bags	Frozen, ship on dry ice	N/A

Standard Operating Procedure - 3

Groundwater Sampling Procedures

1.1.1.1 Scope and Application

This groundwater sampling procedure is designed to ensure that collected groundwater samples will be representative of groundwater present in the aquifer or target formation and that the groundwater samples have not been altered or contaminated by the sampling and handling procedures. These procedures can be applied to permanently, or temporarily, installed monitoring wells, temporary wells constructed using "direct-push" techniques, wells with installed plumbing, remedial groundwater treatment systems, and excavations where groundwater is present. The topics detailed in this Standard Operating Procedure (SOP) include equipment and supply selection, equipment construction materials, and purging and sampling techniques.

1.1.1.2 Acronym List

bgs	below ground surface
C	Celsius
CID	casing inside diameter
DI	deionized
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DTW	depth to water
Eh	redox potential
HASP	Health and Safety Plan
L/min	liters per minute
LNAPL	light non-aqueous phase liquid
mg/l	milligrams per liter
mV	millivolts
NAPL	non-aqueous phase liquid
NTU	Nephelometric Turbidity Unit
ORP	oxygen reduction potential
pH	hydrogen ion activity
PID	Photoionization Detector
PPE	Personal Protective Equipment
PVC	polyvinyl chloride
QAPP	Quality Assurance Project Plan

SOP	Standard Operating Procedure
SU	Standard Units
TD	total depth
TOC	top of casing
µm	micron
µS/cm	microsiemens per centimeter
VOCs	volatile organic compounds

Materials

- Field notebook
- Personal Protective Equipment (PPE)
- Groundwater monitoring data log forms
- Well key(s), as needed
- Adjustable wrench or manhole wrench
- Plastic sheeting
- Air quality monitoring equipment (e.g., photoionization detector [PID]), as needed
- Flashlight or mirror
- Electronic water level indicator or interface probe
- Pump or bailers, tubing, and associated lanyard materials
- Water quality meter(s) with calibration reagents and standards
- Field test kits, as needed
- Pocket knife or scissors
- Deionized (DI) water
- Power supply, as needed
- Buckets or drum(s) for water storage
- Sample bottles, labels, indelible markers, and clear tape

1.1.1.3 Compulsory Prerequisites and Background Information

Before conducting field activities, review the site Health and Safety Plan (HASP) and determine the proper level of PPE. The reader is encouraged to read this entire SOP before beginning any onsite activities. This SOP is designed to provide the user with a general outline for conducting groundwater sampling and assumes the user is familiar with basic field procedures, such as recording field notes, utility location, sample shipment procedures, sample collection and quality assurance procedures, equipment decontamination, and investigation derived waste management procedures. This SOP does not cover the development of a sampling and analysis plan, nor does it cover the selection of analytical

procedures, or evaluation of the analytical results. These topics require a significant amount of planning and are more appropriately addressed in a site-specific work plan.

Before sampling, be sure to review the project specific work plan or Quality Assurance Project Plan (QAPP) and any applicable state and federal guidelines or sampling procedures. Groundwater samples may be collected from a number of different configurations. Each configuration is associated with a unique set of sampling equipment requirements and techniques. As possible, monitoring wells or borings should be sampled by starting with the upgradient (or clean locations) and proceeding downgradient (in the order from least to most contaminated locations) for the remaining monitoring wells or borings.

1.1.1.4 Equipment Selection Considerations

Use groundwater purging and sampling equipment constructed of only non-reactive, non-leachable materials that are compatible with the environment and the selected analytes. Additional supplies such as reagents, preservatives, and field measurement equipment are often necessary.

Pump Selection

Groundwater sampling is accomplished by using in-place plumbing and dedicated pumps or by using portable pumps/equipment when dedicated systems are not present. In selecting groundwater purging and sampling equipment, give consideration to:

- the depth of the well
- the depth to groundwater
- the volume of water to be evacuated
- the sampling and purging technique
- the analytes of interest

Sampling equipment will usually consist of peristaltic pumps, variable speed electric submersible pumps, bladder pumps, inertial lift pumps, or bailers.

- Restrictions and Precautions:
- Follow all manufacturer's instructions for assembly, operation, and maintenance specific to your equipment
- For wells where the water level is below the limit of suction (approximately 25 feet to 30 feet below ground surface, bgs), and/or where there is a large volume of water to be purged, a peristaltic pump should not be used
- Purging and sampling with an inertial lift pump (e.g., polyethylene tubing with a bottom check valve) are performed by oscillating the tubing at the intake depth to drive a column of water to the surface. Inertial lift pumps cause a surging action that may cause increased turbidity, loss of volatiles, aeration, and degassing of samples. These pumps can be used when collecting non-sensitive samples; however, should not be used for volatile organic compounds (VOCs) or gas-sensitive samples. A peristaltic pump may be attached to the sample tubing and used to pump water to the surface

- VOCs and extractable organics should not be sampled with a peristaltic pump unless a vacuum jug or gravity method is employed and the method was previously approved.
- Bailers may also be used for purging in appropriate situations; however, their use is discouraged by many programs. Because bailers have been shown to increase turbidity while purging and sampling, bailers should be avoided when sampling for trace element, metal, polychlorinated biphenyls, and pesticide constituents. Bailers, if improperly used, may also strip VOCs from the water column being sampled
- Bailers with a controlled flow bottom when should be used when collecting volatile organic samples
- Before use, install a check valve at the end of the purge tubing to prevent backflow
- Pump housing and tubing parts must be compatible with the analytes of interest. Parts can be composed of various materials, usually polyvinyl chloride (PVC), Teflon, polyethylene, polypropylene, and stainless steel, or other non-inert materials; be sure to use non-metallic or stainless steel tubing if sampling for metals
- Electrical cords and any cabling must be sealed in Teflon, polyethylene, or polypropylene, or be stainless steel
- Lanyard material must be non-reactive, non-leachable material (e.g., cotton twine, nylon, stainless steel, Teflon coated, polyethylene, or polypropylene; stainless steel, Teflon coated, polyethylene and polypropylene lanyards may be decontaminated for future use

1.1.1.5 Groundwater Indicator Parameter Monitoring Equipment

WSP regularly conducts field testing for the following in-situ or field-collected parameters: groundwater level elevation, hydrogen ion activity (pH), specific conductance (conductivity), temperature, dissolved oxygen (DO), oxygen reduction potential (ORP), and turbidity. Field testing is usually conducted due to the unstable nature of these parameters; laboratory determinations will likely not be representative of field conditions.

1.1.2 pH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

1.1.3 Specific conductance

Specific conductance, or conductivity, measures the ability of water to conduct an electric current. It is generally reported in microsiemens per centimeter (µS/cm), as natural waters commonly exhibit specific conductance well below 1 µS/cm. Total dissolved solid concentrations may be approximated from specific conductance data; high readings may indicate contamination, especially if the readings are elevated compared to background. Alternatively, elevated specific conductance may indicate inadequate well development, grout contamination, or an inadequate grout seal.

1.1.4 Temperature

Temperature is not necessarily an indicator of groundwater chemical stabilization, and is generally not very sensitive in distinguishing between stagnant casing water and formation water. Nevertheless, temperature is important for data interpretation. For example, stabilized temperature readings that are representative of typical groundwater conditions help demonstrate that the sample was collected in a manner that minimized exposure to elevated temperature variations, e.g., heating from the electric motor of a submersible pump. Elevating the temperature of a sample may result in loss of VOCs or the progression of chemical reactions that may alter the sample quality in an undesirable manner.

1.1.5 DO

DO has been demonstrated to be a reliable indicator of the chemical stabilization of purge water under most groundwater purging and sampling circumstances. Concentrations of DO in groundwater generally range from 1 to 4 milligrams per liter (mg/l) and should only be measured with a flow-through cell. Relatively low DO concentrations (< 1 mg/l) in groundwater may indicate the biodegradation of organic contaminants, including VOCs. DO is a good indicator when sampling for VOCs, because erratic or elevated DO readings may reflect procedures that are causing excessive agitation and aeration of the ground water being drawn from the well and subsequent loss of VOCs. Artificially aerated groundwater may also adversely affect dissolved metals analyses.

1.1.6 ORP

ORP is a numerical index of the intensity of the oxidizing or reducing conditions within an aqueous solution such as ground water. Oxidizing conditions are indicated by positive potentials and reducing conditions are indicated by negative potentials. ORP measurements are generally expressed in millivolts (mV). The ORP of natural (uncontaminated) groundwater typically ranges from +500 to -100 mV. Groundwater contaminated with organic compounds generally exhibits depressed ORP values compared to background conditions and may exhibit ORP values as low as -400 mV. ORP must be measured with a flow-through cell and may not be an appropriate stabilization parameter for some groundwater conditions.

1.1.7 Turbidity

Turbidity, which is the visible presence of suspended mineral and organic particles in a ground water sample, can be useful to measure during purging. Relatively high or erratic measurements may indicate inadequate well construction, development or improper sampling procedures, such as purging at an excessive rate that exceeds the well yield. Purging and sampling in a manner that produces low-turbidity water is particularly important when analyzing for total metals, which may exhibit artificially elevated concentrations in high-turbidity samples. Generally, the turbidity of in-situ groundwater is very low (at or below 10 Nephelometric Turbidity Units, NTUs); however, some groundwater zones may have natural turbidity higher than 10 NTUs.

The groundwater indicator parameters selected for field testing should be determined by the project team. Field testing of groundwater indicator parameters should be conducted with cleaned, calibrated equipment. The following procedures should be followed and all observations and measurements recorded in the field book.

- Select water quality monitoring equipment that meets the project's data quality objectives and applicable regulatory requirements for field testing measurements
- Record manufacturer name, model number, and identifying number such as a serial number for each instrument
- Follow the manufacturer's instructions for assembly, operation, calibration, and maintenance specific to your equipment
- Ensure that all equipment is in proper working condition, not damaged, and that batteries are properly charged before using the equipment for field testing measurements
- Calibration of instruments should occur in the field, as close to the time of use as possible and, at least, be at the frequency suggested by the manufacturer
- Although water quality monitoring equipment may vary in configuration or operation, project specific calibration requirements must be met
- Document the time, date, and instrument reading of acceptable calibration and calibration verification for each instrument unit and field test or analysis, linking this record with affected sample measurements
- Document the date of receipt, expiration date, date of first use for all standards and reagents, grade and concentration (or other value) for the standard in the appropriate measurement units
- Thoroughly rinse the instrument with DI water and fresh standards or reagents when calibrating or verifying the calibration or when taking sample measurements. For in-situ measurements, ensure adequate flushing of the instrument with fresh sample water prior to taking measurements. Residual remaining on the instrument may affect the measurement
- If calibration fails to meet criteria, follow the manufacturer's instructions for corrective action to correct instrument performance; record discontinuation of equipment due to non-compliance with calibration failure

1.1.7.1 Field Measurements of Groundwater Indicator Parameters

Indicator parameters are measured in the field to evaluate well stabilization during purging, provide information on general ground water quality, evaluate well construction, or indicate when well maintenance is needed. Indicator parameter data also may be helpful in evaluating the presence of groundwater contamination.

After calibrating the groundwater quality monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. Field testing measurements should be collected in accordance with the purge and sampling schedules provided in Sections 9.8 and 9.9. The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in a flow-through cell, sample cup or beaker, or at the desired depth in the well or borehole, and wait for stabilization of the reading before recording the measurement
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

- Specific conductance is sensitive to temperature. If the meter is equipped with manual temperature compensation, adjust the conductivity meter to the water temperature per manufacturer's instructions. If the water temperature varies significantly from 25°Celcius (C), measure the temperature with a calibrated device, record the temperature, and correct for temperature according to the manufacturer's specifications
- ORP meters may present redox potential (Eh) values; depending on the desired results, conversion between ORP and Eh may be necessary
- **DO and ORP readings must be obtained in a manner in which the sample is not exposed to air prior to the measurement (e.g., flow-through cell).**

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample (e.g., monitoring well identification number, outfall number, station number or other description)
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

1.1.7.2 Set-Up Procedures

Once the equipment and supplies have been selected, gathered, and calibrated, prepare for groundwater sampling. As the following steps are completed, note all observations and measurements on the groundwater monitoring data log.

- Verify locations of wells or borings, media to be sampled, and analytes
- Record the approximate ambient air temperature, precipitation, wind, tidal conditions, and other field conditions the field book. In addition, any site-specific conditions or situations that could potentially alter the ground water samples or water level measurements should be recorded
- Inspect well for soundness of protective casing and surface ground seal
- Survey around the base of the well and wellhead with a PID, as necessary
- Remove the well cover and all standing water around the top of the well casing before opening the well cap
- New plastic sheeting should be placed on the ground surface around the well or boring to prevent contamination of the pumps, hoses, lanyards, etc., in the event they accidentally come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event; keep the plastic as clean as possible and replace as necessary
- Unlock and carefully remove well cap, if present; allow the groundwater level to equilibrate after removing the cap before conducting any downhole testing or measurements

- Survey well casing with a PID, as necessary
- Survey breathing zone to ensure that the level of PPE is appropriate
- Position fuel powered equipment downwind and at least 10 feet from the well head or boring. Make sure that the exhaust faces downwind

1.1.7.3 Groundwater Level and Depth Measurement Procedures

The measurement of the groundwater level in a well is frequently conducted in conjunction with groundwater sampling to determine the "free" water surface. This potentiometric surface measurement can be used to establish groundwater direction and gradients. Groundwater level and well depth measurements are needed to determine the volume of water in the well casing prior to purging the well for sampling purposes. The following procedures should be followed and all observations and measurements recorded in the field book.

- Measure and record the height of the top of the well riser above the ground; if well is a flush mount, measure and record the top of the well riser below the ground surface
- Measure the casing inside diameter (CID) and record in inches
- From the top of the casing (TOC) at the surveyor's mark, if present, measure the depth to water (DTW) to the nearest 0.01-foot with an electronic water level indicator (record in feet below TOC); if no mark is present, mark a location with a metal file or indelible marker on the north side of the casing for future reference
  - All DTW measurements must be made and recorded to the nearest 0.01 foot
  - Water level measurements from boreholes, piezometers, or monitoring wells used to define the water table or a single potentiometric surface should be collected within less than 24 hours. In certain situations, water level measurements should be made within a shorter interval, if possible:
  - Aquifers influenced by tides, recent precipitation, barometric pressure, river stage, bank storage, impoundments and/or unlined ditches, intermittent pumping of production, irrigation, or supply wells
- Total depth (TD) of the well or boring should not be conducted immediately before purging and sampling; as possible, delay purging and sampling activities for at least 24 hours after TD measurement or for a time sufficient to meet the purge stabilization criterion for turbidity. Alternately, measure TD after sample collection
  - All TD measurements must be made and recorded to the nearest 0.1 foot
  - Because of tape buoyancy and weight effects encountered in deep wells with long water columns, it may be difficult to determine when the tape end is touching the bottom of the well. Sediment in the bottom of the well can also make it difficult to determine total depth. Care must be taken in these situations to ensure accurate measurements
  - As a cautionary note, when measuring TD with an electronic water level indicator, measure and add the length of the probe beneath the circuit closing electrodes to the depth measured to obtain the true TD
- Inspect water surface in the well or boring; use flashlight if necessary. Note any observable floating product (light non-aqueous phase liquids; LNAPLs) and sinking free product layer (dense non-

aqueous phase liquids; DNAPLs). Measure the thickness of the LNAPL and/or DNAPL layer using an appropriate interface gauging probe or a weighted tape coated with the appropriate reactive indicator paste for the suspected NAPL. DO NOT PURGE OR SAMPLE GROUNDWATER IN A WELL CONTAINING LNAPL. NAPL sampling is discussed in Section 9.9

1.1.7.4 Groundwater Purging

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of actual aquifer conditions.

The selection of the purging technique and equipment is dependent on the hydrogeologic properties of the aquifer, especially depth to groundwater and hydraulic conductivity. The intent of proper purging is to stabilize the groundwater level and minimize the hydraulic stress to the hydrogeologic formation. Every attempt must be made to match the pumping rate with the recharge rate of the well before evaluating the purging completion criteria.

In order to determine when a well has been adequately purged, geochemical parameters should be monitored, at a minimum, the pH, specific conductance and turbidity of the groundwater removed during purging and, in the case of permanent monitoring wells, the volume of water removed should be observed and recorded. Document and report the following, as applicable:

- Purging rate
- Drawdown in the well, if any
- Pump or tubing intake placement.
- Length and location of the screened interval

There are several purging strategies that may be used, depending on specific conditions encountered for given sampling situations. Purging options detailed in this SOP are:

- Traditional Multiple Volume Purge
- Low-Flow Purge
- Temporary Well Purge
- Private Water Well or In-Place Plumbing Purge

No-flow groundwater sampling techniques are discussed in Section 9.8.

1.1.7.5 Multiple Volume or 'Traditional' Purge

A multiple volume or 'traditional' purge involves removing a minimum of three well volumes of water before sample collection. Generally, an adequate purge is achieved when three to five volumes have been removed. The following procedures should be followed and all observations and measurements recorded in the field book.

- Calculate the volume of water in a well or boring using the following equation
- Volume (in gallons) = (TD – DTW)*CID2*0.041; where:
- TD = total depth (feet)

- DTW = depth to water (feet)
- CID = casing inner diameter (inches)
- Alternately, the volume of water in a well or boring may also be calculated by multiplying the water column height by the gallons per foot of water for the appropriate well or boring diameter:

CID	Gallons per foot of water	Gallons per foot, three water columns
0.75-inch	0.02	0.06
1-inch	0.04	0.12
1.25-inch	0.06	0.18
2-inch	0.16	0.48
3-inch	0.37	1.11
4-inch	0.65	1.98
5-inch	1.02	3.06
6-inch	1.47	4.41
12-inch	5.88	17.61

- Calculate the total volume of the pump, associated tubing and container for in-situ measurements (flow-through cell), if used, using the following equation:

  Volume (in gallons) = P + ((0.0041)*D2*L) + fc; where:

  P = volume of pump (gallons)

  D = tubing diameter (inches)

  L = length of tubing (feet)

  fc = volume of flow-through cell (gallons)
- Install the pump or tubing to the depth prescribed in the work plan or QAPP, as dictated by the purge and sampling method
  - for wells with a completely submerged screen or if a bailer is being used, the pump, tubing or bailer should be placed above the screen at the top of the water column
  - for wells with a partially submerged screen, the pump or tubing should be placed within the middle of the saturated portion of the screened interval
- Prepare for the collection of purge water into an appropriate storage container
- Begin purging at a rate that will not cause excessive turbulence in the well; commonly less than 1 gallon per minute. Stabilization parameters should be collected every 0.5 to 1 well volume, or more frequently, record field measurements and observations of water color, suspended particulates,

discoloration of casing, casing diameter and material, any unusual occurrences during sampling, and any pertinent weather details in the field book

- The minimum volume removed must be more than the volume of the pump and sampling tube capacity and the flow through cell (if used). After the minimum purge volume has been removed, review the geochemical measurements to ensure that readings have stabilized. Stabilization occurs when at least three consecutive measurements are within:

Traditional Purge Stabilization Parameters	
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- If the geochemical measurements have not stabilized within five volumes, it is at the discretion of the project team whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible, with respect to turbidity
- If drawdown is observed on initiation of pumping, reduce the pump speed and attempt to match the drawdown of the well. If the drawdown stabilizes, maintain that level, however, if it continues to lower, "chase" the water column until the well is evacuated
- If the well or borehole is purged dry before removing three well volumes, allow well or boring to recharge (commonly 90%) and proceed immediately to sample collection. If adequate sampling volume is available immediately upon completion of purging, the well should be sampled immediately. If recovery exceeds 2 hours, sample as soon as sufficient volume is available, or within 3 hours of purging. If possible, sampling of wells which have a slow recovery should be scheduled so that they can be purged and sampled in the same day, after adequate volume has recovered
- Once purging is complete, proceed to sample collection, as described in Section 9.9

Low-Flow Purge

Low-flow purging is generally used in low permeability units, when calculated purge volumes for traditional purging are excessive, or when vertical characterization within a screened interval is desired.

In-line flow-through cells equipped with water quality meters with continuous readout displays are highly recommended. The following procedures should be followed and all observations and measurements recorded in the field book.

- Calculate the total volume of the pump, associated tubing and container for in-situ measurements (flow-through cell), if used, using the following equation:

  Volume (in gallons) = P + ((0.0041)*D2*L) + fc; where:

  P = volume of pump (gallons)

  D = tubing diameter (inches)

  L = length of tubing (feet)

  fc = volume of flow-through cell (gallons)

- Install the pump or tubing to the depth prescribed in the work plan or QAPP, as dictated by the purge and sampling method
  - for wells with a completely submerged screen, the open or screened interval is less than 10 feet, and the aquifer recovery rate is approximately equal to the purge rate, the intake should be placed in the middle of the screen or open interval
  - for wells with a partially submerged screen, the pump or tubing should be placed within the middle of the saturated portion of the screened interval
- Prepare for the collection of purge water into an appropriate storage container
- The pump should be started at the lowest flow volume, and adjusted higher as long as the maximum drawdown is not exceeded. Purging should not exceed 0.1 to 0.5 liters per minute (L/min)
- Water level should optimally be monitored continuously, but at a minimum, at 30 seconds to 5 minutes intervals (depending on the hydraulic conductivity of the aquifer, diameter of the well, and pumping rate) during purging. Ideally, a steady flow rate should be maintained that results in a stabilized water level (less than 0.3 feet of variation). Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. However, care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record each adjustment made to the pumping rate and the water level measured immediately after each adjustment.
- If the recharge rate of the well is very low, care should be taken to avoid loss of pressure in the tubing line, cascading through the sand pack, or pumping the well dry. In these cases, purging should be interrupted before the water in the well reaches a level below the top of the pump. Sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples.
- During purging, monitor and record geochemical parameters at 30 seconds to 5 minutes intervals. Stabilization occurs once at least three equipment volumes have been purged and the following criteria have been met over three successive measurements made at least three minutes apart:

Low-Flow Purge Stabilization Parameters	
Water Level Drawdown	<0.3 feet
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- Once purging is complete, proceed to sample collection, as described in Section 9.9.

1.1.7.6 Temporary Well Purge

Procedures used to purge temporary ground water monitoring wells differ from permanent wells because temporary wells are installed for immediate sample acquisition. Wells of this type may include open bedrock boreholes or standard well screen and riser placed in boreholes created by hand augering, power augering, or by drilling. They may also consist of a rigid rod and screen that is pushed, driven, or hammered into place to the desired sampling interval, such as a direct push Wellpoint®, a Geoprobe® Screen Point 15/16 sampler or a Hydropunch® sampler. As such, the efforts to remove several volumes of water to replace stagnant water do not necessarily apply because stagnant water is not present. However, exposed probes that are driven through the soil to the desired water sample depth must be purged of a minimum of three probe-rod volumes of water before sampling is conducted. The longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply, to the extent possible, monitoring well purging criteria to re-achieve aquifer conditions.

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, resulting primarily in increased turbidity. Therefore, the goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure. Low turbidity conditions in these types of wells that are completed within the limit of suction are typically and routinely achieved by the use of low-flow/low stress purging techniques using variable speed peristaltic pumps.

Once purging is complete, proceed to sample collection, as described in Section 9.9.

1.1.7.7 Private Water Well or In-Place Plumbing Purge

This procedure describes the method for purging domestic or other private water supply wells with in-place plumbing. The following procedures should be followed and all observations and measurements recorded in the field book.

- Obtain information regarding the exact well location, well construction details, static water depth, well depth, casing diameter, well yield, date drilled and drilling company, previous testing, presence of water treatment components, resource aquifer, and well use. Locate the well on a map for future reference
- The sampling point should be as close to the well as possible, and before the water passes through any treatment systems, heating unit, or storage system. If available, identify the cold water tap or spigot for purging and sampling
- If a suitable sampling point is not available, try to find another well at the site or take steps to minimize the impact of any potential interferences to the sample. Document in the field notebook whether you can or cannot collect a water sample before any treatment or storage components.
- If purging from a tap or faucet, try to remove any aerators, filters, or other devices from the tap before purging
- Start to purge the system by running water from a tap or outside spigot. Observe and record the purge rate for the system. Ensure that sufficient volume is pumped to allow for the complete exchange of water into equipment, holding tanks, etc.
- The minimum volume removed must be more than the volume of the pump, holding tanks, conveyance piping, etc., and the flow through cell (if used). After the minimum purge volume has been removed, review the geochemical measurements to ensure that readings have stabilized. Stabilization occurs when at least three consecutive measurements are within:

Stabilization Parameters	
pH	± 0.1 SU
Specific Conductance	± 3%
Temperature	± 3%
DO	± 0.2 mg/l or 10% (flow-through cell only)
Turbidity	± 10% for values greater than 1 NTU
ORP	± 10 millivolts (flow-through cell only)

- Once purging is complete, proceed to sample collection, as described in Section 9.

1.1.7.8 Groundwater Sample Collection

Sampling is the process of obtaining, containerizing, and preserving (if required) a groundwater sample after the purging process is complete.

- Once purging is complete, field measurements performed to fulfill regulatory requirements, beyond those used to measure for stabilization, should be obtained after purging and before samples are collected for analysis
- While collecting samples, reduce the water flow to a thin stream. The flow rate should be high enough to deliver a smooth stream of water without splashing but low enough to permit filling of the sample bottles without turning the valve down during sampling; the sampling pump should be operated at a maximum rate of 0.25 L/min or the rate of the purging activities to avoid agitating the water; if using a bailer, lower the bailer slowly to avoid agitating the water
- Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
- Groundwater samples should be collected in the order of the volatilization (highest ability to volatilize to the lowest). Sample for VOCs first at a rate less than 0.1 L/min, taking care to remove all air bubbles from the vial and minimize agitation
- Collect remaining organic samples then inorganic samples in the following order of volatilization sensitivity:
  - VOCs
  - Purgeable organic carbon
  - Purgeable organic halogens
  - Total organic halogens
  - Total organic carbon
  - Extractable organics
  - Total metals
  - Dissolved metals
  - Phenols
  - Cyanide
  - Sulfate and chloride
  - Nitrate and ammonia
  - Radionuclides
- If submitting samples for dissolved metals analyses or conducting field testing that use filtered samples, the sample may be field filtered (Section 9.9.1)
- Affix a sample tag or label to each sample container and complete all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection)
- Place clear tape over the tag or label
- Samples for temperature-sensitive parameters should be preserved immediately after collection by placement into an insulated cooler maintained at a temperature of approximately 4°C
- Record sample designation, date, time, and the sampler's initials on the sample tracking form and in the field book

- Complete chain-of-custody forms with appropriate sampling information
- Complete sample packing and shipping in accordance with proper procedures

1.1.7.9 Groundwater Filtration Procedures

Filtered groundwater samples are sometimes used for field kit analyses and should only be collected for laboratory use after approval from the appropriate agency and/or project manager. If filtration is necessary, the following procedures should be followed and all observations and measurements recorded in the field book.

- Use a variable speed peristaltic, bladder, or submersible pump with the in-line filter fitted on the outlet end; pressurized bailers could also be used
- At the pump discharge end, attach a clean 0.45-micron (□m) filter (for organics) or 0.1-µm (for inorganics), or appropriate sized filter, to the tubing
- Turn on the pump to a rate less than 100 mL/min, hold the filter upright with the inlet and outlet in the vertical position and pump groundwater through the filter until all atmospheric oxygen has been removed and the minimum volume of water has been flushed through the filter, in accordance with the manufacturer's specifications
- Collect the filtered samples directly into the sample container from the pump-filter assembly
- If sediment is visible in the sample container after filtration, filter break-through has occurred and the sampling and filtering process should be repeated
- Disassemble the pump head and discard the tubing and filter appropriately

1.1.7.10 No-Flow Passive Sampling Techniques

A number of alternate sampling devices are becoming available, including passive diffusion samplers, equilibrated grab samplers, and other in-situ sampling devices. These devices may be particularly useful to sampling low permeability geologic materials, assuming the device is made of materials compatible with the analytical parameters, meet data quality objectives, and have been properly evaluated. However, the site investigator should ensure the diffusion membrane materials are selected for the contaminants of concern present at the site. Comparison tests with an approved sampling method and diffusion samplers should be completed to confirm that the method is suitable for the site. Manufacturer's specifications should be strictly followed for deployment and use of these samplers.

Equilibration time for diffusion samplers depends on the time required for the environment disturbed by deployment of the sampler to return to ambient conditions and time required by the sampler to equilibrate with the ambient water. To account for this, diffusion samplers are generally deployed a minimum of 14 days prior to sample collection. Though the sampler may be deployed for an extended period (e.g., three months or longer); the analytical results will be the average of the sample equilibration time for the analyte of concern (generally the last 1 to 4 days).

No-flow grab samplers are placed in the well before sampling and remain closed. The water is then collected when the sampler is activated. Samples are either transferred to containers at the well head or the sampler is shipped to the laboratory for analysis. Examples of equilibrated grab samples include Hydrosleeve®, Snap SamplerTM, and Kemmerer Sampler.

1.1.7.11 Non-Aqueous Phase Liquid Sampling

Non-aqueous phase liquid (NAPL) is normally sampled for two reasons:

- Documentation for its existence and thickness
- Determination of the type of product so that the proper analyses can be performed to determine extent. This is only feasible for relatively recent releases as it may not be possible to identify weathered product

Disposable plastic (acrylic, clear PVC) bailers are recommended for sampling. Disposable polyethylene and polypropylene bailers are also acceptable. If a NAPL is identified in a monitoring well during the water level measurement, measure its thickness in the well.

- If the thickness of the NAPL is greater than 0.01 foot or product globules are present, collect a sample using a precleaned disposable bailer by slowly lowering a bailer and allowing the bottom to sink to 1 foot below the water surface to capture LNAPL only
- Measure the product thickness to the nearest 0.01 foot after withdrawing the bailer
- Pour a portion of the product into a glass sample container
- Follow proper sampling, packing, and shipping procedures

Before DNAPL sampling, to the extent practical, remove the full thickness of the LNAPL

- Record the quantity of LNAPL removed in the field book
- Use a peristaltic pump, inertial pump, double check valve bailer, or polyethylene tubing equipped with a bottom check valve for DNAPL sample collection
- Slowly lower the sampling equipment to the well bottom and then raise it slowly, causing as little agitation as possible
- Minimize contact of the bailer with the well casing as it is raised
- Pour a portion of the product into a glass sample container; use the bottom check valve of the bailer for transfer
- Follow proper sampling, packing, and shipping procedures

1.1.7.12 Closing Notes

At the conclusion of groundwater sampling, be sure to:

- Maintain the record of all purging and sampling observations and measurements
- Remove all temporary equipment from the well or borehole
- Decontaminate equipment and supplies
- Properly manage and dispose of all investigation derived waste

Groundwater Monitoring Data Log

Well No./Designation _____ Date: _

Site Data

Site Name: _____ WSP Sampling Team _

Site Address: _____ WSP project No.: _____

Weather Conditions: __

Well Description

Well Location: _

Well Security: _

Casing Material: Inner _____ Outer _

Organic Vapors (PID, OVA, TIP): Wellhead ppm

Breathing Zone ppm

Nonaqueous Phase (thickness):

Reference Point (e.g., top of PVC casing):

Purge Data

Purge Method: .

(Note: Allow water level to equilibrate after removing well cap)

Total Well Depth (TD): _____ ft Depth to Water (DTW): .

Casing Inner Diameter (CID): _____ inches

To calculate well volume: Well Vol.(gal)=$(CID)^2(0.04)(TD-DTW)$

Well Volume: _____ gal x 3=Purge Volume _____ gal

Purge Time: Begin _____ End _____

Prepurge Data: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 1: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 2: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 3: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 4: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume 5: Temp _____ pH _____ Spec. Cond._____Turb. _____Other _

Volume Purged: _____ Purged Dry: Yes No

Disposal Method for Purgewater:

Water Description

Odor: Prepurge _____ Postpurge _

Color: Prepurge _____ Postpurge _

Sampling Data

Sampling Method: ____

Sampling Time: Begin _____ End ___
Analytical Parameters (circle appropriate parameters):

VOCs BNA BNE Total (Unfiltered) Metals

Dissolved (Filtered) Metals TPH PCB Cyanide

Other: __

Comments: ___

Standard Operating Procedure - 4

Surface Water and Sediment Sampling (using hand trowel)

Surface Water Sampling

Materials:

Nitrile gloves

Saranex or Tyvek suit

Vinyl gloves

Transparent shipping tape

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Hip-waders or rubber boots, as necessary

Plastic sheeting

Expanding ruler or tape measure

Variable speed peristaltic bladder or submersible pump with in-line filter or pressurized bailers

Surface water sampling method-specific materials (as necessary):

- Dippers (sampling vessel)
- Pumps with associated tubing
- Double check valve bailers
- new 0.45 micron filter

Decontamination supplies

Procedure:

1. Collection of surface water samples should be completed before collection of the stream sediment samples from the same location. This procedure will eliminate the introduction of sediment and turbulence in the surface water that is to be sampled. If possible, find a suitable sampling location by selecting an area that is away from any sources of cross-contamination that could compromise the integrity of the samples. This includes positioning the sample collection area away from fuel-powered equipment, such as drill rigs or excavators, and upwind of other site activities (e.g., purging, sampling, decontamination) that could influence the sample.

2. Once you have arrived on site and are prepared to conduct the surface water sampling, note all observations and measurements in the field book.
3. Perform a quick reconnaissance of the site to identify sampling locations.
4. Record the approximate ambient air temperature, precipitation, wind (direction and speed), tidal, and other field conditions in the field book. In addition, any site-specific conditions or situations that could potentially affect the sediment samples or associated water level measurements at the sample locations should be recorded.
5. Describe the sampling location including width of stream, depth of stream, water color and clarity (transparency), and approximate surface flow (e.g., slow, fast moving, etc.)
6. Label all sample containers prior to sample collection.
7. The sampler should wear hip-waders or rubber boots and gloves, or Saranex or Tyvek sleeves duct taped onto nitrile gloves, to avoid dermal contact with the surface water. Wear a clean pair of new, disposable gloves each time a different sample is collected and don the gloves immediately prior to collection to limit the possibility of cross-contamination from accidental contact.
8. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
9. Avoid wading into surface water, if possible, to avoid sediment suspension and potential cross-contamination. If necessary, extreme caution should be exercised when wading into the stream at the sampling location to minimize disturbance of the fine sediments.
10. Because of possible unseen water hazards, two people should be present during the collection of surface water samples.
11. If collecting several surface water samples from the same surface water body, start sampling at the downstream location and progressively move upstream. The sampler should always face upstream (i.e., upcurrent) when collecting the surface water sample.
12. The surface water sample container should be placed into the flowing water and the sample should be collected from just beneath the stream surface. Surface water should be allowed to slowly enter the sampling vessel until the necessary sample volume has been collected. Position the sampling vessel in the upstream direction of flow to minimize the disturbance to the surface water. For dissolved metals analysis, surface water samples must be filtered using the following procedures:
    - Use a variable speed peristaltic, bladder, or submersible pump with the in-line filter fitted on the outlet end; pressurized bailers can also be used.
    - At the pump discharge end, attach a clean 0.45 micron filter to the tubing.
    - Place the inlet into the sampling vessel, turn on the pump and reduce the flow rate, but maintain a flow rate high enough to deliver a smooth stream of water without splashing or undue agitation, hold the filter upright with the inlet and outlet in the vertical position and pump surface water through the filter until all atmospheric oxygen has been removed and the minimum volume of water has been flushed through the filter.

- Collect the filtered samples directly into the laboratory supplied preserved (nitric acid) sample container from the pump-filter assembly.
- If sediment is visible in the sample container after filtration, filter break-through has occurred and the sampling and filtering process should be repeated.
- Disassemble the pump head and discard the tubing and filter appropriately.

13. The sample container should be labeled before sample collection. After the sample is collected, the outside of the container should be cleaned, dried, and sealed. Complete all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection) on the sample label. Place clear tape over the tag or label. Immediately after collection, preserve samples for temperature-sensitive parameters by placing them into an insulated cooler for shipment to the analytical laboratory.
14. The sampling location should be described, including width of stream, depth of stream, water color, and approximate surface flow (e.g., slow, fast moving, etc.).
15. Sampling locations should be marked with a stake or flagged for future reference. Locations should be recorded with respect to a permanent feature, if available.
16. Complete chain of custody form with appropriate sampling information.
17. Discard the used hose and filter and utilize new hose and filters for each sample location.
18. If collecting sediment samples, proceed to collect the sample from this location.

Surface Water Field Measurements

Materials:

Nitrile gloves

Saranex or Tyvek suit

Vinyl gloves

Sample containers

Sample/Shipping labels and indelible marker

Transparent shipping tape

Bound field log book

PPE

Utility knife

Hip-waders or rubber boots, as necessary

Aluminum foil or heavy-duty zipper-style plastic bags (quart size)

Plastic sheeting

Expanding ruler or tape measure

Variable speed peristaltic bladder or submersible pump with in-line filter or pressurized bailers

Surface water sampling method-specific materials (as necessary):

- Dippers (sampling vessel)
- Pumps with associated tubing
- Double check valve bailers
- 0.45 micron filter

Decontamination supplies

Procedure:

1. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
2. Collect all samples for indicator parameters, including pH, temperature, turbidity, and electrical conductivity in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
3. Record the date and time of the measurement, temperature, field/weather conditions, surface water level measurements, and visual observations including color, flow direction, and approximate gradient.
4. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
5. Field instruments will be calibrated prior to field work in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
6. Temperature, pH, and electrical conductivity will be measured with a Hach 2100Q turbidimeter or equivalent and turbidity will be measured with a Myron L Ultrameter II meter or equivalent
7. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
8. Temperature measurements will be measured with thermometers calibrated using a National Institute of Standards and Technology (NIST) traceable thermometer. NIST traceable thermometers are certified by the manufacturer as indicated in its operations and maintenance manual.
9. Turbidity will be measured with a nephelometric turbidity units (NTU) range turbidimeter calibrated daily to a reference standard in accordance to the manufacturer's operation and maintenance manual. Sample and standards cuvets must be handled with care as fingerprint smudges, scratches, condensation, and agitation may cause measurement errors. Outside of cuvettes should be cleaned to remove any smudges or condensation.
10. If possible, avoid floating debris, coarse sediment, and fine air bubbles when collecting turbidity samples as these may cause inaccurate readings.

Sediment Sampling

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)

Nitrile or vinyl gloves

Saranex or Tyvek suit

Hip-waders or rubber boots, as necessary

Sample containers

Sample/Shipping labels and indelible marker

Transparent shipping tape

Bound field log book

PPE

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Bucket augers, auger extension rods, auger handle, pipe wrenches

Split-spoon samplers, pipe wrenches

Direct push acetate liners

Shelby tube samplers

Decontamination supplies

Procedure:

1. Once you have arrived on site and are prepared to conduct the sediment sampling, note all observations and measurements in the field book.
2. Perform a quick reconnaissance of the site to identify sampling locations.

3. Record the approximate ambient air temperature, precipitation, wind (direction and speed), tidal, and other field conditions in the field book. In addition, any site-specific conditions or situations that could potentially affect the sediment samples or associated water level measurements at the sample locations should be recorded.
4. Describe the sampling location including width of stream, depth of stream, water color and clarity (transparency), and approximate surface flow (e.g., slow, fast moving, etc.)
5. Mark sampling locations with a stake or flag for future reference; if available, record locations with respect to a permanent feature.
6. Label all sample containers prior to sample collection.
7. A hand trowel can be used to sample shallow stream bottom sediments, where the depth of water does not exceed 1 foot, using the same procedures specified in the Standard Operating Procedures for Collection of Soils Samples Using a Hand Trowel. A sediment corer or other appropriate sampling device should be used in water deeper than 1 foot (see SOP-6).
8. The sampler should wear hip-waders or rubber boots and gloves, or Saranex or Tyvek sleeves duct taped onto nitrile gloves, to avoid dermal contact with the water.
9. Extreme caution should be exercised when wading into the stream at the sampling location to minimize disturbance of the fine sediments.
10. If collecting several sediment samples from the stream, start sampling at the downstream location and progressively move upstream. The sampler should always face upstream (into the current) when collecting the sediment sample.
11. Position fuel powered equipment downwind and at least 10 feet from the sampling location; make sure that the exhaust faces downwind.
12. Insert the trowel into the sediment bed and retrieve sediment. Carefully remove the trowel from the water to avoid washing sediment from the blade. The trowel blade should be tilted at a slight angle, if necessary, to drain excess water from the blade before placing the sediment in the mixing tray. Separate clumps of sediment material and mix the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.
13. If more sediment is needed to provide sufficient sample volume, reinsert trowel at the sample location and retrieve as before.
14. After the sample is collected, clean the outside of the sample container to remove excess sediment and seal the container. Record all required information (sample number, date, time, sampler's initials, analysis, preservatives, place of collection) on the sample label. Place clear tape over the tag or label. Immediately after collection, preserve samples for temperature-sensitive parameters by placing them into an insulated cooler maintained at a temperature of approximately 4°Celcius.
15. Prepare the sediment samples for offsite laboratory analysis. Record sample designation, date, time, and the sampler's initials on the sample tracking form and in the field book.
16. Complete chain-of-custody forms with appropriate sampling information.
17. Complete sample packing and shipping in accordance with proper procedures.

Sediment Field Measurements

Materials:

PPE

Hip-waders or rubber boots, as necessary

Hand trowels, shovels, or spoons (stainless steel or Teflon)

pH meter (Horiba D51 or equivalent)

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Decontamination supplies

Procedure:

1. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
2. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
3. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
4. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
5. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
6. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.

7. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
8. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
9. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
   - Depth interval
   - USCS group name
   - USCS group symbol
   - Color, using Munsell chart (in moist condition)
   - Percent of cobbles or boulders, or both (approximate; by volume)
   - Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
   - Particle-size range:
     - Gravel—fine, medium, coarse
     - Sand—fine, medium, coarse
   - Particle angularity: angular, subangular, subrounded, rounded
   - Particle shape: (if appropriate) flat, elongated, flat and elongated
   - Maximum particle size or dimension
   - Hardness of coarse sand and larger particles
   - Plasticity of fines: non-plastic, low, medium, high
   - Dry strength: none, low, medium, high, very high (does not apply to sands)
   - Dilatancy: none, slow, rapid
   - Toughness: low, medium, high
   - Odor (mention only if organic or unusual)
   - Moisture: dry, moist, wet
   - Use the following standard descriptors for the textural percentages:
   - Trace: 0 to 10%
   - Little: 11 to 20%
   - Some: 21 to 35%
   - And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

10. Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units

- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

[1] Note that certain states/regulatory programs may require soil classification under a secondary system (e.g., US Department of Agriculture) or the use of hydrochloric acid to test the reaction with soil (none, weak, strong).

Standard Operating Procedure - 5

Sediment/Sludge Sampling with Ekman or Ponar Dredge

Application:

Sediment/sludge samples can be collected with an Ekman dredge, which is normally used when collecting samples from lagoons or other aqueous systems consisting of sediment of a soft consistency such as muck, me, or fine peat. The ponar type dredge is normally used when collecting samples from lagoons or creeks where the sediment is a hard constancy such as sands, gravels or clays. The dredge consists of a spring-loaded set of jaws with a brass sampling bucket that is lowered to retrieve samples. The dredge can be mounted on a pole or suspended from a line. This may not be used for samples to be analyzed for metals and organics.

Materials:

- Ekman dredge
- Saranex or Tyvek suit
- Overboots or hip-waders, as necessary
- Stainless steel spoons or scoops
- Plastic sheeting or garbage bags
- Nitrile, surgical, or work gloves
- Trays, mixing pans, or Ziploc® plastic bags
- Sample containers
- Sample/Shipping labels and indelible marker
- Bound field log book
- PPE
- Utility knife
- Heavy-duty zipper-style plastic bags (quart or snack size)
- Expanding ruler or tape measure
- Munsell color chart
- Field test kits, as needed
- Decontamination supplies

Procedure:

1. Sampler should don personal protective equipment as required to prevent dermal contact with sample media.
2. Carefully open jaws of the dredge and set trip lines to trigger device.

3. Gently lower the Ekman dredge through the aqueous media until it rests on the sediment.
4. To close the jaws of the dredge, send the messenger down the line; or if the dredge is pole-mounted, strike the spring release button on the top of the pole. The weight will strike the trigger mechanism, causing the spring-loaded jaws to close. This will trap the sludge/sediment inside the sampling bucket.
5. To minimize disturbance and to avoid washing the sample from the dredge bucket, slowly retrieve the dredge through the liquid column above it.
6. The sediment sample should be removed from the dredge bucket with a stainless steel scoop or spoon and transferred directly to the labeled sample container. The sample container should be sealed, labeled, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
7. The sampling locations should be described including width of stream or vessel, depth of stream or vessel, and approximate surface flow rate, if applicable. Locations should be flagged or marked with a stake for future reference.
8. Keep detailed notes in the field log book of the sample location, sample description, sample length, sampler's name, and the requested analytical parameters.
9. Completed chain-of-custody forms with appropriate sampling information.

Sediment Field Measurements

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)

Nitrile gloves

Vinyl gloves

Aluminum foil

Saranex or Tyvek suit

Hip-waders or rubber boots, as necessary

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Bucket augers, auger extension rods, auger handle, pipe wrenches

Split-spoon samplers, pipe wrenches

Direct push acetate liners

Shelby tube samplers

Decontamination supplies

Procedure:

11. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
12. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.
13. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
14. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
15. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
16. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
17. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
18. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
19. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
    - Depth interval
    - USCS group name
    - USCS group symbol

- Color, using Munsell chart (in moist condition)
- Percent of cobbles or boulders, or both (approximate; by volume)
- Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
- Particle-size range:
  - Gravel—fine, medium, coarse
  - Sand—fine, medium, coarse
- Particle angularity: angular, subangular, subrounded, rounded
- Particle shape: (if appropriate) flat, elongated, flat and elongated
- Maximum particle size or dimension
- Hardness of coarse sand and larger particles
- Plasticity of fines: non-plastic, low, medium, high
- Dry strength: none, low, medium, high, very high
- Dilatancy: none, slow, rapid
- Toughness: low, medium, high
- Odor (mention only if organic or unusual)
- Moisture: dry, moist, wet
- Use the following standard descriptors for the textural percentages:
- Trace: 0 to 10%
- Little: 11 to 20%
- Some: 21 to 35%
- And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Standard Operating Procedure - 6

Sediment Sampling Using Hand Corer

Application:

Sampling bottom sediment in shallow surface water bodies and lagoons

Materials:

Hand corer

Extension handle

Analyte-inert core tube liners (glass, Teflon, etc.)

Nitrile, surgical, or work gloves

Protective overboots

Tray, mixing pans, or Ziploc® plastic bags

Stainless steel spoons or trowel

Pipe wrenches

Bound log book

Assembly:

1. Secure the core tube head assembly onto the core tube, as specified by manufacturers' instructions.
2. Remove end liner caps and wrapping from a clean core tube liner and insert the liner into the core tube.
3. Insert a clean core retainer into the bottom of the core tube liner so that its convex end points away from the bottom. Plastic materials should not be used if organic analytes are of concern.
4. Screw the shoe (nose cone) onto the bottom of the core tube. The hand corer is now fully assembled for use.
5. If the hand corer is used from a boat or overhead structure, either of the following two attachments can be used:
   a) 5-foot extension handles can be attached to the top of the core head assembly with a threaded adapter fitting, nut, and bolt. The long handle is used to push the corer into bottom sediment.
   b) The corer can be lowered to the bottom with a rope. The rope can be tied to a clevis pin that mounts on the core head assembly with a nut and bolt.

Use:

1. Place nitrile gloves on hands before collecting samples.

2. Sampler should avoid wading into surface water, if possible. If not possible, the sampler should carefully approach the sample location to minimize disturbance of fine sediments.
3. Place the corer in a vertical position perpendicular to the bottom at the desired sample location.
4. The hand corer should be lowered slowly to minimize disturbance of sediments.
5. If using the corer from a wading position push it down into the sediment while turning the handle. The push and twist technique can also be used with the long extension handle attached to the corer.
6. If the corer is attached to a rope, detachable weights can be put on top of the core head assembly by threading the rope through the holes in the center of the weights. The shoe of the corer should not be resting on the bottom before being advanced. Further penetration will be achieved by allowing the corer to fall freely through the water while in a vertical position to strike the bottom. The weights will increase the force of the corer as it strikes the bottom.
7. When sufficient sample has been collected, it is usually necessary to push it back and forth from different angles to loosen the seal between the bottom and the core tube before pulling it up.
8. Slowly retrieve the core tube and place it on a clean surface in a vertical position. Slowly tilt it toward a horizontal position (this can be done immediately following retrieval of the sampling device). This will open the flapper valve and allow water that entered the tube to leak out.
9. Unscrew and remove the core head assembly and shoe from the core tube. Pipe wrenches may be used to unscrew the core tube assembly. Slide the core tube liner out of the core tube.
10. If undisturbed sediment core samples are needed, do not remove the sediment sample from the core tube. Retain the core sample in the core tube liner (i.e., inner sleeve) and place Teflon end caps on each liner end. Label the inner tube and tape the end caps on the tube liner for shipment to the laboratory. Plastic materials, including end cap, should not be used if organic analytes are of concern.
11. To remove sediment from the core tube, place the core tube liner over a sampling tray and remove its contents by inserting the brass rod into the top end of the tube and pushing. The brass rod should be decontaminated and wrapped in clean aluminum foil between each use.
12. If additional sediment is needed to provide necessary sample volume, reassemble the corer using a separate, clean core tube liner and repeat steps 1-11.
13. For volatile organic compound (VOC) samples, do not mix the sample before sampling for VOCs. Transfer sediment from the tray directly into sample containers.
14. Examine the contents of the tray. After the collection of VOC samples and before filling other sample containers, mix the sediment so a homogeneous particle size and texture remain.
15. Using the stainless steel spoon, transfer the tray contents to the sample containers.
16. The sample container should be labeled before sample collection. After the sample is collected, the container should be sealed, wiped clean of excess sediment, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
17. Complete chain-of-custody form with appropriate sample information.
18. The sample location should be described, including depth of the surface water body and approximate surface flow. The location should be flagged or marked for future reference, if possible.

Sediment Field Measurements

Materials:

Hand trowels, shovels, or spoons (stainless steel or Teflon)

Nitrile gloves

Vinyl gloves

Aluminum foil

Saranex or Tyvek suit

Hip-waders or rubber boots, as necessary

Sample containers

Sample/Shipping labels and indelible marker

Bound field log book

PPE

Utility knife

Mixing tray or bowl

Heavy-duty zipper-style plastic bags (quart or snack size)

Plastic sheeting

Expanding ruler or tape measure

Munsell color chart

Field test kits, as needed

Sediment sampling method specific materials:

Stainless steel trowels, shovels, or spoons

Bucket augers, auger extension rods, auger handle, pipe wrenches

Split-spoon samplers, pipe wrenches

Direct push acetate liners

Shelby tube samplers

Decontamination supplies

Procedure:

20. Collect all samples using either new, disposable equipment, or properly decontaminated sampling equipment. The equipment should be constructed of non-reactive, non-leachable materials (e.g., stainless steel, Teflon®, Teflon®-coated steel, polyethylene, polypropylene, etc.) which are compatible with the chemical constituents at the site.
21. Collect all samples for indicator parameters, including pH in accordance with the project-specific work plan and manufacturer's specifications for field testing equipment.

22. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
23. Record the date and time of the measurement, temperature, field/weather conditions, and visual observations including color, texture, odor, presence of oil sheens, and presence of macrophites or animals.
24. Field samples must be directly transferred from the sampling equipment to the container that has been specifically prepared for that given parameter; intermediate containers should be avoided.
25. Field instruments will be calibrated in accordance with manufacturer instructions and cleaned in the field between samples to prevent cross contamination.
26. pH will be measured with a Horiba D51 or equivalent that has been calibrated to manufacturer's specifications as indicated in its operations and maintenance manual.
27. Prior to sampling in the field, pH meters will be calibrated with buffer solutions to manufacturer specification as indicated in its operations and maintenance manual.
28. Sediment classification should be performed whenever samples are being collected to provide context for the analysis. Unified Soil Classification System (USCS) logging procedures as described in ATSM D2488[1] are preferred . The emphasis of sediment classification in the field must be on describing the sediments using ALL of the required descriptors; categorization of the USCS group name or symbol alone may not provide details about the soils that could later prove useful. Avoid geologic interpretation or the use of local formation names, which are often difficult to determine in the field without the regional framework. Record ALL of the following information for each soil type:
    - Depth interval
    - USCS group name
    - USCS group symbol
    - Color, using Munsell chart (in moist condition)
    - Percent of cobbles or boulders, or both (approximate; by volume)
    - Percent of gravel, sand, or fines, or all three (approximate; by dry weight)
    - Particle-size range:
      - Gravel—fine, medium, coarse
      - Sand—fine, medium, coarse
    - Particle angularity: angular, subangular, subrounded, rounded
    - Particle shape: (if appropriate) flat, elongated, flat and elongated
    - Maximum particle size or dimension
    - Hardness of coarse sand and larger particles
    - Plasticity of fines: non-plastic, low, medium, high
    - Dry strength: none, low, medium, high, very high
    - Dilatancy: none, slow, rapid

- Toughness: low, medium, high
- Odor (mention only if organic or unusual)
- Moisture: dry, moist, wet
- Use the following standard descriptors for the textural percentages:
- Trace: 0 to 10%
- Little: 11 to 20%
- Some: 21 to 35%
- And: 36 to 50%

Example descriptions, using the information listed above, would read as follows:
8-10' – 5YR2/6 fine- to medium-grained sand, trace medium sub-angular rounded gravel (up to 0.5" in diameter); medium dense to dense; wet with slow dilatancy; moderate solvent-like odor between 9' and 10'.

10-12' – 5YR2/6 low plasticity clay with some fine to coarse grained angular to subangular gravels (up to 0.25" in diameter) and trace fine to medium grained rounded sands, very stiff, moist with no dilatancy, no odors.

1. Collect field sediment samples by separating clumps of sediment material and mixing the sediments to a homogeneous particle size and texture. Transfer the contents to the appropriate sample container using a stainless steel spoon.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.

- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Standard Operating Procedure - 7

Sludge Sampling with Sludge Judge

Application:

The sludge judge is useful for obtaining a core sample of semi-liquid sludge or water and sludge. This instrument is a long, narrow polyethylene tube with a check valve on the bottom. The tube is graduated in 6-inch intervals for easy identification of sampling depth. A sludge judge may not be used for organics.

Materials:

Sludge judge

Saranex or Tyvek suit

Sample containers, labels, and indelible marker

Tray or mixing pans

Aluminum foil

Plastic sheeting or garbage bags

Nitrile gloves

Vinyl gloves

Eye protection

Over boots

Stainless steel scoops, trowels, or spoons

Procedure:

1. Sampling personnel should don the appropriate personal protective equipment based on the type of material being sampled and its historical characteristics.
2. Slowly insert the sludge judge into the material being sampled.
3. When the sludge judge has filled with material, pull back on the sampler to close the check valve and retrieve the sludge sample.
4. If a liquid sample is not required, decant any liquid into a separate container or back into the vessel being sampled. Transfer the remaining sludge sample directly into a labeled sample container by pouring the sludge from the top of the sampling tube. If a liquid sample is required, decant any liquid in the tube directly into sample containers; retrieve the sludge from tube.
5. The sample container should be sealed, labeled, and placed in a cooler with ice or freezer packs for shipment to the analytical laboratory.
6. Keep detailed notes in the field log book of the sample location, sample description, sample depth, sampler's name, and the requested analytical parameters.
7. Complete chain-of-custody forms with appropriate sampling information

Standard Operating Procedure - 8

Sludge Sampling with Coring Tube

Materials:

Field log book

Personal protective equipment (PPE)

Coring tube

Hammer

Plastic tube liners

Hack saw and replacement blades

Mixing tray or bowl

Plastic sheeting

Stainless steel spoon or trowel

Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Insert a plastic liner into the coring tube to collect an undisturbed sample. A plastic core catcher may be used to improve sample recovery.
3. The sample is collected by pushing or hammering the coring tube into the material to the desired depth, making sure not to exceed the length of the coring tube. Continuous core samples can be obtained from stable coreholes by re-inserting the coring device into the same core-hole. However, a new plastic liner must be placed in the coring device before each sample is collected.
4. Remove the sampler by pulling and twisting the coring tube until it breaks free from the material. This will result in a core that is approximately 2 feet long.
5. If the sample needs to be collected from a depth greater than 2 feet, place a new plastic liner into the tube and re-insert the sampler into the same core-hole and repeat Steps 3 and 4.
6. Remove the plastic liner from the coring tube and decant any excess water above the core.
7. Cut the plastic liner at the desired sample interval using a hack saw with a decontaminated blade. For VOC samples, extrude the sample material directly into a sample container. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.

8. For all other parameters, a stainless steel spoon or trowel should be used to transfer the sample material into a decontaminated mixing tray or bowl to be homogenized. The decontaminated stainless steel spoon or trowel can be used to remove gummy or thick sludge from the tube, if required.
9. If necessary, screen the sludge with a PID/OVA or perform a headspace analysis in accordance with SOP 22. Record the reading in the field logbook.
10. Record the sample location, sample description, sample depth, sampler's name, and the requested analytical parameters in the field log book.
11. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
12. Complete chain-of-custody forms with appropriate sampling information.
13. Samples should be maintained and shipped in accordance with SOP 20.
14. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure – 9

Soil Sampling Using Bucket Auger

Materials:

Field log book

Personal protective equipment (PPE)

Bucket augers

Auger extension rods

Auger handle

Pipe wrenches (for threaded connections)

Push pins (for snap connections)

Stainless steel spoons or trowels

Mixing tray or bowl

Plastic sheeting

Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Remove all vegetation or other surface material (e.g., gravel) with a hand trowel or other tool (e.g., shovel).
3. Advance the borehole to the desired sampling depth (i.e., the top of the sample interval). Attach a decontaminated auger bucket to collect the soil sample.
4. Place the auger bucket in the borehole. Grip the cross-handle with both hands and twist it clockwise to advance the auger.
5. Withdraw the auger bucket from the borehole and place it on plastic sheeting. For VOC samples, use a decontaminated stainless steel spoon or trowel to transfer the sample material directly into the appropriate sample container. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.
6. Remove the retrieved soil from the bucket with a decontaminated stainless steel spoon or trowel and place the material in a decontaminated mixing tray or bowl. If additional soil is needed to provide sufficient sample volume, repeat Step 4.
7. If necessary, screen the lead end of the auger with a PID/OVA or perform headspace analysis in accordance with SOP 22. Record the reading in the field logbook.

8. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the sample description in the field logbook.
9. For all other parameters, the sample material should be transferred into a decontaminated mixing tray or bowl. Use the stainless steel spoon to separate large clumps of soil material and mix the contents of the tray to a homogeneous particle size and texture.
10. Examine the contents of the tray and remove coarse gravel, organic material (e.g., roots, grass, and woody material) and any other debris with the stainless steel spoon.
11. Transfer the tray contents to the appropriate sample container using a stainless steel spoon.
12. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
13. Measure and record the sample depth in the field logbook, along with the sample location, sampler name, and the requested analytical parameters.
14. Complete the chain-of-custody form with appropriate sampling information.
15. Samples should be maintained and shipped in accordance with SOP 20.
16. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure - 10

Split-Spoon Soil Sampling

Application:

To collect soil samples with a split-spoon sampler that is advanced and retrieved with a drill rig.

Materials:

Field log book

Personal protective equipment (PPE)

Split-spoon samplers

Stainless steel spoons or trowels

Mixing tray or bowl

Pipe wrenches

Expanding ruler or tape measure

Note: Decontamination is not required for dedicated sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Ensure that the soil boring has reached the desired sample depth and that loose soil in the bottom of the boring has been cleaned out.
3. The drillers will attach a decontaminated split-spoon sampler to the lead drilling rod, lower it to the bottom of the boring, and advance the split-spoon into the undisturbed soil. Record in the field logbook the number of blows for every 6 inches the sampler is advanced.
4. When the split-spoon has reached the desired depth, the driller will retrieve the split-spoon and disconnect the split-spoon from the drilling rod.
5. Remove the head and shoe from the split-spoon and note which end of the sampler is the top and bottom. Separate the split-spoon sampler into two halves keeping all of the soil in one of the halves (if possible).
6. Measure the length of material recovered in the sampler with respect to the penetration depth and record this ratio in the field logbook.
7. If field screening for organic vapors is required, break or cut the soil core every 3 to 4 inches and quickly scan the breaks in the core material with a PID/OVA. Headspace analyses, if required, should be performed in accordance with SOP 22. Record the reading in the field logbook.
8. For VOC samples, transfer the soil directly into the sample containers with a decontaminated stainless steel spoon. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect sludge samples for VOC analysis using EPA Method 5035 for preservation.

9. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the information in the field logbook.
10. Note: the top of the split-spoon often contains float material, which is not to be included in the soil sample or sample description.
11. If it is necessary to collect soil samples for non-volatile parameters, transfer the recovered soil to a decontaminated mixing tray or bowl with a decontaminated stainless steel spoon. Soil material lodged within the split-spoon shoe should also be placed in the mixing tray or bowl for sampling.
12. Examine contents of the tray and remove pebbles, organic material, (e.g., roots, grass, and woody material), and other debris with the stainless steel spoon. Use the same spoon to chop apart clumps of soil and mix the contents of the tray to a homogeneous particle size and texture.
13. Transfer the soil material to the appropriate sample containers using the stainless steel spoon.
14. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
15. Record the sample location, sample depth, sampler name, and the requested analytical parameters in the field log book.
16. Complete the chain-of-custody form with appropriate sampling information.
17. Samples should be maintained and shipped in accordance with SOP 20.
18. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

BORING LOG WSP Environment & Energy 11190 Sunrise Valley Drive, Suite 300 Reston, Virginia 20191	**PROJECT**	Boring No. Sheet ___ of Date Drilled

Drilling Co. Driller Geologist	Boring Location Ground Elevation TOC Elevation

Boring	Casing/S	Sampler
Method Hole Diameter Inside Diameter Total Depth	Type Diameter Screen Length Screen Slot Size	Method Length (ft) Hammer (lb) Fall (in)

Depth	P.I.D. (ppm)	Percent Recovery	Sample Depth	Blows/6"	Well Design	Sample Description

Standard Operating Procedure - 11

Soil Sampling Using Hand Trowel

Application:

To collect surface or shallow subsurface soil samples with a hand trowel.

Materials:

Field log book

Personal protective equipment (PPE)

Stainless steel trowels or spoons

Mixing tray or bowl

Plastic sheeting

Note: Decontamination is not required for dedicated (disposable) sampling equipment.

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Remove any vegetation or other surface material (e.g., gravel) from the sample location with a trowel or other tool (e.g., shovel).
3. Push the trowel into the soil to the desired sampling depth. If sampling a stiff silty or clayey soil, it may be necessary to remove and reinsert the trowel to loosen the soil. Shallow subsurface soil samples can be collected by digging a hole (e.g., with a shovel or trowel) and collecting a soil sample at the desired depth.
4. Transfer the soil to a mixing tray or bowl using a decontaminated stainless steel trowel or spoon. If required, screen the recovered soil with a PID/OVA or perform headspace analyses in accordance with SOP 22. Record the reading in the field logbook.
5. For VOC samples, transfer soil directly into the sample container with the stainless steel trowel. A closed-system sampler (e.g., Encore Sampler) should be used, if necessary, to collect soil samples for VOC analysis using EPA Method 5035 for preservation.
6. Describe the remaining sample material in accordance with ASTM International Standard D 2488 and the Unified Soil Classification System. Record the sample description in the field logbook.
7. If soil samples will be collected for non-volatile parameters, use the stainless steel spoon to chop apart clumps of soil material and mix the contents of the tray to a homogeneous particle size and texture.
8. Examine contents of the tray and remove pebbles, organic material, (e.g., roots, grass, and woody material), and other debris with a stainless steel trowel or spoon.
9. Transfer the tray contents to the appropriate sample container using a stainless steel spoon.

10. Label the containers, cover the labels with tape, and immediately place the containers in a cooler maintained at an ambient temperature of 4* Celsius with wet ice. Freezer packs or dry ice should not be used for sample preservation.
11. Record the sample location, sample depth, sampler name, and the requested analytical parameters in the field log book.
12. Complete the chain-of-custody form with appropriate sampling information.
13. Samples should be maintained and shipped in accordance with SOP 20.
14. Properly manage all PPE and investigation-derived wastes in accordance with state and federal requirements.

Standard Operating Procedure - 12

Chip, Wipe, and Sweep Sampling

Application:

These methods of sample collection are intended for monitoring surficial contamination of nonvolatile species of analytes on porous surfaces for chip samples and smooth surfaces for wipe samples. Also included is a method for collecting dust samples from porous or non-porous surfaces.

Materials:

Lab-clean sample containers of proper size and composition

Field and travel blanks

Site log book

Sample analysis request forms

Chain of custody forms

Custody seals

Sample seals

Disposable surgical gloves

Sterile wrapped gauze pad (3 in. X 3 in.)

Appropriate pesticide (HPLC) grade solvent

Medium-sized, laboratory-cleaned paint brush

Medium-sized, laboratory-cleaned chisel

Autoclaved aluminum foil

Camera

Distilled/deionized water

Procedure:

1.1.8 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.
2. Obtain necessary sampling and monitoring equipment. Decontaminate or pre-clean the equipment, and ensure that it is in working order.
3. Perform a general site survey and mark all sampling locations. Measure off the designated area and photo document.

1.1.9 Chip Sample Collection

1. Don a new pair of disposable surgical gloves. Remove aluminum foil wrap from the laboratory cleaned chisel or equivalent sampling device.
2. Chip the sample area horizontally, then vertically to an even depth of approximately 1/8 inch. Collect the chipped fragments using a decontaminated dustpan and bristle brush and transfer the sample directly into the labeled sample bottle. Cap the container, attach the label and custody seal, and place in a double plastic bag. Store the samples out of direct sunlight and cool to 40 Celsius.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete chain-of-custody forms with appropriate sampling information.

1.1.10 Wipe Sample Collection

1. Don a new pair of disposable surgical gloves. Open a new sterile package of gauze pad. Soak the pad with the appropriate solvent (one in which the contaminant is most soluble). Wipe the marked surface area using firm strokes. Wipe vertically, then horizontally to ensure complete surface coverage.
2. Place the gauze pad in an appropriately prepared sample container with a Teflon-lined cap. Cap the sample container, attach the label and custody seal, and place in a double plastic bag.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete the chain-of-custody forms before taking the next sample.
4. A blank sample should be collected for each sampling event. This consists of a sterile gauze pad, wet with the appropriate solvent, and placed in a prepared sample container. The blank will help identify potential introduction of contaminants via the sampling methods, the pad, solvent, or sample container.
5. Store samples out of direct sunlight and cool to 40 Celsius.

1.1.11 Sweep Sample Collection

1. Don a new pair of disposable surgical gloves. Sweep the measured area using a dedicated brush and collect the sample in a dedicated dustpan (pre-cleaned).
2. Transfer the sample from the dustpan to the sample container. Cap the sample container, attach the label and custody seal, and place in a double plastic bag.
3. Record all pertinent data (sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters) in the site logbook. Complete the chain-of-custody form before taking the next sample.
4. Store samples out of direct sunlight and cool to 40 Celsius.

Standard Operating Procedure - 13

Concrete Core Sampling

Materials:

Concrete corer and drill
Tap water or containers of water
Concrete chisel
Hammer
Aluminum foil
Ionic detergent
Wet/dry vacuum
Stiff-bristled brushes
Sandpaper
Nitrile gloves
Respiratory protection
Tyvek suits
Paper towels
Eye protection
Indelible marker
Kimwipes
Generator (i.e., electrical power)
Extension cord (heavy duty)
Pipe wrenches
Work gloves
Garden hose

Procedure:

1. Clean surface area to be sampled by scrubbing it using ionic detergent (e.g., Whisk solution) and stiff-bristled brush. Rinse the surface with deionized water, then remove the water by vacuuming surface with wet/dry vacuum.
2. Remove any sealant coatings from the surface area using sandpaper or a grinder. Wash, rinse, and vacuum the surface area.
3. Move the drill into position and connect the decontaminated concrete corer to the drill. Connect the water supply and pressure distribution tank.

4. Establish vacuum pressure to hold core drilling rig to the floor.
5. Lower the corer to within 2 inches of the surface, start the drill, and slowly lower the corer to the surface.
6. Keep a steady stream of water flowing through the corer to cool the concrete, prevent the concrete core from splitting, and control fugitive dust. Do not remove corer from the hole until the required depth is reached. However, should the concrete corer become lodged, shut off power immediately to avoid injury.
7. With the drill still operating, remove the corer from core hole and shut off. Observe the hole to see if core was removed. If the concrete core is still in the floor or the core barrel, use a concrete chisel and rubber mallet to break the core loose.
8. When removing the core from the corer, handle the core with clean nitrile gloves.
9. Place a label on the top of the core written with a permanent marking pen. DO NOT WRITE on the core itself, especially if volatile organic compound analysis is requested. Measure the total length of core and record it in the field log book. Wrap the core in aluminum foil and place it in a labeled sample container.
10. Keep detailed notes in the field log book of the sample location, sample description, sample depth, sampling personnel, and the requested analytical parameters.
11. Complete chain-of-custody forms with appropriate sampling information

Standard Operating Procedure - 14

Waste Pile Sampling

Application:

As with soil sampling, waste pile samples can be collected at the surface or at depth, and different equipment is required in each instance. Surface samples can be collected most efficiently with a trowel or scoop. For samples at depth, a decontaminated, clean bucket auger may be required. For a sample core, waste pile samplers may be used. To collect representative samples of a waste pile, composite samples may be required. The objectives of the sampling program will dictate the need to collect composite samples. A grid system set-up on the waste pile may also be used to collect individual or composite samples.

Materials:

- Stainless steel trowels or scoops
- Stainless steel spoons
- Bucket augers
- Extension rods
- Auger handle
- Pipe wrenches
- Teflon tape
- Work, nitrile, and surgical gloves
- Saranex or Tyvek suit
- Eye protection
- Respirator (if necessary)
- Analytical buckets
- Tray, mixing pans
- Ziploc® plastic bags
- Aluminum foil
- Stakes and string (grid system)
- Plastic sheeting
- HNu or other photoionization detector (PID)

Procedure:

1. At the desired sampling location, clear away the accumulated surface debris.

2. The sampler should put on the correct personal protective equipment as dictated by the medium to be sampled and the project. Care should be taken to ensure that contact between the waste material and the skin, eyes, and face of the sampling personnel is prevented.
3. If a grid system is being employed for sampling the waste pile, lay out the grid according to the developed sampling plan.
4. Assemble the sample equipment (e.g., bucket augers) if necessary, and collect the samples at the chosen designations.
5. Collect an adequate volume of waste from a depth of 0-6 inches using the bucket auger, trowel, or scoop at the desired sampling locations. Record the PID measurement in the open borehole.
6. If using a bucket auger, advance the decontaminated bucket auger by turning the bucket auger clockwise into the sample medium. To remove the auger, turn the auger counter-clockwise and pull it from the borehole. This procedure is repeated until sampling is complete. If the sampled waste pile is heterogeneous, a clean, decontaminated bucket must be used every time to collect the sample of the waste material, if necessitated by the objectives of the sampling plan.
7. If composite samples are to be collected, transfer the waste material to a clean tray or mixing pan for compositing. Report the PID measurement of the material in the tray. Use a clean, decontaminated spoon, scoop, or trowel to homogenize the sample. Remove any foreign matter (e.g., rocks, sticks, or other debris) from the tray. Transfer the waste sample directly into a prelabeled sample container. A wide-mouth sampling container is preferable for containing the sample, as it requires less disturbance of the waste sample as the sample is being transferred into the labeled sample container.
8. Keep detailed notes in the field log book of the sample location, details about the grid spacing, sample description, sample depth, PID measurements, sampler's name, and the requested analytical parameters.
9. Complete the chain-of-custody forms with the appropriate sampling information..

Standard Operating Procedure - 15

Decontamination of Drilling Equipment

Materials:

Canvas or plastic tarp(s)

4-mil polyethylene liner

Pressurized steam cleaner (steam jenny)

55-gallon steel drums with bung (closed) tops

55-gallon steel drums with open tops, rings, lids, ring-nut and ring-bolt

Hammer, nails, duct tape, extension cord(s)

Wood boards - 4" x 4", 2" x 4" or 2" x6"

Portable wet/dry vacuum

Shovel, funnel, and squeegee

Construction of Decontamination Basin:

1. Place tarp(s) on flat, firm surface in an accessible area of the site away from areas of surface contamination. Use enough tarp to accommodate the rear of the drilling rig and hollow stem augers and to prevent overspray from the steam jenny from falling onto adjacent soil surfaces. If necessary, place more than one tarp on the ground. Overlap tarp edges and secure with duct tape. Area should be slightly inclined toward one corner so that the decontamination water will pool in one corner for easier pumping to the containment drums.
2. Place a layer of polyethylene liner on top of the tarp(s). If one sheet cannot completely cover the tarp, use another one. Overlap the sheets at the edges and secure with duct tape.
3. Place 4" x 4" boards along the tarp's outer edges to form a square or rectangular basin. Roll each 4" x 4" board toward the center so the tarp and polyethylene wrap completely around it at least once. Secure the tarp and liner to the top of the boards with nails, tacks or heavy-duty staples.
4. Place the drums, steam cleaner, and wet/dry vacuum adjacent to one side of the basin on the outside.

Decontamination Procedure:

1. Unload drilling equipment from the drilling rig and place in one side of the basin.
2. Activate the steam cleaner. Personnel performing steam cleaning should don rubber boots, Tyvek or Saranex suits, rubber gloves, and a hard hat with a face shield for splash protection.
3. Clean each piece of drilling equipment, including auger bits, drill bits, portable power augers, hollow stem augers, auger holders, split spoons, rod lifters, and drilling rods, by holding the nozzle of the steam cleaner a few inches away. Wood 2" x 4"s can be placed on the basin floor to

prevent drilling equipment from coming into contact with solids that will build up beneath it as it is being steam cleaned.

4. After each piece is cleaned, place it on rows of 2" x 4" boards in a separate area of the basin.
5. If space allows, position the rear of the drill rig in the basin and use the steam cleaner to clean off rig surfaces and the hoist and derrick as needed.
6. Reload drilling equipment onto rig and drive it out of the basin.
7. Vacuum up liquids on the basin floor with the flexible hose of the portable wet/dry vacuum. A long-handled squeegee can be used to pool liquid together to aid vacuuming.
8. Remove accumulated solids from the basin floor with a shovel and place in open-top drums. During removal of the accumulated solids, be careful so that the polyethylene liner is not torn, cut, or punctured with the shovel.
9. Empty the canister of the wet/dry vacuum into a bung-top drum using a funnel.
10. Secure and tighten tops of drums and apply appropriate hazardous waste or nonhazardous waste labels to each drum. The accumulation date should be placed on each drum. An inventory of all onsite drums should be entered into the field log book by field personnel. All drums should be marked, numbered, or labeled with an indelible marker for future reference.
11. On completion of onsite work, the properly labeled and inventoried drums should be stored within a newly constructed pad or basin until disposal is arranged. This containment area should be constructed of wooden boards with a polyethylene liner, as described above.
12. Materials used in construction of the decontamination basin or pad should be disassembled and placed into a properly labeled drum for future disposal.
13. All drilling equipment and the drill rig should be decontaminated on arrival onsite and before the start of any drilling activity. On completion of site work, the drilling equipment and rig should be decontaminated by the drilling contractor before departure from the site.

Standard Operating Procedure - 16

Decontamination of Submersible Pumps

Materials:

Field logbook

Personal protective equipment (PPE)

Polyethylene sheeting

Garbage bags

Nonphosphate detergent (e.g., Liquinox or Alconox)

Tap water

Deionized water

Two containers (e.g., garbage cans, buckets, plastic tubs)

Nylon brushes

Isopropanol

Spray bottles

Paper towels

Note: To limit the potential for cross-contamination between wells, wells should be pumped in the order of increasing constituent concentrations whenever possible. This SOP assumes that dedicated tubing is being used at each well. If dedicated tubing is not being used, the tubing should also be decontaminated using the following procedures.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Prepare a decontamination area by spreading polyethylene sheeting on a firm, flat surface (if possible). Create a berm around the decontamination area to contain inadvertent spillage. A berm can be created by rolling under the edges of the polysheeting or by draping the plastic over a wooden frame, etc.
3. Place two clean containers (e.g., garbage cans, buckets, plastic tubs) on the polysheeting. Place tap water in one container with non-phosphate detergent. Place only tap water in the second container. The containers may also be lined with garbage bags.
4. If an oily film or residue is observed on the pump or leads when they are removed from the well, the pump should be sprayed with isopropanol to remove the oil and then wiped clean with paper towels before proceeding with Step 5 below. The oily rinsate should be contained in a separate container for proper disposal.
5. Place the pump and wire leads in the container of non-phosphate detergent and tap water and scrub the exterior of the pump with a brush. Circulate the soapy solution through the pump for at

least 5 minutes. Rinse the exterior of the pump and leads with additional tap water to remove excess soap (if necessary) before proceeding with Step 6.

6. Place the pump and leads in the container of tap water and run the pump for a least 5 minutes. Run water through the pump until all residual detergent has been removed. The soapy solution and rinse water should be changed when it becomes oily or too silty.
7. Remove the pump and leads from the rinse water. Spray off the pump thoroughly with deionized water and wipe it dry with clean paper towels. Wipe off the wire leads with a paper towel soaked with deionized water.
8. Wrap the pump and leads in plastic sheeting or a new plastic garbage bag to prevent possible contamination during transportation. Label the sheeting or bag with the date of decontamination for future reference.
9. Properly manage all PPE and decontamination rinsate in accordance with state and federal requirements (See SOP 26). The spent wash water and rinse water can potentially be placed in the facility's waste water treatment system. However, field personnel should obtain approval from facility personnel and from the local POTW.

Standard Operating Procedure - 17

Decontamination of Water Level Indicators

Materials:

Field logbook

Personal protective equipment (PPE)

Non-phosphate detergent (e.g., Liquinox or Alconox)

Deionized water

Isopropanol

Two buckets

Spray bottles

Paper towels

Note: To limit the potential for cross-contamination between wells, wells should be gauged in the order of increasing constituent concentrations whenever possible.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. If the groundwater is grossly contaminated (i.e., LNAPL or DNAPL is present), the tape should be pulled out of the well, NOT reeled up, and placed directly into a bucket of nonphosphate detergent and tap water. The tape and probe should be scrubbed with a brush to remove visible contamination. The tape and probe should then be rinsed in a second bucket of tap water before proceeding with Step 3. If persistent stains or oily films remain, apply isopropanol to a paper towel and wipe the tape and probe until clean.
3. Thoroughly wet a paper towel with deionized water from a spray bottle. Fold the paper towel over the tape and wipe it as the tape is reeled up.
4. The water level probe should also be sprayed with deionized water and wiped dry with a clean paper towel.
5. Place water level indicator in the clean carrying case or in a clean plastic bag to prevent contamination during transportation.
6. Properly manage all PPE, used paper towels, and decontamination rinsates in accordance with state and federal requirements (See SOP 26).

Standard Operating Procedure - 18

Decontamination of Interface Probe

Materials:

Field logbook

Personal protective equipment (PPE)

Nonphosphate detergent (e.g., Liquinox or Alconox)

Deionized water

Isopropanol

Two buckets

Spray bottles

Paper towels

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. If the groundwater is grossly contaminated (i.e., LNAPL or DNAPL is present), the tape should be pulled out of the well, NOT reeled up, and placed directly into a bucket of nonphosphate detergent and tap water. The tape and probe should be scrubbed with a brush to remove visible contamination. The tape and probe should then be rinsed in a bucket of tap water before proceeding with Step 3. If persistent stains or oily films remain, apply isopropanol to a paper towel and wipe the tape and probe until clean.
3. Thoroughly wet a paper towel with deionized water from a spray bottle. Fold the paper towel over the tape and wipe it as the tape is reeled up.
4. The interface probe should be sprayed with deionized water and wiped dry with a clean paper towel.
5. Place the interface probe in the clean carrying case or in a clean plastic bag to prevent contamination during transportation.
6. Properly manage all PPE, used paper towels, and decontamination rinsates in accordance with state and federal requirements (See SOP 26).

Standard Operating Procedure - 19

Decontamination of Sampling Equipment

Materials:

Field logbook
Personal protective equipment (PPE)
Deionized water
10% nitric acid solution
Nylon brushes
Hand trowels, shovels, or spoons (stainless steel or Teflon)
Bowls (stainless steel or Teflon)
Hand Trowel (stainless steel or Teflon)
Containers (e.g., garbage cans, buckets, plastic tubs)
Nonphosphate detergent (e.g., Liquinox or Alconox)
Isopropanol
Aluminum foil
Polyethylene sheeting
Plastic garbage bags
Paper towels
Spray bottles
Duct tape

Note: All sampling equipment must be decontaminated before shipment to the office.

Decontamination Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Prepare a decontamination area by spreading polyethylene sheeting on a firm, flat surface (if possible). Create a berm around the decontamination area to contain inadvertent spillage. A berm can be created by rolling under the edges of the polysheeting or by draping the plastic over a wooden frame, etc.
3. Prepare a solution of nonphosphate detergent and tap water in a container.
4. Wipe sampling equipment with paper towels to remove residual soil or gross contamination. Heavy oils or grease may be removed with paper towels soaked with isopropanol.

5. Disassemble sampling equipment (e.g., split-spoon samplers and bailers). Wash equipment thoroughly in a nonphosphate detergent and hot tap water (if available) solution. Teflon bailers must be disassembled and the inside washed with a long-handled bottle brush or short-handled brush pulled through the bailer with rope.
6. Rinse the equipment with hot tap water (if available).
7. If the equipment will be used to collect samples for metals analysis, follow the tap water rinse with a 10% nitric acid solution rinse. Carbon steel equipment (e.g., bucket augers, split-spoons) should be rinsed with 1% nitric acid solution to reduce the potential for oxidizing the metal surfaces. Collect the nitric acid rinse in a separate bucket for proper disposal. Rinse the equipment with tap water.
8. Thoroughly rinse the equipment with deionized water.
9. Spray the equipment with isopropanol and allow to completely air dry. The solvent rinse must be collected in a separate bucket. Isopropanol is the recommended solvent for organic contaminants because it is readily available and is not a Department of Transportation hazardous material. However, other solvents (e.g., acetone, hexane, methanol) may be more effective in removing certain contaminants, such as oils or PCBs. Please note that many state programs and USEPA regions specify the solvents to be used for equipment decontamination.
10. Rinse the equipment with deionized water using at least five times the volume of solvent used in the previous step.
11. After the equipment has been allowed to completely air dry, each piece must be individually wrapped with aluminum foil (shiny side out), and then wrapped in plastic.

Note: Decontamination solvents may introduce contaminants to environmental samples. It is very important to ensure that the equipment has completely dried before use or storage.

12. After the final decontamination event on a project, label each piece of equipment with the date of decontamination, the initials of decontamination personnel, and the type of decontamination solutions used.
13. Note any discrepancies from standard decontamination procedures in the field logbook.
14. Field decontamination presents unique problems in disposal of decontamination solutions. The spent wash water and rinse water can potentially be placed in the facility's waste water treatment system. However, field personnel should obtain approval from facility personnel and from the local POTW. If no wastewater treatment system is present onsite, or if approval cannot be obtained from the facility and local POTW, the wash water should be containerized for offsite disposal in accordance with state and federal requirements. The volume of spent solvent generated during field decontamination should be minimal. Solvents should be collected in separate buckets and allowed to evaporate. See SOP 26 for information on managing investigation-derived wastes.
15. Paper towels soaked with solvent should be allowed to air dry and be disposed of with the general trash. Under no circumstances should any decontamination solution be disposed of on soil surfaces..

Standard Operating Procedure – 20

Sample Shipping Procedures

Materials:

Suitable shipping container (e.g., plastic cooler or lab supplied styrofoam cooler)

Chain-of-custody forms

Custody seals

WSP mailing labels

Strapping, clear packing, or duct tape

Ziploc® plastic bags

Knife or scissors

Permanent marker

Latex or nitrile gloves

Large plastic garbage bag

Wet ice

Bubble wrap or other packing material

Universal sorbent materials

Sample container custody seals (if required)

Federal Express form (with WSP account number)

Vermiculite (or commercially available cat litter)

Procedures:

1. For shipping purposes, samples are segregated into two classes; environmental samples and restricted articles (i.e., hazardous materials). Environmental samples can also be categorized based on expected or historical analyte levels (i.e., low or high). An environmental sample is one that is not defined as a hazardous material by the Department of Transportation (DOT, 49 CFR Part 171.8). The DOT defines a "hazardous material" as a substance which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Any material of a suspected hazardous nature, previously characterized as hazardous, or known to be hazardous is considered a restricted article.
2. In general, the two major concerns in shipping samples are protecting the samples from incidental breakage during shipment and complying with applicable DOT and courier requirements for restricted article shipments.
3. Protecting the samples from incidental breakage can be achieved using "common sense." All samples should be packed in a manner that will not allow them to freely move about in the cooler or shipping container. Glass surfaces should not be allowed to contact each other. When

possible, repack the samples in the same materials that they were originally received in from the laboratory. Each container should be cushioned with plastic bubble wrap, styrofoam, or other nonreactive cushioning material. Shipping hazardous materials should conform to the packaging, marking, labeling, and shipping instructions identified in 49 CFR Parts 172 & 173.

4. Environmental samples shall be packed for shipment using the following procedures:
5. Line the shipping container with a large, heavy-duty plastic garbage bag. Place universal sorbent materials (e.g., sorbent pads) between the cooler and the heavy-duty plastic bag. The amount of sorbent material should be sufficient to absorb the volume of wet ice and aqueous samples. If using a plastic cooler, securely tape the drain plug closed on the outside of the cooler.
6. Place 2-4 inches of bubble wrap or other packing material inside the heavy-duty plastic bag in the bottom of the cooler.
7. The sample packer should wear latex or nitrile gloves when handling the samples during the packing process.
8. Place the bottles in the cooler with sufficient space to allow for the addition of more bubble wrap or other packing material between the bottles. Large or heavy sample containers should be placed on the bottom of the cooler with lighter samples (i.e., VOAs) placed on top to eliminate breakage.
9. Place the "wet ice" inside two sealed heavy-duty zipper-style plastic bags and package the bags of ice on top of or between the samples. Pack enough ice in the cooler to chill the samples during transit. If the cooler is shipped on a Friday or Saturday for Monday delivery, double the amount of ice placed in the cooler (Monday delivery should be used only as a last resort). Fill all remaining space with bubble wrap or other packing material. Securely close and seal with tape the top of the heavy-duty plastic bag.
10. Place chain-of-custody form (and, if applicable, CLP traffic reports) into a Ziploc® plastic bag and affix to the cooler's inside lid, then close the cooler. Securely fasten the top of the cooler shut with tape. Place two signed and dated chain-of-custody seals on the top and sides of the cooler so that the cooler cannot be opened without breaking the seals.
11. Once cooler is sealed, shake test the cooler to make sure that there are no loose sample containers in the cooler. If loose samples are detected, open the cooler and repack the samples.
12. Using clear tape, affix a mailing label with WSP's return address to the top of the cooler.
13. Ship samples via priority overnight express to the contracted analytical laboratory for next morning delivery. If applicable, check the appropriate box on the airbill for Saturday delivery.
14. Declare value of samples on the shipping form for insurance purposes. The declared value should reflect the cost to recollect the samples.
15. Record the tracking numbers from the Federal Express forms in the field notebook and on the chain of custody form. Also, retain the customer's copy of the Federal Express airbill.

Hazardous materials should be packed according to the above procedures with the following additions:

1. Place samples in individual Ziploc® plastic bags and secure with a plastic tie or tape.

2. Place samples in paint cans in a manner which would prevent bottle breakage (i.e., do not place glass against glass).
3. Place vermiculite or other absorbent packing material in the paint can around the samples. The amount of packing material used should be sufficient to absorb the entire contents of the sample if the container is broken during shipment.
4. Secure a lid to the paint can with can clips and label the outside of the can with sample numbers and quantity. Mark the paint can with "This End Up" and arrow labels that indicate the proper upward position of the paint can.
5. Package the paint cans in DOT-authorized boxes or coolers, with appropriate DOT shipping labels and markings on two adjacent sides of the box or cooler.
6. Ship the restricted articles via overnight courier following the courier's documentation requirements. A special airbill must be completed for each shipment. Retain a copy of the airbill for WSP records and tracking purposes, if necessary.

Standard Operating Procedure – 21

Field Quality Assurance/Quality Control Samples

Materials:

Field logbook

Personal protective equipment (PPE)

Sample containers

Sample labels

Clear tape

Laboratory analyte free water

Clean or dedicated sampling equipment

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Select the appropriate glassware for the field Quality Assurance/Quality Control (QA/QC) samples. Refer to the WSP Standard Operating Procedure for Sample Container, Preservatives, and Holding Times to determine the appropriate bottles to use.
3. Field QA/QC samples include the following:
   a. trip blanks
   b. duplicate samples
   c. equipment blanks
4. Trip blanks should be provided by the analytical laboratory for all projects where samples are being collected for analysis of volatile organic compounds (VOCs). Trip blanks should accompany the sample bottles from the analytical laboratory to the site, accompany the sample containers at all times during the sampling event, and return to the laboratory with the sample containers. One trip blank should be submitted to the analytical laboratory with each shipment containing samples for VOC analysis. The trip blank should be analyzed only for VOCs.
5. One duplicate sample should be collected for every 20 samples of each matrix (e.g., soil and groundwater) collected during each sampling event. Duplicate samples of soil and other solid matrices (sediments) should be collected by dividing the sample material in half and alternately filling the two sample bottle sets. Duplicate samples of groundwater and other aqueous matrices should be collected by alternately filling the two sample bottle sets from the same sampling vessel (e.g., bailer). The appropriate SOP should be followed for the collection of each sample type (soil, groundwater, sediment, sludge). Duplicate samples should be analyzed for all the analytes that are being analyzed for during the sampling event.
6. One equipment blank should be collected in the field at a rate of one per type of equipment per decontamination event not to exceed one per day. If dedicated sampling equipment is used, the equipment blanks should be prepared in the field before sampling begins. If field decontamination

of sampling equipment is required, the equipment blanks should be prepared after the equipment has been used and field-decontaminated at least once. Equipment blanks should be prepared by filling or rinsing the precleaned equipment with analyte-free water and collecting the rinsate in the appropriate sample containers. The samples should be labeled, preserved, and filtered (if required) in the same manner as the environmental samples. Equipment blanks should be analyzed for all the analytes for which the environmental samples are being analyzed. Decontamination of the equipment following equipment blank procurement is not required.

7. All QA/QC samples should be submitted to the analytical laboratory with unique sample numbers. Therefore, the QA/QC samples should be labeled as separate environmental samples following the same numbering scheme used during that particular sampling event. However, the QA/QC samples should be clearly identified on WSP's copy of the chain-of-custody form and in the field logbook.

Standard Operating Procedure - 22

Soil Head Space Screening (Field Technique)

Materials:

PPE

Field logbook

Photoionization detector (PID) and/or Flame Ionization Detector (FID)

Aluminum foil

Clear 8-oz to 16-oz glass large-mouth containers with lids

Stainless steel spoon

Zipper-style plastic bags

Procedure:

1. Use appropriate PPE as specified in the site-specific health and safety plan.
2. Check PID to ensure that it is working properly.
3. Using WSP's standard operating procedure (SOP) for collecting soil, sludge, or sediment, half-fill a clean glass jar with sample. Place a piece of aluminum foil over the top of the jar and tightly seal the jar. Label the jar indicating the sampling location, depth, and date. Store the jar upside down until the sample is analyzed.
4. If jars are not available, collect the sample using a zipper-style plastic bag (e.g., Ziploc®). Seal and label the bag as specified in item 3.
5. Shake the sample vigorously for approximately 15 seconds.
6. If necessary, warm the sample to room temperature (70*F) by placing the jar in a heated room or vehicle. This step is very important when the ambient temperature is below 32*F.
7. After waiting approximately 15 minutes, carefully unscrew the lid of the jar without disturbing the aluminum foil. Pierce a hole through the aluminum foil using the tip of the PID. If using zipper-style bags, open the bag slightly and place the tip of the probe into the opening. Do not insert the probe into the soil and avoid the uptake of water droplets.
8. Following probe insertion, record the highest meter response. Using the foil seal/probe insertion method, maximum response should occur between 2 seconds and 5 seconds. Erratic PID response may result from high organic vapor concentrations or elevated headspace moisture. If these conditions exist, the headspace data should be qualified or discounted.
9. Record the sample location, depth, soil texture (i.e., clay or sand), and PID reading in the field notebook. Also record the ambient temperature, humidity, and whether moisture was present in the jar or plastic bag. These points are important because on very cold days volatilization of organic compounds is reduced and water vapor present in the jar may cause the PID to give a false reading. Be consistent in your procedure and in your recording of the data.

10. Duplicate 10 % of the headspace samples by collecting two samples from the same location and following items 2 through 9 above. The headspace screening data from both jars should be recorded and compared. Generally, replicate values should be consistent to plus or minus 20%.
11. Samples collected for headspace screening should not be retained for laboratory analysis. Dispose of the soil and jar appropriately

Standard Operating Procedure – 23

Underground Utility Locating

Application:

The purpose of this procedure is to ensure that all required and appropriate procedures are followed to locate and mark subsurface utilities (e.g., electrical lines, natural gas lines, communication lines) before initiating any intrusive field activities (e.g., drilling, test pits, trenching). Compliance with this procedure is mandatory before intrusive work can be conducted on a WSP project. This procedure is intended to allow the work to proceed safely and will minimize the potential for damaging underground utilities. Intrusive work includes all activities that require WSP's employees or their subcontractors to penetrate the ground surface. Examples of intrusive work include, but are not limited to probing, drilling, injection, test pit excavations, trenching, and remedial excavations.

Materials:

Record of the communication utility locating form (Attachment 1)

Field logbook

Wooden stakes

Spray paint

Flagging tape

As-built drawings for sub grade utilities (if available)

Hand auger or post-hole digger

Hand-held magnetometer or cable locator (optional, if and only if private utility locator has cleared the area and personnel have been properly trained in the use of the equipment)

Procedure:

Pre-site Mobilization

1. Gather the necessary information to complete the record of communication utility locating form (Attachment 1).
2. Contact the state utility locating service (e.g., One-Call, Miss-Dig). It is imperative to contact the locating service with sufficient lead-time to allow all utility providers to visit the site location. In each case, the state utility locating service will provide the caller with a legal dig date. Under no circumstances will intrusive work begin before the legal dig date provided by the call center. The telephone numbers for the locating service in selected states are listed in Table 1. However, the telephone number is typically listed in the area Yellow Pages. Provide the utility locating service with any information they request concerning the site and work activity in order to locate utilities at the site. Several states, including California, require that the proposed drilling locations be marked with white spray paint before contacting the locating services. The following information provided by the locating service should be documented in a record of communication utility locating form (Attachment 1): utility providers that will be contacted, and a utility clearance ticket

number. The ticket number will be used by the various utility companies to reference the clearance request and to contact the caller with clearance verifications (see note below).

Note 1: Generally, the public utility companies will mark underground lines up to the private property boundary. However, you should request that the utility companies mark their utilities in the work areas on the site. If the utility companies will not provide that service, a private utility locating service MUST be contracted.

Note 2:Some utilities (e.g., sewer, water, cable TV) may not be included by the State locating service. The State locating service will provide you with a list of utilities that will be notified based on the information provided regarding the sites location. Compare this list with utilities generally expected at all sites (e.g., sewer, water, gas, communication, electric). If any expected utilities are absent from the contact list, you MUST contact the utilities directly for clearance before the start of intrusive activities. Record all contacts on the utility locating record of communication form.

3. Identify a site contact familiar with the utilities on the property (e.g., plant manager, facility engineer, maintenance supervisor), and provide this individual with a site plan showing the proposed locations of all soil borings, monitoring wells, test pits, and other areas where intrusive activities will be conducted. Ask the site contact for all drawings concerning underground utilities in the proposed work areas.
4. No intrusive work should be done before the legal dig date provided by the State utility locating service. No intrusive activities should be conducted along or near public right-of-ways until all utilities have been marked and visually verified in the area of investigation. In addition, NO field activities shall be conducted on private property unless the State locating service or a private utility locating service has confirmed the presence or clearance of onsite utilities.

Site Mobilization

1. Locate all proposed drilling and trenching locations, both onsite and offsite, with spray paint, stakes, or other appropriate markers.
2. Verify that ALL utility companies listed by the municipal locating service, and any contacted directly by WSP, have either marked the underground lines in the specified work areas or have responded with "no conflict." Document on the utility record of communication form as each utility mark is visually confirmed.

   Note: When receiving verbal clearances by telephone from utility companies, or their subcontractors, it is imperative that you verify which utilities are being cleared, particularly when dealing with subcontractors that may be marking more than one utility.
3. Review all available as-built utility diagrams and plans with the site contact to identify potential areas where underground lines may be present. The review should confirm the locations marked by the locating services and identify utilities that may have been omitted by the locating services. If the as-built drawings do not confirm utilities marked by the locating services, follow instructions in Section 6. If possible, obtain a copy(s) of the utility plans for future reference in the field.

   Conduct a site walk with the site contact. During the site walk, attempt to obtain a general knowledge of the types of utilities present in the work areas. Furthermore, survey your surroundings to identify features that require electricity (e.g., parking lot lights, pad-mounted transformers) or suggest the presence of underground utilities, such as linear depressions in the ground. Check these items against the utility locating record of communication form checklist. For

example, check to see whether major electrical lines are aboveground, or locate underground sewer lines by using the locations of manholes and storm water grates. Keep in mind that many sewer lines can be offset from catch basins.

4. A minimum of 4 feet clearance should exist between utilities and proposed drilling locations, and a minimum of 6 feet between utilities and proposed trenching locations. A minimum distance of 15 feet should be maintained by heavy equipment (e.g., excavator buckets, drill rig towers and rods) from overhead power lines. A safe distance of 25 feet should be maintained from high tension overhead power lines. In the event that work must be conducted within 25 feet of high tension wires, the lines should be wrapped and insulated by the local utilities. If a utility conflict is identified, adjust the proposed location(s) using the criteria given above. These minimum distances should be increased whenever possible to offer additional assurance that utilities will not be encountered.

5. A private utility locating service MUST be used for work on private property in cases where the public utility locating service does not mark utilities on the subject property. It is NOT ACCEPTABLE to rely on as-built drawings or verbal utility clearances. A private locator may not be necessary in rare instances; however, these cases must be discussed with the project manager AND a partner or executive partner of WSP before work may proceed.

   A listing of several private subsurface utility locating firms is provided in Table 2. In addition, a hand-held magnetometer or magnetic-cable locating device can be used to augment, but not replace, clearance for each work area. Use of this equipment is restricted to employees with proper training on the use of hand-held utility locating equipment. Proper training is defined as having working knowledge of the manufacturer's operating procedures, and the completion of at least one successful location under the supervision of a qualified person.

6. In some cases, state and private locating services may not be able to identify all utilities. In areas where uncertainty still exists concerning the presence of underground utilities after clearance by state and private locating services, a hand auger or post-hole digger can be used to probe the shallow subsurface before using any heavy equipment (drill rig, backhoe). The probe hole should be advanced a minimum of 4 feet below ground surface at each proposed drilling or excavation location. A sufficient number of probe holes should be completed so that the area is cleared for the proposed intrusive activity. For drilling, a minimum of three holes installed in a triangular pattern should be advanced at each location. The use of hand digging methods in NO WAY replaces the need State and private utility locating services. Hand digging techniques should only be employed if uncertainty regarding the location of underground lines still exists after clearances by the State locating service and a private locating service.

7. Discuss the site conditions with the subcontractor and recommend that care be used at the start of the intrusive activities. Field personnel should always consider the presence of unidentified utilities at each work area. In addition, field personnel have the authority and responsibility to postpone intrusive activities if insufficient information, as stipulated in this SOP, is available, or if onsite reconnaissance identifies inconsistencies in the findings of utility locators. In these instances, field personnel should contact the project manager or a member of the health and safety committee, and an executive partner or partner of WSP before proceeding with the proposed work. The first priority on every project is to ensure that the work is conducted safely.

   Again, it is the requirement of this SOP to obtain site utility clearances from the State utility locating service. If the State locating service does not provide onsite (i.e., work area) utility clearance, a private locating service must be contracted to clear the work areas before digging,

drilling, or probing begins. Although certain instances and site conditions may appear to allow intrusive work without prior clearance, ALL deviations from this SOP MUST be approved by the project manager and a partner or executive partner BEFORE beginning intrusive work.

8. If the scope of the intrusive activity locations changes, the scope of intrusion expands or includes a new onsite or offsite area(s), review the existing information to determine whether the area(s) can be safely cleared of all potential underground utilities. If necessary, contact the state locating service and request another clearance for the new area(s) of investigation and retain a private locator in accordance with Item 5 above. Remember, the new request will provide a new legal dig date before which NO INTRUSVE WORK CAN BEGIN. Additionally, if a clearance ticket will expire while the work is ongoing (typically after 14 days), a new clearance must be requested at before the first ticket expires so that work can continue uninterrupted. Refer to the communication utility locating form for the legal dig date time frame required by the State locating service.

PUBLIC and PRIVATE UTILITY LOCATING FORM

Project:_____ Project Manager:_____

Project Number:_____

Project Start Date:_____ Duration of Project:_____

Project Location (Site Address):_____

Project Site Description (complete the following with all information available *before* calling):

Work Being Done For: (*Company or Individual Name*): _____

State: _____County:_____ City/Place: _____

Street: _____(*Only one street per ticket)* Zip Code:_____

Nearest Intersecting Street: _____

Lat/Long:_____Parcel/Tax map ID:_____

Description of the area to be marked *(Provide the following: Street working on, which side of street, how far in which direction from nearest intersecting street; etc.)*:

_____

_____

Locations for proposed borings or digging identified with paint and/or stakes? (circle one): YES NO N/A

NOW, MAKE THE CALL:

Call Placed to Phone No.: _____

Date of Call:_____Time of Call:_____a.m. / p.m.

Ticket No. Assigned to Location Request:_____

Assigned Legal Dig Date: ___ / ___ / ___ Assigned Legal Dig Time: ____ AM / PM

The Following Table Must Be Completed Before Work Can Begin:

CALL BACK/FAX BACK INFORMATION RECORD						
	Gas	Communication	Electric	Water	Sewer	Other
Responsible Company (provided by One-Call operator)						
Date Notified						
Time Notified						
Notified By						
Phone Number						
Marks Complete						
No Conflict						
No Facilities						

Calls Made By:_____ Form Completed By:_____

Project Manager Notified of Results (initial if completed):_____

Onsite Underground and Overhead Utility Clearance Checklist

Visual Confirmation of Marked Public Utilities:

Utility Type	Visual Cues	Marks Confirmed (initial)	No Markings Seen (initial)
Water	Blue Markings, fire hydrant, manholes; water meter, sprinkler heads, AST, hose bib		
Gas	Yellow Markings, gas meter, manholes; yellow bollards		
Electric	Red Markings, parking lot lights, overhead lines (telephone poles), underground vaults, manholes; conduit on buildings		
Sewer (sanitary/storm)	White or Blue Markings, underground vaults, manholes, drain grates		

Communication	Red or White Markings, red bollards, telephone poles; manholes; conduit on buildings		

Visual Confirmation of Marked Private Utilities (at onsite drilling/digging locations):

Utility Type	Visual Cues	Marks Confirmed	No Markings Seen (initial)	Not Applicable (initial)
Water	fire hydrant, manholes; water meter, ASTs, interior connections, hose bib, valve box			
Irrigation	sprinkler heads, hose bibs			
Gas	gas meter, manholes; yellow bollards, interior connections, valve box			
Electric	parking lot lights, interior connections, overhead lines, underground vaults, manholes, transformers/switchgear; conduit on buildings			
Sanitary/Storm/ Septic System	underground vaults, manholes, drain grates, leach field, sand mound, no evidence of sanitary sewer (for septic system			
Production Equipment	USTs (fill pipes and vent pipes), ASTs (overhead and underground pipelines), manholes/valve pits; pump islands			
Communication	Red/orange bollards, telephone poles, interior connections; manholes; conduit on buildings			

If any Utilities have "No Marking Seen" checked, private locating must be conducted to clear each drilling/digging area.

Site Visit Made By:_____

Form Completed By:_____

Standard Operating Procedure – 24

Soil Sampling Using GeoProbe® System or Equivalent

Application:

To perform depth-discrete soil sampling with 2-foot or 4-foot long samplers using hydraulically-driven soil sampling equipment (GeoProbe® System or Equivalent).

Materials:

Stainless steel soil sampler (2-foot or 4-foot long)

Clear acetate liners

Tape measure or expandable ruler

Utility knife

Photoionization detector (PID)

Stainless steel spoons

Aluminum tray or stainless steel mixing bowla

Nitrile or latex gloves

Field notebook

Procedure:

1. Calibrate the PID in accordance to the manufacturer's instructions. Decontaminate all down-hole sampling equipment and the utility knife, spoons, and mixing bowl per SOP 19 before initiating any boring activities. Ensure that the location is clear of all underground utilities and pipelines.
2. Attach a decontaminated 2-foot or 4-foot long stainless steel sampler fitted with a new, clear acetate liner and a decontaminated removable cutting shoe to small-diameter rods. Lower the stainless steel sampler to the top of the desired sampling depth.
3. Advance the stainless steel sampler through the desired sample interval. Record in the dedicated field notebook the interval through which the sampler was pushed.
4. After the sampler has reached the desired depth, retrieve the sampler by first removing the rods and then disconnecting the sampler. Remove the cutting shoe and acetate liner containing the soil column from the sampler. Measure the length of the material recovered relative to the interval the sampler was advanced, and record this information in the field notebook.
5. Cut the acetate liner using a utility knife to expose the recovered soil. Quickly scan the recovered soil with the PID and if necessary, immediately collect samples for VOC analysis. If the plan indicates the collection of samples for headspace analysis, collect this sample after obtaining the sample for VOC analysis per SOP 22. Record the PID readings in the field notebook.

6. For VOC samples, transfer soil directly from the acetate liner into the sample containers with a clean, stainless steel spoon. Fill the VOC sample container with a representative sample from the entire length of the recovered sample core, or other designated sample intervala. Fill the VOC container completely, leaving no headspace.
7. Describe the recovered soil using the Unified Soil Classification System or standard geological descriptions. Record the sample description in the field notebook.
8. If it is necessary to mix the sample, transfer the soil from the acetate liner to a clean aluminum tray or decontaminated stainless steel mixing bowl with a decontaminated stainless steel spoonb.
9. Examine contents of the tray/bowl and remove rock fragments and organic debris, such as roots, grass, and woody material, with the stainless steel spoon. Use the same spoon to chop apart clumps of dirt and mix the contents of the tray to a homogeneous particle size and soil texture. Transfer the tray/bowl contents to the appropriate sample containers using the stainless steel spoon.
10. The sample container(s) should be sealed, labeled, and placed in a cooler with ice or freezer packs to maintain 4° Celsius for shipment to the analytical laboratory.
11. Complete the chain-of-custody form with the appropriate sampling information.
    a. NJDEP's Field Sampling Procedures Manual requires the collection of soil samples for VOC analysis from the 0.5-foot interval that exhibits the highest reading during the field (PID) screening.
    b. U.S. Environmental Protection Agency (EPA) Region 4 requires a glass bowl for homogenizing soil for sample collection.

Standard Operating Procedure – 25

Groundwater Sampling Using Geoprobe® System or Equivalent

Application:

To perform groundwater sampling using hydraulically-driven screen point sampling equipment (GeoProbe® System or Equivalent).

Materials:

Stainless steel probe rods with treads sealed with Teflon® tape or O-rings

Stainless steel screen point sampler

Stainless steel mini-bailer

Teflon®-coated stainless steel wire or thin nylon line

Polyethylene tubing (3/8-inch) fitted with a stainless steel check valve

Silicone tubing

0.45-micron filter

Peristaltic pump

Sample bottles, labels, indelible markers, and clear tape

Nitrile or latex gloves

Procedure:

1. Decontaminate all down-hole equipment before conducting sampling activities at each location. Ensure that the sampling location has been cleared of all underground utilities.
2. Drive the stainless steel point sampler into the subsurface material. The design of the sampler should allow the screen to remain retracted within the probe rods until it is driven to the appropriate sampling depth.
3. After reaching the desired depth, pull back on the stainless steel sheath to expose the screen. The point on the probe rods will be displaced and is not recoverable.
4. Purging is not required for probes that are sealed and opened at the target depth for sample collection. Exposed probes that are driven through the soil to the desired water sample depth must be purged of a minimum of three probe-rod volumes of water before sampling is conducted.
5. Groundwater samples can be collected using a mini-bailer lowered on Teflon®-coated stainless steel wire or nylon line inside the probe rods. Another method of sample collection involves the use of a clean section of 3/8-inch polyethylene tubing fitted with a stainless steel bottom check valve. The polyethylene tubing is inserted down the probe rods to the desired sampling depth. Oscillate the polyethylene tubing up and down to drive a column of water to the surface. A peristaltic pump may be attached to the sample tubing and used to pump water to the surface. The peristaltic pump should not be used to collect samples for VOC analysis.

6. Immediately collect samples for VOC analysis, if required. Transfer the groundwater directly from the sampling equipment (mini-bailer, polyethylene tubing) to the sample containers. If analyzing for dissolved metals, the sample must be filtered in the field. See note below with regards to field filtering of metal samples.
7. Seal and label each sample container and place in a cooler with ice or freezer packs to maintain 4° Celsius for shipment to the analytical laboratory.
8. Complete the chain-of-custody form with appropriate sampling information.
    a. Where samples are collected from depths greater than 15-20 feet below the water table, a water level indicator may be inserted into the rods before exposing the screen to determine whether water is entering through the rod joints or disposable probe point.

Field Filtering of Metal Samples:

1. Assemble peristaltic pump per operating manual instructions that accompany the pump. Silicone tubing is generally used though the head of the pump.
2. Attach polyethylene tubing to the inflow end of the silicone tubing. The polyethylene tubing should be long enough to extend to the bottom of the screen point. Attach a clean filter to the outflow end of the silicone tubing.
3. Turn on the pump and slowly draw the water from the sampling equipment, through the pump and filter, and into the sample container. If sediment is visible in the sample container, filter break-through has occurred and the sampling and filtering process will need to be repeated.
4. Disassemble the pump head and discard the tubing and filter.

Standard Operating Procedure – 26

Managing Investigation Derived Waste

Application:

The purpose of this SOP is to provide instructions for handling, storing, and sampling Investigation Derived Waste (IDW) pending disposal. All IDW should be handled as hazardous waste unless information exists which would allow it to be classified as non-hazardous waste. IDW generated during a Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) response action must be managed in compliance with applicable or relevant and appropriate requirements (ARARs) to the extent practicable and with applicable requirements of the CERCLA offsite policy. (EPA Guidance Document OERR Directive 9345.3-02)

IDW includes soil cuttings, development water, purge water, drilling fluids, decontamination fluids, personal protective equipment, and sampling equipment.

Materials:

Non-Hazardous and Hazardous Waste Labels

Investigation Derived Waste Log (Figure 1)

Permanent Ink Marking Pen Paint Stick/Pen

Sampling Equipment (Refer to Sampling SOPs)

Sample Jars

Chain of Custody Forms

Cooler

Procedure:

1.1.12 Hazardous IDW

1. All IDW should be handled as hazardous waste unless information exists which would allow it to classified as non-hazardous waste. New or existing site data (i.e., soil and groundwater results) and generator knowledge can be used to classify the IDW.

- If site data or generator knowledge indicates that the IDW is determined to be hazardous the following procedures will apply:
- The IDW must be placed in DOT approved containers (55-gallon drum, roll-off container, or temporary storage tank).
- The containers must remain closed except when adding, sampling, or inspecting the material.
- All containers must be labeled with the words "Hazardous Waste".
- An accumulation start date and the contents of the container must be included on the label.

- Investigation Derived Waste Logs (Figure 1) must be completed before leaving the site. One copy of the log should be presented to the site contact and the original provided to the project manager. Once the material has been removed from the site, the IDW log should be stamped "Removed" and placed in the project file.
- The IDW containers must be stored in a secure onsite location (facility hazardous waste storage area if one exists).
- Disposal of the IDW must be completed within 90 days of the date the waste was generated. If the facility is a small quantity generator, 180 days is allowed for shipment of the waste offsite.
- Onsite disposal may be allowed or appropriate under certain conditions. Refer to OERR Directive 9345.3-02 for guidance, especially for CERCLA sites.
- WSP personnel should notify the site contact that weekly inspections of the IDW must be conducted and documented.
- WSP personnel should also instruct the site contact that this waste must be included in the facilities annual or biannual reports.

2. If the IDW is presumed to be hazardous and sampling is required to confirm its classification, it should be labeled Hazardous Waste-Pending Analysis. The waste should be sampled before leaving the site (See sampling SOPs). It should be noted that EPA methods 8260 and 8270 may be more cost effective than running the full Toxicity Characteristic Leaching Procedure (TCLP) scan. TSD Facilities will usually specify the required analysis for their waste profiles.

1.1.13 Non-Hazardous IDW

1. If information exists to classify the IDW as non-hazardous waste, the following procedures can be implemented:

1.1.14 Soil Cuttings

- Spread around the borehole or other onsite location with the approval of facility personnel
- Place back in the boring
- Containerize and dispose offsite

1.1.15 Groundwater

- Pour onto ground next to well to allow infiltration
- Containerize and dispose offsite
- Discharge to POTW with approval of facility personnel
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.16 Purge water from onsite well development

- Collect purge water from well development
- Containerize water in steel drums
- Profile waste water through laboratory chemical analysis
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.17 Decontamination Fluids

- Pour onto ground (from containers) to allow infiltration
- Containerize and dispose offsite
- Discharge to POTW with approval of facility personnel
- Discharge to onsite wastewater treatment plant with approval of facility personnel

1.1.18 PPE

- Double bag and deposit in site dumpster
- Containerize and dispose offsite

If the IDW is containerized and is non-hazardous, the following procedures will apply:

- The non-hazardous IDW must be placed in DOT approved containers (55-gallon drum, roll-off container, or temporary storage tank).
- The containers should remain closed except when adding, sampling, or inspecting the material.
- All containers must be labeled with the words "Non-Hazardous Waste".
- An accumulation date and the contents of the container should be included on the label.
- Complete the IDW log (Figure 1). One copy of the log should be presented to your site contact and the original should be given to the project manager.
- The IDW containers must be stored in a secure onsite location.
- Arrangements for disposal should be completed within 90 days of the accumulation start date.

Investigation Derived Waste Log

Date: _____

Site Information

Site Name: _____ Site EPA ID #: _____

Site Contact: _____ Site Address: _____

Contact Telephone No: _____

Waste Identification

Type of Waste Generated (check one of the following):

- ☐ Soil Cuttings ☐ PPE ☐ Decontamination Water
- ☐ Groundwater ☐ Storm Water ☐ Drilling Fluids
- ☐ Other (Describe): _____

Field Activities that generated the Waste:

- ☐ Soil Borings ☐ Well Sampling ☐ Well Installation
- ☐ Decon ☐ Excavation ☐ Pumping Tests
- ☐ Other (Describe): _____

Generation Date: _____ ***90-Day Deadline:*** _____

Quantity of Waste Generated and Container Type: _____

_____

_____

Storage Location: _____

Waste Identification (Check One of the Following);

- ☐ Non Hazardous Waste (pending analysis)
- ☐ Non Hazardous Waste (based on site information or generator knowledge)
- ☐ Hazardous Waste (pending analysis)
- ☐ Hazardous Waste (based on site information or generator knowledge)

If generator knowledge or site information was used for identification, explain: _____

_____

_____

Type of Label Applied to Container: ☐ Non Haz ☐ Hazardous ☐ PCB ☐ Used Oil

WSP Information (Note: One copy to site contact - the original in project file)

Personnel/Contact: _____ Project No.: _____

Telephone _____

Standard Operating Procedure – 27

Soil Sampling Using a Rotosonic Sampler

Application:
To perform soil sampling with a rotosonic drill rig sampler advanced ahead of the temporary casing.

Materials:

Rotosonic samplers
Rotosonic sample bags (plastic)
Knife
Photoionization detector (PID)
Ziplock plastic bags (quart size)
Nitrile or latex gloves
Sample containers
Field Notebook
Marker

Procedure:

1. Decontaminate all downhole drilling and sampling equipment in accordance with the appropriate procedures before initiating any intrusive work.
2. Ensure that the temporary rotosonic casing has been advanced to immediately above the desired sample depth and loose soil in the bottom of the borehole has been removed.
3. The drillers will attach a decontaminated rotosonic sampler to the drilling rods, lower it to the bottom of the borehole, and advance the sampler through the soil using the rotosonic rig. (Note: the standard rotosonic sampler is capable of collecting a 10-foot long continuous core although 5-foot samplers may also be available. It may be desirable to advance the sampler less than 10 feet to avoid penetrating relatively thin confining layers.)
4. After the sampler has penetrated the desired sample interval, the drillers will retrieve the sampler and disconnect it from the drilling rods.
5. The drillers will place a 4-inch diameter plastic bag over the bottom end of the sampler and vibrate the rotosonic drill head until the sample is extruded from the core barrel and fills the bag. After filling, remove the bag and place it on plastic sheeting for examination and screening. Note the orientation of the sample (i.e., which end is "up") and the depth interval. Be sure the examination/screening area is far enough away from the drilling rig exhaust pipe or other potential sources of contaminants that could affect the screening results.
6. Wearing new gloves, cut the sample bag lengthwise using a decontaminated knife. Immediately scan the recovered soil material with a PID at approximately 2-foot intervals and record the readings in the field notebook.
7. Collect samples for soil headspace analysis using the procedure described in WSP's SOP 22 or other approved method/guidance. Record all headspace readings in the field notebook.a
8. If soil samples are being collected for VOC analysis, immediately transfer soil directly from the recovered core into the appropriate sample container(s). Collect the sample from the desired depth based on the sampling plan or field screening results. Label the container(s) with the

sample name, depth, date and time of collection, sampler name, and analysis requested. Immediately place the containers in a cooler with ice to maintain a temperature at 4oC.

9. Collect samples for other parameters, as needed, in accordance with the sampling plan.
10. Describe the recovered soil using the Unified Soil Classification System or standard geological descriptions, and include PID measurements, sample number(s) and depth(s), and observations such as staining or odor. Write all descriptive information in the field notebook.
11. Complete the chain-of-custody form with the appropriate sampling information.

Standard Operating Procedure – 28

Sampling of Private Water Supply Wells

Scope:
This procedure describes the method for collecting representative groundwater samples from a domestic or other private water supply well.

Materials:

Business cards
Field notebook
Temperature, pH, and conductivity meter(s)
Sample containers
Cooler
Labels
Indelible markers
Nitrile or latex gloves
Chain of custody forms
Bucket or pail (1 or 2-gallon)
Paper towels or Kimwipes

Pre-Sampling Activities:

1. Verify the location of the well to be sampled and analytical parameters as specified in the sampling plan.
2. Contact the well owner and explain the scope of work. If no information has been obtained about the well, try to obtain information from the owner regarding the exact well location, well construction details, static water depth, well depth, casing diameter, well yield, date drilled and drilling company, previous testing, presence of water treatment components, resource aquifer, and well use. Locate the well on a site or USGS topographic map for future reference.
3. If insufficient well information is obtained from the owner, consider performing a water well survey to obtain the information.

Sampling Procedures:

1. Prepare the field notebook with description of site, well location and owner, weather conditions, sampling personnel, and other relevant observations.
2. Inspect the well to verify construction and other information concerning the well.
3. Identify a cold water tap or spigot for sample collection. The sampling point should be as close to the well as possible, and before the water passes through any treatment systems, heating unit, or storage system. If a suitable sampling point is not available, try to find another well at the site or take steps to minimize the impact of any potential interferences to the sample. Document in the field notebook whether you can or cannot collect a water sample before any treatment or storage components.
4. If sampling from a tap or faucet, try to remove any aerators, filters, or other devices from the tap before purging. If the sampling point is outside, consider removing any hoses connected to the spigot. (However, hoses may be allowed to remain attached during purging to divert large volumes of water from buildings, septic systems, etc).

5. Start to purge the system by running water from a tap or outside spigot. Use a small bucket or pail and watch to determine the purge rate for the system. If the water sample is to be collected from a tap located after a pressurization or holding tank, a sufficient volume should be pumped from the well to allow for the complete exchange of water into the holding tank and the point where the sample is to be collected.
6. Geochemical measurements (pH, conductivity, and temperature) of the water should be collected at regular intervals (every 1-2 minutes) after the start of purging. Record all measurements in the field notebook.
7. Review the geochemical measurements to ensure that readings have stabilized (within 10% of the previous reading). If the geochemical measurements have not stabilized, continue to purge the well until the measurements do not vary more than 10% between two successive measurements (if possible).
8. Collect the groundwater samples after purging is complete. While collecting samples, reduce the water flow to a thin stream. The flow rate should be high enough to deliver a smooth stream of water without splashing but low enough to permit filling of the sample bottles without turning the valve down during sampling. Collect the samples in accordance with the procedures described in WSP's SOP 2 – Sample Containers Preservatives and Holding Times. Label each container with the site name, sample name, date and time of collection, samplers, preservatives (if added), and analyses. Place the sample containers in a rigid cooler with ice maintained at a minimum of 40C.
9. Decontaminate all field meters and other non-disposable equipment, and properly dispose of any used paper towels, gloves, etc.
10. Complete chain-of-custody forms with appropriate sampling information.

RECORD OF COMMUNICATION
UTILITY LOCATING FORM

Date of Call: _____/ _____/ _____ Time of Call: _____ a.m. / p.m.

Name of Utility Service Protection Center Called: _____

Call Placed to Phone No.: ( ) - _____ Call taken by operator: _____

Ticket No. Assigned to Location Request: [ ]

Co. ID # (*if assigned by Utility Service Protection Center*): _____

Person Calling _____ Company Phone: ( ) - _____

Alternate Contact Name: _____ Alternate Contact Phone: ( ) - _____

Company Address: _____

Assigned Start Date: _____ / _____ / _____ a.m. (8:00 – 12:00) / p.m. (12:00 – 5:00)

Type of Work: _____ Use of Explosives: Y / N

Potential Boring Locations Identified with White Paint and/or Stakes: Y / N / Not Applicable

Work Being Done For: (*Company or Individual Name*): _____

State: _____ County: _____ City/Place: _____

Street: _____ *(Only one street per ticket)* Zip Code: _____

Nearest Intersecting Street: _____

Map Page: _____ Grid(s): _____

Description of the area to be marked *(Provide the following: Street working on, which side of street, how far in which direction from nearest intersecting street; etc.)*:

_____

_____

_____

Map of the area to be marked faxed to the Utility Service Protection Center: Y / N / Not Accepted

Utility Service Protection Center Fax No.: ( ) - _____

CALL BACK INFORMATION RECORD

	Gas	Tele.	Elec.	Wtr.	Swr.	Cable TV	Other
Date Notified							
Time Notified							
Notified By							
Phone Number							
Marks Complete							
No Conflict							
No Facilities							

Standard Operating Procedure – 29

Aquifer Pumping Tests

Scope:

This standard operating procedure (SOP) describes the methods and techniques used to perform aquifer pumping tests on partially- or fully-penetrating test wells.

Objective:

Aquifer tests are generally performed to measure the hydraulic characteristics of a water bearing zone that has been identified and partially characterized (e.g., total depth of the water-bearing unit, groundwater flow direction, texture of the matrix, etc.) by investigations at the site. The tests generally include four separate phases: a background monitoring phase; a stepped-rate pumping test; a constant-rate pumping test; and a recovery test. Aquifer tests typically include a pumping well where the aquifer is stressed by removing formation water, and at least one observation well where the response to pumping is measured.

This SOP is designed to provide the user with a general outline for conducting each phase of the aquifer test and assumes the user is familiar with basic field procedures, such as recording field notes (SOP 1), well gauging (SOP 3) and equipment decontamination (SOPs 16 through 19). This SOP does not cover aquifer test planning (e.g., layout of the pumping and observation wells, estimation of pumping rates, selection of pump types, etc.) nor does it cover the analysis of the aquifer test results for determining the characteristics of the water-bearing zone. These topics require a significant amount of planning and are more appropriately addressed in the work plan or a dedicated aquifer test plan.

These procedures are oriented towards In situ-brand transducers and data loggers, which are the current state-of-the-art and the brand most often used by WSP. While some specifics may be slightly different for other brands of monitoring equipment, the overall sequence of work should be similar and readily adaptable to any equipment set-up.

Because of the inherent complexity and the number of different tests involved, this SOP is divided into four sections. Each section provides an overview of the objective of the test and a brief description of the methods to be used. The set-up procedures are cumulative; each phase of the aquifer test uses equipment or procedures that were used in the previous phase of the test. The reader is encouraged to read the entire SOP before beginning any onsite activities.

Preliminary Procedures

Materials:
Field notebook

Laptop computer with appropriate ports and adapters

Vented pressure transducer with internal or external data loggers

Barometric pressure monitoring equipment

Electronic water level meter or oil/water interface probe (if necessary)

Photoionization detector (PID)

Groundwater extraction pump and appropriate tubing

Power supply

Water storage tank

Flow meter(s) and/or flow gauge(s)

Stopwatch

Small (1 to 5 gallon) container with known capacity

Nitrile gloves

Heat resistant gloves

Tape measure

Exacto, pocket knife, or scissors

Pump lift line (polypropylene rope or other suitable material)

Duct tape

Indelible marker or metal file

Paper towels

5-gallon bucket(s)

Distilled water (decontamination of downhole equipment)

Plastic sheeting

Waste labels (if necessary)

1. Review site Health and Safety Plan (HASP) before mobilizing to the field. Discuss groundwater contamination constituents and specific HASP issues with Project Manager and determine the proper level of PPE.
2. Prepare field book (SOP No. 1) daily with site description, weather conditions, participants, and other relevant observations, including all data necessary to complete the hydraulic conductivity tests. Pay careful attention to the weather conditions (e.g., changes in temperature, precipitation, wind, etc.) both locally and regionally as these can adversely affect the data.
3. Survey the ambient (background) air around the base of the well casing and wellhead for organic vapors (if appropriate) using a PID, or equivalent. Record air monitoring measurements in the field book.
4. Inspect extraction well and observation/test wells (e.g., monitoring wells or piezometers) for soundness of protective casing, surface ground seal, and locking mechanism; record findings in the field book.
5. Unlock protective casing or flush mounted curb box. Remove well cap or plug, place PID probe in wellhead, and record PID response in field book. Survey breathing zone to ensure that PPE level is appropriate.
6. Place plastic sheeting around the head of the extraction well or observation well before initiating any down well measurements or testing activities.
7. Allow static water level to equilibrate, as appropriate, before gauging the wells.

8. Measure the inside diameter (ID) of the casing and record in inches. From the top of the casing, measure the depth to water (DTW) and total depth (TD) of the well (in hundredths of a foot with an electronic water level indicator and record in the field book). Water levels should be measured from the surveyor's mark at the top of the casing, or if no mark is present, from the north side of the casing. Be sure to mark a location with a metal file or indelible marker on the north side of the casing if the surveyor mark is absent and record in the field book for future reference.
9. Thoroughly decontaminate (SOP No. 17) the water level indicator cable during retrieval using distilled water and paper towels.
10. Do not create an airtight seal by replacing the well caps at the locations that will be used during the pumping tests. It is important that they be open to the atmosphere. Take steps, if necessary, to protect the openings by loosely covering the well opening with the protective steel cover or other material to prevent rain, snow, or debris from entering the well during the test.

Background Monitoring Test

This portion of the aquifer test involves monitoring each of the wells (pumping and observation wells) selected for the aquifer test for a period of time, usually a minimum of 24 hours, to identify any naturally-occurring (e.g., changes in barometric pressure, earth tides, rain events, etc.) or man-made (e.g., artificial recharge, nearby pumping wells, trains, etc.) forces that might systematically alter or potentially mask water level changes during the pumping portions of the aquifer test. Data collected from the background monitoring phase establishes a baseline that can be used to correct the pumping test data by analytically removing the non-pumping trends. Background data can also be used to help determine if the water-bearing unit is confined or unconfined.

The physical placement of the downhole equipment for this portion of the test will be used for the remaining phases of the aquifer test.

1. Assemble all of the monitoring gear (transducers, data loggers, cables, etc.). Verify the transducers have power (for transducers with onboard loggers) and sufficient cable length to reach the intended monitoring zone. Each cable should be fitted with a desiccant cartridge (provided by equipment supplier) to prevent moisture from building up in the transducer vent line. Also, verify that the transducers are rated for the intended water depths; Table 1 provides the criteria for selecting the appropriate transducer. Transducers with the lowest pressure rating appropriate for the anticipated depths should be selected as the device accuracy decreases as the pressure rating increases.

Table 1

Maximum Depth Below Water Table for Pressure Transducer (a)

Pressure Transducer (PSI)	Depth Below Water Table (feet)
10	23
20	46
30	69
50	115

a/ PSI=pounds per square inch

2. Mark each transducer (on the sonde itself) with the well identification number to avoid confusion during data recovery after all phases of the aquifer test have been completed.
3. Familiarize yourself with the data logging software. Transducers with onboard data loggers usually require an interface with a laptop or a hand-held computer running the appropriate proprietary software. Additional cables or adapters (e.g., RS-232 [serial] to Universal Serial Bus [USB]) may be necessary to physically link the computer to the data loggers.
4. Thoroughly decontaminate the pressure transducers using a non-phosphate detergent and distilled water before placement of equipment into the extraction well or observation wells. Decontaminated equipment should be placed on or wrapped in clean plastic sheeting until inserted into the monitoring well or piezometer.
5. Determine the depth below ground surface at which you will be setting the individual transducers for the pumping and observations wells:
    a. The most critical placement will be the transducer installed in the pumping well. This transducer is typically positioned below the pump (which will be installed in the next phase of the aquifer test) to allow water level measurements during periods of significant drawdown. If the well is shallow enough and the depth rating of the transducer is sufficient to withstand the pressure (Table 1), the transducer can simply be lowered to a depth just above the bottom of the well; take care to not allow the transducer to contact the bottom of the well during installation. If the transducer is to be positioned below the pump and above the bottom of the well, measure the length from the transducer bottom up the cable and mark the cable for easy reference when lowering into the well. In either case, be sure the transducer position is such that the bottom of the pump will not contact the top of the transducer.

    b. The placement of transducers in the observation wells is less critical. The units need only be positioned below the level of expected drawdown in each well (you should have some idea of this based on your site characterization and aquifer test design). As with the pumping well transducer placement, the easiest positioning is to place the transducer at a depth just above the bottom of the observation well (see Table 1).
6. Practice defining, running, stopping, and downloading data using the laptop or hand-held computer before installing any of the transducers in the pumping or observation wells. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand held computer are synchronized before programming any tests. This step is critical to ensuring a simultaneous start (and stop) of the test, contemporaneous data collection, and synchronized data logs.
7. Follow manufacturer's specifications to prepare the barometric monitoring equipment. The unit should be placed in the same general area as the pumping well.
8. Program the background monitoring test into each data logger and the barometric pressure monitoring equipment. The background monitoring data is recorded on a linear time scale (e.g., once every 15 minutes). As barometric pressure, tides, and other natural forces typically yield changes in the wells that occur over the space of 1 to 2 hours, data collected on a schedule of once every 15 to 30 minutes should provide a data set that is sufficiently dense to follow any systematic trends. Water levels that can change on more rapid scales (e.g., karst terrain) may require shorter sampling intervals. Check the aquifer test plan to verify the appropriate timing.

9. Secure the transducer cable to the wellhead, protective casing, or curb box using duct tape, "S-hooks", or other appropriate methods. The transducers should be secured so that they do not move vertically within the water column during the aquifer tests. Be careful not to pinch or kink the cable, which could block the transducer's vent to the atmosphere.
10. Allow the groundwater elevation in the test wells to re-equilibrate with the surrounding formation after the insertion of the transducers. The amount of time required for this step will be dictated by the characteristics of the formation. Wells with recovery times of less than a minute should be allowed to stand a minimum of fifteen minutes before initiating the background test.
11. Run the background monitoring test for a minimum of 24 hours. Do not disturb the pumping or observation wells during the monitoring period. Be sure to record any changes in the weather, especially rain or snow events, and any other environmental or physical changes (e.g., tides, train schedules, etc.) that may potentially alter the water levels in the wells. Aquifer tests conducted in karst terrain should also note regional precipitation events (i.e., within a 20 mile radius) because even distant meteoric water can quickly be transmitted over long distances through infiltration in karstic terrain.
12. Do not stop the barometric data monitoring equipment at the end of the background monitoring test. Barometric data should be collected for all phases of the aquifer test.
13. Download the groundwater and barometric pressure data to the laptop or hand-held device and create a back-up copy of the data. It is not necessary to erase the data loggers, as most contain sufficient memory to store millions of data points; leaving data on the loggers provides a secondary back-up system. Verify that all of the data has been transferred to the laptop or another storage media (e.g., compact discs) before erasing the data files from the loggers.

Stepped-Rate Pumping Test

The stepped-rate, or variable-rate, pumping test is designed to stress the aquifer with a series of short-term pumping periods with incrementally higher pumping rates to determine the maximum sustainable pumping rate. The primary purpose of the stepped rate test is to establish the pumping parameters for a constant-rate test; however, data from the stepped-rate tests can be used, if sufficient data is collected, as support for the constant-rate analysis. The stepped-rate test usually involves a minimum of three pumping periods or "steps" to establish the response of the aquifer.

The following procedures assume that the background monitoring test has been completed and the monitoring equipment remains in place.

1. The groundwater extraction method should be selected based on the depth to water, the anticipated flow rate, and the availability of power. Submersible pumps should incorporate a backflow check-valve(s) that prevents water within the riser pipe from flowing back into the well when the pump is shut off. Check the aquifer test design to verify the pump requirements and appropriate type of tubing.
2. Before installation, measure the length to the pumping intake depth specified in the aquifer test plan; the length includes the lift line and attached submersible pump or the length of tubing for non-submersible pumps (e.g., peristaltic pumps). Accurate measurements are critical to avoid contact with the transducers during installation and when using electric submersible pumps in low yield aquifers. Dry pumping electric submersible pumps, even for short periods of time, may damage the device.

3. Install and secure the tubing or submersible pump and tubing in the pumping well to the specified depth.
4. Attach a flow through meter to the discharge/conveyance line. Digital and optical styles are available, depending upon the predicted flow rates. Be sure to check the manufacturer's recommendations for calibration and ensure that the meter is equipped with the properly sized fittings. The flow meter measurements should be periodically verified by manually estimating the time necessary to fill a container of known capacity.
5. Extracted groundwater exiting the flow meter should be piped to a water storage tank with sufficient capacity. The tank should be constructed of materials that are compatible with contaminants that may be present in the groundwater and any applicable state or federal regulations (i.e., Department of Transportation), especially if the material will be managed as investigation-derived waste (SOP 26).
6. Define the stepped-rate tests in each data logger and the barometric pressure monitoring equipment. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The stepped-rate test is a logarithmic test. Data is collected rapidly in the first portion of the test, often several times per second, with the time between successive water level measurements becoming progressively longer as the test proceeds. This approach yields dense data in the early portion of the test where changes in water levels are occurring rapidly and less data as the water levels approach a steady-state. Most data loggers will convert the logarithmic measurement schedule to a linear schedule at a pre-designated time, which can be programmed during the test set-up. Typical log-linear conversion times range from 1 to 5 minutes for the short-term (i.e., one hour or less) steps; however, the actual conversion time should be estimated based on the aquifer test design and adjusted (for the constant-rate pump phase to follow) in the field based on observations during the individual stepped-rate tests. Table 2 provides typical log-linear conversion times for various test durations.

Table 2

Maximum Log-linear Conversion Times for Various Pumping Test Durations

Duration of Testing (Minutes)	Maximum Log-linear Conversion Time (Minutes)
10-15	1
15-60	5
60-300	30
300-1440	60

1. Gauge the pumping and observation wells prior to beginning the tests to establish the groundwater elevation at static (i.e., non-pumping) conditions (need to ensure that the pumping well has re-established static conditions following insertion of the pump). These data will be used to determine the amount of drawdown during the tests. Wells should be gauged manually and using the transducers. Record the data in the field notebook.
2. Variable-rate tests typically consist of at least three pumping sessions with different pumping rates, one of which is the anticipated maximum extraction rate estimated from the existing site characterization data. Pumping strategies often begin at some fraction of the maximum anticipated rate (i.e., one-half) with increasing steps towards the maximum. An alternate strategy

begins with the anticipated maximum pumping rate and adjusts the steps up or down based on the drawdown observed in the pumping well. In either case, it is important that at least one of the pumping sessions be conducted at a rate that is greater than the initial estimate to verify that the maximum sustainable pumping rate has been achieved.

3. Begin the variable-rate test sequence. Careful synchronization of the start of the test and the pump are critical to ensure optimal data capture during the initial drawdown phase. Begin pumping the well 0.5 to 5 seconds after the data collection has started; starting pumping before the data loggers begin collecting data will result in the loss of important early drawdown data.
4. Monitor the pumping rate and groundwater elevation frequently during the test. Pumping rates should be maintained within 5 percent of the target extraction rate. Groundwater elevations in the pumping well should be monitored regularly using both the transducer and an electronic water level meter to ensure that the well is not dewatered during the test. Record discharge rates, meter readings, and manual water level measurements in the field book.
5. Continue each step for the prescribed amount of time, or until the water level in the extraction and observation wells reach stabilization. It is important to run the initial step long enough to establish that the effects of well storage (i.e., water stored in the well casing and surrounding filter pack) have dissipated and formation water is being drawn into the well. If the water levels do not stabilize and the continued operation of the test will expose an electric submersible pump, discontinue the test and reevaluate the pumping rate.
6. Water levels should be allowed to recover to static conditions (i.e., within 95 percent of the initial elevations) between each step. Do not remove the transducers or the pump from the test wells at the end of the test.
7. Download the groundwater elevation and barometric pressure data to the laptop or hand-held device and create a back-up copy of the data. It is not necessary to erase the data from the loggers as most contain sufficient memory to store millions of data points; leaving data on the loggers also provides a secondary back-up system. Verify that all of the data has been transferred to the laptop or another storage media (e.g., compact discs) before erasing the data files from the loggers.

Constant-Rate Pumping Test/Recovery Test

The constant-rate pumping phase of the aquifer test is designed to stress the water bearing zone over an extended period of time to evaluate the hydraulic conductivity and storage parameters of the unit. Typical constant-rate tests are run for 48 to 72 hours using the maximum pumping rate established during stepped-rate tests. The constant-rate test includes a recovery phase which occurs immediately after the pump is turned off. The recovery test is a passive test performed to monitor the response of the water-bearing zone as it recovers from the stress induced by the constant-rate test. Recovery is typically measured for 24 hours.

1. The set-up for the constant-rate test is identical to that of the stepped-rate tests. Groundwater levels should be at static, pre-pumping conditions before starting the constant-rate test.
2. Program the constant-rate test into the loggers and barometric pressure monitoring equipment. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The constant rate test is a logarithmic test. Caution should be used when setting the conversion

time for the test. High sampling frequencies that are important in the early phase of pumping become less desirable as the test proceeds and the water levels stabilize. Typical constant-rate tests performed in low to moderate yield unconsolidated units have conversion times that range between 15 and 60 minutes.

3. Begin the constant-rate test. Careful synchronization of the start of the test and the pump are critical to ensure optimal data capture during the initial drawdown phase. Begin pumping the well 0.5 to 5 seconds after the data collection has started; starting pumping before the data loggers begin collecting data will result in the loss of important early drawdown data.
4. Monitor the pumping rate and groundwater elevations periodically during the test. As with the stepped-rate test, pumping rates should be maintained with ± 5 percent of the target extraction rate and the water levels above the pump should be checked to ensure that the well is not being dewatered. Groundwater elevations in the pumping well should be monitored regularly using both the transducer and an electronic water level meter to ensure that the well is not dewatered during the test. Record discharge rates, meter readings, and manual water level measurements in the field book. Monitoring should be more frequent at the beginning of the test with reductions in the monitoring schedule, as appropriate. Once stabilization has been achieved, the monitoring schedule can be reduced further; however, monitoring should be conducted, at a minimum, every two hours after the water levels have stabilized to verify that the pumping rate remains constant, the water levels have not decreased due to changes in the water-bearing unit storage, and other equipment is operating as designed. Record the monitoring intervals and results in the field book.
5. Using the data recorded by the data loggers and barometric monitoring equipment, continue the constant-rate test for the prescribed amount of time or until the groundwater level in the pumping and observation wells reach stabilization.
6. At the conclusion of the constant-rate pumping test, suspend all data recording but do not shut off the pump or remove any equipment from the wells. Download the constant-rate test data from the data loggers.
7. Program a recovery test into each transducer using a laptop or hand-held device. Be sure that the internal time settings in the transducers, loggers, barometric pressure monitoring equipment, laptop, and/or hand-held computer are synchronized before programming any tests. The recovery phase is a logarithmic test. Log-linear conversion times should be the same for the recovery period as those used for the constant-rate pumping test.
8. Begin the start sequence for the recovery test. Careful synchronization of the start of the data loggers and the pump shutdown is critical for capturing the early response data that occurs as soon as the pumps are shut down (this is the inverse of the drawdown data collected during the pumping phases of the test). The pump should be shut down approximately 0.5 to 5 seconds after the data loggers begin recording the test.
9. Continue the recovery test for the prescribed amount of time or until the water level in the extraction and observation wells has returned to 95 percent of the pre pumping static water level. Do not remove the downhole equipment (e.g., pump, transducers) until the completion of the recovery test.
10. Download the groundwater elevation and barometric pressure data to the hand-held device and create a back-up copy of the data on a laptop computer at the conclusion of the recovery test. It is not necessary to erase the data from the loggers as most contain sufficient memory to store millions of data points; leaving data on the loggers also provides a secondary back-up system.

Verify that all of the data has been transferred to the laptop or other storage media (e.g., compact discs) before erasing the data files from the data loggers.

11. Remove the pump, tubing, piping, transducer cables, and transducers from the test wells and place them on plastic sheeting around the well or in a bucket. Replace well cap, locking expandable plug, and secure test wells with locking mechanism.
12. Decontaminate transducers, transducer cables, and pump with distilled water and paper towels (SOP No. 17). Decontaminated equipment should be placed on, in, or wrapped in plastic until further use. Properly dispose of decontamination water, PPE, lift line, and other disposable supplies in accordance with the IDW plan.

Standard Operating Procedures – 30

In Situ Single Well Hydraulic Conductivity Test (Slug Test)

Scope:
This standard operating procedure describes the methods and techniques to be employed for performing in situ single well hydraulic conductivity tests, or slug tests, on partially- or fully-penetrating monitoring wells or piezometers.

Objective:
The objective of performing in situ single well hydraulic conductivity tests, or slug tests, is to measure the hydraulic characteristics of the aquifer (water-bearing zone) in the immediate vicinity of the monitoring well or piezometer for aquifer characterization. The hydraulic conductivity test is performed by creating an instantaneous change in the water level by either inserting (falling head) or withdrawing (rising head) a slug from a monitoring well or piezometer that is at equilibrium.

Materials:
Field book
Monitoring well lock keys
Hand tools (wrenches), if necessary
Water level indicator
Pressure transducer-internal/external logger (Troll, PDA, or Hermit, see Table 1)
Oil/water interface probe, if necessary
Photoionization detector (PID), if necessary
Closed volume slug (polyvinyl chloride [PVC] pipe filled with sand, cement, or other inert material)
Nitrile or heat resistant gloves
Tape measure
Exacto, pocket knife, or scissors
Rope
Duct tape
Indelible marker or metal file
Paper towels
5-gallon bucket(s)
Distilled water (decontamination of downhole equipment)
Plastic sheeting

Procedures:

1. Review site Health and Safety Plan (HASP) before mobilizing to the field. Discuss groundwater contamination constituents and specific HASP issues with Project Manager.
2. Prepare field book (SOP No. 1) with site description, weather conditions, participants, and other relevant observations, including all data necessary to complete the hydraulic conductivity tests, or slug tests. Verify monitoring well or piezometer locations.
3. With field personnel in Level D personal protective equipment (PPE), unless historical data warrants upgrading to Level C PPE, survey ambient (background) air around the base of the well casing and wellhead for organic vapors using a photoionization detector (PID) or equivalent. Record air monitoring measurements in field book.

4. Inspect monitoring well or piezometer for soundness of protective casing, surface ground seal, and locking mechanism.
5. Unlock protective casing or flush mounted curb box. Remove well cap or plug, place PID probe at wellhead, and record PID response in field book. Survey breathing zone to ensure that PPE level is appropriate. Record air monitoring measurements in the field book.
6. Place plastic sheeting around the monitoring well or piezometer head before initiating any down well measurement or testing activities. After plastic is placed around the monitoring well covering the ground surface, measurement and testing procedures may commence. If necessary, place tape on well casing to prevent cutting or slicing of rope or cable.
7. Allow static water level to equilibrate, as appropriate, before measuring or determining the presence or thickness of a floating product layer (LNAPL) or a sinking free product layerc (DNAPL). Measure thickness using an oil/water interface probe, if necessary, in accordance with EPA RCRA Groundwater Monitoring Technical Enforcement Guidance Document [November 1992], state guidance, or site work plan documents (SOP No. 3a). Record observations in the field book. If LNAPL or DNAPL are present, do NOT perform hydraulic conductivity tests in the monitoring well/piezometer.
8. Measure the monitoring well or piezometer casing inside diameter (ID) and record in inches. From the top of the casing, measure the depth (in hundredths of a foot) to water (DTW) with an electronic water level indicator and record in the field book. Static water level measurements must be recorded from the surveyor's mark at the top of the casing, if present. If no mark is present, mark a location with a metal file or indelible marker on the north side of the casing and record in the field book for future reference. Measure and record the total depth (TD), in hundredths of a foot, of the monitoring well or piezometer.
9. Thoroughly decontaminate (SOP No. 17) the water level indicator cable during retrieval from the monitoring well or piezometer using distilled water and paper towels.
10. Thoroughly decontaminate the pressure transducer and closed volume slug using distilled water before placement of equipment into the monitoring well or piezometer. Decontaminated equipment should be placed on or wrapped in clean plastic sheeting until inserted into the monitoring well or piezometer. Record pertinent data logger and pressure transducer information (model or serial number and pressure transducer rating) in field book.
11. Using the tape measure, measure the length from the transducer bottom up the cable to a point where the transducer will be positioned in the monitoring well or piezometer. Mark the transducer cable at the appropriate length for reference. The transducer must be positioned at a depth below the static water level so upon total insertion of the slug into the water column (slug completely enters the water column), the slug bottom does not contact the top of the transducer.
12. Position the transducer in the monitoring well or piezometer below the static water level to the point where mark is aligned with the monitoring well or piezometer survey mark. Allow transducer to equilibrate for a minimum of 10 minutes before starting test.
13. Secure transducer cable to the wellhead, the well casing, protective casing (i.e., duct tape), or the curb box (i.e., "s" hooks). Note, the transducer must be secured such that the transducer can NOT move vertically within the water column during performance of the hydraulic conductivity tests. If using an external data logger, connect the transducer cable to the automatic data logger.

14. Power the data logger/transducer and check for proper operation and battery life. Enter monitoring well/piezometer information, transducer reference data, and testing parameters. Generate a test file for each planned individual slug test; the number of tests to be performed should be determined by the Project Manager, but should be no less than three (two falling and one rising) per monitoring well/piezometer. Verify that data logger/transducer collection mode is programmed to record on the logarithmic scale, with a maximum asymptotic interval of 10 minutes.
15. Evaluate water level stabilization by reviewing pressure head readings from the transducer. Once pressure readings have stabilized, record the transducer reference point (zero or reference head).
16. Verify that the slug is full and closed, as applicable. Closed volume slug should be of an appropriate diameter and length to displace an appropriate water volume on insertion into the water column. Secure rope to the top of the closed volume slug; checking knot to be certain rope will not untie during testing process. Using the tape measure, measure the length from the bottom of the closed volume slug up the rope to a point where the slug will be positioned immediately above the recorded static water level. Mark this location.
17. On the rope, mark a second depth measurement corresponding to the depth at which the slug should be lowered into the water column (falling head). The slug must be positioned below the static water level upon total insertion into the water column, but above the level of the transducer. Secure the closed volume slug rope to well head, the well casing, protective casing, or the curb box using duct tape or some other means.
18. In preparation for the first test, lower the closed volume slug into the monitoring well or piezometer so that the slug bottom is suspended immediately above the static water column. This position should correspond to the first mark (No. 16 above).
19. Falling-Head Tests
    Start the test on the data logger and, approximately 0.5 second to 1 second later, smoothly but quickly lower the closed volume slug into the water column (falling- head test, slug in) until the second mark is positioned equal to the wellhead survey mark. Be sure that the slug does not move the transducer cable.
20. Rising-Head Tests
21. Start the data logger and approximately 0.5 second to 1 second later, smoothly, but quickly, remove the closed volume slug from the water column (rising-head or bail-down test, slug out) until the first mark is positioned equal to the wellhead survey mark (ensuring that the slug is above the original static water level). Be sure that the slug does not move the transducer cable.
    20. During each hydraulic conductivity test, monitor (review) the water level data collected on the data logger to ensure proper operation. Record weather changes, heavy equipment movement, or other conditions that may affect water level measurements in the field book. In addition, verification should also be conducted to determine that the volume of the slug is roughly equal to the volume of water displaced, as measured by the data logger. Using the data recorded by the data logger, continue the hydraulic conductivity test, or slug test, until the water level in the monitoring well or piezometer returns to the smaller of either 0.02-foot from the original static water level or within five percent of the change in head.
22. Using the data logger, stop collecting measurements when the static water level stabilizes as described in Step 20.

23. After completion of the testing procedures at a location, remove closed volume slug, transducer cable, and transducer from the monitoring well or piezometer and place on plastic sheeting around the well or in a bucket. Replace well cap, expandable plug, curb box top and secure monitoring well or piezometer with locking mechanism.
24. Decontaminate transducer, transducer cable, and closed volume slug with distilled water and paper towels (SOP No. 17). Decontaminated equipment should be placed on, in, or wrapped in plastic until further use. Properly dispose of decontamination water, PPE, rope, and other disposable supplies in accordance with the investigation-derived waste (IDW) plan.
25. In the field, periodically, and/or before concluding the field activities, download data files from the transducer/logger to a computer diskette or laptop computer.

General Notes:
If groundwater quality data is available for the well(s) being tested, conduct the slug tests in the order of increasing contaminant levels to reduce the potential for cross-contamination.
Single well hydraulic conductivity test, or slug test, data should be analyzed using appropriate aquifer type (unconfined, confined, etc.), method (Bouwer and Rice, Hvorslev, etc.), monitoring well or piezometer construction parameters (fully- or partially-penetrating), pre-testing water level measurements, and HydroSOLV's AQTESOLV for Windows (or equivalent) software package.
Hydraulic conductivity (K) values are calculated (length/time) as centimeters per second (cm/sec), meters per second (m/sec), or feet per second (ft/sec).

Pressure transducer selection criteria:

Table 1
Maximum Depth Below Water Level for Pressure Transducer (a)

Pressure Transducer (PSI)	Depth Below Water Table (feet)
11	23
21	46
31	69
51	115

a/ PSI=pounds per square inch

Standard Operating Procedure - 31

Screening of Soils for Metals Using a Delta Handheld X-Ray Fluorescence Analyzer

Materials:

Personal protective equipment (PPE)
Sample containers
Sample labels and indelible marker
Stainless steel trowels or spoons
Field log book
Delta XRF
Battery pack and charger
Shielded case
Calibration standards
Test platform
Ziploc bags
Test guard
Baby wipes
Sample tray

Note: Prior to obtaining a rental XRF unit, determine the requirements for radioactive material licensing and training from the State's Radiological Materials Department.

Calibration:

1. Every time the XRF is turned on or reset, the instrument performs a self-calibration. In addition, several known standards are provided to check the calibration of the instrument. Calibrate the XRF at least daily, preferably each morning prior to conducting soil screenings, in the middle of the day, and at the end of day following soil screenings. Calibrate the XRF in accordance with the manufacturer's specifications using the low, medium, and high calibration standards.

2. Allow the instrument 15 minutes to warm up prior to field use.

In-situ Field Screening:

1. Select a measurement site. Always use the appropriate personal protective equipment based on the site conditions.

2. Clear any surface debris or vegetation from the measurement location, and ensure a flat surface to the soil. If necessary level the surface with a stainless-steel trowel or spoon.

3. The surface will be tamped to increase soil density, based on the SW-846 protocol; this process will allow better repeatability and ensure that the sample is representative.

4. Place the test guard on the ground.

5. Hold the Delta in one hand, and push the safety slide (that locks the shutter release) out from under the shutter release.

6. Place the Delta on the test guard so that the rectangular opening on the test guard is under the window of the Delta. Squeeze the shutter release and firmly press the instrument flat against the surface of the test guard (if you don't squeeze the shutter release, the plunger will not depress and the shutter will not fully open causing an inaccurate measurement). **Do not put your hand on the end plate of the unit while measuring. Never point the unit at yourself or anyone else when the shutter is open.**

7. Watch for indications to decide when the test has reached the desired level of accuracy. A typical screening will last 30-60 seconds.

7. The instrument will provide a reading in ppm plus a measurement precision (+/-). Add the precision to the reading of concern to determine the metal concentration at that location.

8. Clean the bottom (soil contact side) of the Delta Test Guard with a baby wipe prior to the next screening location.

Note: In the unlikely event that the plunger gets stuck in the open position, simply push it closed. Then call the Delta Service Department at (866) 446-6689.

<u>*Ex-situ* Field Screening:</u>

1. Using the trowel loosen and homogenize the soil to the desired depth.

2. Place 50-100 grams of soil in a new plastic bag and close securely.

3. Label each bag with the sample location/ID.

4. Break down large clumps of soil within the plastic bag while further homogenizing the sample.

5. Shape the bag of soil to form a continuous uniform layer of at least one centimeter thickness and analyze the sample using the *in-situ* screening procedure (steps 3-8).

Note: Test results will tend to be 10-15% lower than results obtained using the *in situ* screening procedure.

<u>Soil Moisture Content Screening:</u>

The XRF device does not perform well for saturated soils. Therefore, WSP will take the following precautions during XRF sampling:

1. WSP will not conduct XRF sampling unless there has been no precipitation at the site for at least 48 hours.
2. WSP will not conduct XRF sampling during a storm event.
3. WSP field representatives will ensure soil is not saturated prior to XRF analysis by conducting soil moisture measurements at each sampling location using a soil moisture meter (e.g., General DSMM500 Soil Moisture Meter).

4. If soil moisture content is greater than 20%, a new location within the sampling grid will be selected and the soil moisture content will be measured. This procedure will continue until a location within the grid is identified as having a soil moisture content of less than 20%.
5. If a viable sampling location is not available within the selected grid, a soil sample from within the grid will be collected in accordance with WSP SOP #2 for fixed-laboratory analysis.

Standard Operating Procedure - 32
Composite Soil Sampling

Application:

As with soil sampling, composite samples can be collected at the surface or at depth, and different equipment is required in each instance. Surface samples can be collected most efficiently with a trowel or scoop. For samples at depth, a decontaminated, clean bucket auger may be required. For a sample core, waste pile samplers may be used. To collect representative samples of a waste pile, composite samples may be required. The objectives of the sampling program will dictate the need to collect composite samples. A grid system set-up on the waste pile may also be used to collect individual or composite samples.

Materials:

Stainless steel trowels or scoops

Stainless steel spoons

Bucket augers

Extension rods

Auger handle

Pipe wrenches

Teflon tape

Work, nitrile, and surgical gloves

Saranex or Tyvek suit

Eye protection

Respirator (if necessary)

Analytical buckets

Tray, mixing pans

Ziploc® plastic bags

Stakes and string (grid system)

Procedure:

1. Soil samples collected from bucket augers, stainless steel spoons, trowels or scoops, will be placed in plastic bags to be homogenized with the additional samples intended for compositing. Each bag of soil shall be transferred to a clean tray or mixing pan for compositing. Use a clean, decontaminated spoon, scoop, or trowel to homogenize the sample. Remove any foreign matter (e.g., rocks, sticks, or other debris) from the tray. Transfer the homogenized sample directly into a pre-labeled sample container. The laboratory should be contacted in advance so they can provide the appropriate sample containers for the analysis required. A wide-mouth sampling

container is preferable for containing the sample, as it requires less disturbance of the waste sample as the sample is being transferred into the labeled sample container. Enough soil will be collected in total to fill the sample container.

2. Keep detailed notes in the field log book of the sample location, details about the grid spacing, sample description, sample depth, sampler's name, and the requested analytical parameter(s).
3. Complete the chain-of-custody forms with the appropriate sampling information.

Standard Operating Procedure - 33

Interstitial Pore Water Sampling and Analysis

Materials:

Personal protective equipment (PPE)

Sample containers

Sample labels and indelible marker

Stainless steel trowels or spoons

Field log book

Sediment Sampling for Interstitial Pore Water Laboratory Analysis

Below is an explanation of interstitial pore water and recommend procedures if the scope of work requires the extraction and analysis of interstitial pore water in sediment samples.

Sediment interstitial water, or pore water, is defined as the water occupying the spaces between sediment particles. Interstitial water might occupy about 50% (or more) of the volume of a depositional (silt-clay) sediment. The interstitial water is in contact with sediment surfaces for relatively long periods of time and therefore, might become contaminated due to partitioning of the contaminants from the surrounding sediments. In addition, interstitial waters might reflect ground water – surface water transition zones in upwelling or downwelling areas. In these areas their chemistry might be more reflective of ground or surface waters at the site. Therefore, flow, residence time and other physicochemical factors (e.g., pH, temperature, redox potential, organic carbon, sulfides, carbonates, mineralogy) might have varying roles in determining whether interstitial waters are contaminated.

In many depositional sediments, interstitial waters are relatively static, and therefore contaminants in the interstitial water and in the solid phase are expected to be at thermodynamic equilibrium. This makes interstitial waters useful for assessing contaminant levels and associated toxicity. Interstitial water is often isolated to provide either a matrix for toxicity testing and/or to provide an indication of the concentration and/or partitioning of contaminants within the sediment matrix.

Interstitial water sampling is most suitable for sediment types ranging from sandy to uncompacted silt-clays. Such sampling is not typically performed on sediments with coarse particle size (such as gravel) or on hard, compacted clays, as the potential for interstitial water contamination in the sediment types is relatively low.

When field indicator parameters require pH measurements for sediment, below is a brief discussion on procedures to follow.

Field Measurements for PH

pH is a measure of the effective concentration (or activity) of hydrogen ions and is expressed as the negative base-10 logarithm of the hydrogen-ion activity in moles per liter. Uncontaminated groundwater typically exhibits a pH ranging from 5 to 9 Standard Units (SU). Changes in pH from background may indicate the presence of groundwater contamination or that existing contamination has spread and can be very useful in identifying well construction or maintenance problems.

After calibrating the monitoring equipment, follow the manufacturer's instructions to select the display for readings the necessary geochemical parameters. The Horiba D51 equipped with a Horiba 9615-10D or 9625-10D pH electrode should be able to penetrate soft sediments. However, penetration into clays, sands and gravel conditions may not be achievable.

The following procedures should be followed and all observations and measurements recorded in the field book.

- Collection of field parameters should be completed within 15 minutes of sample collection
- Immerse the probe in the sediment (in-situ or within the sample container) and wait for stabilization of the reading before recording the measurement.
- If collecting field measurements ex-situ, agitate or swirl the sample during the stabilization period.

Record all field-testing measurement data, to include the following:

- Project name and location
- Date and time of measurement or test (including time zone, if applicable)
- Source and location of the measurement or test sample
- Analyte or parameter measured
- Measurement or test sample value
- Reporting units
- Initials or name of analyst performing the measurement
- Unique identification of the specific instrument unit(s) used for the test(s)

Ex-situ Extraction of Interstitial Water

Ex-situ interstitial water collection methods are often necessary when relatively large volumes of interstitial water are required (such as for toxicity testing), when *in-situ* collection is not viable or when a brief sampling time is critical. While these extraction methods can be done in the field or in the laboratory, extraction in the laboratory, just prior to analysis or testing, is preferable so that the sample is maintained as close to its original state as much as possible during transport and storage.

General Procedures

Centrifugation and squeezing are the two most common techniques for collecting interstitial water, and are generally preferred when large volumes are required. Other methods include

pressurization (e.g., vacuum filtration) devices, which can be used to recover small volumes of interstitial water.

Regardless of the method used, interstitial water should be preserved immediately for chemical analyses, if appropriate, or analyzed as soon as possible after sample collection if unpreserved (such as for toxicity testing). Significant chemical changes can occur even when interstitial water is stored for periods as short as 24 hours.

If sediments are anoxic, as most depositional sediments are, sample processing, including mixing of interstitial water that has separated from the sediment, should be conducted in an inert atmosphere or with minimal atmospheric contact. Exposure to air can result in oxidation of contaminants, thereby altering bioavailability. Air exposure can also result in loss of volatile sulfides, which might increase the availability of sulfide-bound metals. In addition, iron and manganese oxyhydroxides are quickly formed upon exposure to air. These compounds readily complex with trace metals, thus altering metals-related toxicity. Maintaining anoxic processing conditions is not necessary when study objectives are concerned with exposures to aerobic sediments, or if target contaminants are unaffected by oxidation in short-term toxicity or bioaccumulation testing.

Interstitial water filtration should be avoided. Numerous studies have shown that filters reduce toxicity and contaminant concentrations by retaining contaminant-associated particles and also by contaminant sorption onto the filter matrix. If filtration is stipulated by a test method, treated filters (e.g., pre-soaked in distilled, deionized water, or combusted at 400° C overnight for glass fiber filters) should be used, and an unfiltered sample should also be tested for toxicity and contaminant concentrations. The characteristics of filters and the filtering apparatus should also be carefully considered, as different filters have different sorptive capacities for different contaminants.

Extraction of interstitial water

- *Centrifugation is the generally preferred laboratory method for the extraction of interstitial water.*
- *Extraction of interstitial water should be completed as soon as possible.*
- *Interstitial water that has accumulated on the surface of the homogenized sediment sample should be mixed into the sediment before the sample is partitioned among centrifuge bottles.*
- *Unless site-specific information suggests otherwise, centrifuging should be at 4° C to minimize temperature-mediated biological and chemical processes.*
- *Interstitial water should be preserved immediately for chemical analyses or analyzed as soon as possible after extraction, unpreserved. For toxicity testing, interstitial water should be stored at 4° C for not longer than 24 hours, unless the test method dictates otherwise.*
- *Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead.*

Centrifugation

Centrifugation is the generally preferred laboratory method for collection of interstitial water. It is a relatively simple procedure that allows rapid collection of large volumes of interstitial water. It also facilitates the maintenance of anoxic conditions (if required). However, centrifugation, like other *ex-situ* procedures might yield chemical and/or toxicological artifacts due to the extraction procedures themselves, which might alter the natural equilibrium between interstitial water and sediment.

Prior to centrifugation, the sediment sample is homogenized and partitioned among centrifuge bottles. If the homogenized sample is stored prior to centrifugation, interstitial water might accumulate on the surface of the sediment. This overlying water should be mixed into the sediment before subsampling for centrifugation. Samples are then partitioned among centrifuge bottles. In general, approximately 50% of sediment moisture content can be extracted as interstitial water. If interstitial water volume requirements are lower, smaller sediment subsamples may be used.

Centrifugation has been performed at various temperatures. ASTM (2000a) recommends that the centrifugation temperature reflect the in situ sediment temperature to ensure that the equilibrium between the particulate and interstitial water is not altered. Alternatively, a temperature of 4° C may be preferred to minimize temperature-mediated chemical and biological processes.

When centrifuging coarse sand, it might be desirable to use a modified centrifuge bottle to aid interstitial water recovery. The modified bottle is equipped with an internal filter that can recover 75% of the interstitial water, as compared to 25 - 30% recovery from squeezing.

Below is an example of laboratory procedures for Centrifugation of Sediment for Pore Water collection provided by RTI Laboratories (Michigan).

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ANALYTICAL STANDARD OPERATING PROCEDURE

Centrifugation of Sediments

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Director, Environmental Services: Yemane Yohannes

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STANDARD OPERATING PROCEDURE

Centrifugation of Sediments SOP#: PWCEN_022714_R0

TABLE OF CONTENTS

Section		Page
1.0	Scope and Application	1
2.0	Safety Precautions	1
3.0	Sample Requirements and Sample Handling Procedure	1
4.0	Apparatus and Materials	2
5.0	Reagents	2
6.0	Sample Preparation	2
7.0	Preventative Maintenance and Routine Cleaning	3
8.0	Pollution Prevention and Waste Management	3
9.0	References	3

1.0 Scope and Application:

1.1 Introduction

RTI Laboratories, Inc. has prepared this document to detail the Standard Operating Procedure (SOP) for the centrifugation of sediments

A detailed description of the instrumentation, calibration, run parameters and quality control procedures is included in this SOP. For definition of terms not specifically defined in this document refer to RTI QAP Section 16.

1.2 Summary of Method

1.2.1 This standard operating procedure (SOP) provides general technical requirements and operational guidelines for collecting pore water samples from sediment. The requirements of this SOP are applicable to sampling of pore water from sediment collected in dredging, streams, rivers, lakes, ponds, swamps, wetlands and drainage ditches.

2.0 Safety Precautions

2.1 Waste materials submitted for analyses may contain hazardous components. Uncharacterized waste samples should be handled in a manner that minimizes exposure and personal contact. Analysis should be performed in a fume hood.

2.2 Chemicals used should be regarded as a potential health risk. Sample preparation and analysis is performed in a manner designed to minimize exposure using routine good laboratory practices.

3.0 Sample Requirements and Sample Handling Procedures

3.1 Samples are received in accordance with RTI Laboratories, Inc. Standard Operating Procedure for Sample Receipt and Custody (SRC001-A). Appropriate U. S. Environmental Protection Agency (EPA) sample handling procedures and sample preservation recommendations are followed.

3.1.1 Samples are collected in a clean container with sufficient material to yield enough water after centrifugation to perform the requested analyses.

3.1.2 Sample holding time is 28 days from collection.

3.1.3 Holding time for the generated water sample is dependent on the analytical method being performed.

4.0 Apparatus and Materials

4.1 Centrifuge capable of >1000 g.

4.2 Centrifuge buckets capable of holding at least 1 L

4.3 Balance capable of measuring up to 5 kg to the nearest gram.

4.4 Syringe and 0.45 μm syringe filters (disposable)

4.5 Clean, disposable pipettes.

4.6 Timer (0 to 30 minutes)

4.7 Scrub brushes

5.0 Reagents

5.1 DI Water-for cleaning purposes

5.2 Laboratory soap

6.0 Sample Preparation

6.1 Divide sample by weight into each of the two centrifuge buckets (for balancing purposes) to the nearest gram.

6.2 The centrifuge should be set at 1000-1500 rpm. These settings have been determined to be adequate to generate maximum pore water from samples

6.3 The sample will be spun for up to 30 minutes; however 15 minutes is usually sufficient to achieve pore water separation from the sediment. All samples will be centrifuged for the same period of time to ensure consistency.

6.4 Once the centrifuge rotor has come to a complete stop, the buckets should be carefully removed from the swing are to avoid unsettling the sample.

6.5 Carefully pour the supernatant into pre-cleaned, labeled wide-mouth jars. Clean, disposable pipettes may be used.

6.6 Filter appropriate amount of sample using a disposable syringe and 0.45 μm disposable filter.

6.7 Store all samples, supernatants and filtrates at 4 °C.

6.8 If no liner is used for the centrifuge buckets, they must be scrupulously cleaned using DI water and laboratory soap. Additionally, before each use they must be rinsed several times with DI water and dried thoroughly.

6.9 pore water preparations blank will be processed through all of the steps in this procedures using laboratory DI water as the sample matrix.

7.0 Preventative Maintenance and Routine Cleaning

7.1 If no liner is used for the centrifuge buckets, they must be scrupulously cleaned using DI water and laboratory soap. Additionally, before each use they must be rinsed several times with DI water and dried.

8.0 Waste Management and Pollution Prevention

8.1 Pollution Prevention

8.1.1 Reagents are purchased and quantities used to reflect anticipated usage and minimize disposal of expired or unused chemicals.

8.1.2 Chemicals are handled in a manner that minimizes the potential for release or spill of the material.

8.2 Waste Management.

8.2.1 Acidic materials (pH<2) are treated as hazardous waste and placed in the appropriate waste drum for disposal.

8.2.2 Soil samples are composited in a drum designated for incineration. Disposal of materials is addressed in the laboratory QAP (Section 5.6).

9.0 References

9.1 EPA Test Methods for Evaluating Solid Waste. Laboratory Manual Physical/Chemical Methods, Method 8330B, Revision 2 October 20EPA 8272.

9.2 RTI Laboratories, Inc. Quality Assurance Plan.

9.3 RTI Laboratories, Inc. SOP SRC001-A, Sample Receipt and Custody.

9.4 RTI Laboratories, Inc. Chemical Hygiene Plan

9.5 RTI Laboratories, Inc. Employee Handbook

Standard Operating Procedure - 34

Multi-habitat (MHAB) Benthic-Macroinvertebrate Collection Procedure

Modified from Indiana Department of Environmental Management (IDEM)

Technical Standard Operating Procedure S-001-OWQ-W-BS-10-T-R0 (IDEM, 2010)

TABLE OF CONTENTS

1.0 Scope of Operations 1

2.0 Checklist of Equipment for Macroinvertebrate Sampling 1

3.0 Definitions 2

4.0 Training and Experience Requirements 2

5.0 Field Procedures 3

5.1 Stream Sampling Locations 3

5.2 Pond Sampling 6

6.0 Interferences 6

7.0 Quality Assurance/Quality Control 7

8.0 Sample Labeling and Handling 7

9.0 Equipment Cleaning 7

10.0 References 7

Attachment A - IDEM Qualitative Habitat Evaluation Index (QHEI) form

1.0 Scope of Operations

The purpose of this technical standard operating procedure is to ensure that the sampling of benthic macroinvertebrate communities using the Multi-Habitat (MHAB) benthic-macroinvertebrate sampling method is completed in a consistent manner. This TSOP was modified from Indiana Department of Environmental Management (IDEM) Technical Standard Operating Procedure (S-001-OWQ-W-BS-10-T-R0). The macroinvertebrates are being collected for the purpose of analyzing the organism's tissue for Resource Conservation and Recovery Act (RCRA) metals using EPA method 6020.

2.0 Checklist of Equipment for Macroinvertebrate Collection

- Insect repellent
- IDEM Qualitative Habitat Evaluation Index (QHEI) form and maps
- Biological Samples Field Chain of Custody form

- Clipboard
- Field notebook
- Permanent marker
- Lead pencils
- D-frame dipnet (with extra net and clips) consisting of a wooden 52-inch handle attached to a 1 foot x 1 foot D-shaped frame fitted with a net with a mesh size of 500 micrometers
- Two No. 35 Sieves mesh size of 500 micrometers (0.0197 inches)
- Two plastic sample buckets, 10 quart capacity with handle and pouring spout
- White plastic sample tray (18 inches x 14 inches x 3.5 inches)
- Two pairs of forceps 4A or "fine-tip" forceps
- Chest waders
- Wading boots
- Hip boots (optional)
- Arm length rubber gloves
- Safety goggles or glasses
- Ice chest filled with wet or dry ice
- Chain of Custody forms
- Ziplock bags or other sampling containers
- 25-meter measuring tape
- Brush for cleaning sampling equipment

3.0 Definitions

"Glide" – An area common to most modified stream channels that do not have distinguishable pool, run, and riffle habitats; the current and flow is similar to that of a canal; the water surface gradient is nearly zero (Ohio EPA 2006).

"Pool" – An area of the stream with slow current velocity and a depth greater than riffle and run areas; the stream bed is often concave and stream width frequently is the greatest; the water surface slope is nearly zero (Ohio EPA 2006).

"Riffle" – The shallow parts of the stream where water flows swiftly over completely or partially submerged pebble to boulder sized rocks to produce surface agitation (Ohio EPA 2006).

"Run" – Areas of the stream that have a rapid non-turbulent flow; runs are deeper than riffles with a faster current velocity than pools and are generally located downstream from riffles where the stream narrows; the stream bed is often flat beneath a run and the water surface is not visibly broken (Ohio EPA 2006).

"X-Point": The point in the stream or pond where the sample was proposed. It should be included sampling.

4.0 Training and Experience Requirements

The field team should consist of at least two people. The person in charge of collecting the benthic invertebrate samples should have previous field experience with the collection and identification of aquatic macroinvertebrates. A familiarity with local flora and fauna (beside macroinvertebrates) in order to recognize poisonous species is highly recommended.

Sampling on surface waters requires safety consciousness of staff members. Team members should be familiar with site-specific hazards and emergency response.

5.0 Field Procedures

5.1 Stream Sampling Locations

If multiple samples are to be collected in the stream, work from the downstream-most location first and move upstream for each additional sample.

Document conditions of each sampling location in the field notebook on the Qualitative Habitat Evaluation Index (QHEI) form (Attachment A). The field notebook can reference the QHEI form and does not have to duplicate the information. Information to capture includes:

General Information

Stream Name/Location: The name of the stream and the road nearest to the stream (include GPS coordinates)

NPS Pollution: Note any potential sources of pollution in the sampling reach (litter, aboveground or below ground storage tanks, high traffic, etc)

Predominant Surrounding Land Use: Describe the predominant land use in the area surrounding the sampling reach.

Canopy Cover (% Open): Enter an estimation of the percent of stream area in the sampling reach not shaded by riparian vegetation.

Engineering: Note if the stream has been channelized or if a dam is present.

Riparian Zone/In-stream Features

Erosion: Describe any significant bank erosion in the sampling reach.

High Water Mark: If there is evidence of recent high flow (debris collected in tree limbs, traces of mud left on riparian vegetation) enter (in meters) an estimation of the maximum height of flow, above the current stream level.

Stream Depth: Enter an estimation (in meters) of the average riffle, run, and pool depths, if they were present at the site.

Stream Width: Enter (in meters) the width of the stream at the x-point.

Distances: Enter an estimation (in meters) of the distance between riffles or bends if more than one riffle or bend are present within the stream reach visible from the x-point.

Sediment

Sediment Odors: Note any odors that were detected during the sampling event.

Sediment Deposits: Check any sediment deposits that were encountered in the sampling reach.

Sediment Oils: Check one to provide an estimation of sediment oils (if any) that were encountered during the sampling event. If the undersides of un-embedded stones are black, check the appropriate box.

Substrate Components

Inorganic Substrate Components: Enter in percentages of multiples of 5 (0%, 5%, 10%, etc.) the amount of substrate composed by Bedrock, Boulders, Cobble, Gravel, Sand, Silt, or Clay.

Organic Substrate Components: Enter in percentages of multiples of 5 (0%, 5%, 10%, etc.) the amount of substrate composed by Detritus (as sticks or wood), Detritus (as CPOM), Muck/Mud, or Marl.

Water Quality

Water Odors: Check any water odors that were detected during the sampling event.

Water Surface Oils: Check any water surface oils that were detected in the sampling reach.

Stream Type: Check whether the stream was warm or cold water. Criteria for this designation can be found in Aquatic Research Center of the Indiana Biological Survey 2007.

Turbidity: Provide an estimation of the streams turbidity in the sampling reach.

Document GPS coordinates of sampling locations in field notebook and on field map. If a sample could not be collected note the reason.

Take upstream and downstream pictures of the sampling location.

Visually survey the in-stream and surrounding riparian habitat of the right and left banks of each stream reach 25 meters upstream and 25 meters downstream of the x-point. These 25-meter areas of the stream should be accurately measured using a 25-meter measuring tape or a rangefinder. Ideally, a good quality, wadeable riffle would be located within 25 meters of the x-point; if this is not the case, then a riffle located no more than 15 times the stream's wetted width (to a maximum of 500 meters) from the x-point may be sampled. If a riffle is not available, the crew leader should locate a good quality wadeable run or glide habitat to be sampled during the kick portion of the MHAB sampling procedure. A 1-minute kick sample is not collected in deep U-shaped (or bowl shaped) streams and in

streams where low-flow has reduced the stream to interstitial flow between a series of pools; in these instances the 25-meter MHAB sweep by itself is sufficient.

Identify the 25-meter section of stream shoreline habitat that will potentially yield the most diverse macroinvertebrate sample. The quality of the sample can be improved by selecting the section of stream featuring the largest number of productive in-stream macroinvertebrate habitat types, avoiding collecting samples downstream of bridges and tributaries flowing into the stream and attempting to collect the sample from an area that contains a riffle-run-pool complex. The x-point (which is fixed and must, under normal circumstances, be included in the macroinvertebrate sample) may be located at the "top" of the sampling reach (by sampling a 25-meter stretch of one bank downstream of the x-point), at the "bottom" of the sampling reach (by sampling a 25-meter stretch of one bank upstream of the x-point), or may be in the middle of the sampling reach (for instance, sampling 10 meters upstream and 15 meters downstream of the x-point on one bank). If necessary, the sampling reach may also be defined as two alternating or parallel sections of opposite banks that, combined, total 25 meters and includes the x-point.

If the actual x-point is located on a section of the stream that is currently dry or inaccessible, then the sample may be collected in a section of the stream containing water that is within 15 times the wetted width of the stream from the x-point. This adjustment to the sampling method should be used with reservation and only if the resulting sample location will provide similar habitat to what would have been found at the actual x-point. The reason for such a modification and the area of the stream sampled should be described in the field notebook and on the drawing of the site made in the QHEI form.

In the riffle kick portion of the MHAB sample locate an area in the lower (downstream) area of the riffle or run habitat that is to be sampled. While holding the d-frame net handle, the flat end of the d-frame net is placed securely onto the substrate so that there are no gaps under the frame. Disturb (by kicking) the riffle/run substrate one foot directly in front of the d-frame net so that any dislodged macroinvertebrates are carried by the current into the net. Move in an upstream or diagonal upstream direction across the riffle/run, continuously disturbing the substrate in front of the d-frame net, for a distance of about 10 feet (this is roughly equivalent to sampling one square meter of riffle/run habitat). After completion of the kick, inspect the contents of the net. Any stones (gravel-sized or smaller) or debris (leaves or small sticks) that are kicked into the net are retained. Cobble and large sticks kicked into the net should be visually inspected and any attached macroinvertebrates removed and placed in the net; the cobble or large sticks are then returned to the stream. Streams with little or no flow may require the crew leader to disturb the substrate and sweep the d-frame net through the disturbed water column above the substrate to collect this portion of the sample. After completion of the kick the contents of the d-frame net are emptied into a plastic bucket filled half full with water. This bucket should be left at the sample processing location to reduce the risk of losing the macroinvertebrate sample and may be covered to prevent the escape of any flying species (mostly adult *Coleoptera* and *Hemiptera* species).

The sweep portion of the sample begins with a team memeber at the lower (downstream) portion of the 25 meters of shoreline habitat that is to be sampled. The d-frame net is swept over the shoreline habitat so that the net dislodges any macroinvertebrates on the substrate surface and pushes them into the water column. The d-frame net is then swept through this disturbed area above the substrate two or three times to collect any dislodged macroinvertebrates. Sweeps are conducted at roughly 1.5 to 2-meter intervals for the entire length of the 25 meters of shoreline habitat for a total of about 25-30 sweeps. Sample as many microhabitats (rootwads, emergent vegetation, woody debris, leaf packs, sandy and silty substrates, and cobble and gravel substrates) as possible, sampling these microhabitats in relative proportion to their occurrence in the 25 meters of shoreline habitat. Care should be taken to avoid collecting large amounts of debris in the net as this will slow down the elutriation and picking processes.

After completion of the 25-meter habitat sweep, empty the contents of the net into the same bucket with the contents of the kick component of the sample.

Remove and visually inspect any cobble, large gravel, woody debris, and large leaves that were emptied into the bucket. During this and the following portion of the sample, the crew member should wear elbow length rubber gloves to minimize the risk of injury. Any macroinvertebrates found on these items are removed with forceps and placed into the white plastic tray. After inspection, these debris items are discarded. Due to the conservation status of many species, any freshwater mussels of the family *Unionidae* collected in the sample should be returned to a suitable area of the stream as soon as possible (their presence and species, if known, should be recorded on the stream data sheet and in the field notebook).

After removal of large items, the crew member will use their arm to swirl the contents of the bucket and then pour the water through a No. 35 sieve. The water flow from the bucket should be continuous but stopped before any sand is poured into the sieve. After each pour, the contents of the sieve are emptied into the white plastic tray. The bucket containing the sample is then refilled with stream water using a separate bucket (taking care to not add any additional macroinvertebrates via the refill bucket). This process is repeated until the water poured through the sieve is clear and free of silt. After the elutriations, the crew leader will inspect the contents of the bucket and remove any heavier organisms that were not emptied into the sieve (e.g., crayfish, snails, and Asian or fingernail clams). Handfuls of any remaining sand, leaves or fine gravel are then removed, visually inspected for any remaining organisms, and discarded.

The objectives of the pick are to collect enough organisms to meet the laboratory's minimum required sample mass (5 grams) and also if possible collect as diverse a community of organisms as possible. If known, identify the species collected in the field notes and their relative abundance. Use a pair of forceps to individually pick macroinvertebrates from the contents of the tray and deposit them into a Ziplock bag (doubled-bagged) and placed immediately on ice.

The bags should be pre-labeled with an indelible waterproof marker. Preserving the organisms for later identification of the species is not within the scope of this procedure therefore an ethanol solution will not be used. A laboratory courier will pick samples up daily and freeze them.

Fill out a sample label and insert it into the macroinvertebrate sample bags. If multiple sample containers are used for a single sample, the labels should also include "1 of 2" and "2 of 2."

5.2 Pond Sampling

Sampling for the invertebrates in a pond should be should be conducted in a similar manner to sampling in the stream except that because water is not moving rift sample techniques will not be possible. It may be necessary to disturb more of the bottom sediments to collect enough organisms. Sampling should start on the x-point and move away slowly in a spiral, expanding the size of the sample area just enough to collect the required sample mass.

6.0 Interferences

Heavy rains of more than one or two inches can often scour the stream substrate, removing many macroinvertebrates. Sampling a stream soon after a scouring event may result in a sample of poorer quality than what would be normal. A stream should not be sampled less than two weeks after a scouring event to allow for natural macroinvertebrate recolonization by drift.

7.0 Quality Assurance/Quality Control

If enough organisms are present to meet the minimum mass required for analysis; ten percent of sites sampled for macroinvertebrate community are re-sampled for QA/QC purposes. These duplicate samples are collected during the same sampling event as the original sample.

8.0 Sample Labeling and Handling

Each sample label should have the following information and should be copied in the field notebook and chain of custody form.

- Sample ID
- Sample Location
- Sampler name
- Date
- Time
- Analyses required
- Number of containers (bags)

An indelible waterproof marker can be used to write the sample information directly on the outer-most sample bag. Once labeled the samples should be place immediately in an ice chest filled with wet or dry ice. Samples should be covered in ice and not just placed on top. Samples should be double-bagged to prevent leakage. Contact the laboratory courier to pick the samples up. The courier should take the samples directly to the local laboratory where they will be checked in and placed in a freezer within 2 hours of receipt. The samples will not be allowed to thaw until they are ready to be processed by the laboratory.

9.0 Equipment Cleaning

After collecting the sample the sample tools (i.e., nets, forceps, tray, sieves, buckets) should be washed clean in the stream or pond to remove any trapped sediment. A second rinse of the equipment should be performed at the next sampling locations just prior to collecting the next sample.

10.0 References

IDEM. 2010. Multi-habitat Macroinvertebrate Collection Procedure, Technical Standard Operating Procedure S-001-OWQ-W-BS-10-T-R0, October 30, 2010

Ohio EPA. 2006. Methods for Assessing Habitat in Flowing Waters: Using the Qualitative Habitat Evaluation Index (QHEI). Ohio EPA Technical Bulletin EAS/2006-06-1. Web address as of September 16, 2010: http://www.epa.state.oh.us/portals/35/documents/QHEIManualJune2006.pdf

Sobat, S.L. 2006. Biological Studies Section Qualitative Habitat Evaluation Index (QHEI). IDEM Standard Operating Procedure S-001-OWQ-A-BS-06-S-R1. Biological Studies Section, Assessment Branch, Office of Water Management, Indiana Department of Environmental Management, Indianapolis, Indiana.

SOP 34 Attachment A

OWQ Biological Studies QHEI (Qualitative Habitat Evaluation Index)

Sample #	bioSample #	Stream Name	Location

Surveyor	Sample Date	County	Macro Sample Type	☐ Habitat Complete	QHEI Score:

1] *SUBSTRATE* Check ONLY Two predominant substrate TYPE BOXES; estimate % and check every type present

BEST TYPES

PREDOMINANT P	R		PRESENT P	R	TOTAL %
☐	☐	BLDR/SLABS [10]	☐	☐	____
☐	☐	BOULDER [9]	☐	☐	____
☐	☐	COBBLE [8]	☐	☐	____
☐	☐	GRAVEL [7]	☐	☐	____
☐	☐	SAND [6]	☐	☐	____
☐	☐	BEDROCK [5]	☐	☐	____

OTHER TYPES

PREDOMINANT P	R		PRESENT P	R	TOTAL %
☐	☐	HARDPAN [4]	☐	☐	____
☐	☐	DETRITUS [3]	☐	☐	____
☐	☐	MUCK [2]	☐	☐	____
☐	☐	SILT [2]	☐	☐	____
☐	☐	ARTIFICIAL [0]	☐	☐	____

(Score natural substrates; ignore sludge from point-sources)

NUMBER OF BEST TYPES: ☐ 4 or more [2] ☐ 3 or less [0]

ORIGIN (Check ONE (Or 2 & average))

- ☐ LIMESTONE [1]
- ☐ TILLS [1]
- ☐ WETLANDS [0]
- ☐ HARDPAN [0]
- ☐ SANDSTONE [0]
- ☐ RIP/RAP [0]
- ☐ LACUSTRINE [0]
- ☐ SHALE [-1]
- ☐ COAL FINES [-2]

QUALITY

SILT
- ☐ HEAVY [-2]
- ☐ MODERATE [-1]
- ☐ NORMAL [0]
- ☐ FREE [1]

SEDIMENTATION
- ☐ EXTENSIVE [-2]
- ☐ MODERATE [-1]
- ☐ NORMAL [0]
- ☐ NONE [1]

Substrate Maximum 20

Comments

2] *INSTREAM COVER* Indicate presence 0 to 3 and estimate percent: **0**-Absent; **1**-Very small amounts or if more common of marginal quality; **2**-Moderate amounts, but not of highest quality or in small amounts of highest quality; **3**-Highest quality in moderate or greater amounts (e.g., very large boulders in deep or fast water, large diameter log that is stable, well developed root wad in deep/fast water, or deep, well-defined, functional pools.)

% Amount		% Amount		% Amount	
___ ___	UNDERCUT BANKS [1]	___ ___	POOLS > 70cm [2]	___ ___	OXBOWS, BACKWATERS [1]
___ ___	OVERHANGING VEGETATION [1]	___ ___	ROOTWADS [1]	___ ___	AQUATIC MACROPHYTES [1]
___ ___	SHALLOWS (IN SLOW WATER) [1]	___ ___	BOULDERS [1]	___ ___	LOGS OR WOODY DEBRIS [1]
___ ___	ROOTMATS [1]				

AMOUNT (Check ONE (Or 2 & average))

- ☐ EXTENSIVE > 75% [11]
- ☐ MODERATE 25 - 75% [7]
- ☐ SPARSE 5 - < 25% [3]
- ☐ NEARLY ABSENT < 5% [1]

Cover Maximum 20

Comments

3] *CHANNEL MORPHOLOGY* Check ONE in each category (Or 2 & average)

SINUOSITY	DEVELOPMENT	CHANNELIZATION	STABILITY
☐ HIGH [4]	☐ EXCELLENT [7]	☐ NONE [6]	☐ HIGH [3]
☐ MODERATE [3]	☐ GOOD [5]	☐ RECOVERED [4]	☐ MODERATE [2]
☐ LOW [2]	☐ FAIR [3]	☐ RECOVERING [3]	☐ LOW [1]
☐ NONE [1]	☐ POOR [1]	☐ RECENT OR NO RECOVERY [1]	

Channel Maximum 20

Comments

4] *BANK EROSION AND RIPARIAN ZONE* Check ONE in each category for EACH BANK (Or 2 per bank & average)

River right looking downstream

L	R	EROSION
☐	☐	NONE/LITTLE [3]
☐	☐	MODERATE [2]
☐	☐	HEAVY/SEVERE [1]

L	R	RIPARIAN WIDTH
☐	☐	WIDE > 50m [4]
☐	☐	MODERATE 10-50m [3]
☐	☐	NARROW 5-10m [2]
☐	☐	VERY NARROW [1]
☐	☐	NONE [0]

L	R	FLOOD PLAIN QUALITY
☐	☐	FOREST, SWAMP [3]
☐	☐	SHRUB OR OLD FIELD [2]
☐	☐	RESIDENTIAL, PARK, NEW FIELD [1]
☐	☐	FENCED PASTURE [1]
☐	☐	OPEN PASTURE, ROWCROP [0]

L	R	
☐	☐	CONSERVATION TILLAGE [1]
☐	☐	URBAN OR INDUSTRIAL [0]
☐	☐	MINING / CONSTRUCTION [0]

Indicate predominant land use(s) past 100m riparian.

Riparian Maximum 10

Comments

5] *POOL/GLIDE AND RIFFLE/RUN QUALITY*

MAXIMUM DEPTH (Check ONE (ONLY!))

- ☐ > 1m [6]
- ☐ 0.7 - < 1m [4]
- ☐ 0.4 - < 0.7m [2]
- ☐ 0.2 - < 0.4m [1]
- ☐ < 0.2m [0]

CHANNEL WIDTH (Check ONE (Or 2 & average))

- ☐ POOL WIDTH > RIFFLE WIDTH [2]
- ☐ POOL WIDTH = RIFFLE WIDTH [1]
- ☐ POOL WIDTH < RIFFLE WIDTH [0]

CURRENT VELOCITY (Check ALL that apply)

- ☐ TORRENTIAL [-1]
- ☐ VERY FAST [1]
- ☐ FAST [1]
- ☐ MODERATE [1]
- ☐ SLOW [1]
- ☐ INTERSTITIAL [-1]
- ☐ INTERMITTENT [-2]
- ☐ EDDIES [1]

Indicate for reach – pools and riffles.

Recreation Potential (Circle one and comment on back)

- ☐ Primary Contact
- ☐ Secondary Contact

Pool/ Current Maximum 12

Comments

Indicate for functional riffles; Best areas must be large enough to support a population of riffle-obligate species:

☐ NO RIFFLE [metric = 0]

RIFFLE DEPTH

- ☐ BEST AREAS > 10cm [2]
- ☐ BEST AREAS 5 - 10cm [1]
- ☐ BEST AREAS < 5cm [metric = 0]

RUN DEPTH (Check ONE (Or 2 & average))

- ☐ MAXIMUM > 50cm [2]
- ☐ MAXIMUM < 50cm [1]

RIFFLE/RUN SUBSTRATE

- ☐ STABLE (e.g., Cobble, Boulder) [2]
- ☐ MOD. STABLE (e.g., Large Gravel) [1]
- ☐ UNSTABLE (e.g., Fine Gravel, Sand) [0]

RIFFLE/RUN EMBEDDEDNESS

- ☐ NONE [2]
- ☐ LOW [1]
- ☐ MODERATE [0]
- ☐ EXTENSIVE [-1]

Riffle/ Run Maximum 8

Comments

6] *GRADIENT* (ft/mi)

- ☐ VERY LOW - LOW [2 – 4]
- ☐ MODERATE [6 - 10]
- ☐ HIGH - VERY HIGH [10 - 6]

%POOL: ____ %GLIDE: ____

DRAINAGE AREA (mi^2)

%RUN: ____ %RIFFLE: ____

Gradient Maximum 10

IDEM 07/06/10

IDEM

OWQ Biological Studies QHEI (Qualitative Habitat Evaluation Index)

COMMENT _____

A-CANOPY

- ☐ > 85% - Open
- ☐ 55% - < 85%
- ☐ 30% - < 55%
- ☐ 10% - < 30%
- ☐ < 10% - Closed

B-AESTHETICS

- ☐ Nuisance algae
- ☐ Invasive macrophytes
- ☐ Excess turbidity
- ☐ Discoloration
- ☐ Foam/Scum
- ☐ Oil sheen
- ☐ Trash/Litter
- ☐ Nuisance odor
- ☐ Sludge deposits
- ☐ CSOs/SSOs/Outfalls

C-RECREATION

	Area	Depth
Pool:	☐ > 100 ft^2	☐ > 3 ft

D-MAINTENANCE

☐ Public ☐ Private
☐ Active ☐ Historic
Succession: ☐ Young ☐ Old
☐ Spray ☐ Islands ☐ Scoured
Snag: ☐ Removed ☐ Modified
Leveed: ☐ One sided ☐ Both banks
☐ Relocated ☐ Cutoffs
Bedload: ☐ Moving ☐ Stable
☐ Armoured ☐ Slumps
☐ Impounded ☐ Desiccated
☐ Flood control ☐ Drainage

E-ISSUES

☐ WWTP ☐ CSO ☐ NPDES
☐ Industry ☐ Urban
☐ Hardened ☐ Dirt & Grime
☐ Contaminated ☐ Landfill
BMPs: ☐ Construction ☐ Sediment
☐ Logging ☐ Irrigation ☐ Cooling
Erosion: ☐ Bank ☐ Surface
☐ False bank ☐ Manure ☐ Lagoon
☐ Wash H_2O ☐ Tile ☐ H_2O Table
Mine: ☐ Acid ☐ Quarry
Flow: ☐ Natural ☐ Stagnant
☐ Wetland ☐ Park ☐ Golf
☐ Lawn ☐ Home
☐ Atmospheric deposition

Looking upstream (> 10m, 3 readings; < 10m, 1 reading in middle); Round to the nearest whole percent

	Left	Middle	Right	Total Average
% open	___ %	___ %	___ %	___ %

Stream Drawing:

Attachment C – Laboratory Standard Operating Procedures

Pace Analytical Services, Inc
1241 Bellevue Street, Suite 9
Green Bay, WI 54302
Phone: 920 469 2436
Fax: 920 469 8827

STANDARD OPERATING PROCEDURE

SAMPLE MANAGEMENT

Reference Methods: N/A

Local SOP Number:	S-GB-C-010-Rev.05
Effective Date:	Date of Final Signature
Supersedes:	S-GB-C-010-Rev.04
SOP Template Number:	SOT-ALL-C-001-rev.04

APPROVALS

Nils Melberg, Laboratory General Manager — Date: 05/13/13

Kate Grams, Laboratory Quality Manager — Date: 5/6/13

Alee Her, Department Manager — Date: 2/25/13

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 3
5. Limits of Detection and Quantitation 3
6. Interferences 3
7. Sample Collection, Preservation, Shipment and Storage 4
8. Definitions 4
9. Equipment and Supplies (Including Computer Hardware and Software) 5
10. Reagents and Standards 5
11. Calibration and Standardization 6
12. Procedure 6
13. Quality Control 14
14. Data Analysis and Calculations 15
15. Data Assessment and Acceptance Criteria for Quality Control Measures 15
16. Corrective Actions for Out-of-Control Data 15
17. Contingencies for Handling Out-of-Control or Unacceptable Data 15
18. Method Performance 15
19. Method Modifications 15
20. Instrument/Equipment Maintenance 15
21. Troubleshooting 15
22. Safety 15
23. Waste Management 16
24. Pollution Prevention 16
25. References 16
26. Tables, Diagrams, Flowcharts, and Validation Data 16
27. Revisions 17

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to outline the procedures involved with the receipt, login, storage, and disposal of samples received by Pace Analytical Services, Inc, Green Bay, WI.

2. Summary of Method

2.1. Samples are delivered to the laboratory via several delivery mechanisms. Samples received are checked for adherence to the Sample Acceptance Policy with any discrepancies noted. Discrepancies are communicated to the client for their acknowledgement and decision making.

2.2. The Laboratory Information Management System (LIMS) assigns all samples with a unique sample number and manages the analyses assigned to each sample.

2.3. Samples are labeled with the appropriate information and staged in refrigerated sample storage coolers if temperature preservation is required or on open shelves for samples not requiring sub-ambient temperature preservation. Samples will remain under these conditions until prepared and/or analyzed.

2.4. Samples and associated sub-samples (digestates, extracts, etc.), with the exception of Air cans, are maintained for a minimum of 45 days from receipt of samples unless otherwise requested by the client or other regulatory agency.

2.5. Samples are disposed of in accordance with local laboratory regulatory requirements and the laboratory's waste handling procedures.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP apply to all personnel involved in the receipt, login, storage, and disposal of samples.

3.2. A sample acceptance policy is contained within the Procedure section that outlines guidelines for acceptable sample conditions. Any deviation from these guidelines requires detailed documentation within the report, usually as a footnote, or on the chain-of-custody, or SCURF and may require client contact.

3.3. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Samples may be prone to cross contamination from others within the same delivery group or from other client projects. The sample receiving personnel must make every effort to minimize cross- contamination.

6.2. Preservation checks are one of the most likely situations where cross-contamination may occur. Materials used in the process must be specific to each sample and may not used for multiple samples.

6.3. Samples are stored under specific conditions and in specific locations, typically by container type. However, consideration must be given to samples that are uniquely different from others. Samples that are anticipated to be severely contaminated should be segregated from others in anticipation that the high levels of contaminants may cross-contaminate others in close proximity.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Acceptable sample preservation, containers, and hold times are listed in Attachment IV of this SOP. They may also be located in the Pace Quality Assurance Manual, the laboratory's method SOPs or in the applicable test method. Samples are stored separately from all standards and reagents and any known highly contaminated samples.

7.2. **NOTE**: To avoid contamination, no food or drink products can be located near the areas where samples are unpacked, labeled, or staged.

7.3. Sample Storage – See Section 12.3 for general storage guidelines.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **COC - Chain-of-Custody:** a form used to record the field identification of samples collected, analyses requested, date and time of collection, sample preservation used, and traceability of samples from time of collection until delivery to the laboratory. This is a legal document.

8.3. **LIMS - Laboratory Information Management System:** a computer system used to manage the flow and traceability of environmental samples and associated data within the laboratory.

8.4. **Matrix:** the bulk characteristics of a sample. See Table 8.1 below.

8.5. **SDS - Safety Data Sheet:** contains information on chemicals used in the laboratory.

8.6. **Sample Custody:** a sample is considered to be in someone's custody if:

8.6.1. It is in one's physical possession;

8.6.2. It is in someone's view, after being in someone's physical possession;

8.6.3. It is kept in a secured area, restricted to authorized personnel only.

8.7. **SCURF - Sample Condition Upon Receipt Form:** a form used to record the condition of samples received in the laboratory.

8.8. **SRF – Sample Receipt Form:** form generated by LIMS system after a project is logged in. Contains sample and project information.

8.9. **UN Number** - identification numbers preceded by the letters UN are associated with proper shipping names considered appropriate for international and domestic transportation. These shipping names along with the identification numbers are located in the Federal Register (49CFR172.101).

Table 8.1

NELAC/TNI defined matrix	Pace Analytical defined matrix
Aqueous: any liquid sample not defined as drinking water or saline. Includes surface water, groundwater, effluents, TCLP, and other extracts.	Waters: includes groundwater, wastewaters, drinking waters, effluents, and any free-flowing liquids.
Drinking water: any aqueous sample that has been designated as potable or potentially potable.	Not assigned as a separate matrix, but samples are assigned to drinking water methods.
Saline/Estuarine: any aqueous sample from an ocean, estuary or other salt water source	Not assigned as a separate matrix from waters.
Non-aqueous liquid: any organic liquid with <15% settleable solids.	Other: Not assigned as a separate matrix from waters.
Biological tissue: any sample from a biological origin such as fish tissue or plant material.	Tissue: would include tissue and plant samples.
Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids	Soils: includes soils, sediments, sludges; other solid materials such as wood, metal, etc. may fall under another heading.
Chemical waste: a product or by product of an industrial process that results in a matrix not defined above.	Oil: includes any non-solid material not classified as waters.
Air: vapor samples including those contained within sorbent tubes, filters or other devices.	Air: vapor samples including those contained within sorbent tubes, filters, or other devices.
No corresponding matrix to wipe; wipes would be included in with solids.	Wipe: includes wipe samples or swabs taken to check for surface contamination.

9. Equipment and Supplies (Including Computer Hardware and Software)

Table 9.1

Equipment/Supplies	Description/ Comments
Sample Labels	
Thermometers	Jacketed, NIST traceable, readable at 0.5°C
Sample storage cooling units	Capable of holding required storage temperatures
Chain-of-Custody forms	
Sample Condition Upon Receipt Form (SCURF)	
pH Paper	
Label Printer	
LIMS computer system	EPIC Pro
Disposable pipettes	
Sample containers	C&G, QEC, Fisher
Custody Seals	QEC or equivalent

10. Reagents and Standards

10.1. All reagents used in this procedure must be labeled with:

10.1.1. Laboratory reagent identification number;

10.1.2. Unless otherwise noted, the name and concentration of the reagent;

10.1.3. Date the reagent was received, opened and, as needed, prepared;

10.1.4. Person preparing reagent;

10.1.5. Expiration date.

10.2. **Reagents: Table 10.1**

Reagent	Formula	Concentration
Sulfuric Acid	H_2SO_4	1:1
Nitric Acid	HNO_3	1:1
Hydrochloric Acid	HCl	1:1
Sodium Hydroxide	NaOH	50% or Pellets
Sodium Thiosulfate	$Na_2S2O_3 \cdot 5H_2O$	
Zinc Acetate Solution (for sulfide)		
Methanol	MeOH	Purge and Trap Grade
Ascorbic Acid (for VOA)		
Sodium Bisulfate		
a,a,a-Trifluorotoluene	$C_6H_5CF_3$	2.5mg/L

10.3. For acids, bases and other reagents obtained from other laboratory departments, this information is located in the department reagent preparation log. In the event that these reagents are managed within the Sample Receiving group, the department must maintain its own reagent preparation log.

10.4. Some Pace labs use pre-preserved sample containers. In this case, documentation must be maintained for bottleware and preservation traceability.

11. Calibration and Standardization

11.1. Thermometers, IR-Guns, and other equipment used for measuring temperatures must be calibrated according to SOP S-GB-Q-030 **Support Equipment**, or its equivalent revision or replacement.

12. Procedure

12.1. **Sample Receipt**

12.1.1. The laboratory receives client samples via three major methods: mail/commercial delivery service, Pace Analytical courier/field services and hand delivery.

12.1.2. Pace labs use courier services that pick up client samples on either a regular schedule or on an as-needed basis as communicated by Project Managers or by the client.

12.1.2.1. When the client is present during courier pick-up, the client signs the chain-of-custody (COC) relinquishing custody to the courier. The courier signs the COC as accepting the samples and provides the client with a copy of the COC. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab.

12.1.2.2. If the client is not present during courier pick-up, the courier signs the COC as accepting the samples and leaves a copy of the COC for the client. If a client also has a sample log in use, the courier must sign and date the log when the samples are picked up. When the courier returns to the lab with the client samples, the courier signs the COC as relinquishing the samples to the lab. The date/time of delivery to the lab by the courier is the official date/time received by the lab (analogous to the official date/time of receipt by an outside commercial carrier or courier).

12.1.2.3. To ensure the sample security, the Pace courier custody seals all the coolers being picked up and the courier vehicle is locked at each client pick-up location. IMPORTANT: Pace Analytical courier/field services personnel must open the sample coolers and verify there is adequate ice in the coolers before transporting or shipping to the laboratory.

12.1.2.4. The lab must provide pertinent information to the person or persons responsible for taking and transporting samples. This information includes sampling procedures (where applicable), and information on storage and transport of samples, including any information on factors that may influence the test results.

12.1.3. The chain of custody (COC) (see example Attachment I) is signed immediately upon receipt of the samples from the client. If the client drops off the samples or they are picked up by the Pace courier, a copy of the signed COC is given to the client at that time. If samples are received via commercial carrier or mail delivery, the COC should be signed immediately when the cooler or package is opened and ultimately placed in the project file. The delivery date and time is considered the date/time received.

12.1.4. Sample receiving personnel should review the COC for any evidence of rush turnaround requests and analyses with short hold times. Projects that fall under these conditions should be given immediate attention. The project manager responsible for that client should be alerted in the event that they have not already alerted the laboratory to the project as it may be possible that the client did not pre-schedule the project. Once the samples are received and logged into the LIMS, the sample technician and project manager will coordinate the notification and delivery of samples to the laboratory.

12.1.5. **Sample Acceptance Policy** - This section constitutes the laboratory's sample acceptance policy. Copies of this policy should be provided, in the form of a letter, fax, or e-mail to each client or sampler, as necessary. Samples are considered acceptable if they meet the following criteria:

12.1.5.1. There is proper, full, and complete documentation (e.g., chain-of-custody) including:

12.1.5.1.1. Unique client sample identification. Sample containers are labeled using unique client sample identifications (traceable to the chain-of-custody or other documentation) on durable, waterproof labels or equivalent;

12.1.5.1.2. Location of sampling (site), time and date of sample collection;

12.1.5.1.3. Sampler's name and signature;

12.1.5.1.4. Preservative used (if any);

12.1.5.1.5. Sample type (matrix);

12.1.5.1.6. Requested analyses;

12.1.5.1.7. Any special analysis requirements.

12.1.5.2. Appropriate sample containers have been used;

12.1.5.3. Holding times have not been exceeded upon receipt (holding times are available in Attachment IV). If they have been exceeded, client permission to proceed is required;

12.1.5.4. Adequate sample volume has been received for all tests requested (if not, client permission to proceed is required). For data packages requiring quality control samples to be analyzed on client specific samples, the client must submit adequate sample volume to complete the QC samples as well;

12.1.5.5. When there is insufficient sample to complete the QC samples and the client does not wish to send more sample or more sample cannot be obtained because of sample volume available or holding time issues, the lack of appropriate volume or weight is noted as a sample acceptance policy

deviation on the final report. Batch quality control samples will be used in place of project-specific quality control samples;

12.1.5.6. Samples that require sub-ambient thermal preservation are considered acceptable if they are within +/- 2.0 °C of the required temperature (for samples needing to be at 4.0 °C, the acceptable range is just above freezing to 6.0 °C, as defined by NELAC/TNI). Biological Tissue samples are considered acceptable if they are received $\leq 0°C$. Temperatures of samples received from the State of Wisconsin are recorded as ROI (received on ice) unless client/project required the actual temperature be taken and recorded. For a cooler temperature to be recorded as ROI, actual ice must be present in the cooler. If ice is not present, the temperature is recorded directly on the COC. The sample cooler (ice chest) temperature is recorded directly on the COC. Samples received outside of this criterion must have a notation on the COC and/or on the Sample Condition Upon Receipt Form (SCURF) indicating that the temperature was outside of criteria;

12.1.5.7. Samples that are delivered to the lab on the same day they are collected are considered acceptable if the samples are received on ice. If samples arrive at temperatures that are outside these requirements, the client will be notified and analysis will NOT proceed unless otherwise directed by the client. Data will be appropriately qualified:

12.1.5.7.1. If sample analysis is begun within fifteen minutes of collection, thermal preservation is not required.

12.1.5.7.2. Thermal preservation is not required in the field if the laboratory receives and refrigerates the sample within fifteen minutes of collection.

12.1.5.8. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified

12.1.6. Open the cooler and verify the temperature of the samples by taking the temperature of the temperature blank. If there is no temperature blank in the cooler, measure the temperature of a representative sample bottle using the infrared (IR) thermometer gun. A representative sample will reflect an "average" condition of the samples in the cooler and, depending on the manner in which they are packed, may not necessarily be in direct contact with the cooling material.

12.1.6.1. If an IR gun is not used, the temperature of the cooler should be taken using a NIST-traceable, jacketed thermometer. The jacketed thermometer is placed into the temperature blank, or if there is no temperature blank, the jacketed thermometer is placed into the melt water of the cooler for approximately 5 minutes. After 5 minutes, the thermometer is read to the nearest 0.5°C increment and recorded. If no ice is present, a sample aliquot (non-volatile) is poured into a small container and the temperature of the sample is taken.

12.1.6.2. NOTE: If an IR gun is used, the temperature should be taken from an opaque surface such as the bottle label. Measurements taken through a transparent surface (clear or amber glass) may not be reliable and should incorporate a specific temperature correction factor for that surface reading.

12.1.7. Record the temperature on the COC (example in Attachment I) and/or SCURF (example in Attachment II). In addition, record the type of "ice" used for packing the cooler (e.g., wet ice, "blue ice", gel packs, etc.).

12.1.8. If samples within a project are spread over multiple coolers and one or more of the coolers are outside of the temperature criteria, then the contents of the cooler must be itemized and the samples and sample containers affected by the out-of-control temperature must be listed on the SCURF for inclusion with the final report. This itemization must be retained in the project file for future reference.

12.1.9. Unpack the cooler and chain of custody (COC). Organize the samples, grouped by client sample ID, according to the order on the COC. Review COC against samples to make sure the bottles received match the analysis requested. All anomalies must be recorded on the SCURF.

12.1.9.1. If the lab receives coolers late or cannot unpack a cooler until the following day the following is performed:

12.1.9.1.1. The cooler is visually inspected and opened.

12.1.9.1.2. The COC is removed and signed.

12.1.9.1.3. The temperature of the cooler is taken and recorded on COC.

12.1.9.1.4. The COC is checked for short hold time samples. If short hold time samples are present, the cooler is processed immediately.

12.1.9.1.5. If short hold time samples are not present, the cooler and contents are placed into the designated Walk-in Cooler to be processed the following day.

12.1.10. Discard any ice or water that remains in the cooler and the packing material used to secure the samples. Water or ice should be discarded down a drain that connects to the local sewer. Packing materials should be placed in the garbage. If a sample container was broken, the contents remaining in the cooler MUST be discarded in a manner consistent with the hazardous waste handling standard operating procedure.

12.1.11. **pH Verification Instructions**

12.1.11.1. The pH of the sample must be verified on all preserved sample bottles requiring pH preservation (see exceptions below).

12.1.11.2. Open each preserved bottle (except as noted below). Use a new disposable pipette, a stirring bar or another inert utensil to withdraw a small portion of the sample. Dispense the aliquot on a sample specific narrow-range pH strip and check the pH.

12.1.11.3. NOTE: Do not check the pH of samples for coliform, volatiles, TOC, WI-DRO, oil and grease, or hexane extractables (method 1664A). These analyses will be checked by the analyst at the bench and should not be opened by sample management personnel.

Table 12.1 – General pH Preservation Requirements by Preservative

Sample Preservatives	Sample pH Requirement
HCl	must be less than 2
HN03	must be less than 2
H2SO4	must be less than 2
NaOH	must be greater than 12
ZnAcetate & NaOH	must be greater than 9

12.1.11.4. If the pH is not within the required range, indicate the anomaly on the SCURF form or on the COC. If all bottles are unpreserved for a sample, write N/A in the section of the SCURF.

12.1.12. **pH Preservation Adjustments**

12.1.13.1. If a sample container does not meet the pH preservation required, the pH of the sample must be recorded on the COC or SCUR. Additional preservative is added so that the preservative content is <1% of the sample container volume. The sample is mixed and the pH is taken again. The new pH reading is also recorded on the COC or SCUR along with the amount, type and lot number of the preservative added. In addition, the sample container is marked with the preservative added, volume added, date, time and initials of the technician.

12.1.13.2. If unpreserved sample is received by lab and requires preservation, a clean container is used with appropriate preservation. An aliquant of unpreserved sample is poured out into preserved containers. Sample container is marked with a yellow sticker to notify lab that preservation was done in-house.

12.1.13. **Total Residual Chlorine Verification Instructions** - Total residual chlorine must be verified at the bench as required by the method or individual state regulatory agency for certain analyses (see Table 12.2). Do not check the sample bottles for those analyses listed in 12.1.10.

12.1.14.1. Open the appropriate sample container. Utilizing a new disposable pipette, stirring bar or other inert utensil; withdraw a small portion of the sample. Dispense the aliquot on a sample specific residual chlorine test strip.

12.1.14.2. If any chlorine is detected, regardless of amount, note the information on the COC, SCURF or analytical bench sheet.

Table 12.2 – Analyses requiring Residual Chlorine Verification

Analyses
Ammonia (NH3)
Nitrate (NO3)
Biochemical Oxygen Demand (BOD)
Cyanides
TKN
Dioxin 1613B
PBDE 1614
PCBs 1668A

12.1.14. Note any discrepancies pertaining to samples as defined by the sample acceptance policy detailed above on the COC or SCURF. Any discrepancies involving temperature, preservation, hold time, collection dates and times, sample volume, sample containers, and unclear analysis, must be reported to project management as soon as possible.

12.1.15. For short hold samples, the laboratory is notified and the samples are staged per section 12.3.

Table 12.3 – Analyses with Hold Times Less Than 72 Hours

Short Hold Time	Analyses	Details
15 minutes	Field Parameters	pH, Dissolved Oxygen, Residual Chlorine
8 Hours	Total / Fecal Coliform (MPN, MF)	
24 Hours	Heterotropic Plate Count	
24 Hours	Hexavalent Chromium	Aqueous Samples Only
24 Hours	Odor	
30 Hours	Total Coliform (Presence / Absence)	
48 Hours	Color	
48 Hours	MBAS	
48 Hours	Nitrate (unpreserved)	If Preserved, reported as NO3+NO2
48 Hours	Nitrite (unpreserved)	If Preserved, reported as NO3+NO2
48 Hours	Ortho –phosphate	
48 Hours	Settable Solids	
48 Hours	Turbidity	
48 Hours	VOA - Soils by Unpreserved EPA5035	Jars, Encores, Sleeves
48 Hours	Gross Alpha (NJ 48hr method)-waters	EPA NJAC 7:18-6
72 Hours	3030C Metals	

12.2. **Sample Login**

12.2.1. Rush projects and/or projects with short holds should be logged in first. After these projects have been addressed, projects should be addressed on a first in, first out basis.

12.2.1.1. Samples must be logged into the LIMS so the samples can be uniquely identified (Lab sample identification numbers). These lab sample ID numbers are used to track the prep and analysis activities of the samples, as well as identify the sub-samples, digestates, extracts, and other sample byproducts. This laboratory code maintains an unequivocal link with the unique client field sample ID code assigned to each sample.

12.2.1.2. Clients and Project Profiles are created in EPIC Pro as per Training Document T-ALL-IT-002 *Epic Pro 02: Client Setup.* Projects are logged as per Section 1 of Training Document T-ALL-IT-005 *Epic Pro: Login*

12.2.1.3. Tests are assigned as per Training Document T-ALL-IT-002 *Epic Pro02: Client Setup,* and as per Section 1 of Training Document T-ALL-IT-005 *Epic Pro: Login*

12.2.2. Generate sample labels and Sample Receipt Form (SRF) (see Attachment III).

12.2.2.1. Local SRF generation is as per Section 3 of Training Document T-ALL-IT-005 *Epic Pro: Login*

12.2.2.2. Local sample label generation is as per Section 2 of Training Document T-ALL-IT-005 *Epic Pro: Login*

12.2.3. Attach the sample labels to the appropriate sample bottles.

12.2.3.1. Sample Management must make sure all sample ID's match COC, Samples are staged on the counter in rows by Sample ID. All sample containers are recorded on COC.

12.2.3.2. Sample labels are printed in Sample Management. One label is printed per sample container.

12.2.3.3. The label is place onto the side of the container

12.2.4. The project manager must review and verify the following information by comparing the COC to SRF. Some of this information may not be provided by the client and those fields should be left blank:

12.2.4.1. Report Recipient;

12.2.4.2. Invoice Recipient;

12.2.4.3. Additional Report Recipient;

12.2.4.4. PO#;

12.2.4.5. Project Name;

12.2.4.6. Project Number;

12.2.4.7. Requested Due Date;

12.2.4.8. Sample ID;

12.2.4.9. Matrix;

12.2.4.10. Collection Date & Time;

12.2.4.11. Received Date & Time;

12.2.4.12. Analysis: Double check compound lists;

12.2.4.13. Price;

12.2.4.14. Region Codes;

12.2.4.15. Work Region % Split (for Pace internal subcontracted work).

12.2.5. If any samples require analyses performed outside of the laboratory, prepare the samples for subcontracting according to the procedures listed in the SOP describing the subcontracting of analytical services, S-GB-C-009 **Subcontracting Samples**, or equivalent revision or replacement.

12.3. **Sample Storage**

12.3.1. While awaiting login on the day received, samples may remain in the shipping cooler as received prior to login. Once unpacked, samples will be logged into the LIMS in a timely manner and returned to appropriate storage conditions as soon as possible. For the exceptional case where samples are not logged in the day they were received, they must be stored under appropriate temperature-controlled conditions until login takes place. In all cases, the same temperatures must be taken as soon after receipt as possible and the samples stored so as to maintain the required storage conditions while awaiting log-in.

12.3.2. Once logged into the LIMS and labeled, samples are placed in the appropriate storage areas. Specific temperature requirements are outlined in the analytical methods, but general guidelines are outlined below:

12.3.2.1. Short hold samples are placed in the short hold storage area or delivered directly to the laboratory.

12.3.2.2. Biological tissue samples are staged by receiving date or project number on shelves in a freezer for all types of analyses.

12.3.2.3. Summa canisters and Tedlar bags are stored on designated shelving at ambient temperature.

12.3.2.4. Volatiles- Aqueous samples are stored by project number in a segregated volatiles cooler. Associated trip blanks are stored with the samples.

12.3.2.5. Volatiles- Soil and other solid samples received preserved in methanol are stored by project number in a segregated volatile cooler. Associated trip blanks are stored with the samples.

12.3.2.6. Volatiles – Soil and other solid samples received preserved with sodium bisulfate and a stir bar, are stored by project number in a segregated volatiles cooler. Associated trip blanks are stored with samples.

12.3.2.7. Volatiles- Soil and other solid samples received preserved with deionized water and a stir bar, are stored by project number in a segregated volatiles freezer. Associated trip blanks are stored with samples.

12.3.2.8. Volatiles- Soil and other solid samples received in 4oz containers or similar bottleware must be preserved within 48 hours. In order to preserve these samples, it is necessary to collect a 5g aliquot of the sample and transfer it to a 40mL vial. One of the following preservation options must be utilized:

12.3.2.8.1. The 5g aliquot is preserved with 5mL of deionized water and a stir bar and stored in a freezer until analysis, or;

12.3.2.8.2. The 5g aliquot is preserved with 5mL of sodium bisulfate and a stir bar and stored in a cooler until analysis, or

12.3.2.8.3. Within 48 hours of collection in the field, the 5g aliquot must be immediately extracted with 5mL of methanol and stored in a segregated volatiles cooler until analysis, or;

12.3.2.8.4. Within 48 hours of collection in the field, the 5g aliquot can be preserved with 5mL of deionized water and a stir bar, stored in a segregated volatile cooler and analyzed within 48 hours of collection.

12.3.2.8.5. Within 48 hours of collection in the field with 10mL aliquot can be preserved with 10mL of methanol, stored in a segregated volatile cooler and analyzed within 14 days of collection.

12.3.2.9. Volatiles- Soil and other solid samples received in Encore samplers should be managed within 48 hours of collection by freezing the Encore or extruding it into a 40mL VOA vial, 2 oz jar or 4 jar with tare weights.

12.3.2.9.1. If extruding the sample into a 40mL vial containing a stir bar or a stir bar and 5mL of deionized water, then the sample is stored in the segregated volatile freezer until analysis.

12.3.2.9.2. If extruding the sample into a 40mL vial containing a stir bar and 5mL of sodium bisulfate, then the sample is stored in the segregated volatile cooler until analysis.

12.3.2.9.3. If extruding the sample into methanol, then the sample is preserved within 48 hours of collection and the sample is stored in a segregated volatile cooler until analysis. If sample is 5g Encore, extract into 5mL MeOH: if 25g, extract into 20mL of MeOH 2 or 4 oz jar (tare weight).

12.3.2.9.4. NOTE: if samples are not received within 48 hours of collection or are not received with enough time to process the samples correctly within 48 hours of collection, this must be noted in a way that will be visible on the final report (e.g., footnote in LIMS).

12.3.2.10. General Chemistry/Semi-volatiles- Waters and other liquid samples are staged by project number on the shelves in the appropriate sample storage cooler.

12.3.2.11. General Chemistry/Semi-volatiles- Soils and other solid samples are staged by project number on the shelves in the appropriate sample storage cooler.

12.3.2.12. Metals Solids and Liquids: These samples are staged by project number on designated shelving in the laboratory or appropriate designated area. These samples may be stored at ambient temperature unless Hexavalent Chromium analysis is needed. If Hexavalent Chromium analysis will be performed, the samples are staged by project number in the appropriate sample storage cooler. Samples requiring low level mercury analysis by Method 1631 are taken to the clean room for preservation and ambient storage.

12.3.2.13. Samples Containing Free Product need to be stored in a separate Free Product cooler.

12.4. **Sample Retention and Disposal**

12.4.1. If samples must be returned to customers, the lab must take special care to ensure that the samples are not damaged during any handling, testing, storing, or transporting processes.

12.4.2. Samples may need to be retained longer than the normal sample retention time. Reasons for this extended sample retention include: customer requests due to the possibility of future tests or confirmations; safety concerns prior to disposal; or contract requirements so that samples can be retained in a secure location for the customers.

12.4.3. Disposal of Unconsumed Samples: Refer to the laboratory standard operating procedure for waste handling and disposal S-GB-S-001 *Regulated Soil Handling (Green Bay Location)* and S-GB-W-001 *Waste Handling and Management.*

13. Quality Control

13.1. For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by project management and advised of the situation. For any sample received at the laboratory that does not meet the sample acceptance, hold time or preservation criteria, the client must be contacted by project management and advised of the situation.

13.1.1. If the client instructs the laboratory to proceed with the analysis, all appropriate personnel/departments must be informed and the client approval must be documented on the SCURF or COC. Data will be appropriately qualified.

13.1.2. The client may also instruct the laboratory to preserve the samples at the laboratory prior to proceeding with analysis. This must be documented on the COC or the SCURF, and should be noted in the final laboratory report.

13.2. All supporting documentation related to sample custody must be retained by the laboratory. This includes; memorandums, fax transmissions, all paperwork received with the COC and copies of email transmissions.

13.3. Documenting Discrepancies during receipt of samples:

13.3.1. The following are examples of client discrepancies that need to be documented on the appropriate paperwork (e.g, SCURF):

13.3.1.1. Lost samples/insufficient sample volume;

13.3.1.2. Broken or missing bottles;

13.3.1.3. Missing COC;

13.3.1.4. Mislabeled bottles;

13.3.1.5. Preservation error;

13.3.1.6. Missing sample related details (date, time, sample type).

13.3.2. Pace sample management discrepancies will be documented on the SCURF, COC or within the project files. Discrepancies attributable to errors and omissions on the part of the laboratory will be addressed and resolved through the formal corrective action process.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Hazards and Precautions - Use extreme caution in handling samples and wastes as they may be hazardous. Each reagent and chemical used in this method should be treated as a potential health hazard.

Reduce exposure by the use of gloves, lab coats, safety glasses, and ventilation hoods. Safety Data Sheets (SDS) are on file and available to all personnel.

22.2. All personnel involved in sample management are responsible for complying with OSHA and DOT regulations. These regulations pertain to the safe handling and/or shipping of the chemicals specified in this procedure. Refer to the Sample Control Supervisor for any questions or concerns related to the safe handling and shipment of hazardous materials.

22.3. Other laboratory safety requirements are contained in the Chemical Hygiene Plan/Safety Manual. Immediate questions can also be addressed with the local Safety Officer.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. SW-846, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA, current revision.

25.5. American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 1995, Standard Methods for the Examination of Water and Wastewater, A.E. Greenberg, L.W. Clesceri, A.D. Eaton and M.A.H. Franson, eds., 19th ed., American Public Health Association, Washington D.C.

25.6. U.S. Environmental Protection Agency, 1983, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

25.7. U.S. Environmental Protection Agency, 1988, Methods for Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio.

25.8. Code of Federal Regulations- most recent version.

25.9. Method Update Rule- most recent version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Chain of Custody, Form F-ALL-Q-020.

26.2. Attachment II – Sample Condition Upon Receipt Form, Form F-ALL-C-003.

26.3. Attachment III – Sample Receipt Form (SRF), example.

26.4. Attachment IV – Pace Approved Sample Containers, Preservation.

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-C-001-rev.03	Section 3.4: revised the minimum sample retention time to match the Quality Manual. Section 11.1.12: added section to be compliant with 2010 DoD QSM gray box 41. Section 11.1.2.4: added section with language from 2009 TNI standard (V1M2, section 5.8/ISO 5.8.4 note 2. Section 11.1.2.5: added red-letter section for lab-specific courier requirements. Section 11.2.1.1: added language to be compliant with 2009 TNI standard (V1M2, sections 5.8.5.a and b). Section 11.4: changed to "Sample Retention and Disposal" and moved original section 11.4 for disposal to subsection 11.4.3. Section 11.4.1: added subsection for sample return and added language from 2009 TNI standard (V1M2 5.8/ISO 5.8.4 note 1). Section 11.4.2: added subsection for sample retention and added language from 2009 TNI standard (V1M2 5.8/ISO 5.8.4 note 3). Section 15.4: added DoD QSM reference. Attachment IV: revised to match Quality Manual.	11Jan2012
S-GB-C-010-Rev.03	Throughout document: Added Pace – GB specific information.	08Oct2012
S-GB-C-010-Rev.04	Section 11.1.5.6: Updated Tissue receipt temperature to ≤6°C Section 11.3: Updated hold time for HPC	04Dec2012
SOT-ALL-C-001-rev.04	General: changed to new format. Sections 8.5, 22.1: changed MSDS to SDS based on updated Hazard Communication Standard Table 10.1: added sodium bisulfate and aaa-TFT. Table 12.2: added more examples in red text. Section 12.2.6 and 12.2.7: added two new sections in red text. Added new sections 12.1.4.1, 12.4, and 12.5 in red text regarding internal COC procedures. Section 25.10: added Method Update Rule to references. Attachment IV: changed hold times listed as '6 months' to '180 Days' to match actual practice. Added information for LL Mercury soils, pH soils, ORO, and saturated hydrocarbons	13Feb2013
S-GB-C-010-Rev.05	Throughout document: Added Pace – GB specific information	06May2013

Attachment I – Example Chain-of-Custody Form

Pace Analytical
www.pacelabs.com

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:
Company:	Report To:	Attention:
Address:	Copy To:	Company Name:
		Address:
Email To:	Purchase Order No.:	Pace Quote Reference:
Phone: Fax:	Project Name:	Pace Project Manager:
Requested Due Date/TAT:	Project Number:	Pace Profile #:

Page: of

REGULATORY AGENCY: ☐ NPDES ☐ GROUND WATER ☐ DRINKING WATER ☐ UST ☐ RCRA ☐ OTHER _____

Site Location: STATE: _____

Section D Required Client Information

Valid Matrix Codes

MATRIX	CODE
Drinking Water	DW
Water	WT
Waste Water	WW
Product	P
Soil/Solid	SL
Oil	OL
Wipe	WP
Air	AR
Other	OT
Tissue	TS

SAMPLE ID (A-Z, 0-9 / ,-) Sample IDs MUST BE UNIQUE

ITEM #	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED COMPOSITE START DATE	COLLECTED COMPOSITE START TIME	COLLECTED COMPOSITE END/GRAB DATE	COLLECTED COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test (Y/N)	Requested Analysis Filtered (Y/N)	Residual Chlorine (Y/N)	Pace Project No./Lab I.D.
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS

SAMPLER NAME AND SIGNATURE:

PRINT Name of SAMPLER:

SIGNATURE of SAMPLER: DATE Signed (MM/DD/YY):

Temp in °C | Received on Ice (Y/N) | Custody Sealed Cooler (Y/N) | Samples Intact (Y/N)

*Important Note: By signing this form you are accepting Pace's NET 30 day payment terms and agreeing to late charges of 1.5% per month for any invoices not paid within 30 days.

F-ALL-Q-020rev.08, 12-Oct-2007

Attachment II – Example Sample Condition Upon Request Form

Pace Analytical Services, Inc.
1241 Bellevue Street, Suite 9
Green Bay, WI 54302

Pace Analytical™

Sample Condition Upon Receipt

Client Name: _____ Project # _____

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client ☐ Commercial ☐ Pace Other _____

Tracking #: _____

Custody Seal on Cooler/Box Present: ☐ yes ☐ no **Seals intact:** ☐ yes ☐ no

Custody Seal on Samples Present: ☐ yes ☐ no **Seals intact:** ☐ yes ☐ no

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☐ None ☐ Other _____

Thermometer Used _____ **Type of Ice:** Wet Blue Dry None ☐ Samples on ice, cooling process has begun

Cooler Temperature Uncorr: _____ /Corr: _____ **Biological Tissue is Frozen:** ☐ yes ☐ no

Temp Blank Present: ☐ yes ☐ no

Person examining contents:
Date: _____
Initials: _____

Temp should be above freezing to 6°C for all sample except Biota.
Frozen Biota Samples should be received ≤ 0°C.

				Comments:
Chain of Custody Present:	☐Yes	☐No	☐N/A	1.
Chain of Custody Filled Out:	☐Yes	☐No	☐N/A	2.
Chain of Custody Relinquished:	☐Yes	☐No	☐N/A	3.
Sampler Name & Signature on COC:	☐Yes	☐No	☐N/A	4.
Samples Arrived within Hold Time:	☐Yes	☐No	☐N/A	5.
- VOA Samples frozen upon receipt	☐Yes	☐No		Date/Time:
Short Hold Time Analysis (<72hr):	☐Yes	☐No	☐N/A	6.
Rush Turn Around Time Requested:	☐Yes	☐No	☐N/A	7.
Sufficient Volume:	☐Yes	☐No	☐N/A	8.
Correct Containers Used:	☐Yes	☐No	☐N/A	9.
-Pace Containers Used:	☐Yes	☐No	☐N/A	
-Pace IR Containers Used:	☐Yes	☐No	☐N/A	
Containers Intact:	☐Yes	☐No	☐N/A	10.
Filtered volume received for Dissolved tests	☐Yes	☐No	☐N/A	11.
Sample Labels match COC:	☐Yes	☐No	☐N/A	12.
-Includes date/time/ID/Analysis Matrix:				
All containers needing preservation have been checked. (Non-Compliance noted in 13.)	☐Yes	☐No	☐N/A	13. ☐ HNO3 ☐ H2SO4 ☐ NaOH ☐ NaOH +ZnAct
All containers needing preservation are found to be in compliance with EPA recommendation. (HNO3, H2SO4 ≤2; NaOH+ZnAct ≥9, NaOH ≥12)	☐Yes	☐No	☐N/A	
exceptions: VOA, coliform, TOC, TOX, TOH, O&G, WIDROW, Phenolics, OTHER:	☐Yes	☐No		Initial when completed / Lab Std #ID of preservative / Date/Time:
Headspace in VOA Vials (>6mm):	☐Yes	☐No	☐N/A	14.
Trip Blank Present:	☐Yes	☐No	☐N/A	15.
Trip Blank Custody Seals Present	☐Yes	☐No	☐N/A	
Pace Trip Blank Lot # (if purchased): _____				

Client Notification/ Resolution: If checked, see attached form for additional comments ☐

Person Contacted: _____ **Date/Time:** _____

Comments/ Resolution: _____

Project Manager Review: _____ **Date:** _____

Pace Analytical Services, Inc.
1241 Bellevue Street, Suite 9
Green Bay, WI 54302

Sample Condition Upon Receipt

Client Name: _____ Project # _____

Additional Comments/Resolution: _____

Project Manager Review: _____ Date: _____

Attachment III – Example Sample Receipt Form

Sample Receipt Form

Pace Analytical Services, Inc.
Indiana

Pace Analytical
www.pacelabs.com

Sample Acknowledgement Recipients:

Email:

Bill to:

Final Report Recipients:

Line Item Descriptions:
[4] Water Short VOC

Client P O No:
Phone: 1(317)875-5894
Project Manager: Phaedra Ducksworth
Client Project ID:

Lab Project No: 5018664
Project Deliverables Type: Standard Report
Project Report Due Date: 09/25/08
Profile: 907

Lab Smp ID: 5018664001 Client Smp ID: RW-3:G080908 Collected Date: 09/09/08 11:25
Proj Smp No: 1 Matrix: Water Smp Type: PS Line Item: 4 Received Date: 09/11/08 10:18
Location: OH BUSTR

Auxiliary Data:

Consultant Project Number:	NA	ENFOS Project (AAAAA-0000):	
Site ID/Facility Number:		Phase:	01
Gem Portfolio:	CENTRAL	SubPhase/Task:	03
State of Sample Collection:	OH	Cost Element:	05
Consultant Name:	URS	RCOP:	no
Rush Charges:	0		

PARAMETER	METHOD	UNIT PRICE	WR	SPL	%
8260 WUST - 8260 MSV UST	EPA 8260	$27.00			

COMPOUND	PQL	UNITS
Benzene	5	ug/L
Ethylbenzene	5	ug/L
Methyl-tert-butyl ether	4	ug/L
Toluene	5	ug/L
Xylene (Total)	10	ug/L

Sub Total - Sample 208364 $27.00

Lab Smp ID: 5018664002 Client Smp ID: RW-4:G080908 Collected Date: 09/09/08 11:00
Proj Smp No: 2 Matrix: Water Smp Type: PS Line Item: 4 Received Date: 09/11/08 10:19
Location: OH BUSTR

PARAMETER	METHOD	UNIT PRICE	WR	SPL	%
8260 WUST - 8260 MSV UST	EPA 8260	$27.00			

COMPOUND	PQL	UNITS
Benzene	5	ug/L
Ethylbenzene	5	ug/L
Methyl-tert-butyl ether	4	ug/L
Toluene	5	ug/L
Xylene (Total)	10	ug/L

Attachment IV – Pace Analytical Approved Sample Containers, Preservation

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Acidity	SM2310B	Water	Plastic/Glass	≤ 6°C	14 Days
Actinides	HASL-300	Water		pH<2 HNO_3	180 Days
Actinides	HASL-300	Solid		None	180 Days
Alkalinity	SM2320B/310.2	Water	Plastic/Glass	≤ 6°C	14 Days
Alkylated PAHs		Water		≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved
Alkylated PAHs		Solid		≤ 10°C	1 Year/40 Days
Total Alpha Radium (see note 3)	9315/903.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Total Alpha Radium (see note 3)	9315	Solid		None	180 days
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0/300.1/SM4110B	Water	Plastic/Glass	≤ 6°C; EDA if bromate or chlorite run	All analytes 28 days except: NO_2, NO_3, o-Phos (48 Hours); chlorite (immediately for 300.0; 14 Days for 300.1). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4, bromate, chlorite, chlorate)	300.0	Solid	Plastic/Glass	≤ 6°C	All analytes 28 days except: NO_2, NO_3, o-Phos (48 hours); chlorite (immediately). NO_2/NO_3 combo 28 days.
Anions (Br, Cl, F, NO_2, NO_3, o-Phos, SO_4	9056	Water/ Solid	Plastic/Glass	≤ 6°C	28 days
Aromatic and Halogenated Volatiles (see note 1)	8021	Solid	5035 vial kit	See note 1	14 days
Aromatic and Halogenated Volatiles	602/8021	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days (7 Days for aromatics if unpreserved)
Acid Volatile Sulfide	Draft EPA 1629	Solid	8oz Glass	≤ 6°C	14 Days
Bacteria, Total Plate Count	SM9221D	Water	Plastic/WK	≤ 6°C; $Na_2S_2O_3$	24 Hours
Base/Neutrals and Acids	8270	Solid	8oz Glass	≤ 6°C	14/40 Days
Base/Neutrals and Acids	625/8270	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Base/Neutrals, Acids & esticides	525.2	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C; Na sulfite if Cl present	14/30 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Biomarkers		Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Biomarkers		Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
BOD/cBOD	SM5210B	Water	Plastic/Glass	≤ 6°C	48 hours
BTEX/Total Hydrocarbons	TO-3	Air	Summa Canister	None	14 Days
BTEX/Total Hydrocarbons	TO-3	Air	Tedlar Bag or equivalent	None	48 Hours
Cation/Anion Balance	SM1030E	Water	Plastic/Glass	None	None
Cation Exchange	9081	Solid	8oz Glass	None	unknown
Chloride	SM4500Cl-C,E	Water	Plastic/Glass	None	28 Days
Chlorine, Residual	SM4500Cl-D,E,G/330.5/Hach 8167	Water	Plastic/Glass	None	15 minutes
Chlorophyll	SM10200H	Water	Opaque bottle or aluminum foil		
COD	SM5220C, D/410.4/Hach 8000	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Coliform, Fecal	SM9222D	Water	100mL Plastic	≤ 6°C	6 Hours
Coliform, Fecal	SM9222D	Solid	100mL Plastic	≤ 6°C	6 Hours
Coliform, Total and Escherichla (E. coli)	SM9223B	Water	100mL Plastic	≤ 10°C	48 Hours after collection; results from samples analyzed 30-48 Hours after collection must be qualified as analyzed >30 hours
Color	SM2120B,E	Water	Covered Plastic/Acid Washed Amber Glass	≤ 6°C	24 Hours
Condensable Particulate Emissions	EPA 202	Air	Solutions	None	6 Months
Cyanide, Reactive	SW846 chap.7	Water	Plastic/Glass	None	28 Days
Cyanide, Reactive	SW846 chap.7	Solid	Plastic/Glass	None	28 Days
Cyanide, Total and Amenable	SM4500CN-A,B,C,D,E,G,I,N/9010/9012/335.4	Water	Plastic/Glass	pH≥12 NaOH; ≤ 6°C; ascorbic acid if Cl present	14 Days (24 Hours if sulfide present-applies to SM4500CN only)
Diesel Range Organics- Alaska	AK102	Solid	8oz Glass	≤ 6°C	14/40 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
DRO					
Diesel Range Organics- Alaska DRO	AK102	Water	1L Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- TPH DRO	8015	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Diesel Range Organics- TPH DRO	8015	Tissue	1L Amber Glass	≤ - 10°C	1 Year if frozen/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Diesel Range Organics- NwTPH-Dx	Nw-TPH-Dx	Water	1L Amber Glass	pH <2 HCl; ≤ 6°C	14/40 Days; 7 Days from collection to extraction if unpreserved
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Solid	Tared 4oz Glass Jar	≤ 6°C	10/47 Days
Diesel Range Organics- Wisconsin DRO	WI MOD DRO	Water	1L Amber Glass	≤ 6°C	14/40 Days
Dioxins and Furans	1613B	Solid	8oz Glass	≤ -10°C	1 year
Dioxins and Furans	1613B	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 year
Dioxins and Furans	1613B	Fish/ Tissue	Aluminum foil	< -10°C	1 year
Dioxins and Furans	8290	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	30/45 Days
Dioxins and Furans	8290	Solid	8oz Glass	≤ 6°C	30/45 Days
Dioxins and Furans	8290	Fish/ Tissue	Not specified	< -10°C	30/45 Days
Dioxins and Furans	TO-9	Air	PUF	None	30/45 Days
EDB/DBCP (8011) EDB/DBCP/1,2,3-TCP (504.1)	504.1/8011	Water	40mL vials	≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Explosives	8330/8332	Water	1L Amber Glass	≤ 6°C	7/40 Days
Explosives	8330/8332	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Water	1L Amber Glass	pH<2 HCl; ≤ 6°C	14/40 Days
Extractable Petroleum Hydrocarbons (aliphatic and aromatic)	MA-EPH	Solid	4oz Glass Jar	≤ 6°C	7/40 Days
Ferrous Iron	SN3500Fe-D	Water	Glass	None	Immediate
lashpoint/Ignitability	1010	Liquid	Plastic/Glass	None	28 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Fluoride	SM4500Fl-C,D	Water	Plastic	None	28 Days
Gamma Emitting Radionuclides	901.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Gasoline Range Organics	8015	Water	40mL vials	pH<2 HCl	14 Days
Gasoline Range Organics	8015	Solid	5035 vial kit	See note 1	14 days
Gasoline Range Organics-Alaska GRO	AK101	Solid	5035 vial kit	See 5035 note*	28 Days if GRO only (14 Days with BTEX)
Gasoline Range Organics-Alaska GRO	AK101	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Water	40mL vials	pH<2 HCl; ≤ 6°C	7 Days unpreserved; 14 Days preserved
Gasoline Range Organics-NwTPH-Gx	Nw-TPH-Gx	Solid	40mL vials	≤ 6°C; packed jars with no headspace	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days
Gasoline Range Organics-Wisconsin GRO	WI MOD GRO	Solid	40mL MeOH vials	≤ 6°C in MeOH	21 Days
Gross Alpha (NJ 48Hr Method)	NJAC 7:18-6	Water	Plastic/Glass	pH<2 HNO_3	48 Hrs
Gross Alpha and Gross Beta	9310/900.0	Water	Plastic/Glass	pH<2 HNO_3	180 Days
Gross Alpha and Gross Beta	9310	Solid	Glass	None	180 Days
Haloacetic Acids	552.1/552.2	Water	40mL Amber vials	NH_4Cl; ≤ 6°C	14/7 Days if extracts stored ≤ 6°C or 14/14 Days if extracts stored at ≤ -10°C
Hardness, Total ($CaCO_3$)	SM2340B,C/130.1	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Heterotrophic Plate Count (MPC)	SM9215B	Water	100mL Plastic	≤ 6°C	24 Hours
Herbicides, Chlorinated	8151	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Herbicides, Chlorinated	8151	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Herbicides, Chlorinated	515.1/515.3	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	14/28 Days
Hexavalent Chromium	7196/218.6/SM3500Cr-C,D	Water	Plastic/Glass	≤ 6°C	24 Hours
Hexavalent Chromium	7196 (with 3060A)	Solid		≤ 6°C	24 Hours after extraction
Hydrogen Halide and Halogen Emissions	EPA 26	Air	Solutions	None	6 Months
Ignitability of Solids	1030	Non-liquid Waste	Plastic/Glass	None	28 Days
ead Emissions	EPA 12	Air	Filter/Solutions	None	6 Months

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Lipids	Pace Lipids	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen
Mercury, Low-Level	1631E	Solid	Glass (wide-mouth)	≤ 6°C	28 Days; 1 Year if frozen
Mercury, Low-Level	1631E	Water	Fluoropolymer bottles (Glass if Hg is only analyte being tested)	12N HCl or BrCl	48 Hours for preservation or analysis; 28 Days to preservation if sample oxidized in bottle; 90 Days for analysis if preserved
Mercury, Low-Level	1631E	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Mercury	7471	Solid	8oz Glass Jar	≤ 6°C	28 days
Mercury	7470/245.1/245.2	Water	Plastic/Glass	pH<2 HNO_3	28 Days
Mercury	7471/245.6	Tissue	Plastic/Glass	≤ - 10°C	28 Days if frozen
Metals (GFAA)	7000/200.9	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Metals (ICP)	NIOSH 7300A/7303	Air	Filters	None	6 Months
Metals (ICP/ICPMS)	6010/6020	Solid	8oz Glass Jar	None	6 months
Metals (ICP/ICPMS)	6010/6020/200.7/200.8	Water	Plastic/Glass	pH<2 HNO_3	6 Months
Metals (ICP/ICPMS)	6020	Tissue	Plastic/Glass	≤ -10°C	6 Months if frozen
Methane, Ethane, Ethene	8015 modified	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	RSK-175	Water	40mL vials	HCl	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Summa Canister	None	14 Days
Methane, Ethane, Ethene	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
Methanol, Ethanol	8015 modified	Water	40mL vials	≤ 6°C	14 Days
Methanol, Ethanol	8015 modified	Solid	2oz Glass	≤ 6°C	14 Days
Nitrogen, Ammonia	SM4500NH3/350.1	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	351.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Kjeldahl (TKN)	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrate	SM4500-NO3/352.1	Water	Plastic/Glass	≤ 6°C	24 Hours preferred
Nitrogen, Nitrate & Nitrite combination	353.2	Solid	Plastic/Glass	≤ 6°C	28 Days
Nitrogen, Nitrate & Nitrite combination	SM4500-NO3/353.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Nitrogen, Nitrite or Nitrate separately	SM4500-NO2/353.2	Water	Plastic/Glass	≤ 6°C	48 Hours
Nitrogen, Organic	SM4500-Norg/351.2	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Non-Methane Organics	EPA 25C	Air	Summa Canister	None	14 Days
Non-Methane Organics	EPA 25C	Air	Tedlar Bag or equivalent	None	48 Hours

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Odor	SM2150B	Water	Glass	≤ 6°C	24 Hours
Oil and Grease/HEM	1664A/SM5520B/9070	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Oil and Grease/HEM	9071	Solid	Glass	≤ 6°C	28 Days
PBDEs	1614	Water	1L Amber Glass	≤ 6°C	1 Year/1 Year
PBDEs	1614	Solid	Wide Mouth Jar	≤ 6°C	1 Year/1 Year
PBDEs	1614	Tissue	Aluminum Foil	≤ -10°C	1 Year/1 Year
PCBs and Pesticides, Organochlorine (OC)	TO-4/TO-10	Air	PUF	None	7/40 Days
PCBs and Pesticides, Organochlorine (OC)	608	Water	1L Amber Glass		Pest: 7/40 Days; PCB: 1 Year/1 Year
Pesticides, Organochlorine (OC)	8081	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Pesticides, Organochlorine (OC)	8081	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organochlorine (OC)	8081	Tissue	8oz Glass Jar	≤ -10°C	1 Year if frozen/40 Days
Pesticides, Organophosphorous (OP)	8141	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Pesticides, Organophosphorous (OP)	8141	Water	1L Amber Glass	pH 5-8 with NaOH or H_2SO_4; ≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
PCBs (Aroclors)	8082	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	1 Year/1 Year
PCBs (Aroclors)	8082	Solid	8oz Glass Jar	≤ 6°C	1 Year/1 Year
PCBs (Aroclors)	8082	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/1 Year
PCB Congeners	1668A	Water	1L Amber Glass	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Solid	4-8oz Glass Jar	≤ 6°C but above freezing	1 Year/1 Year
PCB Congeners	1668A	Tissue	4-8oz Glass Jar	≤ -10°C	1 Year/1 Year
Oil Range Organics- ORO		Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Oil Range Organics- ORO		Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Oxygen, Dissolved (Probe)	SM4500-O	Water	Glass	None	15 minutes
Paint Filter Liquid Test	9095	Water	Plastic/Glass	None	N/A
Particulates	PM-10	Air	Filters	None	6 Months
ermanent Gases	EPA 3C	Air	Summa	None	14 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
			Canister		
Permanent Gases	EPA 3C	Air	Tedlar Bag or equivalent	None	48 Hours
pH	SM4500H+B/9040	Water	Plastic/Glass	None	15 minutes
pH	9045	Solid	Plastic/Glass	None	Contact local lab
Phenol, Total	420.1/420.4/9065/9066	Water	Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Orthophosphate	SM4500P/365.1/365.3	Water	Plastic	Filter; ≤ 6°C	Filter within 15 minutes, Analyze within 48 Hours
Phosphorus, Total	SM4500P/ 365.1/365.3/365.4	Water	Plastic/Glass	pH<2 H_2SO_4; ≤ 6°C	28 Days
Phosphorus, Total	365.4	Solid	Plastic/Glass	≤ 6°C	28 Days
Polynuclear Aromatic Hydrocarbons (PAH)	TO-13	Air	PUF	None	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Solid	8oz Glass Jar	≤ 6°C	14/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Water	1L Amber Glass	≤ 6°C; $Na_2S_2O_3$ if Cl present	7/40 Days
Polynuclear Aromatic Hydrocarbons (PAH)	8270 SIM	Tissue	Plastic/Glass	≤ -10°C	1 Year if frozen/40 Days
Radioactive Strontium	905.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-226	903.0/903.1	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320/904.0	Water	Plastic/Glass	pH<2 HNO_3	180 days
Radium-228 (see note 3)	9320	Solid			
Residual Range Organics-Alaska RRO	AK103	Solid	8oz Glass	≤ 6°C	14/40 Days
Saturated Hydrocarbons	SOP S-MN-O-567	Water	≤ 6°C; pH<2 1:1 HCl (optional)	14/40 Days preserved; 7/40 Days unpreserved	≤ 6°C; pH<2 1:1 HCl (optional)
Saturated Hydrocarbons	SOP S-MN-O-567	Solid	≤ 10°C	1 Year/40 Days	≤ 10°C
Silica, Dissolved	SM4500Si-D	Water	Plastic	≤ 6°C	28 Days
Solids, Settleable	SM2540F	Water	Glass	≤ 6°C	48 Hours
Solids, Total	SM2540B	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total	SM2540G	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total (FOC, OM, Ash)	ASTM D2974	Solid	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Dissolved	SM2540C	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Suspended	SM2540D/USGS I-3765-85	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4/SM2540E	Water	Plastic/Glass	≤ 6°C	7 Days
Solids, Total Volatile	160.4	Solid	Plastic/Glass	≤ 6°C	7 Days
Specific Conductance	SM2510B/9050/120.1	Water	Plastic/Glass	≤ 6°C	28 Days
Stationary Source Dioxins and	EPA 23	Air	XAD Trap	None	30/45 Days

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
Furans					
Stationary Source Mercury	EPA 101	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source Metals	EPA 29	Air	Filters	None	6 Months, 28 Days for Hg
Stationary Source PM10	EPA 201A	Air	Filters	None	6 Months
Stationary Source Particulates	EPA 5	Air	Filter/Solutions	None	6 Months
Sulfate	SM4500SO4/9036/ 9038/375.2/ASTM D516	Water	Plastic/Glass	≤ 6°C	28 Days
Sulfide, Reactive	SW-846 Chap.7	Water	Plastic/Glass	None	28 Days
Sulfide, Reactive	SW-846 Chap.7	Solid	Plastic/Glass	None	28 Days
Sulfide, Total	SM4500S/9030	Water	Plastic/Glass	pH>9 NaOH; ZnOAc; ≤ 6°C	7 Days
Sulfite	SM4500SO3	Water	Plastic/Glass	None	15 minutes
Surfactants (MBAS)	SM5540C	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Organic Carbon (TOC)	SM5310B,C,D/9060	Water	Glass	pH<2 H_2SO_4 or HCl; ≤ 6°C	28 Days
Total Organic Carbon (TOC)	9060/Walkley Black	Solid	Glass	≤ 6°C	14 Days
Total Organic Halogen (TOX)	SM5320/9020/9021	Water	Glass; no headspace	≤ 6°C	14 Days
Tritium	906.0	Water	Glass	None	180 days
Turbidity	SM2130B/180.1	Water	Plastic/Glass	≤ 6°C	48 Hours
Total Uranium	908.0/ASTM D5174-97	Water	Plastic/Glass	pH<2 HCl	180 days
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Water	40mL vials	pH<2 HCl; ≤ 6°C	14 Days preserved
Volatile Petroleum Hydrocarbons (aliphatic and aromatic)	MA-VPH	Solid	4-8oz Glass Jar	≤ 6°C; packed jars with no headspace	7/28 Days
Volatiles	TO-14	Air	Summa Canister	None	30 Days
Volatiles	TO-14	Air	Tedlar Bag or equivalent	None	48 Hours
Volatiles	TO-15	Air	Summa Canister	None	30 Days
Volatiles	8260	Solid	5035 vial kit	See note 1	14 days
Volatiles	8260	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$ if Cl present	14 Days
Volatiles	8260	Conc. Waste	5035 vial kit or 40mL vials	≤ 6°C	14 Days
Volatiles	624	Water	40mL vials	pH<2 HCl; ≤ 6°C; $Na_2S_2O_3$	14 Days (7 Days for aromatics if

Parameter	Method	Matrix	Container	Preservative	Max Hold Time
				if Cl present	unpreserved)
Volatiles (see note 2)	524.2	Water	40mL vials (in duplicate)	pH<2 HCl; ≤ 6°C; Ascorbic acid or $Na_2S_2O_3$ if Cl present[2]	14 Days

[1] **5035/5035A Note**: 5035 vial kit typically contains 2 vials water, preserved by freezing **or**, 2 vials aqueous sodium bisulfate preserved at 4°C, **and** one vial methanol preserved at ≤6°C **and** one container of unpreserved sample stored at ≤6°C.

[2] Method 524.2 lists ascorbic acid as the preservative when residual chlorine is suspected, unless gases or Table 7 compounds are NOT compounds of interest and then sodium thiosulfate is the preservative recommended.

[3] Methods 9315 and 9320 both state that if samples are unpreserved, the samples should be brought to the lab within 5 days of collection, preserved in the lab, and then allowed to sit for a minimum of 16 hours before sample preparation/analysis.

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STANDARD OPERATING PROCEDURE

DETERMINATION OF SEMI-VOLATILE ORGANICS BY GC/MS

Reference Methods: EPA SW-846 Method 8270C / EPA 625

Local SOP Number:	S-GB-O-049-Rev.05
Effective Date:	Date of Final Signature
Supersedes:	S-GB-O-049-Rev.04
SOP Template Number:	SOT-ALL-O-001-rev.01

APPROVALS

Nils Melberg, Laboratory General Manager — Date: 06/27/13

Kate Grams, Laboratory Quality Manager — Date: 6/27/13

Chris Haase, Laboratory Department Manager — Date: 06/27/13

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION	PAGE
1. Purpose/Identification of Method	3
2. Summary of Method	3
3. Scope and Application	3
4. Applicable Matrices	4
5. Limits of Detection and Quantitation	4
6. Interferences	4
7. Sample Collection, Preservation, Shipment and Storage	4
8. Definitions	5
9. Equipment and Supplies (Including Computer Hardware and Software)	5
10. Reagents and Standards	6
11. Calibration and Standardization	10
12. Procedure	17
13. Quality Control	27
14. Data Analysis and Calculations	28
15. Data Assessment and Acceptance Criteria for Quality Control Measures	29
16. Corrective Actions for Out-of-Control Data	29
17. Contingencies for Handling Out-of-Control or Unacceptable Data	29
18. Method Performance	29
19. Method Modifications	29
20. Instrument/Equipment Maintenance	30
21. Troubleshooting	30
22. Safety	30
23. Waste Management	30
24. Pollution Prevention	30
25. References	30
26. Tables, Diagrams, Flowcharts, and Validation Data	31
27. Revisions	31

1. Purpose/Identification of Method

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Green Bay to determine the concentration of Semi-volatile Organic Compounds (SVOCs) in environmental samples. The laboratory utilizes GC/MS and bases these documented procedures on those listed in EPA SW-846 Method 8270C/ EPA 625. The Green Bay laboratory currently processes water samples by automated separatory funnel using Method SW846 3510C, soil samples by Microwave Extraction using Method SW846 3546 and biota samples by soxhlet extractor using Method SW846 3540C. The latest revision of Pace's SOPs S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile Analysis* (most current revision or replacement), S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic hydrocarbon, Base/Neutral/Acids, and Total Petroleum Hydrocarbons on Solid Matrices* (most current revision or replacement), and GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* (most current revision or replacement) for these extraction techniques are available from the quality office.

2. Summary of Method

2.1. Sample extracts are prepared for analysis by an appropriate sample preparation method. The semivolatile organic compounds are introduced into the gas chromatograph (GC) by injecting an aliquot of the sample extract. The GC conditions are programmed to separate the analytes. The GC effluent is directly introduced to a mass spectrometer (MS) for both identification and quantification of analytes. Analytes are identified by comparison of their mass spectra with spectra of authentic standards. Analytes are quantified by comparing the response of a selected major (quantitation) ion relative to an internal standard using a multi-point calibration curve.

3. Scope and Application

3.1. This procedure may be used to determine concentrations of neutral, acidic, and basic semivolatile organic compounds in extracts prepared from many types of water samples, soil samples and wastes. Analytes must be soluble in dichloromethane and amenable to capillary gas chromatography. Specific compound classes include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons and pesticides, phthalate esters, organophosphate esters, nitrosamines, haloethers, aldehydes, ethers, ketones, anilines, pyridines, quinolines, aromatic nitro compounds, and phenols. A list of applicable compounds is shown in Table 11.1 Calibration Standard Compound Concentrations. Pace Reporting Levels (PRLs) are also shown for water and soil samples. PRLs are subject to change based on current analytical system performance and actual sample matrices.

3.2. This method is applicable to most water and solid samples, regardless of moisture content. Common matrices are ground and surface water, wastewater, aqueous sludge, sediment, soils, and other solid samples. Procedures may need to be adapted to address limits in the method or equipment that might hinder or interference with sample analysis. All adaptations made to address matrix related modifications must be documented within the analytical data.

3.3. This procedure is restricted to use by, or under the supervision of, analysts experienced in the use of semi-volatile configured GC/MS systems and interpretation of GC/MS data. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.4. This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

4. Applicable Matrices

4.1. This SOP is applicable to soils/sediments, solid wastes, tissue, wipes and aqueous matrices.

5. Limits of Detection and Quantitation

5.1. The reporting limit (LOQ) for all analytes is listed in Table 11.1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Interferences may be introduced into sample extracts by contaminants in solvents, reagents, glassware, and any other material that comes in contact with the sample or extract during extract preparation. These interferences must be closely monitored by analyzing Method Blank samples and taking corrective action as required.

6.2. Matrix interferences may result from materials co-extracted from some samples.

6.3. Significant phthalate contamination may result at any time if consistent quality control is not practiced. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

6.4. Contamination by carryover can occur when high concentration extracts are analyzed prior to low concentration extracts. The contamination may also cause degradation of labile analytes. Whenever carryover is suspected, the affected extracts should be re-analyzed. If significant degradation of the GC/MS systems is suspected, system performances samples should be analyzed and corrective action taken as needed.

7. Sample Collection, Preservation, Shipment and Storage

7.1. **Table 7.1 – Sample Collection, Preservation, Storage, and Hold time**

Sample type	Collection per sample	Preservation	Storage	Hold time
Aqueous	One 1L amber glass Samples to be analyzed for EPA 625 must be checked for residual chlorine. If residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well.	None	4 ± 2°C	7 days
Soil/Solid (non-aqueous)	One 8oz wide glass jar	None	4 ± 2°C	14 days
Biota	-	None	≤ -10°C until extraction	1 year when frozen
TCLP	One 1L Amber Glass	None	4 ± 2°C	TCLP Leachates must be solvent extracted within 7 days of the completion process.
Extracts	Two 5mL glass vials, same as used for standard storage	None	≤ -10°C	40 days

8. Definitions

Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary.

8.1. **Toxicity Characteristic Leaching Procedure (TCLP)** – An extraction procedure used to determine if a sample is acceptable for upland disposal. The extraction procedure is meant to simulate the leaching of contaminants under the environmental conditions typically found in a landfill.

8.2. **Run Sequence Log** – A logbook that lists all injections and analyses performed on a particular piece of equipment regardless of the use of the data collected from each analysis.

8.3. **Tune Period** – The period after the DFTPP instrument tune check within which analyses may be performed.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. **Table 9.1 - Instrumentation**

Analytical Instrument/Peripherals	EPIC Pro Name	Serial Number
HP 5890 Series II GC	40MSS1	3336A57925
HP 5972 Mass Selective Detector	40MSS1	3501A02320
HP 7673 AutoSampler Tray	40MSS1	3526A39072
HP 7673 Injector	40MSS1	3009A20936
HP Controller	40MSS1	3526A02233
Alcatel 2005 Rough Pump	40MSS1	265402
HP 5890 Series II GC	40MSS5	3022A29174
HP 5972 Mass Selective Detector	40MSS5	3501A02209
HP 7673 AutoSampler Tray	40MSS5	3319A32067
HP 6890 Injector	40MSS5	DE64502673
HP Controller	40MSS5	3018A22346
Varian DS302 Rough Pump	40MSS5	244968
HP 5890E GC	40MSS6	3310A49571
HP 5972A Mass Selective Detector	40MSS6	3524A03107
HP 18596A AutoSampler Tray	40MSS6	2920A10670
HP 6890 Injector	40MSS6	US0000692
HP 7673 Controller	40MSS6	3113A25880
Edwards E2M2 Rough Pump	40MSS6	53747

9.2. **Table 9.2 - Chromatography Supplies**

Item	Vendor	Model / ID	Catalog #	Description
Analytical Column	Restek	XTI-5 w/ Integraguard	12223-124	30 m, 0.25 mm ID, 0.25 df
Analytical Column	Restek	Rxi-1 MS		20m, 0.18 mm ID, 0.18 df
Fluorocarbon O-rings	Restek		20377	
Vespel/Graphite Ferrules	Restek		20229	1/16" x 0.4 mm ID
Gooseneck Splitless Liner	Restek		20800	4 mm x 6.5 x 78.5 for Aligent GCs
Guard Column	Restek		20231	0.5 MID
Ferrules	Fisher Scientific		5-5	

9.3. **Table 9.3 - Glassware**

Glassware	Description	Vendor / Item # / Description
Volumetric Flasks	10mL, 25mL, 50mL	Class A
Glass Storage Vials	5mL, 10mL, 12mL, with Teflon-lined screw caps	MG Scientific / T102-3-INV, T102-1-INV V138-19, B510-1
Glass Autosampler Vials	2.0mL with Teflon-lined crimp or screw caps	MG Scientific / V300-3 / V300-20N

9.4.

9.4. **Table 9.4 - General Supplies**

Supply	Description	Vendor/Item #
Gas tight syringes	10-µL, 25-µL, 50-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL, as needed, Hamilton or equivalent.	Fisher Scientific/Various
Pipettes	Borosilicate Glass 9" Pipette	MG Scientific / D200-9

10. Reagents and Standards

10.1. **Table 10.1 – Reagents**

Reagent/Standard	Concentration/ Description	Manufacturer/Vendor/Item #
Methylene Chloride (Dichloromethane)	Pesticide Grade or equivalent / MeCl2	MG Scientific / # 9266-8P
Methanol	Purge and Trap Grade or equivalent / MeOH	Burdick & Jackson / VWR Scientific / 232-1

10.2. **Table 10.2 - Standard Definitions**

Standard	Description	Comments
Tune Standard	Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, pentachlorophenol, and benzidine solution in dichloromethane used to verify ion response ratios and system inertness prior to analysis	Must inject no more than 50ng on column
Initial Calibration Standards	Standards prepared at varying levels to determine response and retention characteristics of instrument	Method requires a minimum of 5 levels
Continuing Calibration Verification Standard	A calibration standard prepared at mid-level concentration for all target compounds. This standard is used to verify that the instrument response has not changed significantly since the initial calibration was performed.	
Second Source Verification Standard	A standard prepared from a source other than that used for the initial calibration. This mid-level standard verifies the accuracy of the calibration curve.	
Internal Standard	A solution added all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard is used to adjust response ratios to account for instrument drift.	1,4 Dichlorobenzene-d4 Naphthalene-d_8 Acenaphthene-d_{10} Phenanthrene-d_{10} Chrysene-d_{12} Perylene-d_{12}
Surrogate Standard	A solution added to all samples, spikes, control samples, and method blanks prior to analysis.	Nitrobenzene-d_5 2-Fluorobiphenyl Terphenyl-d_{14} Phenol-d_6 2-Fluorophenol 2,4,6-Tribromophenol
Spiking Standard	This solution contains 70 target analytes and should not be prepared from the same standards as the calibration standards.	

10.3.

10.3. **Table 10.3 - Standard Storage Conditions**

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened ampulated standards. ▪ Stock standards must be replaced 1 year after ampule is opened or on expiration date, whichever is sooner.	▪ Manufacturer's recommended storage conditions ▪ When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	▪ Reference solutions prepared by dilutions of the stock solution	▪ 1 year from preparation or the expiration date listed for the stock source, whichever is sooner. ▪ Working solutions must be checked frequently and replaced if degradation or evaporation is suspected.	▪ Store in amber vials with Teflon lined screw caps ▪ Manufacturer's recommended storage conditions for stock source solution. ▪ If stock source conditions conflict, store according to method requirements.

10.4. **Standard Sources**: Standards are prepared from commercially available multi-compound stock solutions and neat materials by multiple dilutions. The sources of the stock solutions and neat materials, recipes for preparing dilutions and working standards, and concentrations for all compounds are presented in table 9.4. All intermediate standards are prepared using dichloromethane and stored in glass vials with Teflon lined caps or as recommended by the standard manufacturer.

10.5. **Preparation Procedures:**

10.5.1. **Internal Standard Stock solution:** Restek brand Internal Standard Mix, 4000ug/mL, catalog #31006 (contains six internal standards: acenaphthene-d10; chrysene-d12; 1,4-dichlorobenzene-d4; naphthalene-d8; perylene-d12; and phenanthrene-d10), or equivalent. Add 10uL of internal standard solution to 1000uL of every standard, sample, and QC sample injected.

10.5.2. **Surrogate Standard stock solutions:** Restek brand Base Neutral surrogate mix, 5000ug/mL, catalog #31082 (contains four surrogate standards: 2-fluorobiphenyl; nitrobenzene-d5; p-terphenyl-d14; 1,2-Dichlorobenzene-d4 (advisory)). Restek brand Acid surrogate mix, 7500ug/mL, catalog #31083 (contains four surrogate standards: 2-fluorophenol; phenol-d6; 2,4,6-tribromophenol; 2-chlorophenol-d4 (advisory)), or equivalent.

10.5.3. **Surrogate Standard working solution (for extractions)**: dilute 5.0mL of both the Restek Base Neutral stock surrogate solution (#31082) and the Restek Acid stock surrogate solution (#31083) to 50mL with Dichloromethane, or equivalent. This gives a final concentration of 500ug/mL per Base Neutral surrogate compound and 750ug/mL per Acid surrogate compound. The extraction analyst spikes each water and soil sample with 100uL of this working solution.

10.5.4. **DFTPP tuning solution:** dilute 1250uL of Supelco DFTPP stock standard (catalog #47548-U; 1000ug/mL) to a total volume of 25mL with methylene chloride for a final concentration of 50ug/mL, or equivalent. The stock standard also contains 4,4'-DDT, benzidine, and penta chlorophenol for assessing column degradation. Information for the standards preparation and expiration dates are affixed to the outside of the vial, and is easily accessible through Epic Pro LIMS. The standard material will be kept in a freezer at -10°C.

10.5.5. **Initial Calibration curve standards**: the following three stock standards, or equivalent, are used to prepare the initial calibration curve:

10.5.5.1 8270 Custom Mix 1, O2si, 400ug/mL, Catalog #116597-04-05 or Supelco, 400ug/mL, Quote #20447738

10.5.5.2 n-Nitrosodiphenylamine, Supelco, 5000ug/mL, catalog#46702-U

10.5.5.3 1,4-Dioxane, Restek, 2000ug/mL, catalog #30287

10.5.5.4 2,3,4,6-Tetra chlorophenol, Accustandard, 2000ug/mL, catalog #A-029S-D-10X

10.5.5.5 1,2,4,5-Tetrachlorobenzene, Absolute Standards, 1000ug/mL, catalog #70274

10.5.5.6 **Initial Calibration Intermediate Standard:** Dilute 2.5mL of 400ug/mL Supelco 8270 Custom Mix 1, 200uL of the 5000ug/mL n-Nitrosodiphenylamine solution, 500uL of the 2000ug/mL 1,4-Dioxane solution, 500 uL of the 2000 ug/mL 2,3,4,6-Tetrachlorophenol solution, and 1000 uL of 1000ug/mL 1,2,4,5-Tetrachlorobenzene to 5.0mL with dichloromethane, or equivalent. The resulting intermediate standard has a concentration of 200mg/L for each compound.

10.5.6. **Working Standard Preparation**: Working calibration standards are prepared in dichloromethane or a water soluble solvent. Standards made for direct analysis on the GC/MS are made in dichloromethane. Standards made for addition into samples as part of the preparation are made into dichloromethane. Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks or prepared in smaller, glass vials and brought to volume by additions of solvent with micro syringes.

10.5.6.1 **Initial Calibration Verification stock standards (second-source)**

10.5.6.1.1 Restek, custom 8270, catalog #52939, 200ug/mL per compound.

10.5.6.1.2 Supelco, 1,4-Dioxane, catalog #48367, 2000ug/mL.

10.5.6.1.3 Absolute Standards, 2,3,4,6-tetrachlorophenol, catalot #92389, 5000ug/mL, or equivalent.

10.5.6.1.4 Supelco, 1,2,4,5-Tetrachlorobenzene, catalog #40177, 1000ug/mL.

10.5.6.2 **Initial Calibration Verification working standard (second source):** Dilute 250uL of the custom 8270 second source standard #52939, 10uL of 2,3,4,6-Tetrachlorophenol #560028, 25uL of 1,4-Dioxane #48367, 50uL of 1,2,4,5-Tetrachlorobenzene #40177, and 10uL of the stock internal standard solution (9.5.1) to 1mL with dichloromethane, or equivalent. This gives a final concentration of 50ppm.

10.5.6.3 **LCS/MS Standard working solution:** Supelco 70 Component Custom MCS Mix catalog #861389-U, 200ug/mL. Supelco n-Nitrosodiphenylamine, catalog #46702-U, 5000ug/mL, or equivalent. The extraction analyst spikes each LCS/LCSD and matrix spike sample with 250uL of the LCS mix and 10uL of the n-NDPA solution. This produces a concentration of 50ug/mL.

10.5.6.4 **Other calibrations**: Other compounds are analyzed per client requests. Curves are prepared at levels similar to those of the standards above. The calibration standards and the second source standards are as follows: Calibration Standards, Benzidine, Calibration Standard, Supelco Catalog #40005, 5000ug/mL. Second Source, Restek, catalog #31441, 1000ug/mL; EPA CLP SOW OLM4 mix, Calibration Standard, Supelco Catalog#47514-U, 2000ug/mL. Second Source, Absolute Standards, catalog #19253, 2000ug/mL, or equivalent. Minnesota Phenols Samples required Calibration Standard o2si, catalog #114055-05, 500 ug/mL Second source standard Absolute Standards Catalog #92775, 100ug/mL and for 3-Chlorophenol, Absolute Standards Catalog#79050, 1000ug/Ml.

10.5.7. Store at -10°C or less in amber Teflon-sealed containers. The solutions should be checked frequently for stability.

10.6. **Calibration Standard Preparation**: Calibration standards are made into dichloromethane for the purpose of direct analysis by the analytical instrumentation. The standards must be made in a volumetric fashion. Several alternatives exist but the method employed by Pace – Green Bay utilizes glass autosampler vials according to the following procedure. The individual standards can be made according to the details provided in table 10.3.

Table 10.4 – Working Standard Dilutions and Concentrations

Standard	Standard(s) Amount	Solvent	Solvent Volume	Final Total Volume	Final Concentration
Calibration Std 1	25uL	Dichloromethane	975uL	1010uL	5ppm
Calibration Std 2	50uL	Dichloromethane	950uL	1010uL	10ppm
Calibration Std 3	125uL	Dichloromethane	875uL	1010uL	25ppm
Calibration Std 4	250uL	Dichloromethane	750uL	1010uL	50ppm
Calibration Std 5	400uL	Dichloromethane	600uL	1010uL	80ppm
Calibration Std 6	500uL	Dichloromethane	500uL	1010uL	100ppm
Calibration Std 7	600uL	Dichloromethane	400uL	1010uL	120ppm
Continuing Calibration Verification Standard	250uL	Dichloromethane	750uL	1010uL	50ppm

10.7. Traceability of Calibration Standards—The calibration standards purchased from vendors have been manufactured according to the following guidelines

10.7.1. Identity of neat material verified by GC/MS

10.7.2. Purity of neat material determined by GC/FID or GC/ECD. Correction for impurities is made when purity is less than 97%. Standards are prepared gravimetrically to a precision of 0.5%. All weights are traceable to NIST.

10.7.3. Analyte concentration verified by capillary gas chromatography. Standards tested for stability and homogeneity.

10.7.4. Standards are expiration dated.

10.8. Standard Labeling—All working calibration standards will have a label attached to the bottle identifying the following (Epic pro standard labels do not contain all the following)

10.8.1. Name of Solution

10.8.2. PASI, Inc. Standard ID Number

10.8.3. PASI, Inc Lab Lot ID (for Stock standards and reagents)

10.8.4. Preparation Date

10.8.5. Preparer's initials

10.8.6. Concentration

10.8.7. Expiration Date

11. Calibration and Standardization

11.1. **Tune Verification** – The mass spectrometer tune status must be verified prior to initial calibration and at the beginning of each analytical sequence. If the current tune status does not meet the ion ratio criteria in the method (see section 12.2), follow the equipment manufacturers' instructions for re-tuning the mass spectrometer. The tune status must be verified after the tuning procedures. Refer to section 12.2 for details on the analysis and evaluation of this standard.

11.2. **Initial Calibration:**

11.2.1. **Analysis of Standards**: An initial calibration curve using a minimum of five points is analyzed prior to analyzing client samples. The lowest concentration must be at or below the equivalence of the standard reporting limit. The lowest calibration point reflects the practical quantitation limit for that compound, a level below which all reported results must be qualified as estimated values. Refer to table 11.1 for compound concentrations.

Table 11.1: Laboratory PQL and Calibration Standard Compound Concentrations

Analyte	PQL water (ug/L)	PQL soil (ug/kg)	PQL Biota (ug/kg)	Std 1 ug/L	Std 2 ug/L	Std 3 ug/L	Std 4 ug/L	Std 5 ug/L	Std 6 ug/L	Std 7 ug/L
Acenaphthene	5.0	167	330	5.0	10	25	50	80	100	120
Acenaphthylene	5.0	167	330	5.0	10	25	50	80	100	120
Aniline	5.0	167	N/A	5.0	10	25	50	80	100	120
Anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Benz(a)anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(a)pyrene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(b)fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(g,h,i)perylene	5.0	167	330	5.0	10	25	50	80	100	120
Benzo(k)fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Benzoic acid	10	330	N/A	5.0	10	25	50	80	100	120
Benzyl alcohol	10	330	N/A	5.0	10	25	50	80	100	120
4-Bromophenylphenyl ether	5.0	167	330	5.0	10	25	50	80	100	120
Butylbenzylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
Carbazole	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloro-3-methylphenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Chloroaniline	10	333	330	5.0	10	25	50	80	100	120
bis(2-Chloroethoxy)methane	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Chloroethyl) ether	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Chloroisopropyl) ether	5.0	167	330	5.0	10	25	50	80	100	120
2-Chloronaphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Chlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Chlorophenylphenyl ether	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Diphenylhydrazine	5.0	167	N/A	5.0	10	25	50	80	100	120
Chrysene	5.0	167	330	5.0	10	25	50	80	100	120
Dibenz(a,h)anthracene	5.0	167	330	5.0	10	25	50	80	100	120
Dibenzofuran	5.0	167	330	5.0	10	25	50	80	100	120
1,2-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
1,3-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
3,3'-Dichlorobenzidine	10	330	330	5.0	10	25	50	80	100	120
2,4-Dichlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
Diethylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
2,4-Dimethylphenol	5.0	167	330	5.0	10	25	50	80	100	120
Dimethylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-butylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
4,6-Dinitro-2-methylphenol	5.0	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrophenol	10	333	670	5.0	10	25	50	80	100	120
2,4-Dinitrotoluene	5.0	167	330	5.0	10	25	50	80	100	120
2,6-Dinitrotoluene	5.0	167	330	5.0	10	25	50	80	100	120
Di-n-octylphthalate	5.0	167	330	5.0	10	25	50	80	100	120
bis(2-Ethylhexyl)phthalate	5.0	167	330	5.0	10	25	50	80	100	120
Fluoranthene	5.0	167	330	5.0	10	25	50	80	100	120
Fluorene	5.0	167	330	5.0	10	25	50	80	100	120
Hexachloro-1,3-butadiene	10	333	330	5.0	10	25	50	80	100	120
Hexachlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
Hexachlorocyclopentadiene	5.0	167	330	5.0	10	25	50	80	100	120

Analyte	PQL water (ug/L)	PQL soil (ug/kg)	PQL Biota (ug/kg)	Std 1 ug/L	Std 2 ug/L	Std 3 ug/L	Std 4 ug/L	Std 5 ug/L	Std 6 ug/L	Std 7 ug/L
Hexachloroethane	5.0	167	330	5.0	10	25	50	80	100	120
Indeno(1,2,3-cd)pyrene	5.0	167	330	5.0	10	25	50	80	100	120
Isophorone	5.0	167	330	5.0	10	25	50	80	100	120
2-Methylnaphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Methylphenol	10	333	330	5.0	10	25	50	80	100	120
3&4-Methylphenol	5.0	167	330	5.0	10	25	50	80	100	120
Naphthalene	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitroaniline	5.0	167	330	5.0	10	25	50	80	100	120
3-Nitroaniline	5.0	167	670	5.0	10	25	50	80	100	120
4-Nitroaniline	10	333	670	5.0	10	25	50	80	100	120
Nitrobenzene	5.0	167	330	5.0	10	25	50	80	100	120
2-Nitrophenol	5.0	167	330	5.0	10	25	50	80	100	120
4-Nitrophenol	10	333	670	5.0	10	25	50	80	100	120
N-Nitrosodimethylamine	5.0	167	330	5.0	10	25	50	80	100	120
N-Nitroso-di-n-propylamine	5.0	167	330	5.0	10	25	50	80	100	120
N-Nitrosodiphenylamine	5.0	333	330	5.0	10	25	50	80	100	120
Pentachlorophenol	10	330	670	5.0	10	25	50	80	100	120
Phenanthrene	5.0	167	330	5.0	10	25	50	80	100	120
Phenol	5.0	167	330	5.0	10	25	50	80	100	120
Pyrene	5.0	167	330	5.0	10	25	50	80	100	120
1,2,4-Trichlorobenzene	5.0	167	330	5.0	10	25	50	80	100	120
2,4,5-Trichlorophenol	5.0	167	670	5.0	10	25	50	80	100	120
2,4,6-Trichlorophenol	5.0	167	330	5.0	10	25	50	80	100	120
1,4-Dioxane	10	330	N/A	5.0	10	25	50	80	100	120
1,2,4,5-Tetrachlorobenzene	5.0	167	N/A	5.0	10	25	50	80	100	120
2,3,4,6-Tetrachlorophenol	10	167	N/A	5.0	10	25	50	80	100	120
Acetophenone	10	333	N/A	5.0	10	25	50	80	100	N/A
Atrazine	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzaldehyde	10	333	N/A	5.0	10	25	50	80	100	N/A
Benzidine	50	1670	N/A	5.0	10	25	50	80	100	N/A
Caprolactam	10	333	N/A	5.0	10	25	50	80	100	N/A
Biphenyl	10	333	N/A	5.0	10	25	50	80	100	N/A

11.2.2. An analyte must be present and calibration curve in control in order to be reported on the target analyte list. Analytes identified by mass spectral match but not present and in control in the calibration table may be reported as Tentatively Identified Compounds (TICs). Guidelines for identification are listed in Section 12.15. Results for these TICs should be reported only on a present/absent basis. However, quantitative results may be reported provided they are qualified as estimated values.

11.2.3. Calibration Response Factors: Response factors (RF) establish the relationship of the instruments response in comparison with the concentration of any given analyte. The RF includes the concentration and response of the internal standard as well. By relating the IS concentration and response in an inverse manner, the target analyte concentration is adjusted to account for drift in the instrument on a per injection basis. As instrument response increases as indicated by the response of the internal standard, the concentration of the target is mathematically decreased, and vice versa.

11.2.4. To calculate the RF for any given calibration standard (or calibration verification standard), tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. Response factors are calculated using the following equation:

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured.
A_{is} = Area of the characteristic ion for the specific internal standard.
C_{is} = Concentration of the specific internal standard (µg/L).
C_x = Concentration of the compound being measured (µg/L).

11.2.5. Most, if not all modern chromatography data systems are capable of calculating this factor and using it to quantify analyte concentrations. The 8270C method has minimum requirements that these response factors must meet in order to be considered valid. The method uses a subset of the target analyte list to evaluate the performance of the system. These compounds are referred to as the System Performance Check Compounds or the SPCCs. The SPCCs serve as an indicator of instrument sensitivity and, by meeting a minimum value, ensure that the laboratory has adequate sensitivity to analyze and reliably report data for environmental samples.

11.2.6. **Calibration Curve Fit**: The calibration curve is a representation of the relationship of the instrument response and analyte concentration. The curve is used to quantitate the concentration of an unknown based on its response and this known relationship. The curve is produced in several ways depending on the nature of the "goodness of fit".

11.2.7. **Average Response Factor (ARF)**: The average response factor is determined by averaging the response factors calculated for each calibration level for each target analyte. The average RF can be used to calculate the concentration of target analytes in samples provided the criteria are met for consistency in the RFs for any given analyte. An average response factor is the default curve fitting option for calibrations. It is in the most basic sense, a linear regression that is forced through zero at the origin. Because of its simplicity and the interception of the y axis at the origin, this is the preferred technique for curve fitting. A calculation of the percent relative standard deviation (%RSD) is used to determine the acceptability of the use of the ARF (see Table 11.2):

$$\%RSD = \left(\frac{(SD*100)}{ARF}\right)$$

Where:

SD = Standard deviation of the averaged RFs for a given compound

11.2.8. The average response factor is also used to diagnose the integrity of the chromatography system as it relates to calibration linearity. The ***Calibration Check Compounds (CCCs)*** are a subset of the target analyte list that must meet specific criteria (see Table 11.2) for the calibration to be acceptable. For the CCCs, the %RSD for each is compared to the method criteria. If that of any CCC exceeds the criteria, the system needs to be inspected for potential sources of errors and recalibrated.

11.2.9. **Linear Regression**: The linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique

will have the format of y=ax+b where "a" is the slope of the line and "b" is the y intercept. In order to use this curve fit technique, a minimum of 5 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument is linear in nature but does not necessarily intercept the y axis at the origin. However, because the linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. A calculation of the correlation coefficient "r" is used to determine the acceptability of a linear regressed curve (see Table 11.2).

11.2.10. **Non-linear Regression**: The non-linear regression calibration curve is derived from a least squares regression analysis of the calibration points. A calibration curve based on this technique will have the format of $y= ax^2+bx+c$. In order to use this curve fit technique, a minimum of 6 calibration points must be available and the origin cannot be included as one of the points. This technique works well for calibrations where the response of the instrument gradually decreases with increasing concentrations. Using this technique, an analyst may be able to generate calibration curves with correlation coefficients very close or equivalent to 1.000. However, because the non-linear regression is not forced through the origin, very low levels of contaminants below the response of the lowest calibration point may generate erroneous reportable results. Likewise, high levels of contamination may not be able to be calculated due to regression equations with multiple intercepts of either axis on the calibration plot.

11.2.11. A calculation of the coefficient of determination (COD) is used to determine the acceptability of a non-linear regressed curve (see Table 11.2). Either the low or high calibration points may be dropped to meet linearity criteria provided the laboratory meets the minimum 5 calibration point requirements. Points within the center of the curve may not be dropped unless an obvious problem is discovered and documented. The point must be dropped in its entirety. Re-analysis if performed should be within the same 12 hour time window and must occur within 8 hours of the original analysis.

11.3. **Calibration Verification**:

11.3.1. **Second Source Verification**: In addition to meeting the linearity criteria, any new calibration curve must be assessed for accuracy in the values generated. Accuracy is a function of both the "fit" of the curve to the points used and the accuracy of the standards used to generate the calibration points. By meeting the fit criteria, the accuracy relative to the goodness of fit is addressed. However, because all calibration points are from the same source, it is possible that the calibration points may meet linearity criteria but not be accurately made in terms of their true value.

11.3.2. Therefore, to assess the accuracy relative to the purity of the standards, a single standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as ***Secondary Source Verification*** or, alternatively as ***Initial Calibration Verification (ICV)***. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the ICV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\%\,Difference = \frac{(RFccv - AveRFcal)}{AveRFcal} * 100$$

$$\%\,Drift = \frac{(Result\ CCV - True\ Value\ CCV)}{True\ Value\ CCV} * 100$$

11.3.3. **Continuing Calibration Verification (CCV)**: As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as ***Continuing Calibration Verification***. The validity of the initial calibration is checked at the beginning of every analytical sequence and every 12 hours thereafter for as long as the instrument is analyzing samples and is accomplished by analyzing a midpoint calibration standard (CCV).

11.3.4. The values obtained from the analysis of the CCV are compared to the true values and a percent change calculated. The percent change must meet the method specified criteria for the analysis to proceed for an additional 12 hours.

11.3.5. The actual determination of change in instrument response is based on the type of curve fit used for each analyte. Calibration curves based on an average response factor are assessed based on the percent difference of the RF calculated for the CCV from the average RF established in the initial calibration. Calibration curves based on a linear or non-linear regression are assessed based on the percent drift of the calculated result from the known true value of the standard. The equations for these calculations are as follows:

$$\%\,Difference = \frac{(RFccv - AveRFcal)}{AveRFcal} * 100$$

$$\%\,Drift = \frac{(Result\ CCV - True\ Value\ CCV)}{True\ Value\ CCV} * 100$$

Table 11.2: Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Average Response Factor	%RSD ≤ 15%	If not met, try linear regression fit
	Linear Regression	r ≥ 0.99	If not met, try non-linear regression fit
	Non-linear Regression	COD ≥ 0.99	If not met, remake standards and recalibrate
System Performance Check Compounds (SPCCs)	N-Nitroso-di-n-propylamine Hexachlorocyclopentadiene 2,4-Dinitrophenol 4-Nitrophenol	Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05 Avg RF ≥ 0.05	Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, poor purging efficiency, and active sites in the column or chromatographic system.
Calibration Check Compounds (CCCs)	Acenaphthene 1,4-Dichlorobenzene Hexachlorobutadiene N-Nitrosodiphenylamine Di-n-octylphthalate Fluoranthene Benzo[a]pyrene 4-Chloro-3-methylphenol 2,4-Dichlorophenol 2-Nitrophenol Phenol Pentachlorophenol 2,4,6-Trichlorphenol	%RSD < 30%	%RSD for the calibration check compounds (CCC's) must be ≤30% regardless of curve fit used. If the CCCs are not included on a list of analytes for a project, and therefore not included in the calibration standards, then all compounds of interest must meet a ≤15% RSD criterion.
Second Source Verification Standard	Immediately after each initial calibration	% Drift ±30%	Acceptance criteria are ±30% for all analytes, with allowances for 5% of compounds at ±40%. See current revision of S-ALL-Q-025. Additional client specific requirements for the analysis of contract samples requires that all compounds must be within ±20%.
Continuing Calibration Verification	Prior to the analysis of any samples and every 12 hours thereafter		If the requirements for continuing calibration are not met, these corrective actions must be taken prior to reanalysis of standards. Only two injections of the same standard are permitted back to back.
	SPCCs	Must meet response criteria listed above	
	Internal Standard RT Internal Standard Response	RT ± 30 sec 50 – 200%	Use midpoint calibration standard as reference Use midpoint calibration standard as reference
	CCCs	RF ± 20% Diff. Result ± 20% Drift	Use for Avg RF calibration curves Use for linear and non-linear calibration curves
	Non-CCC Targets	EPA 8270 Criteria: RF ± 50% Diff. Result ± 50% Drift EPA 625 Criteria: RF ± 20% Diff. Result ± 20% Drift	Some programs may require control over non-CCC target analytes. In the absence of specified criteria, use those listed

11.4. **Calibration Corrective Actions:**

11.4.1. Calibration Linearity Problems:

11.4.1.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

11.4.1.2 Perform another initial calibration.

11.4.1.3 No data can be reported.

11.4.1.4 Generate Non-Conformance Memo.

11.4.2. Second Source Verification Problems:

11.4.2.1 Check instrumentation/equipment condition. Document instrument maintenance in the logbook.

11.4.2.2 Perform another initial calibration.

11.4.2.3 No data can be reported.

11.4.2.4 Generate Non-Conformance Memo.

11.4.3. Continuing Calibration Verification Problems:

10.4.3.1. Reanalyze the original CCV standard to determine instrument consistency.

10.4.3.2. Prepare and analyze a new CCV standard to determine preparation consistency/standard integrity.

10.4.3.3. Document instrument maintenance.

10.4.3.4. Reanalyze CCV standard to determine if maintenance was effective in restoring performance.

10.4.3.5. Complete recalibration of instrument.

10.4.3.6. If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the injection log with a thorough explanation for the deviation from policy.

10.4.3.7. *Exceptions:* If calibration verification is above the upper control limit, samples non-detected for those analytes may be reported without reanalysis.

12. Procedure

12.1. **Operating Parameters:** Configure the GC/MS system to match the following operating parameters based on instrument configuration. The parameters themselves are saved as a method on the chromatography data system. By loading the last method used, the instrument will auto-configure to match the parameters from the last time the system was operated under that method. Verify that the settings in the software match the appropriate configuration.

Table 12.1: Instruments and Operating Parameters

GC/MS Instrument 40MSS1	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 40°C	Acquisition mode: SCAN
Temp Program: hold 1.0 min at 40°C, ramp at 18°C/min to 100°C, then ramp at 15°C/min to 290°C, hold 5.95min, then ramp at 40°C/min to 320°C and hold for 1 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25um film thickness)w/Integraguard	
Purge Flow: 40mL/min	

GC/MS Instrument 40MSS5 – Screen Instrument only	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 75°C	Acquisition mode: SCAN
Temp Program: hold 0.3 min at 75°C, ramp at 34°C/min to 320°C, then ramp at 40°C/min to 340°C, hold 1.20min	Mass Range: 35-500
Final Temp: 340°C	
Transfer Line Temp: 300°C	
Column: Restek XTI-5 (30m; 0.25mm ID and 0.25um film thickness)w/Integraguard	
Purge Flow: 40mL/min	

GC/MS Instrument 40MSS6 used for Minnesota Phenols Samples	
GC: Hewlett Packard model 5890	MS: Hewlett Packard model 5972A
Operating Parameters:	Operating Parameters:
Initial Temp: 5°C	Acquisition mode: SCAN
Temp Program 50°C, ramp at 18°C/min to 150°C, then ramp at 3°C/min to 167°C , then ramp at 40°C/min to 320°C and hold for 2.5 min	Mass Range: 35-500
Final Temp: 320°C	
Transfer Line Temp: 300°C	
Column: Restek Rxi-1MS (20m; 0.18um ID, 0.18 df)	
Split Flow: 100mL/min	

12.2. **Tune Verification**: At the beginning of each analytical sequence, prior to the analysis of any standards or samples, the mass spectrometer tune conditions must be verified. This is done by analyzing a standard containing DFTPP. The tune verification standard can be combined with the CCV standard provided that the amount of DFTPP introduced into the system meets the method criteria. For semi-volatile analysis, the system must also be verified for inertness. This is done simultaneously by the inclusion of DDT, benzidine and pentachlorophenol. DDT is used to verify breakdown conditions; benzidine and pentachlorophenol are used to check for tailing due to system activity.

12.2.1. After the analysis of this standard, the mass spectrum of DFTPP must be evaluated against the following criteria:

Mass (m/z)	Ion Abundance criteria
51	10.0-80.0% of m/z 198
68	<2.0% of m/z 69
69	Present
70	<2.0% of m/z 69
127	10.0-80.0% of m/z 198
197	<2.0% of m/z 198
198	Base peak, >50% of Mass 442
199	5.0-9.0% of m/z 198
275	10.0-60.0% of m/z 198
365	>1% of m/z 198
441	Present, but less than m/z 443
442	>50.0% of m/z 198
443	15.0-24.0% of m/z 442

12.2.2. To evaluate the tune spectra, following the operating instructions for the chromatography data system to access the data file and obtain mass spectra for DFTPP. If the software has a program or macro for automatically selecting the spectra and evaluating the response ratios, use this option. Otherwise, the spectra must be obtained in one the following manners, in the listed order:

1. **Using an average of three scans, centered on the apex of the peak; or,**
2. **Using an average of all scans across the width of the peak, taken at half height; or,**
3. **Using an average of all scans taken across the width of the peak from baseline to baseline.**

A background scan taken immediately before but not including the peak must be subtracted.

12.2.3. Once obtained, evaluate the ion ratios against the criteria listed above. If the ratios meet the criteria, then analysis may proceed for 12 hours. The window for analysis is 12 hours from the injection date / time for the DFTPP tune verification. After that, the tune must be verified again to establish a new analytical window. The same Ion Abundance Criteria used for the DFTPP tune coupled with the initial calibration must be used for all subsequent analyses associated with that initial calibration.

12.2.4. If the ratios do not meet the criteria, refer to the following corrective actions to address the problem: Any changes made to the system must be followed with the reanalysis of a tune verification standard. Any maintenance performed on the physical mass spec components requires recalibration. "Autotunes" may be performed as long as the following CCV meets all criteria for response, retention time and sensitivity.

12.3. **Tailing Factor Verification-** Benzidine and Pentachlorophenol should be present at their normal responses, and peak tailing should not be to an excess.

12.3.1. **Column performance test for base / neutrals –** At the beginning of each day that the base / neutral fraction is to be analyzed for benzidine, the benzidine tailing factor must be calculated. The benzidine tailing factor must be less than 3.0.

12.3.2. **Column performance test for acids** – At the beginning of each day that the acid fraction is to be determined, the pentachlorophenol tailing factor must be calculated. The pentachlorophenol tailing factor must be less than 5.0.

12.3.3. **Tailing factor calculation** – Refer to Attachment II: Tailing Factor Calculation.

12.3.4. The tailing factor of 3.0 for Benzidine and 5.0 for Pentachlorophenol must not be exceeded. If the tailing factor for either exceeds this amount, corrective action must be taken prior to the analysis of sample. The tailing factor must be verified by the analysis of another tailing factor standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

12.3.4.1 Perform front-end maintenance on the GCMS System.

12.3.4.2 Begin the run again by re-analyzing the DFTPP tune solution.

12.4. **Breakdown Verification-** The GC/MS system must be sufficiently inert such that DDT will not breakdown excessively while in the injection port. The inertness is assessed by calculating the percent breakdown of DDT into the products DDD and DDE. The calculation is performed as follows:

$$\%DDT\ Breakdown = \left(\frac{(DDD + DDE)}{(DDT + DDD + DDE)}\right) * 100$$

12.4.1. The % breakdown **must not exceed 20%.** If the breakdown of DDT exceeds this amount, corrective action must be taken prior to analysis of samples. The breakdown must be verified by the analysis of another breakdown standard after corrective action is taken. Follow the following steps to return the system to an acceptable operating condition.

12.4.1.1 Perform front-end maintenance on the GCMS System

12.4.1.2 Begin the run again by re-analyzing the DFTPP tune solution.

12.5. **Calibration Verification**: After the instrument tune conditions are verified and the system meets tune criteria, the instrument must undergo calibration verification. If it has already been determined that the instrument needs to be recalibrated, follow the procedures listed in section 11.2 (Analysis of Standards). Otherwise, analyze a Continuing Calibration Verification Standard to determine the current calibration status.

12.6. If the CCV meets control criteria, the system is deemed to be in control and analysis of samples may commence. If the CCV does not meet control criteria, follow the corrective action procedures listed section 11.4.3 (Continuing Verification Problems). If the tune verification has been combined with the CCV, the 12 hour analysis window begins from the analysis date / time of the CCV.

12.7. *Note:* In situations where the instrument will run unattended (i.e., overnight), the analyst may load sequential CCVs in anticipation of that the first in the series may fail due to carry over from a previous sample. If so, the CCV must be evaluated according to the protocol set forth in the Quality Assurance Manual within the Equipment and Measurement Traceability section.

12.8. **Sample Preparation-**

12.8.1. **Water Samples**: Aqueous samples are prepared according to EPA 3510C. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-053 *Separatory Funnel Extraction of Water Samples for Semivolatile* (most current revision or replacement) for details on the preparation of aqueous samples.

12.8.1.1 Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 10uL of the internal standard solution.

12.8.2. **Soil Samples**: Solid samples are prepared according to EPA 3546. These procedures are contained in a separate standard operating procedure. Refer to SOP number S-GB-O-045 *Microwave Extraction for the Determination of Polynuclear Aromatic Hydrocarbons, Base/Neutral/Acids, and Total Petroleum Hydrocarbons in Solid Matrices* (most current revision or replacement) for details on the preparation of soil or solid samples.

12.8.2.1 Prior to analysis, each sample, MD, LCS, MS, and MSD is spiked with 10uL of the internal standard solution.

12.8.3. **Biota Samples:** Biota samples are prepared according to EPA Method 3540C. These procedures are contained in a separate standard operating procedure. Refer to S-GB-O-033 *Extraction of Biological Samples for Base Neutral/Acid and PAH-SIM Analysis* (most current revision or replacement) for details on the preparation of biota samples.

12.8.3.1 Prior to analysis, each sample, MB, LCS, MS, and MSD is spiked with 5uL of the internal standard solution.

12.9. **Dilutions**

12.9.1. Dilutions on sample extracts must be prepared in a volumetric fashion. Sample aliquots should be taken in volumetric syringes and brought to volume by the addition of solvent via an appropriate syringe. In the event a dilution is made to bring a target analyte into calibration range, the analyst should make a dilution such that the target analyte is roughly the equivalent of the mid calibration point whenever possible. If dilutions are made on extracts that already contain internal standards, a proportional aliquot of internal standard solution must be added to the diluted extract based on the volume of diluent used.

12.10. **Sample Analysis-**

12.10.1. GC/MS System Preparation

12.10.1.1 Operating Parameters – Set up the instrument parameters shown in Table 12.1

12.10.1.2 System Tuning and GC Performance Checks – Analyze the Tuning Solution and tune the mass spectrometer to meet the criteria shown in Section 12.2. Verify acceptable GC system performance as described in Section 12.2. Print out a tune report.

12.10.1.3 Batch Sequence – Generate a sequence to run a batch of samples.

Initial Calibration – The typical batch for initial calibration should include:

Tune Standard
Calibration Level 1
Calibration Level 2
Calibration Level 3
Calibration Level 4
Calibration Level 5
Calibration and System Performance Solution

Sample Analysis – The typical batch for sample analysis should include the following. Preparation of LCS, MS, MSD, and Duplicate sample extracts is described in the appropriate sample preparation SOP.

Tune Standard
Calibration and System Performance Solution
Instrument Blank
Method Blank
Laboratory Control Sample
Laboratory Control Sample Duplicate
20 samples
Matrix Spike
Matrix Spike Duplicate

Autosampler – Load the Autosampler with standards and samples for the batch created above.

12.10.1.4 Analyze Samples – Analyze all standards, quality control samples, and environmental samples.

12.10.1.5 Process all runs with Target software

12.10.1.6 View sample chromatograms and verify analyte identifications (Section 12.11).

12.10.1.7 Post data to EPIC Pro.

12.11. **Data Reduction**

12.11.1. Qualitative Analysis

12.11.1.1 **Retention Time Comparison:** The relative retention time (RRT) of the sample component must be within ±0.06 RRT units of the component in the calibration verification standard. Extracted Ion Current Plots (EICPs) may be used to provide a more reliable assignment of RT in the presence of co eluting components.

12.11.1.2 **Mass Spectrum Comparison:** The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

- The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
- The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.
- Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times.
- Additional client specific requirements for the analysis of contract samples requires all ions present in the reference mass spectrum at a relative intensity > 10% must be present in the sample spectrum.
- Due to limitations of the "Target" software, analyst discretion is advised.

Table 12.2 Primary and Secondary quantitation ions for target compounds[2]

Analyte	Primary Ion	Secondary Ions
Phenol	94	65, 66
Bis (2-Chloroethyl) ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	46	148, 111
Benzyl Alcohol	108	79, 107
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	108	107, 79
Bis (2-Chloroisopropyl)ether	45	77, 121
3&4-Methylphenol	108	107, 79
N-Nitroso-di-n-propylamine	70	43, 101
Hexachloroethane	117	200.8, 198.8
1,4-Dioxane	88	58, 43
Benzaldehyde	77	106, 105
N-Nitrosodimethylamine	42	74
Aniline	93	66, 65
Acetophenone	105	77, 51
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	107	122, 121
Benzoic acid	122	105, 77
Bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129
4-Chloroaniline	127	129
Hexachlorobutadiene	224.9	118, 190
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Caprolactam	113	56, 55
Hexachlorocyclopentadiene	237	234.9
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	153
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	108, 92
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	109	139, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	63, 89
Diethylphthalate	149	177, 150
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
4-Nitroaniline	138	108, 92
1,2,4,5-Tetrachlorobenzene	216	214, 179
2,3,4,6-Tetrachlorophenol	232	131, 230

Analyte	Primary Ion	Secondary Ions
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	283.8	142, 248.8
Atrazine	200	173, 215
Pentachlorophenol	265.8	263.8, 267.8
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Di-n-butylphthalate	149	150, 104
Fluoranthene	202	101, 203
Benzidine	184	185, 92
Pyrene	202	200, 203
Butylbenzylphthalate	149	91, 206
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Chrysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
Di-n-octylphthalate	149	43
Benzo(b)fluoranthene	251.99	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 25
Indeno(1,2,3-cd)pyrene	276	138, 277
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Carbazole	167	

[2]The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

Analyte	Primary Ion	Secondary Ions
Internal Standards		
1,4-Dichlorobenzene-d_4	152	150, 115
Naphthalene-d_8	136	68
Acenaphthene-d_{10}	164	162, 160
Phenanthrene-d_{10}	188	94, 80
Chrysene-d_{12}	240	120, 236
Perylene-d_{12}	264	260, 265
Surrogates		
2-Fluorophenol (acid)	112	64
Phenol-d_6 (acid)	99	42, 71
Nitrobenzene-d_5 (BN)	82	128, 54
2-Fluorobiphenyl (BN)	172	171
2,4,6-Tribromophenol (acid)	330	332, 141
Terphenyl-d_{14} (BN)	244	122, 212

[2]The information in this table was taken from Method 8270C. Please refer to the method for additional compounds and their applicable ions.

12.11.2. Internal Standard Assignment List (from Method SW-846 8270C-Table 5): this section lists the internal standard compounds and all target compounds that are assigned to each internal for quantitation:

1,4-Dichlorobenzene – d4
Aniline
Benzyl alcohol
Bis (2-chloroethyl)ether
Bis(2-chloroisopropyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2-Fluorophenol (surrogate)
Hexachloroethane
2-Methylphenol
4-Methylphenol
N-Nitroso-dimethylamine
N-Nitroso-di-n-propylamine
Phenol
Phenol-d6 (surrogate)
1,4-Dioxane
Pyridine
2-Chlorophenol-d4 (advisory surrogate)
Benzaldehyde
1,2-Dichlorobenzene-d4 (advisory surrogate)

Naphthalene-d8
Acetophenone
Benzoic acid
Bis(2-chlor ethoxy)methane
4-Chloroaniline
4-Chloro-3-methylphenol
2,4-Dichlorophenol
2,6-Dichlorophenol
2,4-Dimethylphenol
Hexachlorobutadiene
Isophorone
2-Methylnaphthalene
Naphthalene
Nitrobenzene
Nitrobenzene-d8 (surrogate)
2-Nitrophenol
1-Methylnaphthalene
1,2,4-Trichlorobenzene

Acenaphthene-d10
Acenaphthene
Acenaphthylene
1,2-Diphenylhydrazine
2-Chloronaphthalene
4-Chlorophenyl phenyl ether
Dibenzofuran
Diethyl phthalate
Dimethyl phthalate
2,4-Dinitrophenol
2,4-Dinitrotoluene
2,6-Dinitrotoluene
Fluorene
2-Fluorobiphenyl (surrogate)
Hexachlorocyclopentadiene
2-Nitroaniline
3-Nitroaniline
4-Nitroaniline
4-Nitrophenol
Biphenyl
2,4,6-Tribromophenol (surrogate)
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
1,2,4,5-Tetrachlorobenzene

Phenanthrene-d10
Atrazine
Anthracene
4-Bromophenyl phenyl ether
Di-n-butyl phthalate
4,6-Dinitro-2-methylphenol
Carbazole
Fluoranthene
Hexachorobenzene
N-Nitroso-diphenylamine
Penta chlorophenol
Phenanthrene

Chrysene-d12
Benzidine
Benzo(a)anthracene
Bis(2-ethylhexyl)phthalate
Butyl benzyl phthalate
Chrysene
3,3'-Dichlorobenzidine
Pyrene
Terphenyl-d6 (surrogate)
Di-n-octyl phthalate
Indeno(123,cd)pyrene

Perylene-d12
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(g,h,i)perylene
Benzo(a)pyrene
Dibenz(a,h)anthracene

12.12. Quantitative Analysis- Quantitation is based on the integrated abundance of the target analyte's quantitation ion using the internal standard technique.

12.12.1. **Raw Data Results:** The GC/MS data system will calculate the concentration of each analyte as µg/L (or ng/mL). For water samples, no further calculations are necessary unless a dilution of the sample has been performed. If the initial analysis of the sample or a dilution of the sample has a concentration that exceeds the calibration range, the sample must be analyzed at a higher dilution. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

12.13. **Tentatively Identified Compounds (TICs)** – For some samples, identification may be desired for non-target compounds. A mass spectral library search may be conducted to attempt assignment of tentative identifications. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications.

12.13.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum;

12.13.2. The relative intensities of the major ions should agree within ± 20%;

12.13.3. Molecular ions present in the reference spectrum should be present in the sample spectrum;

12.13.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds;

12.13.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.13.6. For additional information on the determination of TICs, please see SOP: S-ALL-O-038, *Processing of TICs for GCMS* (most current revision or replacement).

13. Quality Control

13.1. Table 13.1 – 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 reporting limit.	Qualify results and/or re-extract associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >20x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination; If sample result <20x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Method specified compounds: **Base Neutrals**: 1,2,4-Trichlorobenzene; Acenaphthene; 2,4-Dinitrotoluene; Pyrene; N-nitroso-di-n-propylamine; 1.4-Dichlorobenzene **Acids**: Pentachlorophenol; Phenol; 2-Chlorophenol; 4-Chloro-3-methylphenol; 4-Nitrophenol *OR (alternative)* 70 compound LCS Mix	One per batch of up to 20 samples	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to the TNI standard.	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)	Method specified compounds: **Base Neutrals**: 1,2,4-Trichlorobenzene; Acenaphthene; 2,4-Dinitrotoluene; Pyrene; N-nitroso-di-n-propylamine; 1.4-Dichlorobenzene **Acids**: Pentachlorophenol; Phenol; 2-Chlorophenol; 4-Chloro-3-methylphenol; 4-Nitrophenol *OR (alternative)* 70 Compounds LCS Mix	One per batch of up to 20 samples	Laboratory derived limits Method Specified List: All compounds must pass control criteria, with no exceptions. Full Target List: Marginal exceedances allowed according to the TNI standard.	If LCS and MBs are acceptable, the MS/MSD chromatogram should be reviewed and it may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate *OR (alternative)* Sample Dup	One for every 5% of all environmental samples	Laboratory Derived Limits	Report results with an appropriate footnote.

13.2. **Table 13.2 – Sample Quality Control Criteria**

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Internal Standard	1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Added to all standards, samples, spikes, control samples, and method blanks prior to analysis	**Retention Time:** RT must be ± 30 seconds from last calibration check on all samples	**Retention Time Failure:** If matrix interference is NOT probable, the analytical system must be checked for source of retention time shifting; Affected samples should be reanalyzed in the absence of an obvious instrument or matrix related interference.
Surrogate Standards	Nitrobenzene-d5 2-Fluorobiphenyl Terphenyl-d14 Phenol-d6 2-Fluorophenol 2,4,6-Tribromophenol	Added to all samples, spikes, control samples and method blanks prior to analysis	Laboratory derived limits	1 Base neutral and 1 Acid surrogate are 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. ***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.

14. Data Analysis and Calculations

14.1. **Results Calculation- Aqueous Samples:**

$$\text{Concentration } (\mu\text{g/L}) = \frac{(C_x)(V_x)(DF)}{(V_s)}$$

Where:

C_x = Concentration in extract (µg/mL).
V_v = Volume of final extract (mL).
DF = Dilution factor.
V_s = Volume of water sample extracted (mL).

14.2. **Results Calculation- Soil/Solid Samples:**

$$\text{Concentration } (\mu\text{g/kg}) = \frac{(C_x)(V_x)(1000)(DF)}{(W_s)}$$

Where:

C_x = Concentration in extract (µg/mL).
V_v = Volume of final extract (mL).
DF = Dilution factor.
W_s = Weight of soil sample extracted (g).

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Refer to Tables 11.2, 13.1, and 13.2.

16. Corrective Actions for Out-of-Control Data

16.1. Refer to Tables 11.2, 13.1, and 13.2

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Refer to Tables 11.2, 13.1, and 13.2.

18. Method Performance

18.1. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement) for each matrix per instrument.

18.2. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Orientation and *Training Procedures*, (most current revision or replacement).

18.2.1. Analysis of four (4) replicates of reagent water spiked with 250uL of the 8270 LCS Spiking Solution and 10uL of nNPDA at a concentration of 50ug/L or equivalent to the LCS. The recovery is to be within the current water LCS QC limits for the known concentrations and 30% RSD for all replicates.

18.2.2. Analysis of four (4) replicates of Ottawa sand spiked with 250uL of 8270C LCS Spiking Solution and 10uL of nNPDA at a concentration of 1670ug/kg or equivalent to the LCS. The recovery is to be within the current LCS QC acceptance limits for the known concentration and 30% RSD for all replicates.

19. Method Modifications

19.1. Method modifications for EPA method 8270C are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modification to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- The laboratory follows the DFTPP Tune criteria outlined in EPA 525.2.
- 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.

20. Instrument/Equipment Maintenance

20.1. Please refer to the instrument operations manual or the SOP S-GB-Q-008, *Preventative, Routine, and Non-routine Maintenance* (current revision or replacement).

21. Troubleshooting

21.1. Please refer to the instrument manufacturer operations manual.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. **Equipment:** Portions of the analytical instrumentation operate at high temperatures and under positive pressure. Care must be taken to minimize accidents and injuries when working on or with this equipment. Instruments should be turned off or the heated zone temperatures lowered to reduce the risk of thermal burns. Allow adequate time for the equipment to cool prior to working on these specific zones. The GC pneumatic system uses gas under high pressure. This high pressure introduces the risk of injury due to flying glass and other objects should a vessel or line rupture. Safety glasses are highly recommended at all times when working in, on or around these pieces of equipment. Even instrumentation that is not operating may contain portions of the system under pressure.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-GB-S-006, *Waste Handling and Management* (most current revision or replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

25. References

25.1. USEPA, SW-846, Method 8270C, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), December 1996.

25.2. USEPA, SW-846, Method 8000B, "Determinative Chromatographic Separations", December 1996.

25.3. USEPA, Method 525.2, Revision 2.0 (1995), "Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry".

25.4. Pace Quality Assurance Manual- most current version.

25.5. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.6. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Client Specific Requirements.

26.2. Attachment II: Tailing Factor Calculation

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-O-001-rev.00	Restructured document format. Added tables and other content to create an SOP Template in which local edits are allowed. Modified signature page to include department manager and SOP template number. Updated SOP Template number. Made the tuning criteria red so edits can be made to reflect local tuning criteria used. Updated calibration sections with new calibration policies described in Pace QAM revision 10.0.	28Nov2006
S-ALL-GB-O-001-Rev.00	First Issue using Corporate Template	04Jun2008
S-GB-O-049-Rev.00	Renamed document to follow Document numbering SOP. Renumbered document. Incorporate 1,2,4,5-Tetrachlorobenzene in all sections in document. Included client specific critera and information. Added Attachment I	22Sep2009
S-GB-O-049-Rev.01	Updated Table 10.2 CCC Criteria to 15% RSD acceptance criteria for Non-ccc analytes when CCC analytes are not present Updated SOP references.	11Feb2010
S-GB-O-049-Rev.02	Updated numbering throughout document. Updated references throughout document. Section 14: Updated SOP reference for S-GB-S-006 Waste Handling and Management	06Jul2010
S-GB-O-049-Rev.03	Updated references in sections 10.2.1.2 to 11.6.4, 10.4 to Table 10.2, 10.5.1 to 9.2.4.6, and Section 11.2 to Section 11.2	09Jul2010
SOT-ALL-O-001-rev.01	Converted to current SOT format. Section 6: removed definitions for run sequence and tune period. Section 6.2: added section in red letter text for local edits. Tables 7.1: changed to red text to allow for local lab edits. Table 9.1: changed to Reagents and Stock Standards so that stock standard catalog information could be captured. Table 9.2: changed some sections to red text to allow for local lab edits. Table 10.1: changed compound names to red letter text to allow for local lab edits. Section 10.4.3: changed list of corrective actions to red text to allow for local lab edits. Section 11.13.1: changed "middle" to "upper half". Section 13.3: removed explanation of method modifications section. Section 15: added references 15.3-15.5.	01Aug2011
S-GB-O-049-Rev.04	Throughout Document: Updated References Table 10.2: Added EPA 625 Method Requirement of all compounds requiring 20%.	31Dec2011

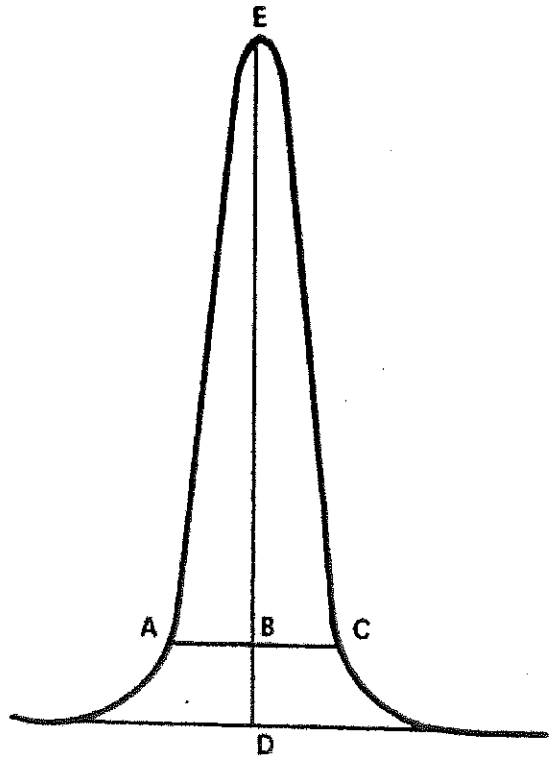
Document Number	Reason for Change	Date
S-GB-O-049-Rev.05	Throughout Document: Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs* Throughout Document: Renamed Tables to be consistent with current Section. Section 11.3: Changed ICV calculation criteria to match CCV calculation criteria. Table 11.Section 12.2.1: Updated DFTPP Tune Criteria to be consistent with EPA 525.2. Section 12.3: Added tailing factor criteria. Section 19: Added Modification in relation to tune criteria. Attachment II: Tailing Factor Calculation added.	30May2013

Attachment I:

Throughout document, reference to Client Specific requirements refers to samples analyzed following: BP Technical Requirements LaMP Revision 10.1, Canadian National Railway Services and Technical Specifications Manual, GE Minimum Standards Revision 2.

Attachment II: DFTPP Tailing Factor Calculation

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$



TAILING FACTOR= $\frac{BC}{AB}$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Figure 13. Tailing factor calculation.

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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Multi EA4000 Instrument

Reference Method: EPA 9060A

SOP NUMBER:	S-GB-I-073-REV.00
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	First Revision

APPROVAL

Nils Melberg, Laboratory General Manager	09/28/12 Date
Kate Grams, Laboratory Quality Manager	9/27/12 Date
Chad Rusch, Department Manager	09/27/2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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Table of Contents

Section **Page**

1. PURPOSE/IDENTIFICATION OF METHOD 3
2. SUMMARY OF METHOD 3
3. SCOPE AND APPLICATION 3
4. APPLICABLE MATRICES 3
5. LIMITS OF DETECTION AND QUANTITATION 3
6. INTERFERENCES 3
7. SAMPLE COLLECTION, PRESERVATION AND HANDLING 3
8. DEFINITIONS 4
9. EQUIPMENT AND SUPPLIES 4
10. REAGENTS AND STANDARDS 5
11. CALIBRATION AND STANDARDIZATION 5
12. PROCEDURE 7
13. QUALITY CONTROL 9
14. DATA ANALYSIS AND CALCULATIONS 10
15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES 12
16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA 13
17. CONTINGENCIES FOR HANDLING OUT-OF CONTROL OR UNACCEPTABLE DATA 13
18. METHOD PERFORMANCE 14
19. METHOD MODIFICATIONS 14
20. SAFETY 14
21. WASTE MANAGEMENT 15
22. POLLUTION PREVENTION 15
23. REFERENCES 15
24. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC. 15
25. REVISIONS 16

1. PURPOSE/IDENTIFICATION OF METHOD

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used to determine the concentration of Total Organic Carbon (TOC) in solid and soil samples using the Multi EA4000 Instrument.

2. SUMMARY OF METHOD

The sample is placed in a combustion furnace where it heats up to 1000 degrees Centigrade The product of the reaction is CO_2 gas, which is then sucked into the non-dispersive infrared detector (NDIR). The NDIR uses electromagnetic radiation or infrared energy to measure the CO_2. This measurement is proportional to the carbon in the sample. In order to analyze for TOC the sample must first have the Inorganic Carbon (IC) removed during the preparation process utilizing acid and heat.

3. SCOPE AND APPLICATION

This SOP is applicable the analysis of Total Organic Carbon (TOC) in soils and solids. The applicable range is MDL to the top standard in the curve

4. APPLICABLE MATRICES

4.1 This SOP is applicable to solid samples.

5. LIMITS OF DETECTION AND QUANTITATION

5.1 Current MDL and RL can be found in the Laboratory Information Management System (EpicPro) or in the Green Bay Wet Chemistry Method Detection Limit Summary.

6. INTERFERENCES

6.1 Carbonate and bicarbonate carbon represent interferences under the terms of this test and must be removed or accounted for in the final calculation.

6.2 This SOP is applicable only to those samples that can be adequately represented with no more than 1g.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Table 1: Sample Collection, Preservation, Shipment, and Storage

Matrix	Method	Container(s)	Preservation	Hold Time	Shipment Conditions	Lab Storage Conditions
Solid	9060A	4 oz Clean glass	None	28 days	On ice, $\leq 6^0$C	$\leq 6^0$C

8. DEFINITIONS

8.1 Total Carbon (TC): The measurement of all the carbon in the sample, both inorganic and organic.

8.2 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by addition of acid and purging: The result is that only organic carbon is left.

8.3 Total Inorganic Carbon (TIC): The measurement of the total inorganic carbon is found by using the TIC module. Acid is added to a sample and measuring the resulting reaction.

8.4 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

9. EQUIPMENT AND SUPPLIES

Table 2: Equipment

Equipment	Manufacturer	Model(s)/Catalog number •
TOC Analyzer	Analytikjena	EA4000 with NDIR detector
Auto-sampler	Analytikjena	TIC Solid Module
Top loading Analytical Balance	Mettler	BA310P
Vented Fume Hood	Hamilton	Fisher
Mortar and Pestle (500mL)	Fisher	S337641
Drying Oven	Yamato	DKN600

- Or equivalent

Table 3: Supplies

Supplies	Manufacturer •	Catalog number
Ceramic Sampling Boats	Analytikjena	886.130
Volumetric Flasks	Fisher	Various
Disposable Transfer pipettes	Eppendorf	Various
Spoonula (Lab Spoon)	Fisher	Various
Gloves – Heat Resistant		

- Or equivalent

10. REAGENTS AND STANDARDS

Table 4: Reagents

Reagent	Concentration	Manufacturer	Catalog #	Directions:	Expiration Date
Nano Pure water	NA	NA	NA	NA	NA
Hydrochloric Acid (HCl)	36-38%	JT Baker	9530-33	NA	1 year from open
Hydrochloric Acid (HCl)	10%	NA	NA	Add 60mL Nanopure water and 20mL 36-38% HCl to Satellite container.	6 month from made date
Compressed Oxygen	Ultra High Purity (UHP)	Airgass		NA	1 year from open

Table 5: Standards

Standard	Concentration	Manufacturer/ Catalog #	Direction:	True Value (Final Conc.)	Acronym
Level 0 Calibration Standard, Initial Calibration Standard, Continuing Calibration Standard, Method Blank	0 g/kg	NA	Ceramic Boat	0 mg/kg	CAL0 ICB CCB MB
Level 1 Calibration Standard and Reporting Limit Verification Standard	120 g/kg CaCO3	Ricca Chemical/ RDCC0050-500B1	Add 5mg CaCO3 to Ceramic Boat	600 mg/kg	CAL1 CRDL
Level 2 Calibration Standard	120 g/kg CaCO3	Ricca Chemical/ RDCC0050-500B1	Add 10mg CaCO3 to Ceramic Boat	1200 mg/kg	CAL2
Level 3 Calibration Standard	120 g/kg CaCO3	Ricca Chemical/ RDCC0050-500B1	Add 50mg CaCO3 to Ceramic Boat	6000 mg/kg	CAL3
Level 4 Calibration Standard and Continuing Calibration Verification Standard	120 g/kg CaCO3	Ricca Chemical/ RDCC0050-500B1	Add 200mg CaCO3 to Ceramic Boat	24000 mg/kg	CAL4 CCV
Level 5 Calibration Standard	120 g/kg CaCO3	Ricca Chemical/ RDCC0050-500B1	Add 400mg CaCO3 to Ceramic Boat	48000 mg/kg	CAL5
Initial Calibration Verification Standard	120 g/kg CaCO3	Fisher Scientific/ C64-500	Add 200mg CaCO3 to Ceramic Boat	1200 mg/kg	ICV
Laboratory Control Spike	120 g/kg CaCO3	Fisher Scientific/ C64-500	Add 200mg CaCO3 to Ceramic Boat	1200 mg/kg	LCS
Matrix Control Spike/Matrix Control Spike Duplicate	120 g/kg CaCO3	Fisher Scientific/ C64-500	Add 200mg CaCO3 to Ceramic Boat	1200 mg/kg	MS MSD

**All neat standards utilize a 5 year from receipt expiration date (if an expiration date is not provided by the manufacturer).

11. CALIBRATION AND STANDARDIZATION

11.1 A new calibration curve should be prepared yearly at minimum or whenever the continuing calibration standard does not pass control criteria.

11.2 Turn on the Furnace unit, main power switch and the **Compressed Oxygen.**

11.3 Enter MultiWin software through the **MultiWin** icon and log in.

11.4 *Activating the instrument.* After software open, initialize analyzer to start furnace.

11.5 *Setting up a new curve.* First select the method being used by going to **Method > Load** if desired method not already in use. Then go to **Measurement > Calibration** and select "yes" to choose a method. Have "calibration with fixed concentration" selected, change number of standards to 5, and put a concentration of 120.000g/kg in the TC field. The blank boat value is going to be the CAL0 point so select measure to set up a new blank. Enter in the exact weights for corresponding ID's, set the boats in the matching number slots on the auto sampler, and hit the Play icon in the menu bar on top to activate all samples. Hit the check mark icon to move to next step. When the temperature is within 50 points of method requirements, the pump will kick in allowing you to hit the **Start** button to begin. Create the following curve:

Table 6: Calibration Information

Standard ID	Volume (mg)	Concentration (g/kg C)	Method ID	ug C (m-Nominal)
CAL0	0	120	TC	0
CAL1	5	120	TC	600
CAL2	10	120	TC	1200
CAL3	50	120	TC	6000
CAL4	200	120	TC	24000
CAL5	400	120	TC	48000

11.6 *Activating the New Curve.* When finished, the window will pop up showing results for each point. Select the points to be used and verify the linear regression for the ug C has a corresponding correlation coefficient > 0.995. The software plots the CAL1 through CAL5 standards using ug C (m-Nominal) versus Integral (I-Net in Area Units (AU)) minus the CAL0. Select the **Link with Method** button, when prompted "Link with the calibration method?" select "No" but then choose the method being used in the window that opens. Hit OK at the bottom of the next screen. Have the circle filled to overwrite the previous calibration and hit the **Accept Values** option on the bottom. Hit close, the calibration is saved and active.

11.7 *Running the New Curve.* To run the curve desired, go to by to **Method > Load** and select the method. The new curve should already be linked.

11.8 *Initial Calibration QC.* Select **Measurement > Start** Measurement. Enter in a folder name and when prompted "Open available rack table" hit no. Enter the Sample names and exact weights and continue as previously did for Calibration to run samples. Analyze the applicable Quality Control (QC) to verify the curve: ICV, ICB, CRDL, CCV, and CCB.

11.9 The calibration report and all analyses should be printed out with all standards traced back to COA and scanned to a file on the desktop labeled the same as the calibration ID.

12. PROCEDURE

12.1 Two methods are used for soil samples, 9060A Quads and 9060A Mods. For 9060A Quads four replicates analyses of the sample are performed; with each replicate, the RSD, and the average being reported. For 9060A Mods two replicate analyses of the sample are performed; with each replicate, the RPD, and the average being reported.

12.2 No injections can be used if they are above the calibration curve. If a sample exceeds the upper end of the calibration curve, a smaller volume of sample must be used and the analysis re-performed.

12.3 Soil Sample Preparation

12.3.1 Weigh the sample directly into a combustion boat and note the weight into the rack table.

12.3.2 Add 10% HCl drop by drop until no further reaction occurs to each pre-weighted sample. Add 2 additional drops to verify the reaction is complete.

Note: The sample must be weighed prior to adding HCl!

12.3.3 Dry the samples in the oven at 103°C for at least 3 hours to remove of excess moisture and HCl.

12.4 Epic Pro/LimsLink.

12.4.1 Samples are batched in Epic Pro.

12.4.2 LimsLink is used as a data reduction tool to evaluate and post data to the LIMS.

12.4.3 Sample weights and instrument raw results are recorded in the instrument software.

12.4.4 Instrument QC and replicate precision are evaluated in LimsLink.

12.4.5 Acceptable data is then posted to EPIC Pro.

12.5 Sample Analysis.

12.5.1 Turn on the furnace unit, main power switch and the Compressed Oxygen

12.5.2 EnterMultiWin software through it's icon.

12.5.3 *Activating the instrument.* After software is open, initialize analyzer to start furnace.

12.5.4 *Each analysis is performed as follows:* Select **Measurement > Start** Measurement. Enter in a folder name and when prompted "Open available rack table" hit no. Enter the Sample names and weights. After samples have dried, load up the auto sampler and hit the play icon for samples set up, then hit the check mark. When the temperature is within range, the pump will start. Hit the **Start** button to begin.

12.5.5 *CCV Analysis:* Weigh 200mg of CAL4 (Primary source CCV standard) in ceramic boat and load into the autosampler. Enter the exact weight into the software program.

12.5.6 *CCB Analysis:* Place a blank boat on the autosampler and enter the weight 1000mg (the maximum weight used). All CCB analysis which detect Integral Area Units (AU) less than the Integral (AU) in the CAL0 of the calibration curve will result in an Integral Area Unit (AU) of 0"B!". The B! signifies this on the Analyses Table raw data print-out.

12.5.7 *CRDL Analysis:* Run a Reporting Limit Verification Standard (CRDL) at the beginning of each batch before the MB/LCS by weighing 5mg of primary standard.

12.5.8 *Batch QC:* When running a Method Blank (MB), load a blank boat and enter 1000mg. A Laboratory Control Spike (LCS) is run by weighing 200mg of secondary stock standard. Matrix Spike / Matrix Spike Duplicate (MS/MSD) are run by adding 50mg of secondary stock standard, after sample has been acidified and dried, along with a variable weight of the parent sample for each the MS and MSD.

12.5.9 *Samples:* For each field sample in an analytical batch, weigh 1g or less into the boat. Run a CCV/CCB every 10 samples and at the end of the run.

12.5.10 While running samples, no injections can be used if they are above the high point in the calibration curve. If an injection is above the curve, weigh out a smaller amount of the sample and reanalyze the injection.

12.5.11 To determine that the sample concentration is less than the reporting limit, at least 1.0000 grams must be utilized.

12.5.12 *Continuing Analytical QC:* Run a CCV/CCB every 10 samples and at the end of the run.

12.5.13 *Printing data:* When the run is complete, the results will open. Select the samples desired to print and hit the print icon. This will print the summary of each rep. Each file selected can be printed seperatly giving a detailed analysis by selecting **Analysis Table > Print Analysis Report.**

12.6 Shutdown

12.6.1 Turn off the furnace unit, main power switch, and the **Compressed Oxygen.**

13. QUALITY CONTROL

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV)** must be run immediately following the instrument calibration. The ICV must be prepared from stock standard obtained from a different source than the stock standard used to prepare the initial calibration and must be prepared at a level at or near the mid range of the calibration curve. The result of the ICV must fall within the range of 90%-110% - if not rerun or recalibrate.

13.3 **Initial Calibration Blank (ICB)** – An ICB is analyzed after the ICV and must meet the acceptance criteria before samples are analyzed. The absolute value of the ICB result must be less than the Pace Reporting Limit (PRL) – if not rerun or recalibrate.

13.4 **Continuing Calibration Verification (CCV)** - Each analytical run must begin and end with a CCV and a CCV must be run every 10 samples. The CCV must be prepared from the primary source as the mid-point calibration standard and the result must fall within the range of 90%-110%. If outside the range, rerun once. If still outside the range, the problem must be corrected before continuing and the affected samples reanalyzed.

13.5 **The Continuing Calibration Blank (CCB)** – A CCB is analyzed after the CCV and must meet the acceptance criteria before samples are analyzed. The absolute value of the CCB result must be less than the PRL. If outside the range, rerun once. If still outside the range, correct the problem and rerun all samples back to the last passing CCB. All CCB analysis which detect Integral Area Units (AU) less than the Integral (AU) in the CAL0 of the calibration curve will result in an Integral Area Unit (AU) of 0"B!". The B! signifies this on the Analyses Table raw data print-out.

13.6 **Method Blank** - A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples whichever is more frequent. The data obtained is used to assess contamination from the laboratory environment. Values that exceed the PRL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. Values that are between the MDL and PRL are evaluated based on the concentration in the samples compared to the concentration in the blank.

13.7 **Laboratory Control Sample (LCS)** - A LCS prepared from an independent source must be performed with every batch or every 20 environmental samples, whichever is more frequent. The LCS must be evaluated for accuracy and must meet the laboratory generated control limits before samples are analyzed. If no in-house limits are in use, then a range of 80 - 120% will be used. If the LCS recovery does not meet the acceptance criteria corrective action must be taken and the LCS recovery must be acceptable before proceeding with sample analysis. A LCSD is performed when requested by the client or when there is insufficient sample volume to perform a matrix spike / matrix spike duplicate. The LCSD must be evaluated to the same criteria as the LCS. The LCS/LCSD must be evaluated for precision and must meet the laboratory generated control criteria. Should the LCS and/or the LCSD fail either accuracy or precision criteria, it can be re-analyzed once. Should they fail a second time, find and correct the problem, then reanalyze the entire batch.

13.8 **Matrix spike/Matrix spike duplicate** - A MS/MSD pair must be prepared and analyzed every 10 samples. The MS must be a duplicate of the aliquot used for sample analysis. Calculate the percent recovery, corrected for concentration measured in the parent sample, and compare to the in-house generated limits. If no in-house limits are in use then a range of 80 - 120% will be used. Calculate the precision between the MS and MSD and compare to the in-house generated limits. If no in-house limits are in use, then an upper limit of 20% will be used.

13.9 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and after the initial CCV/CCB of each new batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.10 **MOD Replicate Precision (RPD)** - The Relative Percent Difference is calculated between the two replicates used to calculate the average. If the RPD resulting from the two reported replicates is 40% or greater, two additional replicates are analyzed. The calculated results of each of these two additional replicates are reported in a qualifier for the reported RPD.

13.11 **Quad Replicate Precision (RSD)** - The Relative Standard Deviation is calculated for the four replicates used to calculate the average. If the RSD resulting from the four reported replicates is 40% or greater, a qualifier for the reported RSD is added.

13.12 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

13.13 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

13.14 See Tables 7 and 8 for a summary of QC.

14. DATA ANALYSIS AND CALCULATIONS

14.1 All data produced by the EA4000 must be processed through the appropriate curve.

$$c = (k1 * I + k0) / V$$

Where:

c is the final concentration (mg/kg)
k0 and k1 are constants derived from the calibration curve
I is the integral area units
V is the sample volume (mg)

14.2 Final concentration is exported to LIMS Link where it is back calculated to get the posted LIMS Link value.

$$\text{Final Concentration (mg/kg)} \times \left(\frac{\text{Initial weight (mg)}}{1000\text{mg}}\right) = \text{posted LIMSLink value (mg)}$$

14.3 Calculations in LIMS

Injection 1:
Soil:

$$\text{LIMS Link result (mg)} \times \left(\frac{1000\ (\text{mg})}{\text{Sample weight (g)}}\right) = \text{TOC (mg/Kg) (TOC1)}$$

Injection 2:
Soil:

$$\text{LIMS Link result (mg)} \times \left(\frac{1000\ (\text{mg})}{\text{Sample weight (g)}}\right) = \text{TOC (mg/Kg) (TOC2)}$$

Injection 3:
Soil:

$$\text{LIMS Link result (mg)} \times \left(\frac{1000\ (\text{mg})}{\text{Sample weight (g)}}\right) = \text{TOC (mg/Kg) (TOC3)}$$

Injection 4:
Soil:

$$\text{LIMS Link result (mg)} \times \left(\frac{1000\ (\text{mg})}{\text{Sample weight (g)}}\right) = \text{TOC (mg/Kg) (TOC4)}$$

14.4 Average sample result reported as mg/kg.

14.4.1 Mod Average result = ((TOC1) + (TOC2))/2

14.4.2 Quad Average result = ((TOC1) + (TOC2)+(TOC3)+(TOC4))/4

14.5 The result in mg/kg is divided by 10,000 to express the result as a percentage (%w/w).

14.6 MS/MSD calculation

$$\%\ \text{Recovery} = \left(\frac{\text{SSR} - \text{SR}}{\text{SA}}\right) \times 100$$

Where:
SSR = Spike sample result
SR = Sample result
SA = Spike added

14.7 Precision (for MOD replicates)

The Relative Percent Difference is calculated between the two replicates used to calculate the average.

$$\text{Replicate \% RPD} = \frac{|\text{TOC1} - \text{TOC2}|}{(\text{TOC1}+\text{TOC2})/2} \times 100$$

14.8 Precision (for Quad replicates)

The Relative Standard Deviation is calculated for the four replicates used to calculate the average.

$$\text{Mean} = (TOC1+TOC2+TOC3+TOC4)/4$$

14.9 Std Deviation = $\sqrt{(((TOC1-Mean)^2+(TOC2-Mean)^2+(TOC3-Mean)^2+(TOC4-Mean)^2)/3)}$

14.10 Relative Standard Deviation = (Standard Deviation/Mean)*100

14.11 Precision (for QC samples):

The precision is calculated based on the % recovery of the matrix spike / matrix spike duplicate (MS/MSD) result.

$$\text{QC Sample \% RPD} = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

15. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURES

Table 7: Quality Control/Acceptance Criteria

Preparation Method ⇨ Quality Control Measure ⇩	**SW846 9060** **Frequency**	**Acceptance Criteria**
Method Blank	One per batch of samples, up to 20m whichever is more frequent.	Project Specific or Less than the RL (Lowest curve standard.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20, whichever is more frequent. An LCSD is required if MS/MSD is not performed.	Project Specific or For solids 80 –120% with 20% RPD
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	Project Specific or For solids 80 –120% with 20% RPD
Initial Calibration	Minimum of 4 standards plus blank at least once per year.	Correlation Coefficient of 0.995
CRDL	After the calibration and following the initial CCV/CCB in each batch..	60-140%
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.	Project specific or Recovery between 90 – 110%
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.	Project specific or Less than RL (Lowest curve standard)

16. CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

Table 8: Data Assessment

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**TOC If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
CRDL standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the MS or MSD fail because the concentration of the spike is less than 25% of the concentration of the parent, use appropriate flag for the parent sample. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD following the above guidance. If the concentration of the spike is greater than 25% of the concentration of the parent, use appropriate flag for the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate precision data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration and following the initial CCV/CCB in each batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

17. CONTINGENCIES FOR HANDLING OUT-OF CONTROL OR UNACCEPTABLE DATA

17.1 See section 16.

18. METHOD PERFORMANCE

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*, most current revision or replacement. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

18.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ*, most current revision or replacement, for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

18.1.4 Linear Calibration Range - The linear calibration range must be determined initially and verified whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. If any verification data exceeds the initial values by $\pm$ 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

18.1.5 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program*, most current revision or replacement, to demonstrate continuing competence. All results are stored in the QA office.

19. METHOD MODIFICATIONS

19.1 Method modifications for SW846 9060A are as follows:

19.1.1 SW846 9060A is an aqueous method, the laboratory has modified the method to accommodate solid matrices.

19.1.2 SW846 9060A requires the use of quadruplicate analysis. The laboratory has modified the method to additionally report duplicate replicates for states which allow for the modification.

20. SAFETY

20.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

20.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

20.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the most current revision or replacement of S-GB-S-006, *Waste Handling and Management.*

20.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

21. WASTE MANAGEMENT

21.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

21.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the most current revision or replacement of S-GB-S-006, *Waste Handling and Management.*

22. POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

24.3 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

23. REFERENCES

23.1 PASI Quality Manual, current revision

23.2 USEPA Test Methods for Evaluating Solid Wastes, SW 846, 3rd Edition, Methods 9060A.

23.3 USEPA Region 9, 9060dqi, Total Organic Carbon in Soil, SW 846 9060.

23.4 Multi EA4000 Operation Manual

24. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

24.1 Attachment A: **Total Organic Carbon Calculation Forms (MOD and QUAD)**

25. REVISIONS

Revision Number	Reason for Change	Date
S-GB-I-073-REV.00	First Revision	20Sept2012

Attachment A: Total Organic Carbon Calculation Sheets (MOD AND QUAD)

Pace Analytical Services, Inc - Green Bay

WETA:
HBN:

Sample ID						
	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	RPD
Re 1		1		#DIV/0!	#DIV/0!	
Re 2		1		#DIV/0!	#DIV/0!	
Average				#DIV/0!	#DIV/0!	
Posted	**#DIV/0!**		**#DIV/0!**			**#DIV/0!**
			Epic Pro Calc =		#DIV/0!	

Sample ID						
	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	RPD
Re 1		1		#DIV/0!	#DIV/0!	
Re 2		1		#DIV/0!	#DIV/0!	
Average				#DIV/0!	#DIV/0!	
Posted	**#DIV/0!**		**#DIV/0!**			**#DIV/0!**
			Epic Pro Calc =		#DIV/0!	

Sample ID						
	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	RPD
Re 1		1		#DIV/0!	#DIV/0!	
Re 2		1		#DIV/0!	#DIV/0!	
Average				#DIV/0!	#DIV/0!	
Posted	**#DIV/0!**		**#DIV/0!**			**#DIV/0!**
			Epic Pro Calc =		#DIV/0!	

Sample ID						
	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	RPD
Re 1		1		#DIV/0!	#DIV/0!	
Re 2		1		#DIV/0!	#DIV/0!	
Average				#DIV/0!	#DIV/0!	
Posted	**#DIV/0!**		**#DIV/0!**			**#DIV/0!**
			Epic Pro Calc =		#DIV/0!	

Sample ID						
	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	RPD
Re 1		1		#DIV/0!	#DIV/0!	
Re 2		1		#DIV/0!	#DIV/0!	
Average				#DIV/0!	#DIV/0!	
Posted	**#DIV/0!**		**#DIV/0!**			**#DIV/0!**

Attachment A: Total Organic Carbon Calculation Sheets (MOD AND QUAD) Cont.

Pace Analytical Services - Green Bay

Sample ID:

	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	Difference from the mean	DifferenceSquared	Standard Deviation	Relative Standard Deviation
Re 1		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 2		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 3		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 4		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Average				#DIV/0!	#DIV/0!			#DIV/0!	#DIV/0!
Posted	**#DIV/0!**		**#DIV/0!**						
			Epic Pro Calc =		#DIV/0!				

Sample ID:

	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	Difference from the mean	DifferenceSquared	Standard Deviation	Relative Standard Deviation
Re 1		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 2		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 3		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 4		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Average				#DIV/0!	#DIV/0!			#DIV/0!	#DIV/0!
Posted	**#DIV/0!**		**#DIV/0!**						
			Epic Pro Calc =		#DIV/0!				

Sample ID:

	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	Difference from the mean	DifferenceSquared	Standard Deviation	Relative Standard Deviation
Re 1		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 2		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 3		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 4		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Average				#DIV/0!	#DIV/0!			#DIV/0!	#DIV/0!
Posted	**#DIV/0!**		**#DIV/0!**						
			Epic Pro Calc =		#DIV/0!				

Sample ID:

	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	Difference from the mean	DifferenceSquared	Standard Deviation	Relative Standard Deviation
Re 1		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 2		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 3		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 4		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Average				#DIV/0!	#DIV/0!			#DIV/0!	#DIV/0!
Posted	**#DIV/0!**		**#DIV/0!**						
			Epic Pro Calc =		#DIV/0!				

Sample ID:

	Initial Weight	Final Volume	Inst Result	Calc Result	Calc RDL	Difference from the mean	DifferenceSquared	Standard Deviation	Relative Standard Deviation
Re 1		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 2		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 3		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Re 4		1		#DIV/0!	#DIV/0!	#DIV/0!	#DIV/0!		
Average				#DIV/0!	#DIV/0!			#DIV/0!	#DIV/0!
Posted	**#DIV/0!**		**#DIV/0!**						
			Epic Pro Calc =		#DIV/0!				

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STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Apollo 9000 Instrument

Reference Method: EPA 9060A

SOP NUMBER:	S-GB-I-059-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-059-REV.03

APPROVAL

Nils Melberg, Laboratory General Manager	09/28/12 Date
Kate Grams, Laboratory Quality Manager	9/26/12 Date
Chad Rusch, Department Manager	09/27/2012 Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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Table of Contents

Section **Page**

1. PURPOSE 3
2. SCOPE AND APPLICATION 3
3. SUMMARY OF METHOD 3
4. INTERFERENCES 3
5. SAFETY 3
6. DEFINITIONS 4
7. SAMPLE COLLECTION, PRESERVATION AND HANDLING 4
8. REAGENTS AND STANDARDS 4
9. EQUIPMENT AND SUPPLIES 5
10. CALIBRATION AND STANDARDIZATION 6
11. PROCEDURE 7
12. QUALITY CONTROL 10
13. METHOD PERFORMANCE 11
14. POLLUTION PREVENTION AND WASTE MANAGEMENT 12
15. REFERENCES 12
16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC. 13
17. REVISIONS 13

1. PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures used to determine the concentration of Total Organic Carbon (TOC) in solid and soil samples using the Apollo 9000 Instrument.

2. SCOPE AND APPLICATION

This SOP is applicable the analysis of Total Organic Carbon (TOC) in soils and solids. The applicable range is MDL to the top standard in the curve. The current Pace Reporting Limit is 250 mg/kg. The current MDL can be found in Epic Pro.

3. SUMMARY OF METHOD

The sample is placed in a combustion furnace where it heats up and reacts with the catalyst that is contained in the furnace. The product of the reaction is CO_2 gas, which is then blown into the non-dispersive infrared detector (NDIR). The NDIR uses electromagnetic radiation or infrared energy to measure the CO_2. This measurement is proportional to the carbon in the sample. In order to analyze for TOC the sample must first have the Inorganic Carbon (IC) removed during the preparation process utilizing acid and heat.

4. INTERFERENCES

4.1 Carbonate and bicarbonate carbon represent interferences under the terms of this test and must be removed or accounted for in the final calculation.

4.2 This SOP is applicable only to those samples that can be adequately ground with mortar and pestle to result in a fairly homogenous sample.

5. SAFETY

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the most current revision or replacement of S-GB-W-001, *Waste Handling and Management.*

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6. DEFINITIONS

6.1 Total Carbon (TC): The measurement of all the carbon in the sample, both inorganic and organic.

6.2 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by addition of acid and purging: The result is that only organic carbon is left.

6.3 Total Inorganic Carbon (TIC): The measurement of the total inorganic carbon is found by subtracting the Total Organic Carbon from the Total Carbon. TIC = TC – TOC.

6.4 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

7. SAMPLE COLLECTION, PRESERVATION AND HANDLING

Table 1. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Solid	9060A	4 oz Clean glass containers	None 28 days	On ice 4+/- 2° Celsius	≤6^{0}C

8. REAGENTS AND STANDARDS

8.1 Reagents

8.1.1 Sulfuric Acid (H_2SO_4) – concentrated

8.1.2 Sulfuric Acid (H_2SO_4) – 10%

To make: Fill 2000mL volumetric flask with 1000mL of Nano Pure Water. Measure out 200mL of concentrated Sulfuric Acid and add to volumetric flask. Dilute to volume. Shelf life = 6 months.

8.1.3 Nano Pure water

8.1.4 Purified Compressed Air

8.2 Standards (Equivalent pre-made standards or Potassium hydrogen phthalate may be purchased and used).

8.2.1 Soil Calibration Standard 0 – 0 mg/L

10mL Nano-Pure water.

8.2.2 Soil Calibration Standard 3 – 1000mg/L

1000 mg/L primary stock standard, purchased. (SCP Science, 250-250-051)

8.2.3 Soil Calibration Standard 1 and Reporting Limit Verification Standard (CRDL) – 250mg/L
Add 2.5 mL of 1000 mg/L primary standard to a 10mL volumetric flask and bring to final volume with Nano-Pure water.
Soil Calibration Standard 2 – 500mg/L

8.2.4 Add 5 mL of 1000 mg/L primary standard to a 10mL volumetric flask and bring to final volume with Nano-Pure water.

8.2.5 Soil Calibration Standard 4– 2000mg/L
2000 mg/L primary stock standard, purchased. (SCP Science, 260-000-043)

8.2.6 Soil Initial Calibration Verification (ICV) –1000mg/L
1000 mg/L secondary stock standard, purchased. (Ultra Scientific, IQC-106-5)

8.2.7 Laboratory Control Spike (LCS) – Spiked from the secondary source standard.

8.2.8 Soil Continuing Calibration Verification (CCV) –1000mg/L
Same as Soil Calibration Standard 3 - 1000 mg/L primary stock standard, purchased. (SCP Science, 250-250-051)

9. EQUIPMENT AND SUPPLIES

9.1 Equipment

9.1.1 Tekmar / Dohrmann Apollo 9000 TOC analyzer with NDIR detector

9.1.2 TOC Boat Analyzer – Rosemount/Dohrmann

9.1.3 Calibrated Mechanical Pipettes –Assorted adjustable air displacement pipettes with disposable tips - Eppendorf or equivalent

9.1.4 Analytical syringe – 500uL

9.1.5 Analytical syringe – 50uL

9.1.6 Hot Block

9.1.7 Analytical balance – accurate to 0.0001gram

9.1.8 Mortar & Pestle

9.1.9 Spatula

9.1.10 Volumetric flasks

9.1.11 Oven

9.2 **Supplies**

9.2.1 Aluminum dishes

9.2.2 40 mL VOA vials

9.2.3 Digestion Tubes

9.2.4 Data printout paper

10. CALIBRATION AND STANDARDIZATION

10.1 Turn on the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

10.2 Enter **TOC/Apollo 9000** software through the **Talk 4.2 Boat** icon.

10.3 *Activating the instrument.* Go to the **Setup** dropdown and select **Instrument**. Go to the **System** portion and select **Ready**. A bubbling sound will be made, signaling the instrument is not hibernating.

10.4 *Setting up a new curve.* Go to **Setup** dropdown and select **Calibration** then **Standards**. This is where the new calibration and the calibration points are set. Through the **Edit** drop down rows can be added, deleted, or copied and pasted. Create the following curve:

Table 2. Calibration Information

Standard ID	Concentration (ppmC)	Method ID	ugC	Comments
0ppmC	0	Boat Sampler	0.0000	
250ppmC	250	Boat Sampler	10.0000	
500ppmC	500	Boat Sampler	20.0000	
1000ppmC	1000	Boat Sampler	40.0000	
2000ppmC	2000	Boat Sampler	80.0000	

The **Cal. Curve ID** will be filled in when **File, Save As** is done. Save the file as the date that the calibration will be done. Click **OK** to exit.

10.5 *Activating the New Curve.* Set the new curve as the active curve by going to the **Setup** dropdown and selecting **Calibration,** then **Set Active,** and **TOC Curves**. From the popup set the **Aux** to the calibration curve just set up. Leave all the other fields set to **Default**. **OK** will close the popup.

10.6 *Running the New Curve.* Click **Run** then **Sample Setup** and change the sample type to **TOC Standard**. In the popup select **Aux** and then click **OK**. In the new popup select the level that is to be run by highlighting it. Number of Repeats should be 1, Method ID is Boat Sampler, Mode is TOC, and the Calibration Curve should reflect the new curve. Click Save/Use. **Click Start** and enter sample weight (40uL for standards and blanks) and follow the prompts to inject the boat. After a calibration point is run a calibration popup will appear showing each point that has been analyzed. From this popup different points can be selected, the curve calculated, and the calibration report printed. This popup is closed by clicking **OK**.

10.7 *Rest of Calibration Points*. Repeat 10. 6 for the rest of the calibration points. For each point **TOC Standard** will need to be selected even though it is already showing. This is done to initiate the next popup.

10.8 When four standards have been read click on menu command **Results, Calibration**. Select the calibration curve ID. This is the same as the popup that shows after each calibration point. Locate and check the calibration points just performed, all others should not be checked. Click "**Recalc**" button. This will create a calibration curve. The curve will be 1st order linear. The r-sqr value must be greater than 0.995, if not, recalibrate.

10.9 *Initial Calibration QC*. Change the **Sample Type** to **Sample** and analyze the applicable QC: ICV, ICB, CRDL, CCV, and CCB.

10.10 The calibration report and all analyses should be printed out with all standards traced back to COA and scanned to a file on the desktop labeled the same as the calibration ID.

11. PROCEDURE

11.1 Two methods are used for soil samples, 9060A Quads and 9060A Mods. For 9060A Quads four replicates analyses of the sample are performed with each replicate, the RSD, and the average being reported. For 9060A Mods two replicate analyses of the sample are preformed with each replicate the RPD, and the average being reported.

11.2 No injections can be used if they are above the curve. A smaller amount of sample must be used and rerun.

11.3 Soil Sample Preparation

11.3.1 Take an approximate sub-sample of 10 g of soil, add 10% Sulfuric Acid and place in a digestion block at 70°C. After the sample does not react with acid, It is filtered and then baked in an oven at 103°C for a minimum of 18 hours.

11.3.2 Crush and grind the sample with a mortar and pestle to form a homogenous sample.

11.4 Epic Pro/LimsLink.

11.4.1 Samples are batched in Epic Pro.

11.4.2 LimsLink is used to aquire and evaluate data as it is generated.

11.4.3 Sample weights and instrument raw results are recorded in LimsLink.

11.4.4 Instrument QC and replicate precision are evaluated in LimsLink.

11.4.5 Acceptable data is then posted to EPIC Pro.

11.5 Sample Analysis.

11.5.1 Turn on the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

11.5.2 Enter **TOC/Apollo 9000** software through the **Talk 4.2 Boat** icon.

11.5.3 *Activating the instrument.* Go to the **Setup** dropdown and select **Instrument**. Go to the **System** portion and select **Ready**. A bubbling sound will be made, signaling the instrument is not hibernating.

11.5.4 *Each analysis is performed as follows:* Click **Run** then **Sample Setup** and change the **Sample ID** to what is being analyzed. Sample Type should be **Sample**. Number of Repeats should be 1, Method ID is Boat Sampler, Mode is TOC, and the Calibration Curve should reflect the curve being used. Click Save/Use. **Click Start** and enter sample weight (40 µL for standards and blanks) and follow the prompts to inject the boat. Once the instrument beeps and displays the result from the injection, slide the boat out of the oven and wait at least one minute for the boat to cool down.

11.5.5 *CCV Analysis:* Inject 40 µL of the 1000mg/L Soil Continuing Calibration Verification (CCV) standard into the boat through the septa using a 50-µL syringe.

11.5.6 *CCB Analysis:* Inject 40µL of Nano Pure H_2O (CCB).

11.5.7 *Batch QC:* Run a Reporting Limit Verification Standard (CRDL) at the beginning of each batch before the MB/LCS by injecting 40µL of prepared 250mg/L CRDL standard. When running a Method Blank (MB), 40 µL of Nano Pure water is injected into the boat. A Laboratory Control Spike (LCS) is run by injecting 40 µL of the secondary stock standard . Matrix Spike / Matrix Spike Duplicate (MS/MSD) are run by injecting 20 µL of the 1000mg/L secondary stock standard along with a variable weight of the parent sample for each the MS and MSD.

11.5.8 *Samples:* For each field sample in an analytical batch, weigh 0.04g or less into the boat. Run a CCV/CCB every 10 samples and at the end of the run.

11.5.9 While running samples, no injections can be used if they are above the curve. If an injection is above the curve, weigh out a smaller amount of the sample and rerun that injection.

11.5.10 To determine that the sample concentration is less than the reporting limit, at least 0.04 grams must be utilized.

11.5.11 *Continuing Analytical QC:* Run a CCV/CCB every 10 samples and at the end of the run.

11.5.12 *Printing data:* After the run has completed go to the **Results** drop down and select **Samples**. From here select the **File** dropdown and **Clear**. Then select the **File** drop down and Open **Results From Day**. Type in the year, month and day the samples were analyzed and then click **OK**. The samples will then appear. From the **File** dropdown select both **Print Summary Report** and **Print Detail Report**. Click **Exit** and **No** to Save Changes.

11.6 Shutdown

11.6.1 Slide boat into oven.

11.6.2 Turn off the **TOC Boat Sampler** power (oven) and the **Compressed Air**.

11.7 Calculations:

11.7.1 All data produced by the Apollo 9000 must be processed through the appropriate curve.

Injection 1:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (ml)}{\text{Injection weight (g)}} \times \frac{1000\ (g)}{1\ (Kg)} \times \frac{1(L)}{1000\ (ml)} = \text{TOC (mg/Kg) (TOC1)}$$

Injection 2:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (ml)}{\text{Injection weight (g)}} \times \frac{1000\ (g)}{1\ (Kg)} \times \frac{1\ (L)}{1000\ (ml)} = \text{TOC (mg/Kg) (TOC2)}$$

Injection 3:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (ml)}{\text{Injection weight (g)}} \times \frac{1000\ (g)}{1\ (Kg)} \times \frac{1\ (L)}{1000\ (ml)} = \text{TOC (mg/Kg) (TOC3)}$$

Injection 4:
Soil:

$$\text{Raw Data Value (mg/L)} \times \frac{0.04\ (ml)}{\text{Injection weight (g)}} \times \frac{1000\ (g)}{1\ (Kg)} \times \frac{1\ (L)}{1000\ (ml)} = \text{TOC (mg/Kg) (TOC4)}$$

11.7.2 Average sample result (mg/kg)

$$\text{Mod Average result} = \frac{(TOC1) + (TOC2)}{2}$$

$$\text{Quad Average result} = \frac{(TOC1) + (TOC2)+(TOC3)+(TOC4)}{4}$$

Note that soils are dried to 100% dry weight before analysis – therefore dry weight correction is not needed.

11.7.3 The result in mg/kg is divided by 10,000 to express the result as a percentage (%).

11.7.4 MS/MSD calculation

$$\%\ \text{Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

SSR = Spike sample result
SR = Sample result
SA = Spike added

11.7.5 Precision (for MOD replicates)

The Relative Percent Difference is calculated between the two replicates used to calculate the average.

$$\text{Replicate \% RPD} = \frac{|TOC1 - TOC2|}{(TOC1+TOC2)/2} \times 100$$

11.7.6 Precision (for Quad replicates)

The Relative Standard Deviation is calculated for the four replicates used to calculate the average.

Mean = (TOC1+TOC2+TOC3+TOC4)/4

Standard Deviation = √(((TOC1-Mean)+(TOC2-Mean)+(TOC3-Mean)+(TOC4-Mean))/3)

Relative Standard Deviation = (Standard Deviation/Mean)*100

11.7.7 Precision (for QC samples):

The precision is calculated based on the % recovery of the matrix spike / matrix spike duplicate (MS/MSD) result.

$$\text{QC Sample \% RPD} = \frac{|MS-MSD|}{(MS+MSD)/2} \times 100$$

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 **Initial Calibration Verification (ICV)** must be run immediately following the instrument calibration. The ICV must be prepared from stock standard obtained from a different source than the stock standard used to prepare the initial calibration and must be prepared at a level at or near the mid range of the calibration curve. The result of the ICV must fall within the range of 90%-110% - if not rerun or recalibrate.

12.3 **Initial Calibration Blank (ICB)** – An ICB is analyzed after the ICV and must meet the acceptance criteria before samples are analyzed. The absolute value of the ICB result must be less than the Pace Reporting Limit (PRL) – if not rerun or recalibrate.

12.4 **Continuing Calibration Verification (CCV)** - Each analytical run must begin and end with a CCV and a CCV must be run every 10 samples. The CCV must be prepared from the same solution as the mid-point calibration standard and the result must fall within the range of 90%-110%. If outside the range, rerun 1X. If still outside the range, the problem must be corrected before continuing and the affected samples reanalyzed.

12.5 **The Continuing Calibration Blank (CCB)** – A CCB is analyzed after the CCV and must meet the acceptance criteria before samples are analyzed. The absolute value of the CCB result must be less than the PRL. If outside the range, rerun 1 time. If still outside the range, correct the problem and rerun all samples back to the last passing CCB.

12.6 **Method Blank** - A Method Blank (MB) must be prepared and analyzed with every sample batch or every 20 samples whichever is more frequent. The data obtained is used to assess contamination from the laboratory environment. Values that exceed the PRL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis. Values that are between the MDL and PRL are evaluated based on the concentration in the samples compared to the concentration in the blank.

12.7 **Laboratory Control Sample (LCS)** - A LCS prepared from an independent source must be performed with every batch or every 20 environmental samples, which ever is more frequent. The LCS must be evaluated for accuracy and must meet the laboratory generated control limits before samples are analyzed. If no in-house limits are in use, then a range of 80 - 120% will be used. If the LCS recovery does not meet the acceptance criteria corrective action must be taken and the LCS recovery must be acceptable before proceeding with sample analysis. A LCSD is performed when requested by the client or when there is insufficient sample volume to perform a matrix spike / matrix spike duplicate. The LCSD must be evaluated to the same criteria as the LCS. The LCS/LCSD must be evaluated for precision and must meet the laboratory generated control criteria. Should the LCS and/or the LCSD fail either accuracy or precision criteria, it can be re-analyzed once. Should they fail a second time, find and correct the problem, then reanalyze the entire batch.

12.8 **Matrix spike/Matrix spike duplicate** - A MS/MSD pair must be prepared and analyzed every 10 samples. The MS must be a duplicate of the aliquot used for sample analysis. Calculate the percent recovery, corrected for concentration measured in the parent sample, and compare to the in-house generated limits. If no in-house limits are in use then a range of 80 - 120% will be used. Calculate the precision between the MS and MSD and compare to the in-house generated limits. If no in-house limits are in use, then an upper limit of 20% will be used.

12.9 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and after the initial CCV/CCB of each new batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

12.10 **MOD Replicate Precision (RPD)** - The Relative Percent Difference is calculated between the two replicates used to calculate the average. If the RPD resulting from the two reported replicates is 40% or greater, two additional replicates are analyzed. The calculated results of each of these two additional replicates are reported in a qualifier for the reported RPD.

12.11 **Quad Replicate Precision (RSD)** - The Relative Standard Deviation is calculated for the four replicates used to calculate the average. If the RSD resulting from the four reported replicates is 40% or greater, a qualifier for the reported RSD is added.

12.12 When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

12.13 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

12.14 See attachments Table A and Table B for a summary of QC.

13. METHOD PERFORMANCE

13.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*(most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Linear Calibration Range - The linear calibration range must be determined initially and verified whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear. If any verification data exceeds the initial values by $\pm$ 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

13.1.5 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

14.2 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the most current revision or replacement of S-GB-W-001, *Waste Handling and Management.*

14.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

15. REFERENCES

15.1 PASI Quality Manual, current revision

15.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version

15.3 USEPA Test Methods for Evaluating Solid Wastes, SW 846, 3rd Edition, Methods 9060A.

15.4 Apollo 9000 TOC Combustion Analyzer User Manual

16. TABLES, DIAGRAMS, FLOWCHARTS, APPENDICES, ADDENDA ETC.

16.1 Table A: **QUALITY CONTROL**

16.2 Table B: **ANALYST/TECHNICIAN DATA ASSESSMENT**

17. REVISIONS

Revision Number	Reason for Change	Date
S-GB-I-059-Rev.00	First Issue.	09Jan2009
S-GB-I-059-Rev.01	Updated Method Reference to SW846-9060A throughout document.	20May2009
S-GB-I-059-Rev.02	Updated Signature Page Section 8.2: Updated calibration information. Section 11.5.13: Removed Section 11.7: Updated calculations to include multiple replicates and manner to average replicates for final TOC value. Section 12.0: Added additional criteria for RSD of Quadruplicate analysis and RPD criteria for Mod analysis. Throughout document: Updated instances of the language "EQL" with "PRL".	08Nov2010
S-GB-I-059-Rev.03	Updated Signature Page Section 8.2: Updated calibration information Throughout document: Updated instances of the language "RLVS" with "CRDL" Throughout document: Updated instances of the language "DI" with "Nano Pure"	24Aug2011
S-GB-I-059-Rev.04	Updated Signature Page Section 12.7 – Added that LCSD must pass corrective action and added corrective action taken Section 13.1.5 – Updated SOP reference Section 15: Added NELAC Reference.	26Sept2012

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**TOC**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. A LCSD is required if MS/MSD is not performed.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.
Initial Calibration	Minimum of 4 standards plus blank.
CRDL	After the calibration and following the initial CCV/CCB in each batch..
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**TOC If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
CRDL standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the concentration of the spike is less than 25% of the concentration of the parent, accuracy need not be calculated. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate (precision) data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifier to reflect the deficiencies.

4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration and following the initial CCV/CCB in each batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

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STANDARD OPERATING PROCEDURE

EVALUATION AND QUALIFICATION OF VENDORS

Reference Methods: N/A

Local SOP Number:	S-GB-Q-018-Rev.02
Effective Date:	Date of Final Signature
Supersedes:	S-GB-Q-018-Rev.01
SOP Template Number:	SOT-ALL-Q-027-rev.04

APPROVALS

Nils K Melberg — 01/07/14
Nils Melberg, Laboratory General Manager — Date

Kate E. Grams — 1/6/14
Kate Grams, Laboratory Quality Manager — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 3
5. Limits of Detection and Quantitation 3
6. Interferences 3
7. Sample Collection, Preservation, Shipment and Storage 3
8. Definitions 3
9. Equipment and Supplies (Including Computer Hardware and Software) 4
10. Reagents and Standards 4
11. Calibration and Standardization 4
12. Procedure 4
13. Quality Control 9
14. Data Analysis and Calculations 9
15. Data Assessment and Acceptance Criteria for Quality Control Measures 9
16. Corrective Actions for Out-of-Control Data 9
17. Contingencies for Handling Out-of-Control or Unacceptable Data 10
18. Method Performance 10
19. Method Modifications 10
20. Instrument/Equipment Maintenance 10
21. Troubleshooting 10
22. Safety 10
23. Waste Management 10
24. Pollution Prevention 10
25. References 10
26. Tables, Diagrams, Flowcharts, and Validation Data 10
27. Revisions 11

1. Purpose/Identification of Method

1.1. The purpose of this Standard Operating Procedure (SOP) is to describe the process by which vendors are qualified for use by Pace Analytical Services (PASI).

2. Summary of Method

2.1. PASI vendors are evaluated to determine their qualifications for providing their goods or services. In general, vendors are first assessed to determine their level of importance based on the degree of impact and analytical results should they fall short in providing quality products or services. Once determined to be a critical or non-critical supplier, a vendor's qualifications are assessed across several categories that include quality, financial stability and business terms, supply chain capabilities and customer service strengths. Separate qualification requirements exist for each of the general vendor categories. These categories are identified in the Procedures section below.

3. Scope and Application

3.1. **Personnel**: These procedures are applicable to all Pace personnel responsible for the evaluation and qualification of vendors.

3.2. This SOP is applicable to the evaluation and qualification of vendors to PASI. These vendors include, but are not limited to, laboratory product suppliers, chemical and standard manufacturers, durable goods providers, support equipment and software providers, repair service providers and analytical laboratory subcontractors.

3.3. **Parameters**: Not applicable to this SOP.

4. Applicable Matrices

4.1. Not applicable to this SOP.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Not applicable to this SOP.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

8.2. **Critical Vendor**: A vendor for whose goods or services may have a direct impact on the quality of analytical results produced by PASI. These vendors would include but are not limited to providers of standards and reference materials, performance testing (PT) solutions, bottleware, chemicals, laboratory supplies, instruments, instrument repair and calibration services.

8.3. **Distributor**: A company that specializes in marketing and dispersing supplies or durable goods manufactured by another company.

8.4. **Non-critical Vendor:** A vendor whose goods or services have no direct impact on the quality of analytical results produced by PASI. Such suppliers include but are not limited to providers of office supplies, facility repair services (e.g., electrical, HVAC, plumbing), computer hardware and software (excluding those applicable to data acquisition, processing and reporting), and other non-analytical related goods and services.

8.5. **Service Provider:** A vendor that supplies non-material services to PASI (e.g., instrument maintenance, balance calibration, climate control service).

8.6. **Subcontract Lab**: A non-PASI (or non-subsidiary) laboratory that provides analytical testing services. These include laboratories that perform tests that can be performed within the network.

8.7. **Supplier**: A vendor that supplies chemicals, standards, consumables or other material goods to PASI.

8.8. **Vendor:** An external company that provides goods or services to the PASI network.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Not applicable to this SOP.

10. Reagents and Standards

10.1. Not applicable to this SOP.

11. Calibration and Standardization

11.1. Not applicable to this SOP.

12. Procedure

12.1. **General Procedures**:

12.1.1. Determining the need for New Vendors: Laboratories may need to evaluate new vendors on a periodic basis as determined by the need for new goods or services, competitive pricing or service related needs. This determination may be made by the laboratory personnel, but the purchasing agent (however named) or the Senior General Manager (SGM) or General Manager (GM) should be consulted to assist in making this determination. Needs for new vendors based on poor performance by existing vendors should be addressed with the vendor prior to PASI spending the resources required to qualify a new vendor.

12.1.1.1. Client Services (Project Management) frequently requires the addition of new providers for laboratory services. Project Managers have the responsibility of alerting the Quality Department before any new subcontract laboratory is used. The Director of Quality or Senior Quality Manager

(SQM) or Quality Manager (QM) can verbally approve the use of the lab if needed due to time constraints.

12.1.2. Classification of Vendors: All vendors need to be categorized as either a critical or non-critical vendor. This, along with the vendor type, will determine if qualification is necessary and, if so, the protocol for qualification.

12.1.2.1. Vendors can be categorized as critical or non-critical based on the definitions in Section 6. In short, those vendors that provide goods or services to PASI that can directly affect data quality must be considered critical and undergo a formal qualification process.

12.1.2.2. At a minimum, the following are to be considered critical vendors:

12.1.2.2.1. Subcontract laboratories;

12.1.2.2.2. Manufacturers of standards and reference materials, PT samples, chemicals, and high purity gases;

12.1.2.2.3. Manufacturers of bottleware and other sample containers;

12.1.2.2.4. Manufacturers and providers of instruments;

12.1.2.2.5. Providers of instrument repair and calibration services;

12.1.2.3. Providers of the items listed above are all critical vendors in that the products supplied can have or do have a direct impact on the quality of laboratory results. However, if the provider is simply a distributor and not a manufacturer, then the degree of qualification will be different than those listed above. Goods and materials received through a distributor must be evaluated on a case by case basis by the users. If the product is deemed unacceptable for use, then an alternative product must be acquired. Under these conditions, the distributor must be notified of the product suitability issues but is still considered a "qualified" vendor for PASI. See 12.2.9 for additional details on qualification of distributors.

12.1.2.4. All other vendors that do not fall into these categories may be considered non-critical vendors and do not require qualification beyond that of simple business terms (i.e. payment terms, credit lines, etc.). The SQMs/QMs or Corporate Quality Office should be consulted if one is unable to make a determination as to the type of vendor.

12.1.3. Responsibilities for Qualification:

12.1.3.1. In general, the responsibility for the qualification of new vendors will be split between the Purchasing Department and the Quality Department. Vendors who will provide goods and services to all PASI laboratories must be qualified by the Corporate Purchasing Manager and the Director of Quality. Vendors who will provide goods and services to a single PASI facility must be qualified by the local purchasing agent and the SQM/QM of the local laboratory as needed.

12.1.3.2. The submission and collection of the Supplier Qualification Form (example in Attachment I) and supporting documentation is the responsibility of the purchasing agent. Once received, the purchasing agent should disseminate the information collected as needed.

12.1.3.3. If the vendor requires a review of quality system documentation, the Director of Quality - or the SQM/QM - must review the materials and comments received in response to the qualification form. The approval of the vendor is based on the professional judgment of the Director of Quality or the SQM/QM. A rejection of the vendor and the justification for doing so will be documented on the vendor approval form.

12.1.3.4. The business terms, customers service and supply chain responses where applicable are to be reviewed by the Corporate Purchasing Manager (or local purchasing agent) or their designee. If

the purchasing agent can make the determination on the business terms then they can approve the vendor. Otherwise, the Accounting department must determine if the company can meet the business terms specified by the vendor. The purchasing agent will exercise professional judgment in determining the acceptability of the supply chain and customer service responses. The SGM/GM should be consulted for concerns related to supply chain management. A rejection of the vendor and the justification for doing so will be documented on the vendor approval form.

12.1.3.5. The paperwork for the supplier qualification will be archived by the Quality department. The purchasing agent may maintain a copy of the documentation for their records. The purchasing agent will maintain a list of vendors approved for use and make it available for others as needed. The Corporate Purchasing Manager will maintain the corporate list of approved vendors and disseminate that list periodically. The local purchasing agents should combine it with their local list of providers and make it available to the laboratory personnel.

12.2. **Supplier Qualification Requirements:** The information collected from a potential critical supplier is essential in helping determine the acceptability of that vendor. In qualifying a supplier, the information gathered will be determined based on the status of the vendor (critical vs. non-critical) and the vendor type.

12.2.1. **PASI Vendors**: All vendors require qualification based on the business terms necessary for supplying goods and services to PASI. This business terms approval requirement does not apply for a "one time use" vendor whose goods or services are purchased via credit card due to an urgent need. Repeated use of this vendor requires that they be officially qualified; the use of a credit card for purchases does not exclude them from this requirement.

12.2.2. **Subcontract Labs**:

12.2.2.1. If a laboratory is accredited under NELAC/TNI, DoD, or ISO, obtain a copy of the laboratory's certificate and list of accredited parameters. This certification, in addition to a copy of their insurance information, qualifies the lab to be used as a subcontract lab for Pace.

12.2.2.2. If a laboratory is certified under a state specific program but not a NELAC/TNI, DoD or ISO laboratory, collect a copy of the state certification and a list of the certified parameters. Successful receipt of these materials, in addition to receiving a copy of their insurance information, qualifies the lab to be used as a subcontract lab for Pace.

12.2.2.3. Subcontract laboratories that are not certified by a recognized outside agency should not be used, unless it is for tests or services that do not have an applicable certification (e.g., materials testing, etc.). In the event that there are no alternative laboratories, an uncertified laboratory may be used only after approval by the Director of Quality or the SQM/QM.

12.2.3. **Standards and Reference Materials Providers**:

12.2.3.1. Standard and reference material providers holding ISO based certifications are considered qualified on collection and retention of their ISO certificates and confirmation that the materials provided are traceable to NIST sources and materials. This confirmation may be included on the certificate or in a separate document from the provider.

12.2.3.2. As a general rule, non-NIST traceable materials should not be used in quantitative applications at PASI facilities. However, in the absence of an alternative vendor, providers supplying non-NIST traceable materials used for quantitative purposes may only be approved for use by the Director of Quality or the SQM/QM after evaluating the vendor's quality manual, the material in question and the manner of usage.

12.2.3.3. Providers supplying similar materials that are used for qualitative purposes only do not require any qualification beyond the business terms discussed in 12.2.1.

12.2.4. **Performance Testing Material Providers**:

12.2.4.1. For the purposes of meeting NELAC/TNI accreditation, any PT provider used must be on the list of approved PT providers by the NELAC Proficiency Testing Oversight Board (PTOB), currently A2LA. To qualify a PT vendor for use, collect the certificate that lists their recognition from the PTOB. Collect any additional certificates currently held as well (e.g., ISO 9001, 17025, NVLAP). Aside from business terms, no other qualification is needed. For the purposes of meeting requirements of non-NELAC/TNI state accreditation programs, any PT provider used must be on the list of approved PT vendors established by the laboratory accreditation program. Aside from business terms, no other qualification is needed.

12.2.4.2. In the event that the purpose of the PT samples needed is not for NELAC/TNI compliance, another PT provider may be used. At a minimum, the vendor must have an ISO accreditation (e.g., 9000, 9001, 17025, 34). Collect the ISO certificate, making sure that the expiration date has not passed. Aside from business terms, no other qualification is needed.

12.2.4.3. PT providers having no recognized quality system (e.g., ISO) are not approved for use by PASI.

12.2.5. **Chemical and High Purity Gas Providers**:

12.2.5.1. Chemical and high purity gas manufacturers may be approved for use if registered under an ISO quality system (e.g., 9001). Collect the ISO certificate and note the expiration date to ensure the expiration date has not passed.

12.2.5.2. Chemical and high purity gas manufacturers that do not have a registered quality system (e.g., ISO) must submit their quality manual for review and approval. The Director of Quality or the SQM/QM must review the manual and approve the vendor prior to use.

12.2.5.3. Regardless of the quality system in place, all chemicals and gases must be evaluated by the user for suitability in the respective application. Chemicals found to be unsuitable for use should be returned to the supplier and the deficiency noted by the purchasing agent to prevent future ordering.

12.2.5.4. Chemical and high purity gas providers having no formal quality system are deemed unacceptable for use by PASI.

12.2.6. **Bottleware and Sample Container Providers**:

12.2.6.1. Sample container providers and standard manufacturers provide critical components to PASI operations. Because of the roles each play in the analytical process and due to the client or regulatory driven requirements placed on the laboratories, each of these providers must address the same types of documentation and traceability issues as the analytical laboratories.

12.2.6.2. Bottleware and sample container providers that have an ISO registered quality system (e.g., 9001) are deemed acceptable for use. Collecting the ISO certificate and note the expiration date to ensure the expiration date has not passed.

12.2.6.3. Bottleware and sample container providers that do have an ISO registered quality system (e.g., 9001) must be approved by the Director of Quality or the SQM/QM prior to use. Collect a copy of the vendor's quality assurance manual and submit for review. The Director of Quality or the SQM/QM may approve the vendor or require a follow up interview or onsite audit at their discretion.

12.2.6.4. Bottleware and sample container providers having no formal quality system are deemed unacceptable for use by PASI.

12.2.7. **Instrument Manufacturers**:

12.2.7.1. Manufacturers of analytical instrumentation and high accuracy support equipment (e.g., gas chromatographs, ICPs, balances, digital burettes, etc.) must have an ISO (or equivalent) registered quality system. To qualify the vendor for use, collect the ISO certificate and note the expiration date to ensure that it has not passed.

12.2.7.2. Manufacturers of non-analytical laboratory equipment (e.g., drying ovens, refrigerators, vacuum pumps) do not require a formal qualification and approval beyond that of the general business terms.

12.2.8. **Instrument Service- Repair or Testing and Calibration Providers**:

12.2.8.1. Instrument Repair Service providers do not require a more extensive qualification and approval process beyond the general business terms referenced in 12.2.1. However, the individual seeking approval for this vendor should collect a list of supported equipment and the formal training records of the vendor's service staff. The vendor should only be used for repairs for equipment on which their staff have been formally trained.

12.2.8.2. Instrument testing and calibration service providers must have a formal, registered quality system and all calibrations must be performed using NIST traceable equipment and materials. Collect a copy of the ISO certificate or National Metrology Institute recognition and review the expiration date to ensure that it has not passed. Vendors providing calibration services on balances, weights and thermometers should have quality systems accredited to ISO/IEC 17025 by a mutually recognized Accreditation Body (e.g., NVLAP, A2LA, ILAC or IAAC). In the event this specific standard is not listed on the certificate, consult the Director of Quality for guidance and approval of the vendor.

12.2.9. **Distributors:**

12.2.9.1. A distributor is a reseller of products; they do not manufacturer the product they sell. Consequently, evaluating a quality system for the purposes of direct impact to the materials provided may not provide any insight to product quality.

12.2.9.2. Distributors can be evaluated on the basis of supply chain management and their overall qualification programs of their primary suppliers.

12.2.9.3. In qualifying and approving a distributor for use, collect any quality system registration certificates held and request documentation on their criteria for approval of their supplier. Preference should be given to a distributor with a quality system that includes or focuses on its supply chain and that has a formal set of criteria or a program for evaluating the quality of the suppliers whose products it distributes. Consult the Corporate Purchasing Manager and/or the Director of Quality for guidance in approving such suppliers.

12.3. **Approved Vendor List Management:**

12.3.1. The corporate vendor list will be maintained by the Corporate Purchasing Manager. This list should be updated and disseminated periodically in electronic format (e.g., Excel) to the Administrative Business Managers (ABMs) and purchasing agents in each of the PASI laboratories. The Excel file name should include a versioning designation to identify the last date updated (e.g., CorpVendorList – 122606.xls). Any hardcopies stored at Corporate must be marked with "SUPERCEDED".

12.3.2. The ABMs and/or purchasing agents should update the corporate vendor list to include any new additions of the locally approved supplies and disseminate the list to the laboratory personnel in their respective facilities. The new version of the approved vendor list is used in the laboratory with the old list marked as "SUPERCEDED".

12.3.3. All vendors currently in use upon the date of final signature of this SOP are considered qualified and are automatically added to the list.

12.3.4. Each qualified vendor should be re-evaluated on an annual basis. This review should focus on the performance over the last year. Vendors with repeated performance issues should be removed from the list of approved vendors. See 12.4 for more details on disqualification.

12.3.5. In cases where the vendor's quality system registration or quality assurance manual has expired, they must be contacted to obtain an updated version. The Purchasing department is responsible for collecting these updated documents.

12.4. **Vendor Disqualification and Reinstatement:**

12.4.1. A vendor may be considered unacceptable if history has demonstrated its inability to meet product specifications or service agreements including the quality system and quality assurance criteria. A vendor will be considered acceptable if it, minimally:

12.4.1.1. Meets product specifications, expectations and delivery schedule;

12.4.1.2. Responds expeditiously in resolving problems and discrepancies, and values customers (timeliness and service response);

12.4.1.3. Provides consistent quality, service and product performance.

12.4.2. In the event it is necessary to disqualify a vendor, then a secondary qualified vendor will be used until a new primary supplier is established. Where it is a corporate supplier, the Corporate Purchasing Manager or their designee will notify each Pace facility of the vendor disqualification. In the event that the disqualification is done at a local level, then the local Purchasing representative will notify Corporate. The vendor file will be clearly marked as "Not-Acceptable" and dated by the SQM/QM. The purchasing agent will update the approved vendor list to indicate the vendor is no longer approved for use.

12.4.3. Vendors rated as "not-acceptable" may request an opportunity to continue doing business with Pace Analytical. In this case, they must demonstrate that adequate corrective/preventative actions have been taken in order to meet product specifications including quality system and quality assurance criteria. A copy of the vendor's corrective action plan should be requested. The Corporate Purchasing Manager must be notified and an audit of the facility may be requested. The Director of Quality and/or the SQM/QM should review and approve the vendor corrective action plan in the event of any quality related issues that could impact data quality.

13. Quality Control

13.1. Not applicable to this SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Not applicable to this SOP.

16. Corrective Actions for Out-of-Control Data

16.1. Not applicable to this SOP.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. Not applicable to this SOP.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

19. Method Modifications

19.1. Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1. Not applicable to this SOP.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Not applicable to this SOP.

23. Waste Management

23.1. Not applicable to this SOP.

24. Pollution Prevention

24.1. Not applicable to this SOP.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I: Example Supplier Qualification Form (F-ALL-Q-034).

26.2. Attachment II: Example Subcontract Lab Qualification Form (F-ALL-Q-008).

27. Revisions

Document Number	Reason for Change	Date
SOT-ALL-Q-027-rev.02	Converted to SOT General: Changed all references to Director of Quality, Safety and Training to Director of Quality or Director of Training and Safety as applicable. General: changed 'vendor' to 'supplier' when referencing the Supplier Qualification Form. Added additional sections in red text (editable) throughout the document (including the DoD reference in section 15). Section 11.1.3.1: changed 'should' to 'must' where applicable Section 11.2.6: changed header title. Replaced Attachments I and II with updated forms.	18Apr2011
SOT-ALL-Q-027-rev.03	General: changed to new format. General: throughout SOP, reworded to account for management structure changes (SQM/QM). Added section 3.1. Attachment II: replaced with current version of form.	03Jul2012
S-GB-Q-018-Rev.01	Updated to Local SOP number	17July2012
SOT-ALL-Q-027-rev.04	Section 12.2.2.1: Added language regarding proof of insurance. Section 12.2.2.2: Added last sentence to clarify when labs are qualified to be subcontract labs. Removed requirement to acquire QAM. Section 12.2.2.3: clarified language for non-certified subcontract lab use.	09Sep2013
S-GB-Q-018-Rev.02	Updated to Local SOP number	06Jan2104

Attachment I: Example Supplier Qualification Form

Pace Analytical	Document Name: Supplier Qualification Form	Document Revised: February 23, 2011 Page 1 of 1
	Document No.: F-ALL-Q-034-rev.02	Issuing Authority: Pace Corporate Quality Office

Supplier Qualification Form

Company Name:	
Service Provided	
Contact:	
Address:	
Telephone:	
Fax:	
E-mail:	
Website:	

ITEM	RESPONSE
# Years in Business	
Ownership (Public/Private)	
Bankruptcy declared (Y/N)? If so, when?	
Early payment discounts (Y/N, % @ Days)	
Rebates for volume purchases (Y/N)	
Payment Terms: Accept NET 40? (Y/N)	
Dedicated Customer Service Rep.? (Y/N)	
Internet catalogs available? (Y/N)	
Online ordering available? (Y/N)	
On Time Delivery tracked? (Y/N)	
Product Lot Set Asides available? (Y/N)	
Backup suppliers for critical items? (Y/N)	

ITEM	RESPONSE
Is a formal Quality Assurance Program in place?	
If so, what is the basis of the Quality System (i.e. ISO)?	
Does the company maintain any formal certifications of the Quality System (i.e. ISO 9001, 17025)?	
Is a formal Corrective Action/ Preventative Action (CAPA) system in place ?	
Does your company have a formal vendor qualification program for your suppliers?	
Does your company have a formal system for capturing, monitoring and tracking customer complaints?	
If required, will your company agree to an on-site audit by a Pace Quality representative?	

Completed By:	
Date:	
Title	

Pace Analytical Use Only

Reviewed By:_____

Date:_____

Approved: Y / N

Quality Documents Received:

- ❑ Quality System registration certificate (s)
- ❑ Quality Assurance Manual
- ❑ _____ Other

Attachment II: Example Subcontract Lab Qualification Form

Pace Analytical®	Document Name: Subcontract Laboratory Qualification Form	Document Revised: January 18, 2012 Page 1 of 1
	Document No.: F-ALL-Q-008-rev.06	Issuing Authority: Pace Corporate Quality Office

Subcontract Laboratory Qualification Form

Company Name: _____

Subsidiary, Division of Facility of (if applicable): _____

Company Address: _____

_____

Quality Contact: _____ Phone: _____ Email: _____

Customer Services Contact: _____ Phone: _____ Email: _____

Is your facility certified/registered/licensed by any federal, state, or professional agency? (specify below):

NELAC/TNI? ☐ Yes ☐ No ISO? ☐ Yes ☐ No DoD? ☐ Yes ☐ No Other_____

Has the laboratory been prosecuted/debarred/de-certified by any state or federal agencies within the last 5 years? ☐ Yes ☐ No

Name of Agencies: _____

Has the company been audited by any regulatory agencies within the last 2 years? ☐ Yes ☐ No

Name of Agencies: _____

If requested, can a Pace Quality representative perform an on-site assessment? ☐ Yes ☐ No

Does your laboratory have a current USDA regulated soil permit? ☐ Yes ☐ No

Please provide the following materials with this form *(PDF/electronic versions preferred)*:

1. NELAC/TNI, ISO and/or DoD Certificates and approved parameter lists (if applicable) and a list of any other certifications.

2. Quality Manual (non-NELAC/TNI-accredited facilities only)

3. Proof of Insurance (e.g., Certificate of Liability, Evidence of Insurance, Certificate of Liability Insurance, or however named)

4. Copy of your USDA regulated soil permit (Pace labs cannot ship soil samples to labs without a permit).

I (We) certify the information contained in this Subcontract Laboratory Qualification Form is accurate and complete as of the date indicated. All trade secrets or other proprietary information obtained about Pace Analytical Services, Inc. or its' customers will be kept confidential. This profile has been made with the permission of the company surveyed. **** Our laboratory also agrees to provide to Pace all quality control results necessary to validate the results for all subcontracted samples.**

Signature: _____ Name: _____ Date: _____

(For Pace use only) Approved by: _____ Date: _____

Pace Analytical Services, Inc.
1241 Bellevue Street Suite 9
Green Bay, WI 54302
Phone: 920 469-2436
Fax: 920 469-8827

STANDARD OPERATING PROCEDURE

The Determination of Total Organic Carbon Using the Walkley-Black Procedure

Reference Methods: Methods of Soil Analysis, Walkley-Black

SOP NUMBER:	S-GB-I-037-Rev.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-037-Rev.03

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 09/21/12

Kate Grams, Laboratory Quality Manager — Date: 8/20/12

Chad Rusch, Department Manager — Date: 08/20/12

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL

Signature	Title	Date

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Table of Contents

Section **Page**

1. Purpose 3

2. Scope and Application 3

3. Summary of Method 3

4. Interferences 3

5. Safety 3

6. Definitions 4

7. Sample Collection, Preservation and Handling 4

8. Reagents and Standards 4

9. Equipment and Supplies 5

10. Calibration and Standardization 6

11. Procedure 7

12. Calculations 8

13. Quality Control: 9

14. Method Performance 12

15. Pollution Prevention and Waste Management 13

16. References 13

17. Tables, Diagrams, Flowcharts, Appendices, Addenda etc. 13

18. Revisions 14

1 Purpose

The purpose of this Standard Operating Procedure (SOP) is to describe the method used to determine the concentration of Total Organic Carbon (TOC) in soil samples compliant with the Walkley-Black procedure.

2 Scope and Application

This SOP pertains to sediments, soils and sludges. This method is based on Methods of Soil Analysis, Walkley-Black. The applicable range is 400 to 32,000 mg/kg. The current Pace Reporting Limit is 400 mg/Kg. The current MDL can be found in Epic Pro.

3 Summary of Method

An aliquot of a dried and homogenized solid sample is put into a COD vial and caped. The sealed tubes are heated in a hot block at 150°C. After two hours, the tubes are removed, cooled and measured spectrophotometrically at 620 nm.

4 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2 Chloride and iron give positive interference. Mercuric sulfate in the digestion tubes complexes the chlorides.

5 Safety

5.1 All samples, standards, and reagents should be treated as hazardous. Safety glasses, gloves, and lab coats are to be worn. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. Exposure to these chemicals must be reduced to the lowest possible level by a safe technique. Special care should be taken when handling the high concentration acids and oxidizing reagents used for sample digestion.

5.2 The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of any chemical. A reference file of Material Safety Data Sheets (MSDS) and a formal safety plan are made available to all personnel involved in chemical analysis and should be consulted prior to handling samples and standards.

5.3 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management.*

5.4 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

6 Definitions

6.1 Total Organic Carbon (TOC): All carbon atoms covalently bonded in organic molecules. To measure TOC, all the inorganic carbon is removed by the addition of acid; the result is that only organic carbon is left.

6.2 Additional definitions can be found in Section 10 of the Pace Analytical Services, Inc. Quality Manual.

7 Sample Collection, Preservation and Handling

Table 1. SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

Matrix	Method	Container(s)	Preservation	Shipment Conditions	Lab Storage Conditions
Solid	Walkley Black	Clean 4oz glass containers	None 28 days	On ice 4+/- 2° Celsius	4°C

8 Reagents and Standards

8.1 Reagents

8.1.1 15,000mg/L COD Vials

8.1.2 Reagent Water – Deionized water (DI water)

8.2 Standards

8.2.1 Pre-made TOC standard

8.2.1.1 One primary source for calibration, CRDL's and CCVs. 1,000 mg/L primary stock standard, purchased. (SCP Science, 250-250-051)

8.2.1.2 Secondary source for ICV and spikes. 1,000 mg/L secondary stock standard, purchased. (Ultra Scientific, IQC-106-5)

8.2.2 Calibration Curve: Made up of the following six standards, 10, 20, 50, 200, 400, 800 mg/L. Calibration standards should be made daily.

8.2.2.1 Calibration Standard 0 – 0 mg/L. 2ml of Nano-Pure water.

8.2.2.2 Calibration Standard 3– 50mg/L. Add 2.5ml of 1,000mg/L primary standard to 50ml volumetric flask and bring to volume with Nano-Pure water.

8.2.2.3 Calibration Standard 1 and Reporting Limit Verification Standard (CRDL) – 10mg/L. Add 2ml of Calibration Standard 3 to 10ml volumetric flask and bring to volume with Nano-Pure water. Shelf life = 3 months

8.2.2.4 Calibration Standard 2 – 20mg/L. Add 4ml of Calibration Standard 3 to 10ml volumetric flask and bring to volume with Nano-Pure water.

8.2.2.5 Calibration Standard 4 – 200mg/L. Add 2ml of 1,000mg/L primary standard to 10ml volumetric flask and bring to volume with Nano-Pure water.

8.2.2.6 Calibration Standard 5 and Continuing Calibration Verification (CCV) – 400mg/L. Add 20ml of 1,000mg/L primary standard to 50ml volumetric flask and bring to volume with Nano-Pure water. Shelf life = 3 months

8.2.2.7 Calibration Standard 6 – 800mg/L. Add 8ml of 1,000mg/L primary standard to 10ml volumetric flask and bring to volume with Nano-Pure water.

8.2.3 Initial Calibration Verification (ICV) - – 400mg/L. Add 4ml of 1,000 mg/L secondary standard to a 10ml volumetric flask and bring to final volume with Nano-Pure water.

8.2.4 Laboratory Control Spike/Laboratory Control Spike Duplicate (LCS/LCSD) – 800mg/L. Add 40ml of 1,000mg/L secondary standard to 50ml volumetric flask and bring to volume with Nano-Pure water. Shelf life = 3 months.

8.2.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) – Use secondary standard created for LCS/LCSD.

9 Equipment and Supplies

Table 2. EQUIPMENT

Equipment	Manufacturer*	Model(s)*
Top loading Balance	OHAUS	AP 110S
Spectrophotometer	Hach	DR2000
COD reactor block	Hach	45600
Adjustable pipettor	Finnpipette	
Adjustable pipettor	Chemisphere	02111245
Sample cells	Hach	Cat# 13537-02
Drying Oven (103-105°C)	Precision Scientific Group	17
Mortar and Pestle	Coors	60316, 60317
Vented Hood	Hamilton or Equivalent	
100 mL volumetric flasks	Pyrex	5660 100
1000 mL volumetric flask	Pyrex	5640 1L
Wire racks	Fisher	Cat# 14802

* Or equivalent

Table 3. SUPPLIES

Supplies	Manufacturer*	Vendor*	Catalog #*
COD digestion vials	Chemetrics	Fisher	K-7370S
Latex gloves	Diamond Grip	Fisher	11-462-67D
Thermal gloves	Fisher	Fisher	19-013-541
Finntip 5mL tips	Thermolabsystems	VWR	53515-886
Eppendorf 1000 uL Pipette tips	Eppendorf	Fisher	21-372

10 Calibration and Standardization

10.1 Prepare a calibration curve using a minimum of five standards and a blank: see standards in Section 8. Add 2.0 mL of each curve standard to a digestion tube and digest per Section 11.

10.2 Turn on Spectrophotometer and set to 620 nm and allow it to warm up for at least 20 minutes.

10.3 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place Level 0 calibration standard into the spectrophotometer and zero the instrument.

10.4 Place the standard cells into the spectrophotometer and record standard absorbance at 620 nm into the prep log . Calculate and lock curve to set as new curve.

10.5 . The data is saved within the program and can be referenced when needed.. Note: Record the HBN# associated to the curve as it needs to be entered for each new preplog created.

10.6 A linear regression analysis of the absorbance values against the corresponding concentrations must yield a correlation coefficient of >0.995. The y-intercept must be less than the Pace Reporting Limit.

10.6.1 A new calibration curve should be prepared yearly at minimum or whenever the continuing calibration standard does not pass control criteria.
The ICV, ICB, and RLVS standards must be run immediately following creation of a new curve and record as in section 11.

10.6.2 ICV – Add 2ml of prepared ICV standard to digestion tube and digest per section 11.

10.6.3 ICB – Add 2ml of Nano-Pure water to digestion tube and digest per section 11.

10.6.4 RLVS (CRDL) – Add 2ml of prepared Calibration Standard 1 to digestion tube and digest per section 11.

11 Procedure

11.1 Sample Preparation.

11.1.1 Physical Preparation

11.1.1.1 Oven-dry overnight and grind to a size that the sample will pass through a 0.5-mm sieve.

11.1.1.2 Mix the sample thoroughly before selecting a portion for analysis.

11.1.1.3 Discard any foreign objects such as sticks, leaves, and rocks.

NOTE: Document all sample sizes, standards and reagents used in the digestion in the electronic Preplog.

11.2 Analytical

11.2.1 Samples are batched in Horizon/Epic Pro (the LIMS) with the appropriate batch QC. See section 13 for frequency, acceptance criteria, and corrective actions.

11.2.2 The electronic Prep Log is used to document the Prep and Analytical steps and to post the data to the LIMS. Use the TOC Walkley Black Prep and WB TOC Walkley Black Analytical templates.

11.2.3 Label Chemetrics COD digestion vials

11.2.4 Unseal the vials and carefully weigh out 0.05 g using a calibrated balance. Place the 0.05 g of soil into a vial. Pipette 1.95 mLs of Nanopure water into a vial such that it forms a layer on top of the reagents contained in the vial.

11.2.5 Prepare CCV, CCB, MB, LCS, MS, and MSD and digest per section 8.. See Section 12 for control limits and frequency.

11.2.5.1 CCV - Add 2.0ml of prepared CCV standard to a digestion tube.

11.2.5.2 CCB – Add 2.0ml of Nano-Pure water to digestion tube.

11.2.5.3 MB - Add 2ml of Nano-Pure water to digestion tube.

11.2.5.4 LCS – Add 1ml of prepared LCS secondary standard and 1ml of Nano-Pure water to digestion tube.

11.2.5.5 MS/MSD – Add 1ml of prepared LCS secondary standard and 0.95ml of Nano-Pure water in addition to the 0.05g of soil to digestion tube.

11.2.6 Carefully seal the vial. During digestion, the reagents and sample are raised to a point just below boiling. Improperly sealed vials may leak or break.

11.2.7 Thoroughly mix the contents of the sealed vial by shaking. CAUTION: The vial will get very hot during mixing. It is recommended that vials be mixed

either in racks or with the use of insulated gloves. Eye protection MUST be worn.

11.2.8 Place the digestion vials in the reactor block at 150°C ± 5°C for 2 hours.

11.2.9 Cool about ½ hours. Then invert vials to mix.

11.2.10 Cool to room temperature. Allow any suspended precipitate to settle.

11.2.11 Turn on Spectrophotometer and set to 620 nm. Let warm up at least 20 minutes.

11.3 Wipe the outside of each vial with tissue paper to remove any fingerprints. Place ICB/CCB into the spectrophotometer and zero the instrument. Then, place the sample vials into the spectrophotometer and record sample and QC absorbance at 620 nm into the prep log.

11.3.1 The prep log will calculate the sample results in mg/L TOC, upload from the prep log and report the final results in LIMS.

12 Calculations

12.1 $$\frac{\text{(mg/L TOC from the curve) X (Final Volume (mL))}}{\text{(Weight in (g))}} = \text{TOC (mg/Kg)}$$

12.2 TOC (%) = (concentration in mg/kg / 10,000)

12.3 Accuracy:

A laboratory control spike / laboratory control spike duplicate is analyzed for each analytical batch of 20 or fewer samples.

$$\text{LCS, \% Recovery} = \frac{\text{TOC, mg/kg}}{\text{True Value mg/Kg}} \times 100$$

12.4 Precision:

The precision is calculated based on the recovery of the sample / sample duplicate result. A sample duplicate is performed at a frequency of 10% or one per batch which ever is more frequent and must meet laboratory specific limits for precision.

Relative percent difference (RPD) calculation:

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

S = Sample Value
SD = Sample Duplicate Value

13 Quality Control:

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 **Initial Calibration Verification (ICV):**

13.2.1 The ICV must be analyzed immediately after calibration.

13.2.2 Concentration must be within ± 10% of the true value. The true value is 400 mg/L. When measurements are outside the control limits, reanalyze once. If the measurement is still outside of the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified.

13.2.3 The source of the purchased 1,000mg/L TOC standard used to make the ICV must be different from that of the calibration curve standards and CCV.

13.3 **Continuing Calibration Verification (CCV):**

13.3.1 The CCV is analyzed after every 10 samples.

13.3.2 Concentration must be within ± 10% of the true value. The true value is 400 mg/L. When measurements are outside the control limits, reanalyze once. If the measurement is still outside of the control limits, the analysis must be terminated, the problem corrected, and the calibration re-verified. If the reset CCV fails recalibrate and reanalyze all samples back to the last acceptable CCV.

13.3.2.1 If the CCV is greater than the control limit and the samples are non-detects, the sample may be reported without a flag.

13.4 **Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB):**

13.4.1 The ICB must be analyzed after the ICV. The CCB must be analyzed after the CCV.

13.4.2 The value must be < PRL. When measurements are above the PRL, reanalyze once. If the measurement is still outside of the control limits, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank.

13.4.2.1 If the sample concentration is greater than ten times the concentration in the ICB or CCB, the samples do not need to be reanalyzed.

13.4.2.2 If the samples are non-detections and the ICB or CCB fails high, the samples do not need to be reanalyzed.

13.5 **Reporting Limit Verification Standard (CRDL)** – A standard prepared at the concentration of the Pace Reporting Limit. It is analyzed after the calibration and also before each new batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

13.6 **Method Blank (MB)**

13.6.1 A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples.

13.6.2 The absolute value must be < PRL.

13.6.2.1 When measurements are above the PRL, reanalyze once. If the measurement is still outside of the control limits, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples associated with the method blank.

13.6.2.2 Method blank criteria is evaluated and flagged with appropriate data qualifiers to the MDL when MDL reporting is required.

13.6.2.3 Qualify data with the appropriate data qualifier if sample results are less than 10 times the absolute value detected in the blank and are not a non-detection.

13.6.2.4 Additionally, method blank acceptance may be based on project specific criteria or regulatory limits .

13.7 **Laboratory Control Sample (LCS):**

13.7.1 The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples. A Laboratory Control Spike Duplicate (LCSD) must be analyzed if there is insufficient sample volume to perform a matrix spike/matrix spike duplicate or if the client requests one.

13.7.2 The recovered concentration must be within default limits of $\pm$ 20%. The true value is 400 mg/L.

13.7.2.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a data qualifier.

13.7.2.2 If no errors are found reanalyze once. If the measurement is still outside of the control limits and sufficient sample is available, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still out side the limits after re-analysis, contact the PM to

determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.7.2.3 If sufficient sample volume is not available, report the sample data with appropriate data qualifier on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.7.3 The precision between the LCS and LCSD must be =/< 20% RPD.

13.7.3.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.7.3.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.7.4 The source of the 1,000mg/L TOC standard used to make the CCV is not the same for the LCS.

13.8 **Matrix Spike (MS) and Matrix Spike Duplicate (MSD):**

13.8.1 One pair at 10% frequency or up to 10 environmental.

13.8.2 Both QC samples must be calculated for accuracy and precision.

13.8.3 The recovered concentration must be within default limits of ± 20%. The spike true value is 400 mg/L.

13.8.3.1 If four times the concentration of the spike is less than the analyte concentration of the parent, accuracy need not be calculated.

13.8.3.2 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a data qualifier.

13.8.3.3 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.4 The precision between the MS and MSD must be =/< 20% RPD.

13.8.4.1.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an

error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.8.4.1.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with appropriate data qualifier.

13.8.5 The sample used for MS/MSD pair is either determined by the client or selected at random from client samples as sample volume allows. No field, filter, trip or equipment blanks can be used for MS/MSD.

13.8.6 The source of the 1,000mg/L TOC standard used to make the CCV is not the same for the MS and MSD.

13.9 **Hold:** When preparation of a sample exceeds 28 days past the time of collection, notify the project manager before proceeding. If a sample is run past 28 days after collection, flag the result with appropriate data qualifier.

13.10 If a sample was diluted due to matrix effects and the result is a non-detect, the result must be qualified with appropriate data qualifier.

13.11 See attachments Table A and Table B for a summary of QC.

14 Method Performance

There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual and specific Standard Operating Procedures.

14.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

14.2 An initial demonstration of capability (IDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

14.3 Continuing demonstration of capability (CDC, YDOC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the CDOC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

14.4 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ,* for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

14.5 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *PE/PT Program,* to demonstrate continuing competence. All results are stored in the QA office.

14.6 Linear Dynamic Range Study - A linear dynamic range study must be conducted at least

once. The study is conducted by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element.

14.7 Once the linear dynamic range study determination is performed, keep the data, and then every three months verify with a single high point.

15 Pollution Prevention and Waste Management

15.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

15.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management.*

16 References

16.1 Methods of Soil Analysis, 1982 Second Edition, Method 29-3.5.2.1 Walkley-Black Procedure.

16.2 Nelson D W, Sommers L E. A Rapid and Accurate Method for Proceedings of the. Indiana Academy of Science, 1975, 84: 456-462.

16.3 EPA Manual 600 4-79-020, March 1983, Method 410.4, 40CFR Part 136

16.4 PASI Quality Manual, current revision

17 Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

17.1 Table A – Quality Control

17.2 Table B – Analyst/Technician Data Assessment

17.3 Flowchart

18 Revisions

Revision Number	Reason for Change	Date
KM-I-004.Rev.00	Converted SOP to PASI format. Added laboratory charted control limits.	8Aug2005
GB-I-037-Rev.00	Converted from Kimberly to Green Bay. 9.1.7 Changed expiration date of Ferrous Sulfate. 11.1.7 Added acceptable range for ferrous sulfate titrant standardization. Clarified 12.1.1.1 and 12.2.1.1.	12Sept2006
S-GB-I-037-REV.01	Updated signature page. Deleted Section 7. Renumbered document. Section 13.3 – Updated Method Blank acceptance criteria Section 14 – Updated SOP references. Updated Attachments	24July2008
S-GB-I-037-Rev.02	Updated body of document to Hach Spec Method Updated Attachments	10Sept2009
S-GB-I-037-Rev.03	Updated SOP references for Waste Management SOP to S-GB-S-006 Waste Management and Handling. Table B,1 MB 20X rule changed to 10X.	13Dec2010
S-GB-I-037-Rev.04	Updated SOP references throughout document. Section 2—Added reference to the LOD and LOQ Throughout document: Incorporated electronic preplog. Section 8, 10 and 11: Updated to pre-made standards.	15Aug2012

Table A. QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**Walkley Black**
Method Blank	One per batch of samples, up to 20 environmental samples.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples. A LCSD is required if MS/MSD is not performed.
Matrix Spike / Matrix Spike Duplicate	One pair per batch of samples, up to 10 environmental samples.
Initial Calibration	Minimum of 5 standards plus blank. Performed once a year at a minimum.
RL Standard	After the calibration and and following the initial CCV/CCB in each batch.
Calibration Verification (ICV/CCV)	ICV – analyzed after calibration but before samples. CCV – analyzed after every 10 samples.
Calibration Blank (ICB/CCB)	ICB – analyzed after ICV. CCB – analyzed after every CCV pair.

Table B. ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**Walkley Black If these conditions are not achieved ⇒**
Method Blank	• 1
Accuracy & Precision Matrix Spike Samples	• 2
Accuracy & Precision Laboratory Control Spikes	• 3
Initial Calibration	• 4
CRDL standard	• 5
Initial / Continuing Calibration Verification	• 6
Initial / Continuing Calibration Blank	• 7

1. In the absence of project specific requirements, sample detects less than 10 times the method blank contamination level is reported with the appropriate data qualifier. Sample detects greater than 10 times the method blank contamination are reported without qualification.

2. In the absence of project specific or method requirements, in-house generated limits will be used. If the concentration of the spike is less than 25% of the concentration of the parent,accuracy need not be calculated. If the parent, MS, or MSD is greater than the top standard in the curve, dilute and reanalyze the parent, MS, and MSD. If the concentration of the spike is greater than 25% of the concentration of the parent, appropriately flag the parent sample if either the MS and/or MSD fails. If the MS and MSD fails precision control limits flag the parent with the appropriate (precision) data qualifier.

3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate accuracy and appropriate precision qualifiers to reflect the deficiencies.

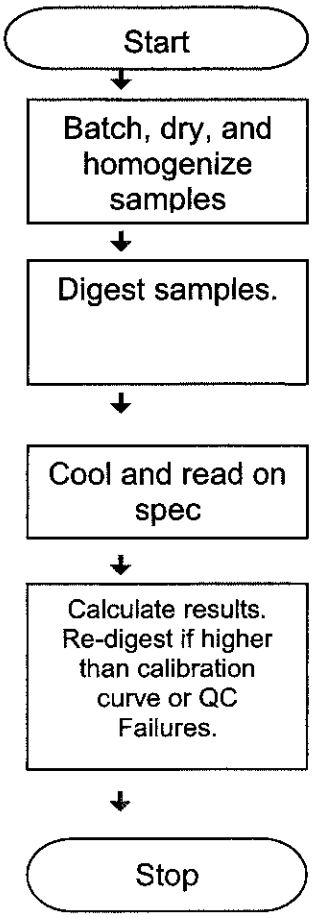
4. If correlation coefficient is less than 0.995 perform maintenance and recalibrate.

5. It is analyzed after the calibration and following the initial CCV/CCB in each batch, recovery 60-140% of true value. If outside the limits, reanalyze once. If still outside the limits, recalibrate.

6. If ICV/CCV is outside the control limits reanalyze the ICV/CCV to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. If the ICV/CCV recovers greater than the control limit and the samples bracketing the out of control ICV/CCV are non-detects, the results may be reported without a flag.

7. If ICB/CCB is outside the control limits reanalyze the ICB/CCB to verify the instrument is out of control. If the 2nd analysis is outside control limits, perform maintenance and recalibrate. Samples that bracket the out of control standards must be reanalyzed. Samples that are > 10X the concentration in the CCB the samples do not have to be reanalyzed.

FLOWCHART


Start
Batch, dry, and homogenize samples
Digest samples.
Cool and read on spec
Calculate results. Re-digest if higher than calibration curve or QC Failures.
Stop

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STANDARD OPERATING PROCEDURE

Measurement of Volatile Solids and Solids in Waters

Reference Methods: Standard Methods 2540-B, -C, -D, -E, -G, ASTM D2974 and EPA 160.4

SOP NUMBER:	S-GB-I-068-Rev.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-I-068-Rev.01

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/24/13

Kate Grams, Laboratory Quality Manager — Date: 12/17/2013

Chad Rusch, Department Manager — Date: 12/16/2013

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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Table of Contents

Section		Page
1.	Purpose /Identification of Method	3
2.	Summary of Method	3
3.	Scope and Application	3
4.	Applicable Matrices	4
5.	Limits of Detection and Quantitation	4
6.	Interferences	4
7.	Sample Collection, Preservation Shipment and Storage	5
8.	Definitions	5
9.	Equipment and Supplies	6
10.	Reagents and Standards	7
11.	Calibration and Standardization	7
12.	Procedure	7
13.	Quality Control	13
14.	Data Analysis and Calculations	16
15.	Data Assessment and Acceptance Criteria for Quality Control Measures	17
16.	Corrective Actions for Out-of-Control Data	18
17.	Contingencies for Handling Out-of-Control or Unacceptable Data	19
18.	Method Performance	19
19.	Method Modifications	19
20.	Instrument/Equipment Maintenance	19
21.	Troubleshooting	19
22.	Safety	19
23.	Waste Management	20
24.	Pollution Prevention	20
25.	References	20
26.	Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.	20
27.	Revisions	21

1. Purpose /Identification of Method

1.1 This Standard Operating Procedure (SOP) describes operations used to measure the parameters in the following table:

Table 1.

Test		Matrix	Method	Reports as
TS	Total Solids	Water	SM 2540B	mg/L
TS	Total Solids	Solid	SM 2540G	%
TSS	Total Suspended Solids	Water	SM 2540D	mg/L
TDS	Total Dissolved Solids	Water	SM 2540C	mg/L
TVS	Total Volatile Solids	Water	EPA 160.4	mg/L
TVS	Total Volatile Solids	Solid	EPA 160.4	%
TVSS	Total Volatile Suspended Solids	Water	EPA 160.4	mg/L
OM	Organic Matter	Solid	ASTM D2974-87(C)	%
FOC	Fractional Organic Carbon	Solid	ASTM D2974-87	%

2. Summary of Method

2.1 Total Solids (TS) – A well-mixed sample is evaporated to dryness and the residue solids are measured gravimetrically.

2.2 Total Suspended Solids (TSS) – A well-mixed sample is filtered. The residue collected by the filter is dried and measured gravimetrically.

2.3 Total Dissolved Solids (TDS) – A well-mixed sample is filtered. The filtrate passing through the filter is evaporated to dryness and the residue solids are measured gravimetrically.

2.4 Total Volatile Solids (TVS, TVSS) – Residue obtained from the determination of TS, or TSS is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids for solid samples and mg/L Volatile Solids for liquid samples.

2.5 Fractional Organic Carbon (FOC) - Residue obtained from the determination of TS is ignited at 440°C in a muffle furnace. The weight loss after ignition is multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

2.6 Organic Matter (OM) - Residue obtained from the determination of TS is ignited at 440°C in a muffle furnace. The weight loss after ignition is reported as % Organic Matter.

3. Scope and Application

3.1 This method is applicable to the determination of Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS), and Total Volatile Solids (TVS) in water samples.

3.2 This method is applicable to the determination of Total Solids (TS) and its Volatile (TVS) fractions in soil and semisolid samples. The terms total solids apply to matter left after drying in an oven at 103-105° C and igniting in a muffle furnace at a 550°C temperature and a defined time interval.

3.3 This method is applicable to the determination of Fractional Organic Carbon (FOC) and Organic Matter (OM) in soil and semisolid samples. The term fractional organic carbon (FOC) applies to the weight loss after ignition, multiplied by a 0.58 factor and reported as % Fractional Organic Carbon.

3.4 This procedure is restricted to use by, or under the supervision of, analysts experienced with the use of laboratory information systems, balances, desiccators, and ovens. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

4. Applicable Matrices

4.1 This SOP applies to surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, soils, and sludges.

5. Limits of Detection and Quantitation

5.1 The reporting limit (LOQ) for all analytes is listed in Table 1 for the listed methods. All current MDLs are listed in the LIMS and are available by request from the Quality Manager.

Table 2.
REPORTING LIMITS

COMPOUND NAME	Aqueous	Solids
	Units mg/L	Units %w/w
	LOQ	LOQ
TS	20	0.1
TSS	10	
TDS	20	
TVS, TVSS	20	
FOC		0.058
OM		0.1

6. Interferences

6.1 Non-representative materials, e.g., leaves and sticks should be removed from the sample prior to measurement unless it is determined that their inclusion is desired.

6.2 Measurements are subject to negative bias for samples containing significant quantities of ammonium carbonate, volatile organics, or other volatile materials that could be lost during drying.

6.3 The residue of samples for TS and TDS that are highly mineralized, especially containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require longer drying, desiccation, and rapid weighing.

6.4 Samples for TS and TDS containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying to ensure that all bicarbonate is converted to carbonate.

6.5 The volumes of aliquots for TS and TDS should be selected to limit the total residue to 200 mg to prevent the residue from crusting over and trapping water during drying.

6.6 Samples for TSS with high TDS, such as saline waters, brines, and some wastes, may be subject to positive bias. Care must be taken to properly rinse the filter to minimize the bias.

7. Sample Collection, Preservation Shipment and Storage

Table 3
SAMPLE COLLECTION, PRESERVATION, HOLD TIME SHIPMENT AND STORAGE

Matrix	Analytical Method	Container(s)	Preservation / Hold Time	Shipment Conditions	Lab Storage Conditions
Aqueous	SM 2540B SM 2540C SM 2540D EPA 160.4	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C
Soils	SM 2540G ASTM D2974-87	Clean Plastic or glass containers	None / 7 Days	On ice ≤6° Celsius	Walk-in cooler ≤6° C

8. Definitions

8.1 Batch – A grouping of 20, or less, environmental samples of similar matrix, which are prepared and/or analyzed together with the same method.

8.2 Blank Sample – A sample analyzed to determine potential contamination attributed to sample handling and analysis.

8.3 Duplicate Sample – A second aliquot of the same environmental sample analyzed in the same manner as the original sample in order to evaluate precision.

8.4 Environmental Sample – An environmental sample or field sample is a representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required.

8.5 Laboratory Control Sample (LCS) – A blank matrix sample spiked with a known concentration of analytes of interest. Laboratory accuracy is evaluated using the LCS.

8.6 Laboratory Information Management System (LIMS) – This is a system for transferring, processing, storing, and reporting analytical results.

8.7 Replicate Samples – Samples collected at the same time, from the same place, for the same analysis, as the original sample in order to determine precision between samples.

8.8 Pace Reporting Limit (PRL) – The lowest level that will be reported by this method. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

8.9 Solid – matrix such as soils, sediments, sludge, organic liquids, oils or aqueous products and by-products of industrial processes, and aqueous samples with more than 10% settleable solids.

8.10 Total Solids Content (TSC) – Total Solids Content is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature of 103-105°C.

8.11 Volatile Solids – Residue obtained from the determination of TS is ignited at 550°C in a muffle furnace. The loss of weight on ignition is reported as % Volatile Solids.

8.12 Refer to Section 10 of the Pace Quality Manual for additional terms used in this SOP.

9. Equipment and Supplies

9.1 Analytical balance – Electronic, capable of weighing to 0.1 mg.

9.2 Vacuum Filtration System – Includes filter holder, membrane filter funnel, vacuum flask and vacuum pump.

9.3 Glass Fiber Filters (for TDS) – GE Water & Process Technologies PreSep Prefiler, Glass, 1.0 Micron, 47mm, or equivalent.

9.4 Glass Fiber Filters (for TSS) – Pre-weighed from Environmental Express, ProWeigh 47mm Glass Fiber Filters, 1.5 µm, with matched aluminum evaporating dishes, or equivalent.

9.5 Graduated Cylinders – 25, 50, 100, 250, 500 and 1000mL.

9.6 Wide bore serological pipettes and pipette helper.

9.7 Drying Ovens – Capable of maintaining 103-105°C and 178-182°C.

9.8 Desiccator

9.9 Indicating Desiccant – Drierite Anhydrous Calcium Sulfate, Stock # 2305, or equivalent.

9.10 Laboratory Scoopulas

9.11 Muffle furnace (for volatile solids) – Capable of maintaining temperature of 440°C and 550°C.

9.12 Evaporating Beaker (TS/TDS/TVS/OM/FOC) – 50 mL

10. Reagents and Standards

10.1 ERA DataPacK Total Volatile Solids Standard, Cat # 913 for TVS, OM, and FOC analysis, or equivalent.

10.2 ERA QC Plus Solids, Cat #4033 for TS, TSS, and TDS analyses, or equivalent.

10.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent.

10.4 Nanopure Reagent Water – ASTM Type II, or equivalent.

11. Calibration and Standardization

11.1 Balance Calibration – The only required calibration is the analytical balance.

11.2 The balance must be calibrated at least annually by an outside agency and checked **daily** across the range of use, using Class 1 or 2 weights as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

11.3 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

11.3.1 1g for TSS and TVSS with a tolerance of +/-0.0100g

11.3.2 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

12. Procedure

12.1 Create the analytical batch in Horizon.

12.2 Open the Electronic Logbook and create a new worksheet for the batch to be analyzed.

12.3 Pre-Weighed Dried Beaker. Ignite the evaporating beaker at 550°C for 1 hour in a muffle furnace. Cool in desiccator and store there until ready to use. Record their weight prior to use.

12.4 Check that the oven temperature is within required specifications before placing samples into oven as per the most current version of SOPS-GB-Q-030 *Support Equipment*. Document temperature in the Electronic Logbook.

12.5 **Weighing Dry/Muffle Weights**

12.5.1 Oven temperatures are the same as those described for the test being conducted.

12.5.2 Place the samples back into the oven for 15 minutes and repeat desiccating, cooling, and weighing steps until weight change is ≤0.0005g.

12.5.3 If the first and second weights agree within 0.0005 g, or if the weight loss between the first and second weighing is less than 4%, the analysis is complete. If not, place them back into the oven for and additional 15 minutes. Cool in the desiccator, and weigh a third time.

12.5.4 If the third weigh is within 0.0005 g of either the first or second weight or if the weight loss between the second and the third weighing is less than 4%, the analysis is complete.

12.5.5 If the weight loss between the second and third weighing is more than 0.0005g and 4%, heat, cool, and weigh the sample a fourth time and use the fourth weight to calculate the result.

12.6 **Total Solids Determination (TS)**

12.6.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.6.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of NSI Solutions (or equivalent) and pour into a tared and dried evaporating beaker. Record the volume for TS-waters and weight for TS-solids analysis in Electronic Logbook.

12.6.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.6.5 Sample

12.5.4.1 For Solid Samples – Weight out 1 to 1.5g of sample in a tared and dried evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.2 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.6.6 Place the samples to dry in the oven overnight at 103-105°C. (Overnight is a period of time ≥ 8 hours.)

12.6.7 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, obtain the weight of the samples as per Section 13.5, and record in Electronic Logbook.

12.6.8 If Total Volatile Solids (TVS) are also to be performed proceed to Section 12.9. Note: TVS utilizes a different LCS than the TS.

12.7 **Total Suspended Solids Determination (TSS)**

12.7.1 Record Filter IDs and weights in Electronic Logbook for samples and batch QC.

12.7.2 Method blank (MB) – Using a graduated cylinder measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.7.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of Alpha-Trol (or equivalent); record the volume in Electronic Logbook.

12.7.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.7.5 For wastewater, the Pace Analytical Services, Inc. - Green Bay Laboratory uses 500mL in order to meet Wisconsin Reporting Limit requirement of 2.0 mg/L.

12.7.6 For South Carolina work a sample volume is chosen to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 L.

12.7.7 Shake the sample thoroughly and measure the chosen aliquot using a graduated cylinder.

12.7.8 Prepare the filter system with a fresh pre-weighed filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.7.9 Filter the sample and wash the filter 3 times with approximately 10mL of Nanopure water. Maintain the filter vacuum for about 1 minute after filtration is complete.

12.7.10 Remove the filter with forceps and place it back into the evaporating dish.

12.7.11 Place the samples (filters and residue) to dry in the oven a minimum of 3 hours at 103-105°C.

12.7.12 After drying, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.7.13 If Total Volatile Suspended Solids (TVSS) are also to be performed proceed to Section 11.10. TVSS utilizes a different LCS than TSS.

12.8 **Total Dissolved Solids (TDS)**

12.8.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.8.2 Method blank (MB) – Using a graduated cylinder measure out 50mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.8.3 Laboratory Control Sample (LCS) – Using a graduated cylinder measure out 50mL of APG Solids APG plus Standard (or equivalent), record the volume in Electronic Logbook.

12.8.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.8.5 Shake the sample thoroughly and measure a 50mL aliquot using a graduated cylinder.

12.8.6 Prepare the filter system with a fresh filter. Handle the filter with forceps only. Wet the filter with a small volume of Nanopure water to seat it. Discard the rinse water.

12.8.7 Filter the sample and transfer the filtrate to a pre-weighed dried evaporating beaker.

12.8.1 Place the samples to dry in an oven that has it temperature under a program. The oven starts out at <100 degrees C and increases in temperature according to the program until it reaches 178 - 182 degrees C. It is then held there for a minimum of 1 hour.

12.8.2 After drying overnight, remove from oven and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.9 **Total Volatile Solids (TVS)**

12.9.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be ≤0.0005g before use.

12.9.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.9.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.9.5 Sample

12.5.4.3 For Solids Samples – Weight out 1 to 1.5g of sample in a pre-dried, tared evaporating beaker. Record the weight in Electronic Logbook.

12.5.4.4 For Aqueous Samples - Shake the sample thoroughly and measure an aliquot (between 1mL and 50mL, typically 50mL) into a pre-dried evaporating beaker using a graduated cylinder. Record the volume in Electronic Logbook.

12.9.6 Follow TS Section to obtain a dried residue.

12.9.7 Place the evaporating beaker or filter and evaporating dish into the muffle furnace at **550°C** for 1 hour.

12.9.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5.

12.10 Total Volatile Suspended Solids (TVSS)

12.10.1 Method blank (MB) – Using a graduated cylinder, measure out 1000mL of Nanopure (or equivalent) water, record the volume in Electronic Logbook.

12.10.2 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker. Record the weight in Electronic Logbook.

12.10.3 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.10.4 Shake the sample thoroughly and measure the chosen aliquot (see sections 11.7.5 and 11.7.6) using a graduated cylinder. Record the volume in Electronic Logbook.

12.10.5 Follow TSS Section to obtain a dried residue.

12.10.6 Place the filter and evaporating dish into the muffle furnace at **550°C** for 1 hour. Write the filter number on the evaporating dish – paper label will burn off.

12.10.7 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.11 Fractional Organic Carbon (FOC) and Organic Matter (OM)

12.11.1 For each sample and QC point place a pre-dried evaporating beaker on the balance and record the weight. The initial weights must be confirmed by obtaining a second weight. The weight change must be $\leq$0.0005g before use.

12.11.2 Method blank (MB) – Using a graduated cylinder, measure out 50mL of Nanopure (or equivalent) water, pour into a tared evaporating beaker, record the weight in Electronic Logbook.

12.11.3 Laboratory Control Sample (LCS) – Using a graduated cylinder, measure out 50mL of ERA DataPacK Total Volatile Solids Standard (or equivalent), pour into a tared evaporating beaker, and record the weight in Electronic Logbook.

12.11.4 Sample Duplicate (DUP) – At least one DUP must be analyzed per ten samples.

12.11.5 Samples – Into a tared evaporating beaker weigh out about 1-1.5g of sample. Record the weight in Electronic Logbook.

12.11.6 Follow TS Section to obtain a dried residue.

12.11.7 Place the evaporating beakers into the muffle furnace at **440°C** for 1 hour.

12.11.8 After 1 hour, remove from muffle furnace and place into the desiccator to cool. After the samples have cooled, record the weight of the samples as per Section 13.5 in Electronic Logbook.

12.12 **Entering Results into Horizon**

12.12.1 Post samples and batch QC using Electronic Logbook.

12.12.2 Evaluate batch QC in Horizon.

13. Quality Control

13.1 Refer to the most current version of the Pace Quality Manual Appendix I *Quality Control Calculations* and SOP S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

13.2 Method Blank (MB) – A MB is carried through all prep procedures and analyzed with a frequency of 5% or one per batch of up to 20 environmental samples.

13.2.1 TS, TDS, TVS, FOC, and OM all use 50mL of deionized water for the method blank.

13.2.2 TSS and TVSS both use 1000mL of deionized water for the method blank.

13.2.3 For tests that are reported in %, the MB is evaluated in mg/Kg. The mg/kg result must be less than 20 mg/kg. Data can be reported if the sample mg/Kg result is 10x greater than the method blank. The value reported in Epic Pro will be adjusted to 0%.

13.2.4 For tests that are reported in mg/L the absolute value must be < PRL.

13.2.5 When measurements are above the PRL, terminate analysis, correct the problem, verify the calibration, and reanalyze all analytical samples analyzed since the last compliant calibration blank

13.2.6 If the analyte is detected in the method blank between the MDL and the PRL, then samples need to be qualified with a "B" when MDL reporting is required and sample results are greater than the MDL and less than 10 times the absolute value detected in the blank. Additionally, method blank acceptance may be based on project specific criteria or determined from analyte concentrations in the sample and are evaluated on a sample-by-sample basis. Other criteria may apply, such as regulatory limit and the analyte concentration in the samples.

13.3 Laboratory Control Sample (LCS) – The LCS is carried through all preparation procedures with frequency of 5% or one per batch of up to 20 environmental samples.

13.3.1 ERA DataPacK Total Volatile Solids Standard, Cat. # 913 is used for TVS, FOC, and OM analysis. Equivalent can be used.

13.3.2 APG Solids APG Plus standard, Cat #4033 is used for TDS analysis. Equivalent can be used.

13.3.3 NSI Solutions, Cat # QCI-084 for TS, TSS, and TDS analysis, or equivalent. Equivalent can be used.

13.3.4 A Laboratory Control Spike Duplicate (LCSD) must be analyzed if the client requests one.

13.3.5 The LCS recovered concentration must be within calculated in-house limits or default limits of $\pm$ 20%.

13.3.6 For tests that are reported in %, the LCS is evaluated in mg/L.

13.3.7 When measurements are outside the control limits, check for errors in calculations, standards preparation, and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.8 If no errors are found, sufficient sample is available, it is within hold, re-prepare the LCS (and/or LCSD) and all associated samples. If the recovery is within the limits in the analysis, accept the second set of data. If the recovery is still outside the limits after re-analysis, contact the PM to determine the resolution. If the client does not require additional work, report the data, applying an appropriate flag to the samples associated with the non-compliant LCS.

13.3.9 If sufficient sample volume is not available or the samples are now outside of hold, report the sample data with a qualifier flag (L0) on each of the samples associated with the non-compliant LCS (and/or LCSD). Contact the project manager regarding the occurrence.

13.3.10 When an LCSD is performed, the precision between the LCS and LCSD must be ≤10% RPD.

13.3.11 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.3.12 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an appropriate data qualifier.

13.4 Duplicate – Analyze one sample in duplicate per 10 samples or less. The sample used for the duplicate is selected at random, unless specified by the client. The relative percent different (RPD) between duplicates must be ≤10%.

13.4.1 When measurements are outside the control limits, check for errors in calculations, standards preparation and spiking. If an error or problem is found and can be corrected by amending the calculations and the results falls within the limits, accept the data and report without a qualifier flag.

13.4.2 If no calculation errors are found when measurements are outside the control limits, flag the parent sample with an "R1" data qualifier

13.5 Constant Weight – Place the samples back into the 103-105°C oven, and/or 440/550°C muffle furnace for 15 minutes and repeat cooling, weighing, and desiccating steps until weight change is ≤0.0005g or the weight loss between the two weights is less than 4%.

13.6 Balance – The balance must be verified daily across the range of use, before use, as per the most current version of SOP S-GB-Q-030 *Support Equipment*.

13.6.1 Each weighing of the batch is bracketed at the beginning and end with either a 1g or 50g verification.

13.6.2 1g for TSS and TVSS with a tolerance of +/-0.0100g

13.6.3 50g for TS, TDS, TVS, OM, and FOC with a tolerance of +/-0.5000g

13.7 Oven – The oven temperature must be within required specifications before placing samples into oven and the temp is recorded in the Electronic Logbook with the correction factor, as per the most current version of SOP S-GB-Q-030 *Support Equipment.*

13.8 Hold – When preparation of a sample exceeds 7 days past the time of collection, notify the project manager before proceeding. If a sample is run past 7 days after collection, flag the result with an "appropriate data qualifier and fill out a LabTrack report.

13.9 See attachments Table A and Table B for a summary of QC

14. Data Analysis and Calculations

14.1 Total Solids (TS), Total Suspended Solids (TSS), Total Dissolved Solids (TDS) in mg/L $= \frac{(A - B) \times 1000 \times 1000}{V}$

Where:	V = sample volume (mL)
Total Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)
Total Suspended Solids	A = final weight (dry weight of residue and filter, g) B = filter weight (g)
Total Dissolved Solids	A = final weight (dry weight of residue and dish, g) B = dish weight (g)

14.2 Volatile Solids (TVS and TVSS mg/L) $= \frac{(A - D) \times 1000 \times 1000}{V}$

14.3 Volatile Solids (TVS %) $= \frac{(A - D) \times 100}{(A - B)}$

14.4 % FOC $= ((1-((D-B) / (A - B))) \times 100) \times (0.58)$

14.5 % OM = ((1-((D-B) / (A-B))) x 100)

Where:

A = dry weight (weight of dried residue and dish, g)
B = dish weight (g)
C = weight of wet sample and dish (g)
D = muffled weight (weight of residue and dish after ignition, g)
V = volume (mL)

14.6 Precision - The relative percent difference (RPD) is calculated as:

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

Where: **S** = sample value
D = duplicate value

14.7 Constant Weight is calculated as:

$$\textbf{Constant Weight (\%)} = \frac{|FW - CW| * 100}{(FW + CW)/2}$$

Where: FW = First Weighing, mg

CW = Confirmation Weighing, mg

If FW – CW is 0.0005 g or less, then there is not a need to calculate as a percentage

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1 See Section 13 and Table 3: Quality Control, and Table 4: Data Assessment for a listing of Quality Control Criteria.

Table 5.
QUALITY CONTROL

Preparation Method ⇨ Quality Control Measure ⇩	**SM 2540G – ASTM D2974-87** **Frequency**	**Acceptance Criteria**
Method Blank	One per batch of samples, up to 20 environmental samples, whichever is more frequent.	For solid samples the MB must be < 20 mg/Kg and is reported as 0%. For aqueous samples the MB absolute value must be <RL. Detections > MDL but < RL must be evaluated for data qualification.
Laboratory Control Spike and Duplicate	One per batch of samples, up to 20 environmental samples, whichever is more frequent. None performed on TVSS.	Recovery must be within +/- 20% of the true value. RPD must be < 10%
Duplicate	One pair per batch of samples, up to 10 environmental samples, whichever is more frequent.	RPD must be < 10%

Table 6.
ANALYST/TECHNICIAN DATA ASSESSMENT

Analytical Method Acceptance Criteria⇨ Data Assessment Measure ⇩	**SM2540G – ASTM 2974-87 If these conditions are not achieved ⇒**
Method Blank	• 1
Duplicate	• 2
Accuracy & Precision Laboratory Control Spikes	• 3

1. In the absence of project specific requirements, sample detects less than 20 times the method blank contamination level are reported with the appropriate data qualifier. Sample detects greater than 20 times the method blank contamination are reported without qualification.
2. In the absence of project specific or method requirements, in-house generated limits will be used. If the DUP fails precision control limits flag the parent with the appropriate data qualifier.
3. If sample volume does not allow re-analysis the entire prep/analytical batch of samples shall be flagged with the appropriate qualifier qualifiers to reflect the deficiencies.

16. Corrective Actions for Out-of-Control Data

16.1 See Section 13 and Table 6.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1 If not specifically listed in Section 13 or Table 6, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers.

18. Method Performance

18.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

18.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

18.1.2 Demonstration of Capability (DOC) – Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC). DOC's must be repeated annually. Analyze 4 replicate environmental sample aliquots. Calculate the mean and standard deviations of the results. The relative percent different (RPD) between replicates must be $\leq$ 5% or the study must be repeated.

19. Method Modifications

19.1 Not applicable to this SOP.

20. Instrument/Equipment Maintenance

20.1 Maintain the analytical balance, ovens, and furnaces according to the most current revision of SOP S-GB-Q-030, *Support Equipment.*

21. Troubleshooting

21.1 Refer to the support equipment operating manual.

22. Safety

22.1 **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2 **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001 *Waste Handling and Management* most current revision or replacement).

23.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1 Pollution prevention encompasses any technique or procedure that reduces or eliminates the quantity or toxicity of waste at the point of generation.

24.2 The laboratory Chemical Hygiene Plan/Health and Safety Plan contains additional information on pollution prevention.

25. References

25.1 Pace Analytical Quality Assurance Manual (most current revision or *replacement).*

25.2 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems" (most current revision or replacement).

25.3 Standard Methods for the Examination of Water and Wastewater, SM Method 2540 B-2011.

25.4 Standard Methods for the Examination of Water and Wastewater, SM 2540 C-2011.

25.5 Standard Methods for the Examination of Water and Wastewater, SM 2540 D-2011

25.6 Standard Methods for the Examination of Water and Wastewater, SM 2540 G-2011.

25.7 ASTM D2974-87, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils, May 29, 1987

25.8 VA30007.00.004, Recommendations Regarding the Sampling and Analysis of Fractional Organic Carbon (FOC) in Soils, Technical Decision Compendium, Rule OAC 3745-300-07, March 2000

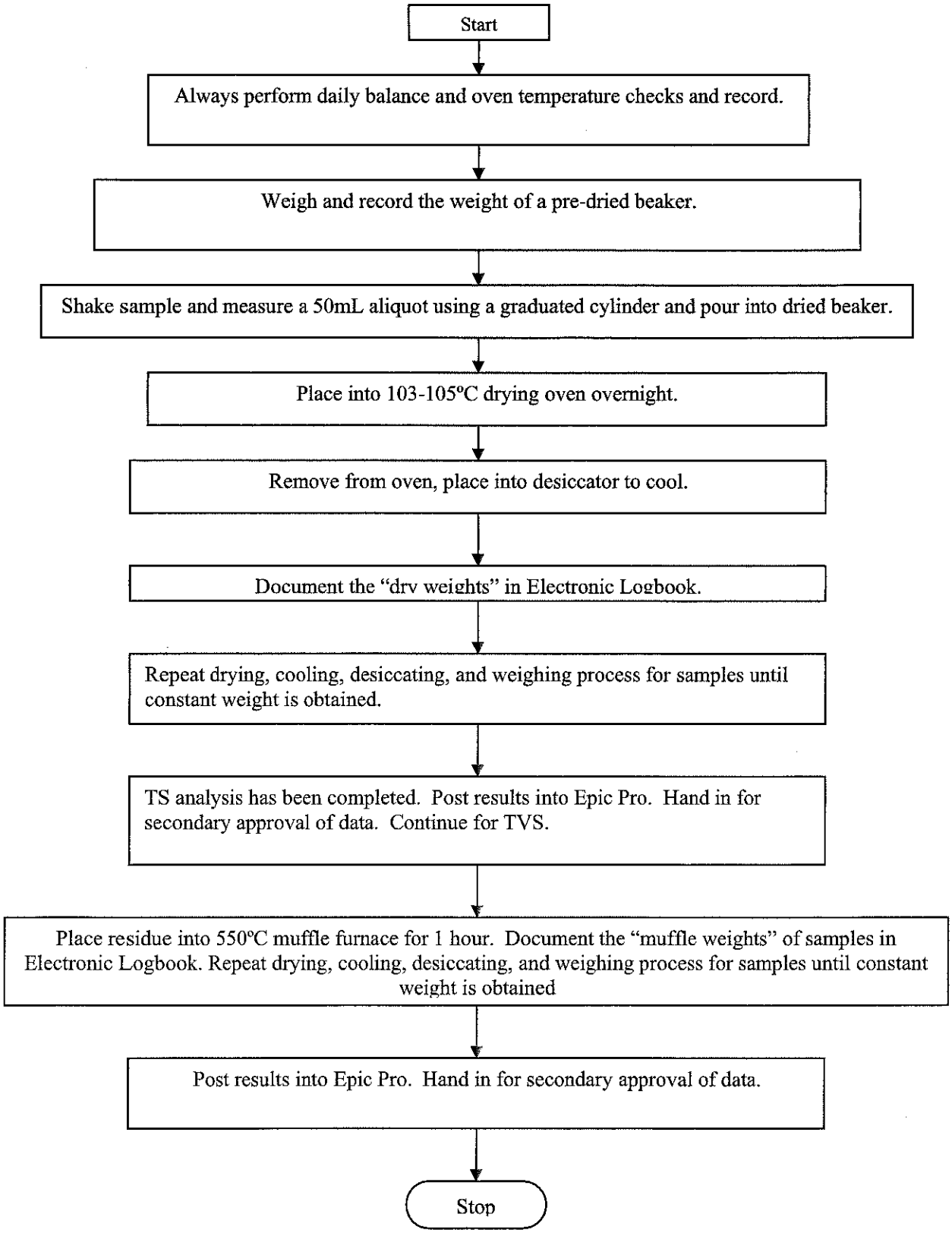
26. Tables, Diagrams, Flowcharts, Appendices, Addenda, etc.

26.1 Flow charts – Attachment I.

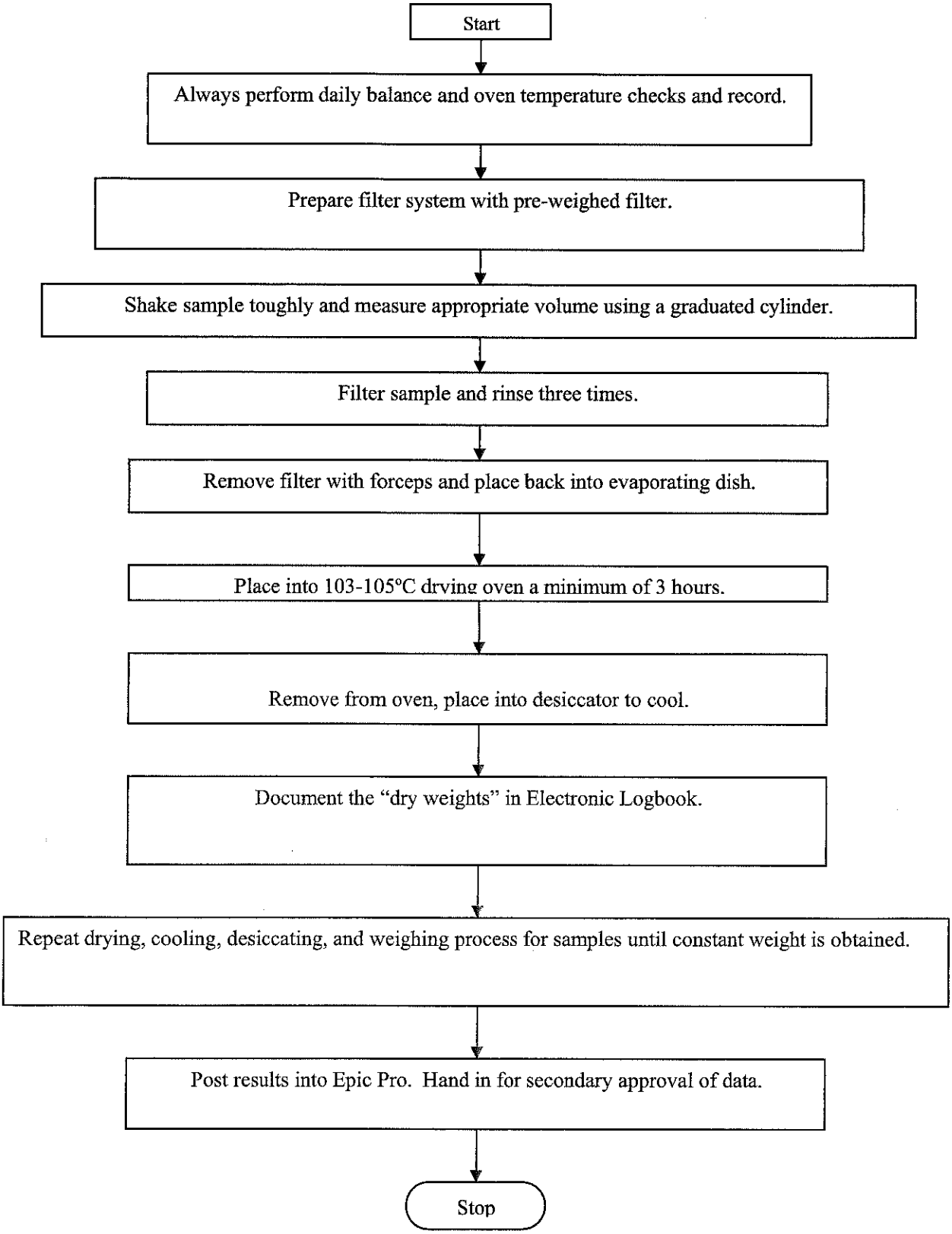
27. Revisions

Document Number	Reason for Change	Date
S-ALL-GB-I-014-Rev.1	Consolidated SOPs ALL-I-014-rev.1, ALL-GB-I-014-rev.1, and GB-I-050-Rev.0. Section 12.6.2 – added South Carolina minimum residue requirements.	24Aug2007
S-ALL-GB-I-014-Rev.2	Corrected FOC and OM calculations and definitions	05Oct2007
S-ALL-GB-I-014-Rev.3	Clarified analysis steps for each test. Changed qualifiers to match Epic Pro.	28Apr2008
S-ALL-GB-I-014-Rev.4	Included notations on how to evaluate MB and LCS for tests reporting in %. Updated Signature Page to Periodic Review.	29May2009
S-GB-I-068-Rev.00	Updated numbering format to match SOP: S-ALL-Q-003, Document Numbering Changed Qualifier from & to L0 and * to R1.	21Sept2010
S-GB-I-068-Rev.01	Updated SOP format to be consistent with SOP: S-GB-Q-017 *Preparation of SOPs* Updated SOP references throughout document. Throughout document: Changed LimsLink to Electronic Prep Log. Sections 2, 3.2, 8.1.2: Removed information relating to Fixed Solids (FS). Sections 2, 3.3, 12.11: Removed information relating to ASH. Section 12.8.1: Changed temperature program to ramp up to 100 deg C. Section 12.10.2: Added LCS requirement to TVSS methods. Tables A and B: Moved and renamed in appropriate sections of SOP.	29Nov2012
S-GB-I-068-Rev.02	Section 25: Updated references to most current revision of the Standard Methods.	16Dec2013

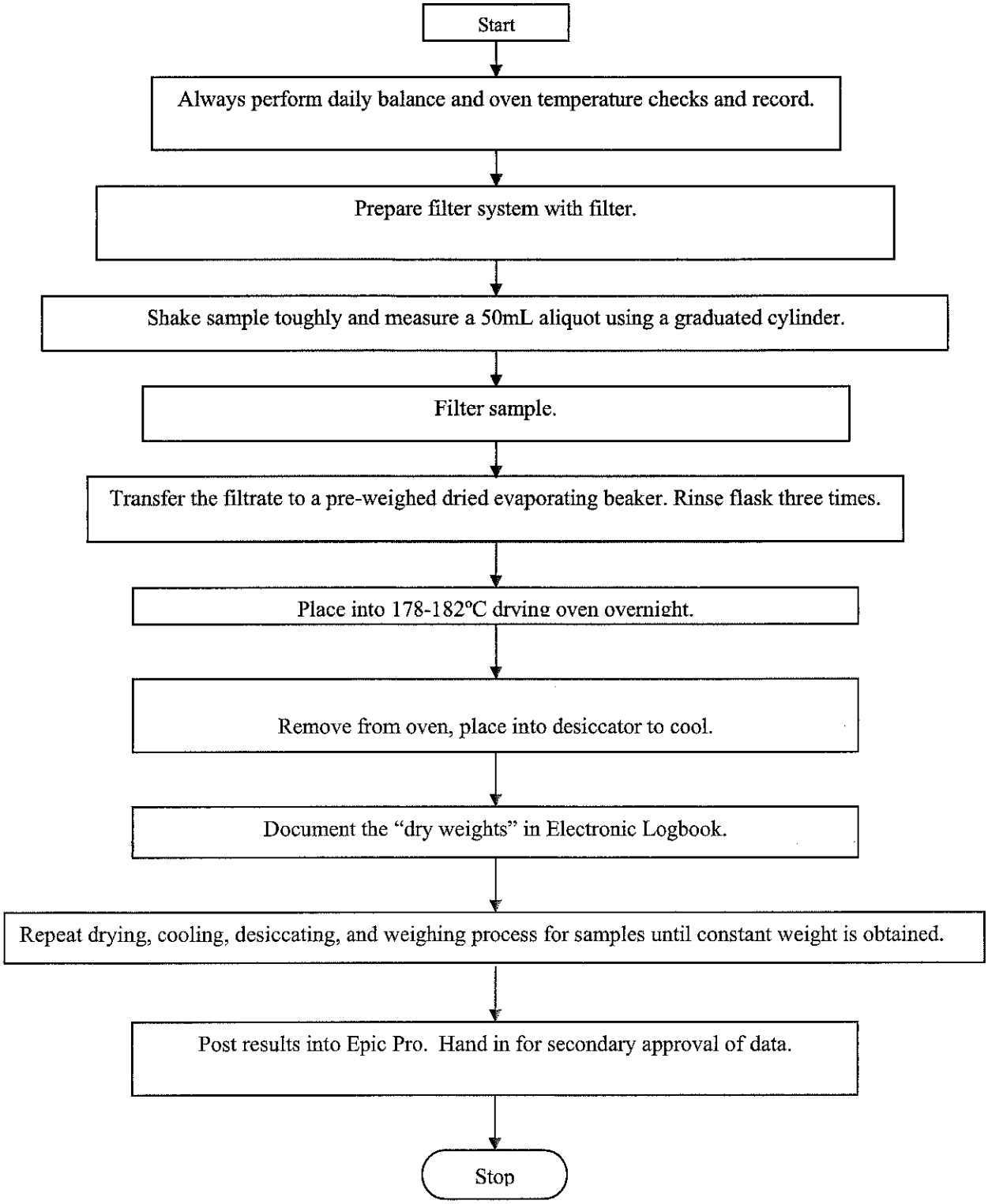
Attachment I – TS and TVS FLOWCHART



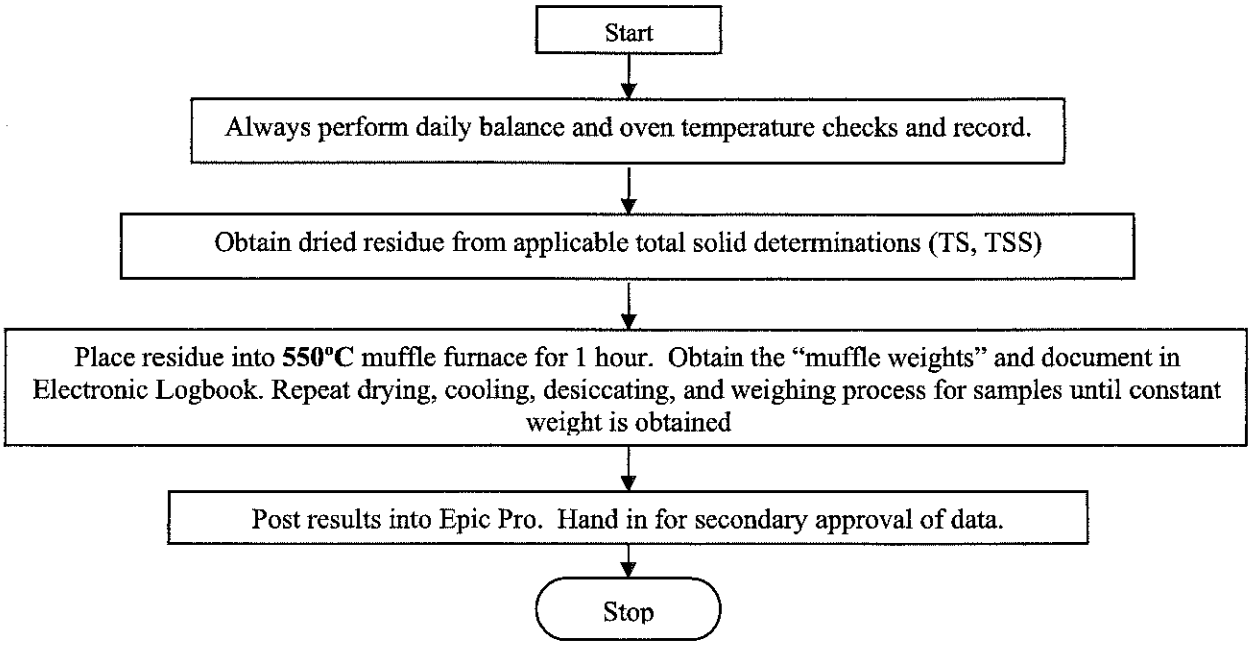
Attachment I – TSS FLOWCHART



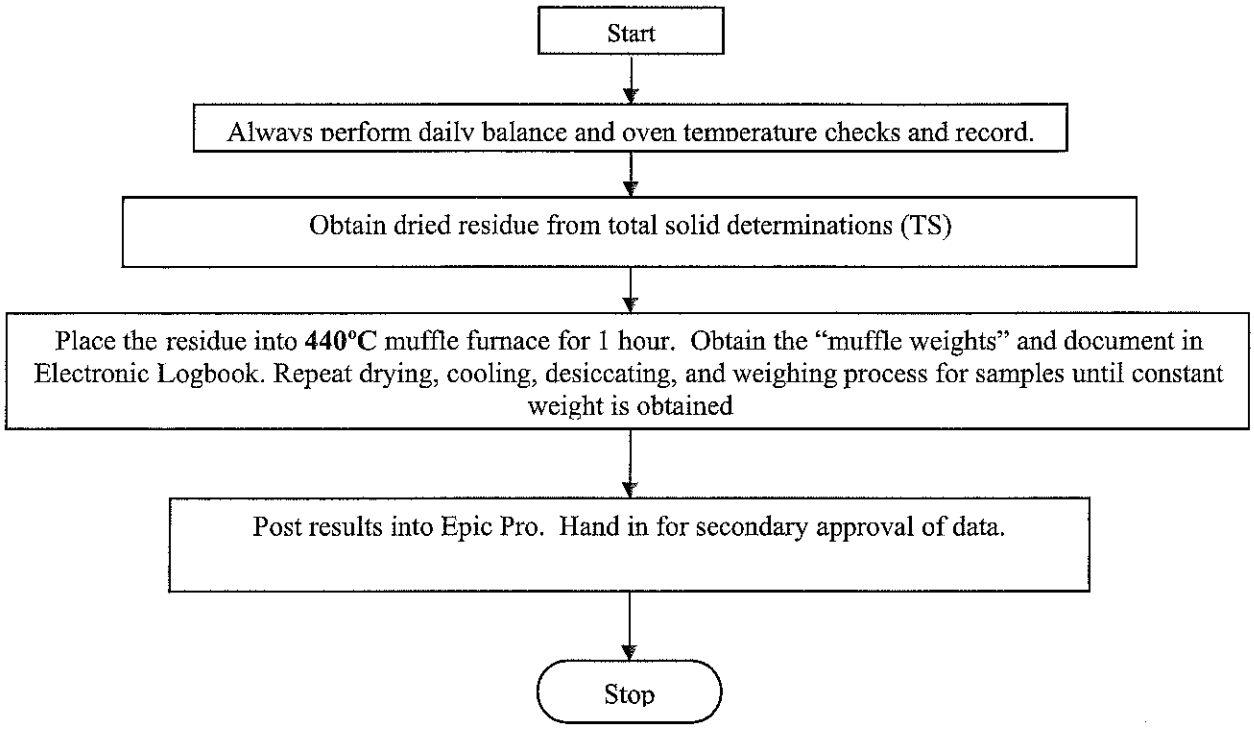
Attachment I – TDS FLOWCHART



Attachment I – TVS and TVSS FLOWCHART



Attachment I – OM and FOC FLOWCHART



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STANDARD OPERATING PROCEDURE

Hardness by Calculation

Reference Methods: Standard Methods for the Examination of Water and Wastewater, 2340 B

SOP NUMBER:	S-GB-M-009-REV.02
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-009-REV.01

APPROVAL

Nils K Melberg — 09/28/11
Nils Melberg, Laboratory General Manager — Date

Kate E. Grams — 9/28/2011
Kate Grams, Laboratory Quality Manager — Date

Chad Rusch — 09/28/2011
Chad Rusch, Department Manager — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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Table of Contents

Section **Page**

1. Purpose 3

2. Scope and Application 3

3. Summary of Method 3

4. Interferences 3

5. Safety 3

6. Definitions 3

7. Sample Collection, Preservation and Handling 3

8. Equipment and Supplies 3

9. Reagents and Standards 4

10. Calibration 4

11. Procedure 4

12. Quality Control 4

13. Method Performance 4

14. Pollution Prevention and Waste Management 5

15. References 5

16. Tables, Diagrams, Flowcharts, Appendices, Addenda etc. 5

17. Revisions 5

1. Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide a consistent format for Hardness by calculation.

2. Scope and Application

The measure to precipitate soap was the original definition of Hardness. The main two ion species that are responsible for hardness are Calcium and Magnesium. Current Total Hardness is defined as the sum of Calcium (Ca) and Magnesium (Mg) concentration expressed as Calcium Carbonate ($CaCO_3$).

3. Summary of Method

Ca and Mg ICP results are put through the equation in section 12 of this SOP, resulting in Hardness as Calcium Carbonate. This SOP does not apply the generation of the Ca and Mg results (see SOPs S-GB-M-0014, *Determination of Metals by Inductively Coupled Plasma (ICP) by 6010B and S-GB-M-005 Determination of Metals by Inductively Coupled Plasma (ICP) by 6010C, most current revisions or replacements*).

4. Interferences

Not Applicable.

5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training

6. Definitions

Refer to Section 10.0 of the most current version of the Pace Quality Manual for the terms used at Pace Analytical. When definitions are not consistent with NELAC defined terms, an explanation will be provided in this SOP.

7. Sample Collection, Preservation and Handling

Not Applicable.

8. Equipment and Supplies

Not Applicable

9. Reagents and Standards

Not Applicable.

10. Calibration

Not Applicable.

11. Procedure

Analyze water samples for Calcium and Magnesium as outlined in SOPs S-GB-M-005, *Determination of Trace Metals by Inductively Coupled Plasma (ICP)by 6010C* (most current revision or replacement) and S-GB-M-014, *Determination of Trace Metals by Inductively Coupled Plasma (ICP)by 6010B* (most current revision or replacement).

11.1 Calculate Total Hardness as:

11.1.1 Hardness as $CaCO_3$ in mg/L = 2.497 * $[Ca_{\text{in mg/L}}]$ + 4.118 * $[Mg_{\text{in mg/L}}]$

12. Quality Control

Follow quality control criteria as outlined in SOPs S-GB-M-005, *Determination of Trace Metals by Inductively Coupled Plasma (ICP) by 6010C* (most current revision or replacement); S-GB-M-014, *Determination of Trace Metals by Inductively Coupled Plasma (ICP) by 6010B* (most current revision or replacement).

13. Method Performance

13.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

13.1.1 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.1.2 An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.1.3 An annual method detection limit (MDL) study will be completed per S-GB-Q-020, *Determination of LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.1.4 Periodic performance evaluation (PE) samples are analyzed per S-GB-Q-021, *Proficiency Testing Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

14. Pollution Prevention and Waste Management

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents, samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management* (most current revision or replacement).

15. References

15.1 Standard Methods for the Examination of Water and Wastewater.

15.2 S-GB-M-005 *Determination of Metals by Inductively Coupled Plasma (ICP) by 6010C* (most current revision or replacement).

15.3 S-GB-M-0014, *Determination of Metals by Inductively Coupled Plasma (ICP) by 6010B* (most current revision or replacement).

16. Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

Not Applicable.

17. Revisions

Document Number	Reason for Change	Date
GB-I-022-Rev.0	Updated and Reformatted SOP body to Pace Analytical format	27Oct2005
S-GB-I-022-Rev.1	Updated Signature Page Updated Section 3 with current SOP references Updated Sections 6, 12, 13, 14, 15 and 16 with current SOP references	11Dec2007
S-GB-M-009-REV.00	Updated signature page. Renumbered document. Deleted Section 7. Updated SOP references in Sections 3, 11, 12 and 15.	04Dec2008
S-GB-M-009-REV.01	Updated Signature Page	26May2009
S-GB-M-009-Rev.02	Section 14: Updated Waste SOP All Sections: Removed References to EPA Method 6020/A; Updated References to EPA Method 6010B/C Section 13.1.1: Updated MDL SOP Section 13.1.4: Updated PT SOP	27Sep2011

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STANDARD OPERATING PROCEDURE

Determination of Trace Metals in Waters and Wastes By Inductively Coupled Plasma Mass Spectroscopy

Reference Methods: SW-846 6020A WITH 3010A AND 3050B

SOP NUMBER:	S-GB-M-006-REV.04
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-006-REV.03

APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/05/11

Kate Grams, Laboratory Quality Manager — Date: 11/10/11

Chad Rusch, Department Manager — Date: 11/15/2011

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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Table of Contents

Section		Page
1.	Purpose	3
2.	Scope and Application	3
3.	Summary of Method	3
4.	Interferences	4
5.	Safety	4
6.	Definitions	5
7.	Sample Collection, Preservation and Handling	5
8.	Equipment and Supplies	6
9.	Reagents and Standards	6
10.	Set Up and Calibration	8
11.	Procedure	9
12.	Quality Control	14
13.	Method Performance	18
14.	Pollution Prevention and Waste Management	18
15.	References	19
16.	Revisions	20

1. Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide a consistent format for acid digestion and analyzing samples using the ICPMS. It applies to the Thermo X Series II ICPMS.

2. Scope and Application

2.1 Inductively coupled plasma-mass spectrometry (ICPMS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in biota, aqueous, and solid extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. Dissolved samples are typically acid digested prior to analysis. Acid digestion prior to filtration and analysis is required for aqueous and solid samples for which total (acid-leachable) elements are required.

2.2 This SOP is applicable to the determination of the analytes listed in Table A, at a minimum. On instrument reporting limits are listed in Tables M, N, and O, based on their matrix and prep method. Please see the QA manager for the most current MDLs for each method.

2.3 If Method 6020A is used to determine any analytes not listed in Table A, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the matrix to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

2.4 Use of this method is restricted to analysts who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

3. Summary of Method

3.1 Prior to analysis samples, which require total ("acid-leachable") values, must be digested using appropriate sample preparation methods. A number of methods are recommended in SW846 for the sample preparation/digestion of various matrices for trace metals analysis by ICPMS. The prep methods utilized are listed below. The analytes they are applicable for are listed in the attachments.

3.1.1 **SW846 3010A**	Wastes, aqueous samples	Found in attachment I
3.1.2 **SW846 3050B**	Soils, Sludges, solid wastes	Found in attachment II
3.1.3 **M3050B**	Biota	Found in attachment IV

3.2 Method 6020A describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

4. Interferences

4.1 Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

4.2 Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. Examples include ArCl+ (mass 75), which interferes with As. ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

4.3 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

4.4 Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.5 It is important to note that matrix matching acid concentrations and compositions between standards, blanks and samples is required and cannot be ignored.

4.6 Chromic acid should never be used to clean any container used in ICP-MS analysis.

4.7 Borosilicate glass in sample containers can lead to interference of Boron concentrations in samples. This is also true of volumetric flasks, thus when dilution in the flask is complete the standards must be removed as soon as possible from the dilution container and placed into a clean plastic container.

5. Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2 A reference file of Material Safety Data Sheets (MSDS) is made available to all personnel involved in the chemical analysis, and is located at the front desk. A formal safety plan has been prepared and is distributed to all personnel with documented training.

5.3 Analysts should take necessary safety precautions when handling chemicals and samples. Proper personal protective equipment may include safety gloves, lab coats, and safety glasses or goggles. Analysts should be familiar with the MSDS sheets for all chemicals and reagents they use for this procedure and the location of the MSDS sheets within the laboratory. Any questions or concerns should be taken to the laboratory Chemical Hygiene/Safety Officer.

6. Definitions

Refer to the most current version section 10 of the Pace Quality Manual.

7. Sample Collection, Preservation and Handling

7.1 All sample containers must be HDPE, glass, or Teflon. The containers are purchased pre-cleaned and documented to be contaminant free.

7.2 Aqueous Samples

7.2.1 Dissolved samples must be filtered through a 0.45-um pore diameter membrane filter at the time of collection or as soon after as practically possible. The laboratory can perform the filtration if the step was not performed in the field. The filtrate is then preserved to a pH<2 with nitric acid, acid not to exceed 2% of the container capacity.

7.2.2 Total samples are preserved to a pH<2 with nitric acid, acid not to exceed 2% of the container capacity.

NOTE: Aqueous samples that react violently to the addition of acid maybe collected without chemical preservation with proper variances approved by the regulatory authority. The responsibility of requesting this variance lies with the sample collector.

NOTE: Samples may be preserved in the lab. The samples may not be processed until 24 hours after preservation with a pH test of <2, unless otherwise noted on the data. Samples that do not attain a pH<2 or samples that are filtered in the lab must be flagged with P4.

7.3 Solids samples require no chemical preservation.

7.4 Shipments of samples to the laboratory require thermal preservation in the form of cubed or block ice. At the time of laboratory receipt, proper thermal preservation is checked by measuring the temperature of melt water or when provided the temperature blank. The Pace Analytical acceptable temperature range is 2 to 6^0 C. All QAPjP and regulatory authority requirements become priority over this requirement.

7.5 There are no thermal storage requirements for preserved aqueous samples. Solids samples require storage at 2 to 6^0 C. All QAPjP and regulatory authority requirements become priority over this requirement.

7.6 Sample hold times:

7.6.1 The hold time for solid and preserved aqueous (total and dissolved) samples, excluding Hg, is 6 months.

7.6.2 The hold time for solid and preserved aqueous (total and dissolved) samples for Hg is 28 days.

7.6.3 The hold time for biota samples, excluding Hg, is 6 months after removal from the freezer.

7.6.4 The hold time for biota samples for Hg is 28 days after removal from the freezer.

8. Equipment and Supplies

8.1 All reusable labware (glass, quartz, polyethylene, PTEFE, FEP, etc.) should be sufficiently clean for the task objectives.

8.2 **See Table B for a summary of Equipment.**

8.3 **See Table C for a summary of supplies.**

9. Reagents and Standards

9.1 Standards are used in the tuning of the instrument through the calibration, calibration verification, sample analysis, and continuing calibration verification. Standard solutions include: instrument tuning solutions, calibration and calibration check standards, ICSA/AB, internal standards, low level check standards (LLC or CRI), and spike standards. **Please see Table D for a list of working standards. Table F lists the directions to making intermediate standards from the stock standards listed in Table E.**

9.1.1 Mass Spectrometer Tuning Solution: A solution containing elements representing all of the mass regions of interest (for example, 10 µg/L of $^{23, 24, 25}$Mg and $^{206, 207, 208}$Pb) must be prepared to verify that the mass resolution and mass calibration of the instrument are within the required specifications. This solution is also used to verify that the instrument has reached thermal stability.

9.1.2 Cross Calibration Solution (X Cal): contains 50 ppb of most method analyte elements. This solution is used to calculate the concentration when the detector changes from pulse mode to analog mode. This enables a large linear range while protecting the detector.

9.1.3 Calibration Standards: These are an increasing gradient of concentration for the analytes of interest. These can be made in the laboratory or purchased from commercial suppliers.

9.1.4 Initial Calibration Verification (ICV): The quality control standard is the initial calibration verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard at approximately half (or less than) of the concentration of the high standard used for instrument calibration. An independent standard is defined as a standard composed of the analytes from a source different from those used in the standards for instrument calibration. The limits for the ICV are +/-10% of the true value.

9.1.5 Reporting Limit Verification Standard (RLVS): With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed and meet established acceptance criteria. The RLVS is analyzed prior to any samples being analyzed and at the end of the analytical sequence. Additional RLVSs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the RLVS are +/- 30% of the true concentration. These standards are the first calibration point for the analytes of interest. The analysis of this standard demonstrates the instruments ability to report down to the reporting limit with known accuracy.

9.1.6 Interference Check Solutions (ICSA and ICSAB). The ICSA and ICSAB are prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interferences and provide an adequate test of any corrections. The ICSA and ICSAB are analyzed prior to any samples and at the end of the analytical sequence. Chloride in the ICS provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl\ ^{16}O$ on ^{51}V, and $^{40}Ar\ ^{35}Cl$ on $^{75}As^{+}$. Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese. Molybdenum serves to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various polyatomic isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system to within quality control limits. The ICS-AB recovery limits are +/-20% of the true value. Table D provides a summary of the ICS-A and ICS-AB solution concentrations used.

9.1.7 Continuing Calibration Verification (CCV): This standard is near (or less than) the midpoint of the curve and used to verify that the instrument is still in calibration. The recovery limits for the CCV are +/-10% of the true value.

9.2 Reagents: **Please see Table G for a list of reagents.**

9.3 All standards, reagents, and spiking solutions are kept at room temperature. Stock standards and reagents can be used until they expire. Refer to the most current version of S-ALL-Q-025 *Standard and Reagent Management and Traceability* for stock standard and reagent expiration rules. Intermediate and working standards are given a six month expiration date. However the expiration date may not extend past the earliest expiration date of any stock standard used to create the solution. The use of any standard or reagent will be terminated if any contamination or problems arise prior to the expiration date.

9.4 Log-in of Standards, reagents, and spike solutions are logged as follows:

9.4.1 Stock standards have a copy of their certificate of analysis logged into the LIMS (Epic Pro). Please see the SOP T-ALL-IT-010 (most current revision or equivalent).

9.4.2 Reagents are logged in the same manner.

10. Set Up and Calibration

10.1 Calibration Curve: Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed and is required periodically throughout sample analysis as dictated by the results of the continuing calibration verification standards. Refer to Section 11.4 for calibration and standardization control criteria. Calibration consists of a calibration blank and four or five non-zero standards (analyte dependent) analyzed after a calibration blank. The ICPMS software creates a curve based on this data which is linear regression using Absolute Standard Deviation weighting and uses the following equation: Y=a(x)+b. In calculating x(concentration) from y(response) the b(intercept) is subtracted. The b represents the intercept of the regression line. The correlation coefficient must be ≥0.998.

10.2 Calibration Levels: **Please see Table D for directions on making the calibration standards and Table H to see the calibration levels used in the calculations.**

10.3 Internal Standards: Internal standardization must be used in all analyses to correct for instrument drift and physical interferences. For full mass range scans, a minimum of six internal standards must be used. Procedures described in this SOP for general applications detail the use of six internal standards; ^{45}Sc-CCT, ^{45}Sc-KED, ^{72}Ge, ^{89}Y, ^{115}In, ^{159}Tb, and ^{209}Bi. Additional internal standards such as Ho may be used if results indicate the presence of one or more of the internal standard elements in the samples or if recoveries for certain elements such as Zn or Cd are poor in some matrices. Internal standards must be present in all samples, standards and blanks at identical levels. This is achieved by directly adding the internal standard stock solution (200ppb in 3% HNO3) to all samples, standards and blanks by on-line addition prior to nebulization using a second channel of the peristaltic pump and a mixing T-connector. The concentration of the internal standard should be sufficiently high that good precision is obtained in the measurement of the isotope used for data correction and to minimize the possibility of correction errors if the internal standard is naturally present in the sample. A final concentration at the nebulizer of approximately 100 ppb will result from the addition of a 200 ppb solution. **Table I provides a list of acceptable internal standards and their suggested associated elements.**

10.3.1 Due to matrix interferences one or more of the internal standards may not be suitable. It is up to the analyst to recognize and correct the problem by diluting the sample(s) for reanalysis. NOTE: The interpolation between 2 internal standards is still possible when one internal standard is present in concentration sufficient to cause recoveries outside the 70-130% range. The sample MUST be diluted to a level where the background concentration of the internal standard saturates the contribution from the sample. This procedure may result in some analytes being diluted below the PRL and must be appropriately flagged.

10.3.2 During the determination, the software uses the ratio of analyte and internal standard intensities to adjust the final concentration values. Ratios are based on the intensities in the sample vs. the calibration blank intensities.

10.3.3 An internal standard without Li must be used if Li is an analyte of interest. If Li is to be used as an internal standard, and Li is an analyte of interest, an isotopically enriched Li standard must be used such that the analyte can be differentiated from the internal standard. (i.e. ^{6}Li enriched internal standard to report ^{7}Li in samples.)

10.3.4 Interference Calculations- Interference equations are used to correct for isobaric elemental and polyatomic interferences. All equations can be adjusted if necessary or added if the analyst determines that any particular correction is insufficient or if an equation is over correcting the data. **Table J shows the recommended Elemental Interference Equations**

10.3.4.1 Acquisition Mode:

Points per Mass:	1
Number Replicates:	3
Integration Time:	10 ms for all elements except Na, As, Se, Kr ; 5 ms for Na, and 50 ms for As, Se, Kr.

10.3.4.2 Example Peristaltic Pump Program:
Uptake time: 20 sec [1]
Stabilization Time (CCT pre experiment):45 sec
Stabilization Time (KED Changeover): 60 sec
Monitored Wash: 15Sec Minimum, 200 Maximum

10.3.4.3 **Acquisition masses and integration times can be found in Table K.**

11. Procedure

11.1 Solubilization and digestion procedures are presented in the Attachments I, II, and III (e.g. Methods 3010A and 3050B both Solids and Biota).

11.2 Instrument Startup:

11.2.1 Verify argon supply and pressure.

11.2.2 Turn on water chiller and exhaust fan.

11.2.3 Insure that the internal standard solution bottle is adequately full

11.2.4 Verify contents of auto sampler rinse port reservoir.

11.2.5 Empty the waste reservoir.

11.2.6 Ignite the plasma and allow at least 25 minutes of warm-up while scanning the mass analyzer. The tuning procedures may then be carried out.

11.2.7 Insure that all peristaltic pump tubes are in good condition and correctly clamped into the peristaltic pumps. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from extreme pulsations by introducing a bubble into each line and observing its progress.

11.3 Mass Tuning: Allow 25 minutes of scanning for the Instrument to achieve thermal stability. Aspirate the 10ppb Tuning Solution inserting both the sample and internal standard delivery lines into the tune solution so as not to dilute the tune solution. Run each of the following performance reports, print the passing report, and save the mode by month, day, year (no commas) into the configurations list by type (e.g. Standard Mode09052008, CCT Mode09052008, and CCTKED Mode09052008).

11.3.1 EPA Performance Report (Xt Standard Mode)

11.3.1.1 Mass Resolution (10 Sweeps, 5 Reps)
Acquisition Parameters: Peak width measured at 5% peak maximum.
Dwell Time (mSec): 10
Point Spacing: 0.05amu
Mass Limits: 0.65 – 0.85 amu (Max error 0.10 amu)
Defined Masses: ^{24}Mg, ^{25}Mg, ^{26}Mg, ^{206}Pb, ^{207}Pb, ^{208}Pb

11.3.1.2 Sensitivity and Stability (35 Sweeps, 5 Reps)
Mass: Dwell (mSec) : %RSD : Counts
(NA=Not Applicable)
^{5}Bkg 500.0 : NA : <1
^{7}Li 10.0 : 2.0 : >60,000
^{24}Mg 10.0 : 2.0 : >10,000
^{25}Mg 10.0 : 2.0 : >10,000
^{26}Mg 10.0 : 2.0 : >10,000
^{59}Co 10.0 : 2.0 : >150,000
^{137}Ba++ 10.0 : NA : NA
^{115}In 10.0 : 2.0 : >400,000
^{137}Ba 10.0 : NA : NA
^{138}Ba 10.0 : NA : NA
^{140}Ce 10.0 : NA : NA
^{156}CeO 100.0 : NA : NA
^{206}Pb 10.0 : 2.0 : >10,000
^{207}Pb 10.0 : 2.0 : >10,000
^{208}Pb 10.0 : 2.0 : >10,000
^{220}Bkg 500.0 : NA : <1
^{238}U 10.0 : 2.0 : >800,000

11.3.1.3 Oxides and Doubly Charged (Ratio Results)
^{137}Ba++/^{137}Ba: <0.0300
^{156}CeO/^{140}Ce: <0.0200

11.3.2 CCT Performance Report (Xt CCT)
Sensitivity and Stability (35 Sweeps, 5 Reps)
Acquisition Parameters: Mass : Dwell (mSec) : %RSD : Counts
^{7}Li 10.0 : 2.0 : >10,000
^{9}Be 10.0 : 2.0 : >2,000
^{11}B 10.0 : 2.0 : >2,000

11.3.3 KED Performance Report (Xt CCT-KED)

11.3.3.1 Sensitivity and Stability (35 Sweeps, 5 Reps)
Acquisition Parameters: Mass : Dwell (mSec) : %RSD : Counts
^{78}Se 100.0 : NA : <20
^{115}In 10.0 : 2.0 : >100,000
^{140}Ce 10.0 : NA : NA
^{156}CeO 50.0 : NA : NA

11.3.3.2 Ratio Results
^{156}CeO/^{140}Ce <0.0200

11.3.4 Introduce the internal standard and aspirate a new rinse blank for 5-10 minutes to eliminate any carry-over into the calibration blank.

11.3.5 The Xcal solution does not need to be run unless a deviation between the pulse counting and analog counting methods is observed in the spectra. The analog counting will appear to sit above the pulse baseline in an observed spectra and indicates that the cross calibration needs to be performed. Also, an indication of when the Xcal needs to be reset is when the calibration loses its linearity (this will likely occur first with High Resolution Mineral elements like Na and K). Aspirate the Xcal solution in Standard Mode, with both the sample delivery and internal standard lines (so as to not dilute the Xcal solution). Follow the options in 11.3.5.1 or 11.3.5.2. Step 11.3.5.3 needs to be followed after a detector change.

11.3.5.1 Cross Calibration Only. This is done when the deviation between the pulse and analog counting methods is observed (from spectra or from linearity observations) and the minimum counts per second listed in the tuning section are achievable. Run the Instrument Calibration wizard in PlasmaLab when deviation of the analog spectra is observed. The detector cross calibration is selected by default in the wizard. This will reset the detector-gating plateau such that the analog spectra (dashed line in the spectra) will sit directly on top of the pulse baseline (solid line in the spectra).

11.3.5.2 Detector Setup and Cross Calibration. This is done when the minimum counts per second listed in the tune section are not achievable. Thus, the detector dynode value will be adjusted and a new cross calibration will be performed with the new detector voltage setting. Launch the Instrument Calibration wizard in PlasmaLab and select detector set up. The detector cross calibration will be checked by default, also select the detector set up portion in the wizard. The voltage applied to the detector will be set first to achieve acceptable sensitivity followed by a detector cross calibration with the new detector setting.

11.3.5.3 Dead Time Experiment. This experiment must be performed after a detector is replaced. The dead time experiment will identify the delay or "dead time" associated with a particular detector. The dead time is the delay between the pulse measurement and analog measurement when the detector gate is dropped. In the experiment a 1ppb and 10ppb solution of Uranium is run, and the ratios between ^{235}U and ^{238}U is calculated for both concentrations. The values for the ratios are adjusted such that the ratio values are as close to identical as possible and that value (dead time in nanoseconds) is entered into PlasmaLab software via the Advanced Page.

11.3.6 Tune Procedure Summary (Use this order of events, some adjustments may be required after an auto tune)

11.3.6.1 Torch Box Alignment

11.3.6.2 Xt Standard Mode Autotune or X Series II Factory auto tune; Mass Calibration

11.3.6.3 EPA Performance Report

11.3.6.4 Detector X Calibration (Note: Return to 11.3.6.2 if the Cross Calibration trips a detector set up sequence, excluding the Mass Calibration)

11.3.6.5 XT CCT Mode Auto tune

11.3.6.6 XT CCT Mode Performance Report

11.3.6.7 XT CCT/KED Auto tune

11.3.6.8 XT CCT/KED Performance Report

11.4 Automated Calibration, Quality Control and Sample Analysis:

11.4.1 Prepare calibration standards, blanks, spikes, samples, and QC samples. **Directions to make up the standards can be found in Tables D and F.**

11.4.2 Build a sequence in the sequence table and apply the repeat run rules to insert the CCV and CCB for every 10 unknowns.

11.4.3 Ensure the RLVS standards, ICSA, and ICS-AB bracket the sample list.

11.4.4 When the sample list and QC checks have been verified for accuracy, queue the experiment to the Technician Queue. Each experiment will require a unique code (e.g. 0905208A, 0905208B, etc.).

11.4.5 Expected values and limits for ICV, CCV, ICS-A, and ICS-AB can be found in Table L.

11.5 Data Analysis and Calculations:

11.5.1 The Plasma Lab software performs all calculations necessary to convert raw counts per second data into quantitative concentration results.

11.5.2 Consideration should be given to the interference equations that are in the method. As a result of changing tune parameters over time, the equations may require adjustment periodically to ensure they are not over compensating or under compensating for the polyatomic or isobaric interferences. This is especially true of ^{52}Cr and ^{51}V as the CCT-KED tune can vary slightly from day to day operation. This variation (primarily in the add gas for the collision cell) will change the counts per second acquired for Cl and ClO ions. This can make the counts per second in the calibration blank for these isotopes artificially high or low (**See Table J**). In the case of Lead, quantitation is based on the sum of isotopes 206, 207, and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

11.5.3 See the QA Manager for Isotopes and the respective laboratory specific method detection limits and reporting limits.

11.5.4 Aqueous Sample Calculation:

$$\frac{\text{Raw Data result } (\mu g/L) * DF * V_F}{V_I} = \text{Final Result } (\mu g/L)$$

Where:
DF = Dilution Factor
V_F = Final Volume (L)
V_I = Initial Sample Volume (L)

11.5.5 Soil Sample Calculation:

$$\frac{\text{Raw Data result } (\mu g/L) * DF * V_F}{W_S \ X \ \%S} = \text{Final Result (mg/kg dry weight)}$$

Where:
DF = Dilution Factor
V_F = Final Volume (L)
V_I = Initial Sample Volume (L)
W_S = Sample weight (grams)

%S = Percent solids/ 100
Example: For a sample that is 97.6% solid use 0.976

11.5.6 Biota Sample Calculation

$$\frac{\text{Raw Data result } (\mu g/L) * DF * V_F}{W_S} = \text{Final Result (mg/kg)}$$

Where:
DF = Dilution Factor
V_F = Final Volume (L)
V_I = Initial Sample Volume (L)
W_S = Sample weight (grams)

NOTE: Results for biological samples are routinely reported on an "as is" or wet weight basis. Dry weight correction is available on request when sufficient sample has been provided.

11.5.7 Hardness as $CaCO_3$ in mg/L = 2.497 * [Ca $_{\text{in mg/L}}$] + 4.118 * [Mg $_{\text{in mg/L}}$]

11.5.8 Silica (SiO_2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO_2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

12 Quality Control

There are three levels of quality control utilized in this SOP. They consist of Method QC, Instrument QC, and Prep/Batch QC.

12.1 Method QC consists of the instrument detection limits, linear ranges, method detection limits, the lower limit quantitation check, and demonstrations of capability. This QC must be completed prior to analyzing any samples.

12.1.1 Instrument Detection limits (IDLs): IDLs in ug/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined quarterly.

12.1.2 Linear Dynamic Ranges (LDR): The LDR is determined by analyzing a standard series ending at a concentration expected to be near the high end of the instruments range. The result must be within +/-10% of the expected concentration, or a lesser concentration tested until acceptable results are obtained. After the initial determination the high point is verified every 6 months (semi annually) using the same +/- 10% of the true value.

12.1.3 Method Detection Limits (MDLs): Method detection limits were obtained by multiplying 3.143 by the standard deviation of seven spikes. The spikes went through the digestion procedures and were spiked at a concentration suspected to be within a factor of ten greater than the MDL. The most current version of the MDL SOP, S-ALL-Q-004, must be followed when determining MDLs. Note: The MDL determined by the MDL study is the theoretical MDL. The MDL used for reporting purposes may be higher than the theoretical MDL.

12.1.4 Lower Limit Quantitation Check (LLQC): The LLQC is a standard at the reporting limit concentration that has gone through all the preparation procedures. It has control limits of +/-30% of the true value. It is analyzed to establish the lower limit of quantitation, verified annually, and whenever new limits are established.

12.1.5 Initial Demonstration of Capability (IDOC): An initial demonstration of capability (IDC) must be performed per S-ALL-Q-020, *Orientation and Training Procedures*. A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

12.1.6 Instrument QC: Prior to the analysis of samples, and in some cases during the analysis run, the following quality control must be generated and/or within limits: Daily Performance Report for each mode of operation, Internal Standards, Interference Correction Solutions A and B (ICSA and ICSAB), Initial Calibration Verification (ICV), Continuing Calibration Verification (CCV), RLVS standards, and Initial and Continuing Calibration Blanks (ICB and CCB).

12.1.7 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 130 percent of the intensity of that internal standard in the blank of the calibration, one of the following procedures is followed. The sample is diluted appropriately and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. If significant deviation cannot be overcome by dilution of the sample in the same analytical run then recalibration is required.

12.1.8 The ICSA and ICSAB solution are used to verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run, prior to the analysis of any samples and at the end of the analytical sequence. The analyst should be aware that precipitation from solution AB may occur with some elements, specifically silver. The control limits for the elements in the ICSAB solution that are in the linear range must be within 80 to 120% of the expected recovery. If an element of interest does not meet these limits, then the problem must be corrected, the instrument recalibrated, and the run reanalyzed.

12.1.9 The ICV is analyzed to check the accuracy of the curve. It must be evaluated before any samples are analyzed. The ICV should be at or near (or lower than) the midpoint of the calibration curve, derived from a source independent of the calibration standards, and must quantitate within +/-10% of the expected value. If these limits are not met for an element of interest, the problem must be corrected, the instrument recalibrated, and the run reanalyzed. If the ICV fails high and the samples are non-detects, then they may be reported.

12.1.10 Reporting Limit Verification Standard (RLVS): With every Initial Calibration, a standard corresponding to the Pace reporting limit (PRL) must also be analyzed and meet established acceptance criteria. The RLVS is analyzed prior to any samples being analyzed and at the end of the analytical sequence. Additional RLVSs may be analyzed throughout the analytical sequence at the analyst's discretion. The limits for the RLVS are +/- 30% of the true concentration. These standards are the first calibration standard for the analytes of interest. The analysis of this standard demonstrates the instruments ability to quantify down to the reporting limit with known accuracy.

12.1.11 The CCV is analyzed to check for calibration drift. The CCV is run after every 10 samples and again at the end of samples. It must quantitate within 10% of the expected value. Any sample analyzed under out-of-control calibration must be reanalyzed, following the successful re-calibration of the instrument. If the CCV fails high and the samples are non-detects, then they may be reported.

12.1.12 The ICB is analyzed to check the accuracy of the curve. The CCBs are analyzed to check for calibration drift. In the absence of project specific reporting limits, the results of the calibration blanks must be less than the Reporting Limit. The ICB is run after the ICV and the CCBs are run after the CCVs. If the ICB or CCB fails high for an analyte and the samples are non-detects or are greater than 10x the blank value, then they may be reported.

12.2 A batch will consist of 20 or fewer samples. Batch Quality Control will include a Method Blank (MB), Chicken Blank (CB) (Biota only), Laboratory Control Spike (LCS), Matrix Spike (MS), Matrix Spike Duplicate (MSD), Post Spike (PS), and a Serial Dilution (SDL). It may also include a Laboratory Control Spike Duplicate (LCSD) and/or Standard Reference Material (SRM).

12.2.1 The Method Blank is used to verify that interferences caused by contaminants in the solvents, reagents, glassware, etc. are known and minimized. The method blank is processed through all clean-ups, etc., which were performed on the samples in the batch. For a method blank to be acceptable, in the absence of project specific criteria, the concentration shall not be higher than the highest of the following: The reporting limit, or ten percent of the regulatory limit of concern for that analyte, or ten percent of the measured concentration in a particular sample of interest. See Flowcharts for a visual guide on applying flags to the above conditions. Each sample in the batch is assessed against the above criteria to determine if the sample results are acceptable. Any sample associated with an unacceptable blank is either flagged, re-prepped for analysis, or if re-prepping is not an alternative, the results are reported with the appropriate data qualifying codes.

12.2.2 The Chicken Blank (CB) must be prepared with every biota batch, and is processed through all clean-ups, etc., which were performed on the samples. It consists of a control matrix (ground chicken) to verify the interferences in the biological tissue is known and minimized. The CB will contain detectable amounts of elements such as K, Ca, Na, Mg, and P etc., and is used to ensure acceptable performance of the laboratory control spike. Any detections in the CB greater than the 40CFR Part 136 statistically derived MDL is subtracted from the LCS/LCSD before recovery limits are evaluated.

12.2.3 A laboratory control sample (LCS) consists of a control matrix, which has been spiked, with the analytes(s) of interest or compounds representative of those analytes. Laboratory Control Samples are analyzed at a minimum of 1 per batch of 20 or fewer samples or preparation method. Results of the LCS are expressed in terms of percent recovery, and are used to determine batch acceptance. Acceptance limits are generated based on laboratory generated data and are not to exceed 80 to 120% of the expected recovery. If these limits are not met with the instrument in control, then the entire batch will be re-digested and re-analyzed.

An LCS Duplicate may be analyzed to evaluate laboratory precision. The LCSD must also meet the criteria for the LCS. The Relative Percent Difference (RPD)

will be calculated between the LCS and LCSD. The RPD is calculated as outlined below:

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

Where:
RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (duplicate)

The control limit for RPD is based on laboratory generated data and is not to exceed 20%. If outside this limit, all associated results are given a R1 data qualifier. Data generated with LCS samples that fall outside the established acceptance criteria are judged to be out-of-control. These data are considered suspect and the corresponding samples are reanalyzed or reported with qualifiers.

For the biota matrix, any detections greater than the MDL must be subtracted from the LCS/LCSD on-instrument value before calculating the recovery limits.

NOTE: In the event where adequate sample is not supplied by the client to perform a Matrix Spike/ Matrix Spike Duplicate, the LCS and duplicate can lend insight on the precision of the analysis.

12.2.4 Matrix spikes (MS and MSD) are performed to evaluate the effect of the sample matrix upon analytical methodology. A separate aliquot of sample is spiked with the analyte of interest and analyzed with the sample. MS and MSD are performed at a minimum frequency of one pair in 20 samples per matrix type per sample preparation method and are done more frequently where regulations require. Matrix spike recoveries are evaluated against in-house control limits. The recovery must not exceed 75 to 125% of the expected recovery. If outside this recovery, the parent is flagged with an M0 data qualifier. If the recovery of an analyte is outside this range but the spike level in not at least 25% the background concentration in the parent sample, the data is flagged with the P6 data qualifier. The RPD between the MS and MSD must be less than 20 %. If outside this limit, the parent is given a R1 data qualifier. The parent sample for the MS/MSD is chosen at random unless specified by a client. Poor performance in a matrix spike generally indicates a problem with the sample composition, and not the laboratory analysis, and results are used to assist in data assessment. A matrix effect is indicated if the LCS data are within acceptance criteria but the matrix spike data exceed the acceptance criteria. Prior to calculating recovery, the parent sample concentration (results <Reporting MDL = 0) is subtracted from the spike aliquot concentrations.

12.2.5 The Post Spike (PS) is run to verify matrix interferences. An analyte spike added to a portion of a prepared sample, or its dilution if above the LDR, must be recovered to within 80 to 120 percent of the known value. If the spike is not recovered within the specified limits with the instrument in control, then the sample has a confirmed matrix effect and the serial dilution test should be performed, and appropriate flags given to the matrix spike and duplicate data (e.g. M0, P6, and or R1). The post spike will be run per project specific requirements or per client request.

12.2.6 The Serial Dilution (SDL) is run to check for matrix interferences. If the analyte concentration is within the linear dynamic range (LDR) of the instrument and sufficiently high (minimally, a factor of at least 10 times greater than the lower limit of quantitation for the diluted sample, or 50 times the RLVS standard for the parent sample) an analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. If these limits are not met then an interference effect must be suspected and the data qualified with an SD flag. If the analytes of interest are greater than the LDR in the parent sample, the sample can be diluted and an SDL done off of the dilution. The serial dilution will be run per project specific requirements or per client request.

12.2.7 The type of SRM is typically prepped and analyzed upon client request. It is a sample of known concentration chosen to resemble the matrix being analyzed. All ICPMS biota batches include the default SRM of lobster hepatopancreas material.

13 Method Performance

13.1 There are several requirements that must be met to insure that this procedure generates accurate and reliable data. A general outline of requirements has been summarized below. Further specifications may be found in the Laboratory Quality Manual.

13.2 The analyst must read and understand this procedure with written documentation maintained in his/her training file.

13.3 An initial demonstration of capability (IDC) must be performed per the most recent version of S-ALL-Q-020, *Orientation and Training Procedures* (most current revision or replacement). A record of the IDC will be maintained in his/her QA file with written authorization from the Laboratory Manager and Quality Manager.

13.4 An annual method detection limit (MDL) study will be completed per the most recent version of S-GB-Q-020, *Determination of the LOD and LOQ* (most current revision or replacement), for this method and whenever there is a major change in personnel or equipment. The results of these studies are retained in the quality assurance office.

13.5 Periodic performance evaluation (PE) samples are analyzed per the most recent version of S-ALL-Q-010, *PE/PT Program* (most current revision or replacement), to demonstrate continuing competence. All results are stored in the QA office.

14 Pollution Prevention and Waste Management

14.1 The quantity of chemicals purchased is based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes reflect anticipated usage and reagent stability.

14.2 Excess reagents samples and method process wastes are characterized and disposed of in an acceptable manner. For further information on waste management consult the current version of S-GB-S-006, *Waste Handling and Management* (most current revision or replacement).

15 References

15.1 USEPA, SW-846, Method 6020A "Inductively Coupled Plasma - Mass Spectrometry", February 2007.

15.2 USEPA, SW-846, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry", December 1996.

15.3 USEPA, SW-846, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", December 1996.

Tables, Diagrams, Flowcharts, Appendices, Addenda etc.

Attachment I -	SW846 3010A DIGESTION
Attachment II -	SW846 3050B DIGESTION
Attachment III -	SW846 M3050B DIGESTION
Flow Chart A -	ICPMS DETERMINATIVE METHOD
Flow Chart B -	MDL or LOD FLAGGING and REPORTING
Flow Chart C -	EQL or PSRL FLAGGING and REPORTING
Table A -	APPLICABLE ANALYTES
Table B -	EQUIPMENT
Table C -	SUPPLIES
Table D -	WORKING STANDARDS
Table E -	STOCK STANDARDS
Table F -	INTERMEDIATE STANDARDS
Table G -	REAGENTS
Table H -	CALIBRATION LEVELS
Table I -	INTERNAL STANDARDS
Table J -	INTERFERENCE EQUATIONS
Table K -	INTEGRATION TIMES
Table L -	INSTRUMENT QC LIMITS
Table M -	AQUEOUS BATCH QC ON INSTRUMENT CONCENTRATIONS AND LIMITS
Table N -	BIOTA BATCH QC ON INSTRUMENT CONCENTRATIONS AND LIMITS
Table O -	BIOTA BATCH QC ON INSTRUMENT CONCENTRATIONS AND LIMITS

16 Revisions

Document Number	Reason for Change	Date
S-GB-M-006-REV.00	First Issue	22Sept2008
S-GB-M-006-REV.01	Fixes to typographical errors Changes to digestion method for Aqueous samples (3010 modification) Changes to Method for Au content (location of) changed to ISTD solution and rinse solution Analyte list additions (Si, Li, and U)	09March2009
S-GB-M-006-REV.02	Updated naming format and signature page Added operating parameters throughout document for Thermo X Series II ICPMS. Fixed typographical errors Changes to digestion method for Aqueous samples(3010modification) Changes to Method for Au content(location of) changed to ISTD solution and rinse solution Analyte list additions (Si and U) Updated Tables and added P and Zr	12Apr2010
S-GB-M-006-Rev.03	Section 10.1: Added the curve is a linear regression using Absolute Standard Deviation weighting. Also included acceptance criteria of correlation coefficient must be ≥0.998. Section 12.2: Added Chicken Blank information and criteria.	13Sept2010
S-GB-M-006-Rev.04	Sections 9.4.1, 9.4.2: Removed language referencing standard login procedures and replaced with EpicPro Standard Log SOP: T-ALL-IT-010 Table J: Added interference equation for Iron.-	10Nov2011

Attachment I:
SW846 3010A DIGESTION

- **This prep is to be used for aqueous (Total and Dissolved) samples and sludges for the determination of Li, Be, B, Na, Mg, Al, Si, P, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Zr, Mo, Ag, Cd, Sn, Sb, Ba, Hg, Tl, Pb, and U.**
- **See Table M for reporting limits, spike concentrations, and spike recovery limits.**

1.0 Water Samples: All water samples must be digested as described in Method 3010A.

2.0 Turn on the Hot Block and allow it to warm up. Verify that the hood is turned on. Before placing samples in the block to digest, verify that the sample well temperature is in the range of 95 ^{0}C +/- 5^{0}C, adjust as required. Record the sample well temperature in the prep logbook along with the thermometer ID.

3.0 Prepare the MB and LCS (an LCSD may also be required) by adding 50.0mL of Nanopure water into digestion vials. One MB and one LCS must be prepared for each batch of 20 samples or less.

4.0 Prepare the parent sample, duplicate and MS, or the MS and MSD by placing 50.0mL aliquots of a selected sample from each batch into digestion vials. One MS and one duplicate or MSD must be prepared for each analytical batch of 20 samples or less. The parent sample is chosen at random or per project specific requests– excluding rinse blanks, equipment blanks, and trip blanks.

5.0 Place 50.0mL of each sample in a digestion vial.

6.0 Spike the LCS (LCSD), MS, and MSD with 1.0 mL of ICP Spike solution.

7.0 Carefully add 5.0mL of concentrated HNO_3 to each sample, MB, LCS (LCSD), MS, and MSD vial, followed by 2.5mL of Concentrated HCl. Heat in the hot block for 4 hours. Do not let sample go to dryness, low sample recovery will result. If this occurs, discard sample and re-prep.

8.0 Remove the digestates from the hot block and allow to cool in the hood.

9.0 Dilute each vial to a final volume of 50.0mL with Nanopure water.

10.0 Place a screw cap on each vial and shake to mix thoroughly. Allow to settle before analysis.

11.0 If any digestate is turbid or there is a concern that insoluble materials may clog the nebulizer, centrifuge to settle or use a filter to remove the solids.

Attachment II:
SW846 3050B DIGESTION

- **This prep is to be used for solid samples and sludges for the determination of Li, Be, B, Na, Mg, Al, Si, P, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Mo, Zr, Ag, Cd, Sn, Sb, Ba, Hg, Tl, Pb and U.**
- **See Table N for reporting limits, spike concentrations, and spike recovery limits.**

1.0 Heat hot block at 95°C +/- 5°C (verify temperature with a calibrated thermometer). Typically, setting the temperature at 102 degrees will achieve this range. Record temperature in prep logbook.

2.0 Verify sample Station IDs and LIMs numbers on containers against backlog.

3.0 Lay out run in digestion logbook. Identify QC samples at frequency stated in the Quality Control Section of this SOP.

4.0 To the extent possible, mix and crush the sample as per SOP S-ALL-Q-021, *Sub-sampling (Sample Homogenization)*, in the sample container with a spatula to achieve homogeneity.

5.0 Prepare Method Blank by weighing 0.500g of Teflon chips in digestion tube.

6.0 Prepare Laboratory Control Spike (LCS) by adding 1.0 mL of the ICP Spike to the LCS tube with Teflon chips.

7.0 Spike Duplicate (LCSD) if there is not enough volume for an MS/MSD or at client request. See Table E for spiking solutions.

8.0 Prepare Matrix Spike / Matrix Spike Duplicate (MS/MSD), by weighing 0.500 to 1.000 grams of parent sample into 3 separate digestion tubes. Record sample weight to nearest 0.001 gram on digestion log. Parent, matrix spike, and matrix spike duplicate should be approximately the same weight. Add 1.0 mL of the ICP Spike to the MS/MSD digestion tubes. See Table E for spiking solutions.

9.0 Weigh 0.500 to 1.000 gram of homogenized sample into a labeled digestion tube. Record sample weight to nearest 0.001 gram on digestion log. Do this for each sample in the digestion group. For samples with high liquid content, a larger sample size may be used, as long as the digestion is complete. For sludges with % solids >10% and <50%, weigh out 2.5-5 grams. Record all information in the Metals Digestion Log (see attached example).

10.0 Preparing Paint Chips for Digestion

11.0 Transfer paint chips into labeled drying tins.

12.0 Dry samples at 103-105 degrees Celsius for 12-24 hours.

13.0 Remove samples from oven and desiccate for 1 hour.

14.0 Weigh out 0.500 to 1.000 gram of sample into a digestion tube. If the sample available is less than 0.5 gram, use the entire sample.

15.0 Always prepare an LCSD for paint Chips.

16.0 Results are always reported as mg/kg dry wt for paint chips.

17.0 CONTINUE DIGESTION IN HOOD.

18.0 Add small amount of Nanopure water (2mL) to digestion tube (to cover bottom). Swirl to keep sample from sticking to the tube.

19.0 Add 10 mL 1:1 HNO_3 using a calibrated repipettor. Cover with watch glass and swirl gently to mix.

20.0 To make 1:1 HNO_3, add 1 part HNO_3 to 1 part Nanopure water.

21.0 Heat the samples at 95°C +/- 5°C for 2 hours. Maintain a covering of solution over the bottom of the tube at all times. Remove from hot block and cool in hood.

22.0 CAUTION: DO NOT BOIL AT ANY TIME DURING DIGESTION.

23.0 This may result in metals volatilizing which would require starting the digestion over.

24.0 Add 2.5 mL 30% H_2O_2 using a calibrated repipettor.

25.0 CAUTION: DO NOT ALLOW LOSS OF SAMPLE DUE TO EXCESSIVE EFFERVESCENCE.

26.0 If sample loss occurs the sample must be re-digested.

27.0 Cover with watch glass, swirl gently to mix, and heat in hot block to start peroxide reaction. Heat the samples at 95°C +/- 5°C until effervescence subsides (no more bubbling). Maintain a covering of solution over the bottom of the tube at all times. Remove from hot block and cool in hood.

28.0 Add 2.5mL of Concentrated HCl and heat until the fuming subsides.

29.0 Rinse watch glass with Nanopure water into digestion tube.

30.0 Allow samples to cool to room temperature.

31.0 Bring samples to final volume of 50 mL with Nanopure water.

32.0 Centrifuge sample for 10 minutes at a setting between 20 and 25 or

33.0 Filter through Whatman #541(or equivalent) filter paper into a labeled digestion tube.

34.0 Rinse digestion tube with Nanopure water into filter paper.

35.0 Bring to final volume of 50 mL with Nanopure water, cap, and mix well.
or

36.0 Samples may also be allowed to settle overnight if not filtered or centrifuged.

37.0 Samples are now ready for analysis.

Attachment III:

SW846 M3050B DIGESTION

- This prep is to be used for biota samples for the determination of Li, Be, B, Na, Mg, Al, Si, P, K, Ca, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Sr, Zr, Ag, Mo, Cd, Sn, Sb, Ba, Hg, Tl, Pb, and U.
- See Table O for reporting limits, spike concentrations, and spike recovery limits.

1.0 Heat hot block at 95°C +/- 5°C (verify temperature with a calibrated thermometer). Record the temperature in prep logbook along with the thermometer ID.

2.0 Verify sample Station IDs and LIMs numbers on containers against backlog.

3.0 Lay out run in digestion logbook. Identify QC samples at frequency stated in the Quality Control Section of this SOP.

4.0 To the extent possible, mix and crush the sample as per SOP S-ALL-Q-021, *Sub-sampling (Sample Homogenization)*, in the sample container with a spatula to achieve homogeneity.

5.0 Prepare Method Blank by leaving one digestion tube empty.

6.0 Prepare Chicken Blank by weighing 0.500 to 1.000 gram of chicken into one digestion tube for the control blank. Record sample weight in digestion log.

7.0 Prepare Laboratory Control Spike (LCS) by weighing 0.500 to 1.000 gram of Chicken into one digestion tube and adding 1.0 mL of the ICPMS Spike. Record sample weight in digestion log.

8.0 Spike Duplicate (LCSD) if there is not enough volume for an MS/MSD or at client request. Prepared the same as the LCS.

9.0 Prepare Standard Reference Material (SRM): If a SRM is requested, weigh out 0.500 to 1.000gram of appropriate reference material. Record in digestion log.

10.0 Prepare Matrix Spike / Matrix Spike Duplicate (MS/MSD), by weighing 0.500 to 1.000 gram of parent sample into 3 separate digestion tubes. Record sample weight to nearest 0.001 gram in digestion log. Parent, matrix spike, and matrix spike duplicate should be approximately the same weight. Add 1.0 mL of the ICPMS Biota Spike to the MS/MSD digestion tubes.

11.0 Weigh 0.500 to 1.000 gram of homogenized sample into a labeled digestion tube. Record sample weight to nearest 0.001 gram on digestion log. Do this for each sample in the digestion group. Record all information in the Metals Digestion Log (see attached example).

12.0 CONTINUE DIGESTION IN HOOD.

13.0 Add small amount of Nanopure water (2mL) to digestion tube (to cover bottom). Swirl to keep sample from sticking to the tube.

14.0 Add 10.0 mL of 1:1 HNO_3 using a calibrated repipettor. Cover with watch glass and swirl gently to mix.

15.0 To make 1:1 HNO_3, add 1 part HNO_3 to 1 part Nanopure water.

16.0 Heat the samples at 95°C +/- 5°C for 2 hours.
CAUTION: DO NOT BOIL AT ANY TIME DURING DIGESTION.
This may result in metals volatilizing which would require starting the digestion over.

17.0 Remove from hot block and cool.

18.0 Add 2.5 mL of 30% H_2O_2 using a calibrated repipettor.

19.0 Swirl gently to mix, and heat in hot block to start peroxide reaction. Heat until effervescence subsides (about 15 min).

CAUTION: DO NOT ALLOW LOSS OF SAMPLE DUE TO EXCESSIVE EFFERVESCENCE.

20.0 Remove from hot block and cool.

21.0 Add 2.5 mL of 30% H_2O_2 using a calibrated repipettor.

22.0 Swirl gently to mix, and heat in hot block to start peroxide reaction. Heat until effervescence subsides.

23.0 **CAUTION: DO NOT ALLOW LOSS OF SAMPLE DUE TO EXCESSIVE EFFERVESCENCE.**

24.0 Remove from hot block and cool.

25.0 Add 2.5 mL Conc. HCl using a calibrated repipettor.

26.0 Bring samples to final volume of 50 mL with Nanopure water or go to step 27.0.

27.0 Centrifuge the samples for 10 minutes at a setting between 20 and 25. OR

28.0 Filter through Whatman #541(or equivalent) filter paper into a labeled digestion tube.

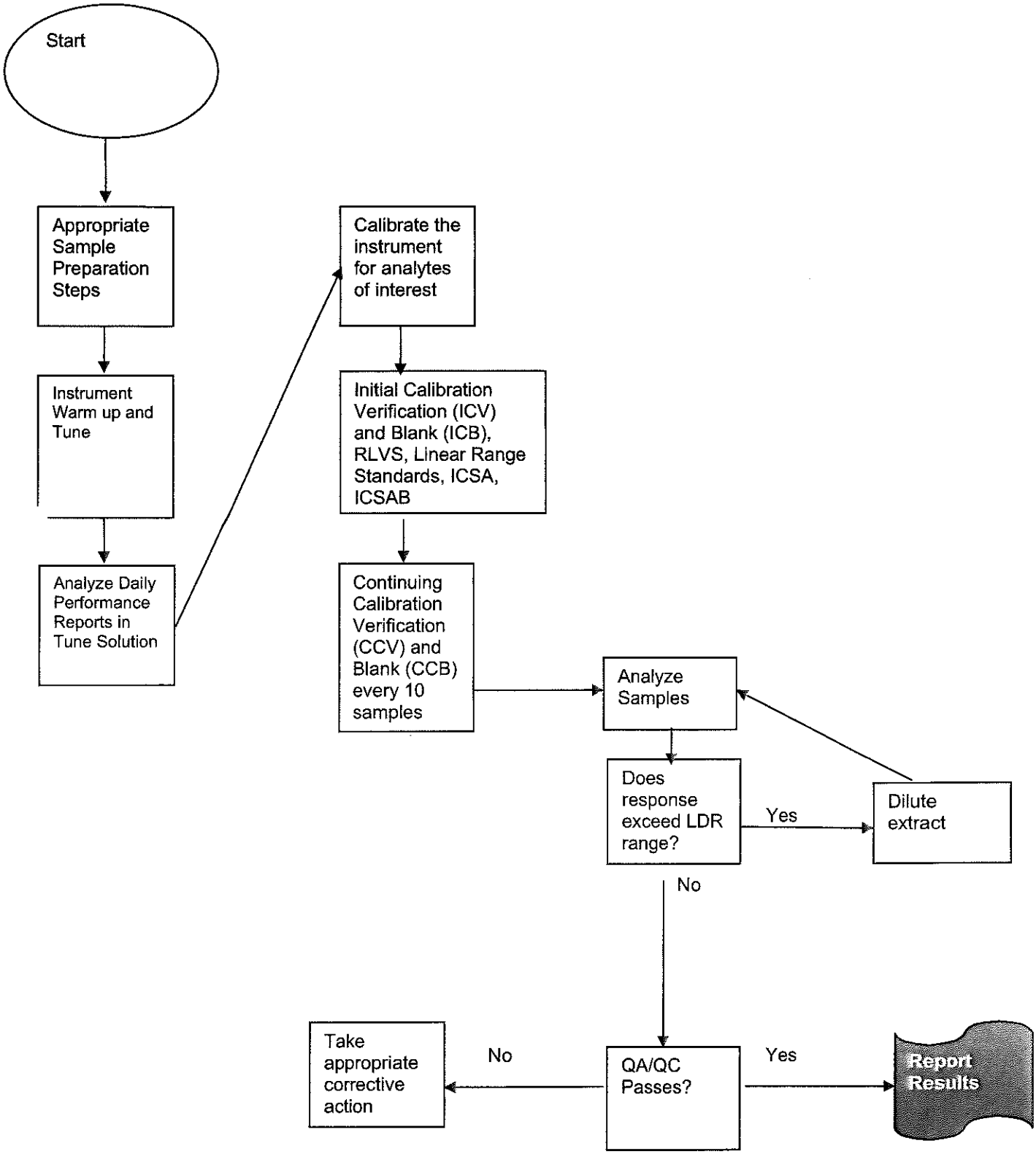
29.0 Rinse digestion tube with Nanopure water into filter paper. Bring to final volume of 50 mL with Nanopure water, cap, and mix well. OR

30.0 Samples may also be allowed to settle overnight if not filtered or centrifuged.

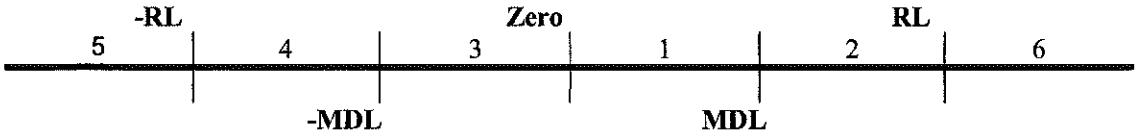
31.0 Samples are now ready for analysis.

Flowchart A

ICPMS DETERMINATIVE METHOD



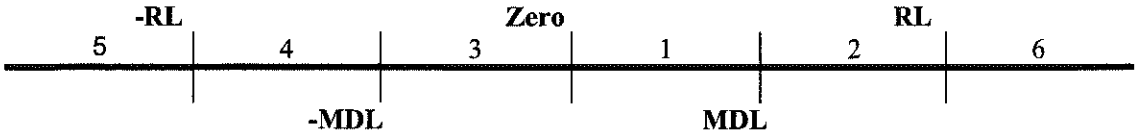
Flowchart B
MDL or LOD FLAGGING and REPORTING



Blank Position> **Sample Position⇓**	5	4	3	1	2	6
1	R	B Flag Report < MDL	Report < MDL	Report < MDL	Report < MDL	Report < MDL
2	R	B Flag Report result w/10X Rule	Report result	Report result	B Flag Report result w/10X Rule	R
3	R	B Flag Report < MDL	Report < MDL	Report <MDL	Report < MDL	Report <MDL
4	R	B Flag Report <MDL	Report <MDL	Report <MDL	B Flag Report result w/10X Rule	Report <MDL
5	R	R	R	R	R	R
6	R	B Flag Report result w/10X Rule	Report result	Report result	B Flag Report result w/10X Rule	Report result w/10X Rule -or- R if result is not 10XMB

R= Analyst discretion to Re-run, Re-calculate, or Re-digest
X= Value in Method Blank (MB).

Flowchart C
EQL or PRSL FLAGGING and REPORTING



Blank Position> **Sample Position⇓**	5	4	3	1	2	6
1	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
2	R	Report < RL	Report < RL	Report result	Report < RL	Report < RL
3	R	Report < RL	Report < RL	Report <RL	Report < RL	Report < RL
4	R	Report < RL	Report < RL	Report < RL	Report < RL	Report < RL
5	R	R	R	R	R	R
6	R	Report result	Report result	Report result	Report result	Report result w/10X Rule -or- R if result is not 10XMB

R= Analyst discretion to Re-run, Re-calculate, or Re-digest
RL= Reporting Limit

Table A: APPLICABLE ANALYTES

Isotope	Element	Symbol	Chemical Abstracts Service Registry Number (CASRN)
27	Aluminum	Al	7429-90-5
121	Antimony	Sb	7440-36-0
75	Arsenic	As	7440-38-2
137	Barium	Ba	7440-39-3
9	Beryllium	Be	7440-41-7
11	Boron	B	7440-42-8
111	Cadmium	Cd	7440-43-9
43	Calcium	Ca	7440-70-29
52	Chromium	Cr	7440-47-3
59	Cobalt	Co	7440-48-4
65	Copper	Cu	7440-50-8
57	Iron	Fe	7439-89-6
7	Lithium	Li	7439-93-2
208	Lead	Pb	7439-92-1
24	Magnesium	Mg	7439-95-4
55	Manganese	Mn	7439-96-5
201	Mercury	Hg	7439-97-6
95	Molybdenum	Mo	7439-98-7
60	Nickel	Ni	7440-02-0
31	Phosphorous	P	7723-14-0
39	Potassium	K	7440-09-7
82	Selenium	Se	7782-49-2
28	Silicon	Si	7440-21-3
107	Silver	Ag	7440-22-4
23	Sodium	Na	7440-23-5
88	Strontium	Sr	7440-24-6
205	Thallium	Tl	7440-28-0
118	Tin	Sn	7440-31-5
47	Titanium	Ti	7440-32-6
238	Uranium	U	7440-61-1
51	Vanadium	V	7440-62-2
66	Zinc	Zn	7440-66-6
90	Zirconium	Zr	7440-67-7

Table B:
EQUIPMENT

Equipment	Manufacturer	Model(#)	Serial #
Thermo Xseries 2 ICPMS	ThermoFisher Scientific	XSeries 2	01301
Rough Pump	Edwards	E2 M28	086 105337
Computer	Dell	Optiplex 755	DJG5L3J
Autosampler	CETAC	ASX520	030867A520
Refrigerated Recirculator	NESLAB	Merlin M75	108009024
Uninterruptible Power Supply (UPS)	Toshiba	1600 EP Series UPS	080400330
Hot Block	Environmental Express	SC100	526CEC0714
Analytical Balance	Denver Instrument	XE-310	718860

Note: Equivalent substitutes may be used.

Table C:
SUPPLIES

Supplies	Manufacturer	Vendor	Catalog #*
Sample Per-Pump Tubing	Analytical West	Analytical West	PT-2100P
Internal Standard Per-Pump Tubing	Analytical West	Analytical West	PT-2100P
Waste Per-Pump Tubing	Analytical West	Analytical West	PT-2160P
Quartz T Connector	Glass Expansion	Glass Expansion	60-808-1185
Concentric Nebulizer	Thermo Electron Corp.	Thermo Electron Corp.	4600294-03
Quartz Torch	Thermo Electron Corp.	Thermo Electron Corp.	3601145
Screen and Bonnet	Thermo Electron Corp.	Thermo Electron Corp.	3601219
Ni Sample Cone	Thermo Electron Corp.	Thermo Electron Corp.	3600812
Ni Skimmer Cone	Thermo Electron Corp.	Thermo Electron Corp.	3600811
Graphite Sample Cone Seal	Thermo Electron Corp.	Thermo Electron Corp.	3004382
15 mL Polypropylene Test Tubes	Fisher Scientific	Fisher Scientific	14-956-7E
50-mL Disposable Digestion Cups	Environmental Express	Environmental Express	SC475
Calibrated Pipette 1000 uL	Eppendorf	Fisher Scientific	21-371-13
Calibrated Pipette 10-50 uL	Thermo	Thermo	21-377-193
Calibrated Pipette 100-1000 uL	Eppendorf	Fisher Scientific	05-402-50
Calibrated Pipette 1000-5000 uL	Eppendorf	Fisher Scientific	05-402-91
Trace Metal Grade Pipette Tips 1 mL	Eppendorf	Fisher Scientific	21-372
Trace Metal Grade Pipette Tips 5 mL	Eppendorf	Fisher Scientific	21-381-198
N-DEX Nitrile Gloves	Best	Fisher Scientific	6005 PFM
pH Indicator Sticks	Whatman	MG Scientific	P114-26

Note: Equivalent substitutes may be used.

Table D:
WORKING STANDARDS

Standard	Stock or Intermediate Standard	Concentration	Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
Cross Calibration (XCal)	ICP-MS-68A Solution A	10 ppm	Al, As, Ba, Be, Bi, B, Cd, Ca, Ce, Cs, Cr, Co, Cu, Dy, Er, Eu, Gd, Ga, Ho, In, Fe, La, Pb, Li, Lu, Mg, Mn, Nd, Ni, P, K, Pr, Re, Rb, Sm, Sc, Se, Na, Sr, Tb, Tl, Th, Tm, U, V, Yb, Y, Zn	0.5 mL	100 mL	50 ppb	All Elements
	ICP-MS-68A Solution B		Sb, Ge, Hf, Mo, Nb, Si, Ag, Ta, Te, Sn, Ti, W, Zr	0.5 mL			
Tune Solution	Tune Int	1 ppm	Mg, Bi, Ge, In, Sc, Tb, Y, Ba, B, Be, Ce, Co, Li, Ni, Pb, U	5 mL	500 mL	10 ppb	All Elements
ICB	Same as Cal. Level 1		------	------	------		------
CCB	Same as Cal. Level 1		------	------	------		------
Calibration Level 1 and Diluent/Blank Solution	Cal 1	---	----	------	50 mL	------	------
	Diluent / Blank	----	----	----	1000 mL	----	-----
Calibration Level 2 and RLVS as applicable (See Table H)		----	----	----	50 mL	-----	-----
	Hg Int	1 ppm	Hg	0.010 mL		0.2 ppb	Hg
	T Int	1.25 ppm / 1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	0.04		1 ppb	All Elements (not Ag, Si)
		1.25 ppm / 61.25 ppm	P (from two sources)				
		0.625 ppm	Ag			0.5 ppb	Ag
		6.25 ppm / 56.25 ppm	Si (from two sources)			5 ppb	Si (not applied)
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.025		250 ppb	Al, Ca, Fe, K, Mg, Na
Calibration Level 3 and RLVS as applicable (See Table H)					50 mL		
	Hg Int	1 ppm	Hg	0.025 mL		0.5 ppb	Hg
	T Int	1.25 ppm / 1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	0.2 mL		5 ppb	All Elements (not Ag, Si)
		1.25 ppm / 61.25 ppm	P (from two sources)				
		0.625ppm	Ag			2.5 ppb	Ag
		6.25 ppm / 56.25 ppm	Si (from two sources)			25 ppb	Si
	M Int	500 ppm	Al, Ca, Fe, K, Mg, Na	0.025 mL		500 ppb	Al, Ca, Fe, K, Mg, Na
Calibration Level 4					50 mL		
	Hg Int	1 ppm		0.05 mL		1 ppb	Hg

	T Int	1.25 ppm	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	2 mL		50 ppb	All Elements (not Ag, Si)
			61.25 ppm	P (from two sources)				
		0.625 ppm		Ag			25 ppb	Ag
		6.25 ppm	56.25 ppm	Si (from two sources)			250 ppb	Si
	M Int	500 ppm		Al, Ca, Fe, K, Mg, Na	0.25 mL		2,500 ppb	Al, Ca, Fe, K, Mg, Na

Table D: Continued
WORKING STANDARDS

Standard	Stock or Intermediate Standard	Concentration		Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
Calibration Level 5						50 mL		
	Hg Int	1 ppm		Hg	0.5 mL		10 ppb	Hg
	T Int	1.25 ppm	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	10 mL		250 ppb	All Elements (not Ag, Si)
			61.25 ppm	P (from two sources)				
		0.625 ppm		Ag			125 ppb	Ag
		6.25 ppm	56.25 ppm	Si (from two sources)			1250 ppb	Si
	M Int	500 ppm		Al, Ca, Fe, K, Mg, Na	1.25 mL		12,500 ppb	Al, Ca, Fe, K, Mg, Na
Calibration Level 6						50 mL		
	Hg Int	1 ppm		Hg	1.25 mL		25 ppb	Hg
	T Int	1.25 ppm	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	20 mL		500 ppb	All Elements (not Ag, Si)
			61.25 ppm	P (from two sources)				
		0.625 ppm		Ag			250 ppb	Ag
		6.25 ppm	56.25 ppm	Si (from two sources)			2500 ppb	Si
	M Int	500 ppm		Al, Ca, Fe, K, Mg, Na	2.5 mL		25,000 ppb	Al, Ca, Fe, K, Mg, Na
CCV						50 mL		
	Hg Int	1 ppm		Hg	0.25 mL		5 ppb	Hg
	T Int	1.25 ppm	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	4 mL		100 ppb	All Elements (not Ag, Si)
			61.25 ppm	P (from two sources)				
		0.625 ppm		Ag			50 ppb	Ag
		6.25 ppm	56.25 ppm	Si (from two sources)			500 ppb	Si
	M Int	500 ppm		Al, Ca, Fe, K, Mg, Na	0.5 mL		5,000 ppb	Al, Ca, Fe, K, Mg, Na
ICV						100 mL		
		1000 ppm		U	0.011 mL		110 ppb	U
	PA-STD-1B	200 ppm		As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	0.055 mL		110 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn
	PA-STD-2B	1000 ppm		Si	0.055 mL		550 ppb	Si
		200 ppm		B, Mo, Sb, Sn, Ti, Zr			110 ppb	B, Mo, Sb, Sn, Ti, Zr
		100 ppm		Ag			55 ppb	Ag
	PA-STD-3B	2000 ppm		Al, Ca, Fe, K, Mg, Na	0.275 mL		5500 ppb	Al, Ca, Fe, K, Mg, Na
	Hg ICV Int	10 ppm		Hg	0.04 mL		4 ppb	Hg
ICSA	4400-061025MB01	500ug/mL		Al, Ca, Fe, K, Mg, Na, P, S,	1.0 mL	10 mL	50,000 ppb	Al, Ca, Fe, K, Mg, Na, S, P
		1,000ug/mL		C			100,000 ppb	C

	Pace	3,600ug/mL		Cl			360,000 ppb	Cl
		10ug/mL		Mo, Ti			1,000 ppb	Mo, Ti
ICSAB	4400-061025MB01 Pace	500ug/mL		Al, Ca, Fe, K, Mg, Na, P, S,	1.0 mL	10 mL	50,000 ppb	Al, Ca, Fe, K, Mg, Na, S, P
		1,000ug/mL		C			100,000 ppb	C
		3,600ug/mL		Cl			360,000 ppb	Cl
		10ug/mL		Mo, Ti			1,000 ppb	Mo, Ti (see Next Section)
	T Int	1.25 ppm	1.25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,	0.8 mL		100 ppb	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn , B, Mo, Sb, Sn, Ti, Zr,
			61.25 ppm	P (from two sources)				
		0.625 ppm		Ag			50 ppb	Ag
		6.25 ppm	56.25 ppm	Si (from two sources)			500 ppb	Si
	Hg Int	1 ppm		Hg	0.05 mL		5 ppb	Hg

Table D: Continued
WORKING STANDARDS

Standard	Stock or Intermediate Standard Used	Concentration	Elements	Amount Used	Final Volume (W/ Diluent)	Final Concentration	Elements
ICPMS Biota SPK 1	PA-STD-1B	200 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	5 mL	100 mL	10 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn
	PA-STD-2B	1000 ppm	Si	5 mL		50 ppm	Si
		200 ppm	B, Mo, Sb, Sn, Ti, Zr			10 ppm	B, Mo, Sb, Sn, Ti, Zr
		100 ppm	Ag			5 ppm	Ag
	PA-STD-3B	2000 ppm	Al, Ca, Fe, K, Mg, Na	25 mL		500 ppm	Al, Ca, Fe, K, Mg, Na
		10,000 ppm	K	15 mL		1500 ppm	K (2000 ppm Total)
		1000 ppm	U	1 mL		10 ppm	U
		1000 ppm	Hg	0.025		0.25 ppm	Hg
	Conc. HNO_3	69-70%		6.0 mL		6%	HNO_3
ICP Water / Soil Spike 2	PA-STD-1B	200 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sr, Tl, V, Zn, P, Li	25 mL	200 mL	25 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se, Sr, Tl, V, Zn, P, Li
	PA-STD-2B	1000 ppm	Si	25 mL		125 ppm	Si
		200 ppm	B, Mo, Sb, Sn, Ti, Zr			25 ppm	B, Mo, Sb, Sn, Ti, Zr
		100 ppm	Ag			12.5 ppm	Ag
	PA-STD-3B	2000 ppm	Al, Ca, Fe, K, Mg, Na	25 mL		250 ppm	Al, Ca, Fe, K, Mg, Na
Internal Standards (ISTD)	PACE-WI-CAL-4A	100 ppm	Bi, Ge, In, Sc, Tb, Y	1 mL	1000 mL	100 ppb	Bi, Ge, In, Sc, Tb, Y
	Conc. HNO_3	69-70%	NA	3.0 mL		3%	HNO_3
	Gold	1000ppm	Au	2.0mL		2000 ppb	Au
Probe Rinse 3	Gold	1000ppm	Au	1.0	2500 mL	400 ppb	Au

Note: Equivalent substitutes may be used.

1 = 1 mL added to LCS/LCSD and MS/MSD, 0.2 mL to PS of 10 mL

2 = 1 mL added to LCS/LCSD and MS/MSD, 0.2 mL to PS of 10 mL

3 = All Matrices contain 10% HNO_3 and 5% HCl

Table E:
STOCK STANDARDS

Standard Type	Manufacturer	Part #	Conc	Analytes	Used In
Calibration Stocks	SPEX CertiPrep	XFSMN-26-250A	100mg/L	As, Ba, Be, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn, Cu	Calibration, CCVs, ICSAB
	SPEX CertiPrep	XFSMN-27-250A	500mg/L	Si	
			100mg/L	B, Mo, Sb, Sn, Ti, Zr	
			50mg/L	Ag	
	SPEX CertiPrep	XFSMN-28-250A	1,000mg/L	Al, Ca, Fe, K, Mg, Na	
	High-Purity Stds	100033-1	1,000ug/mL	Hg	
ICV/Spike Stocks	Inorganic Ventures	PA-STD-1B	200mg/L	As, Ba, Be, Cd, Co, Cr, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn, Cu	ICV/Spike
	Inorganic Ventures	PA-STD-2B	1,000mg/L	Si	
			200mg/L	B, Mo, Sb, Sn, Ti, Zr	
			100mg/L	Ag	
	Inorganic Ventures	PA-STD-3B	2,000mg/L	Al, Ca, Fe, K, Mg, Na	
	Inorganic Ventures	CGHG1-1	1,000ug/mL	Hg	
	High Purity Standards	100064-1	1,000ug/mL	U	
Internal Standard Stocks	Inorganic Ventures	PACEWI-CAL-4A	10mg/L	Bi, Ge, In, Sc, Tb, Y	ISTD, Tune
Single Element Stocks	Ultra Scientific	ICP-112	10,000 mg/L	Mg	Tune / Int
	Ultra Scientific	ICP-056	1,000 mg/L	Ba	Tune
	Ultra Scientific	ICP-005	1,000 mg/L	B	Tune
	Ultra Scientific	ICP-004	1,000 mg/L	Be	Tune
	Ultra Scientific	ICP-058	1,000 mg/L	Ce	Tune
	Ultra Scientific	ICP-027	1,000 mg/L	Co	Tune
	Inorganic Ventures	CGLI10-5	10,000 mg/L	Li	Tune
	Ultra Scientific	ICP-028	1,000 mg/L	Ni	Tune
	Ultra Scientific	ICP-082	1,000 mg/L	Pb	Tune
	Inorganic Ventures	AAU1-1	1,000 mg/L	U	Tune / Cal
	High Purity Standards	100033-1	1,000 mg/L	Hg	Cal
	Ultra Scientific	ICP-119	10,000 mg/L	K	Spike
ICSA	CPI	4400-061025MB01 Pace	500ug/mL	Al, Ca, Fe, K, Mg, Na, P, S,	ICSA & ICSAB
			1,000ug/mL	C	
			3,600ug/mL	Cl	
			10ug/mL	Mo, Ti	

Note: Equivalent substitutes may be used.

Note: Single element standards may be used.

Table F:
INTERMEDIATE STANDARDS

Intermediate Type	Name	Stock Standard(s) Used (Part #)	Concentration	Elements	Amount Used	Acid Used	Final Volume (W/Nanopure H_2O)	Final Concentration
Tune Solution Intermediates	Mg Tune Int		10,000 ppm	Mg	0.1 mL	3 mL HNO_3	100 mL	10 ppm
	Tune Int		10 ppm	Mg	10 mL	2 mL HNO_3	100 mL	1 ppm
			100 ppm	Bi, Ge, In, Sc, Tb, Y	1.0 mL			
			1000 ppm	Ba	0.1 mL			
			1000 ppm	B	0.1 mL			
			1000 ppm	Be	0.1 mL			
			1000 ppm	Ce	0.1 mL			
			1000 ppm	Co	0.1 mL			
			10000 ppm	Li	0.01 mL			
			1000 ppm	Ni	0.1 mL			
			1000 ppm	Pb	0.1 mL			
			1000 ppm	U	0.1 mL			
Calibration Intermediates	Hg Int		1000 ppm	Au	0.2 mL	3 mL HNO_3	100 mL	2 ppm
			1000 ppm	Hg	0.1 mL			1 ppm
	T Int		1000 ppm	U	0.125 mL	3 mL HNO_3 2 mL HCl	100 mL	1.25 ppm
			100 ppm	As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Ni, P, Pb, Se, Sr, Tl, V, Zn	1.25 mL			1.25 ppm
			100 ppm Ag 50 ppm Si 500 ppm	Si, B, Mo, Sb, Sn, Ti, Zr, Ag	1.25 mL			1.25 ppm 2.5 ppm 6.25 ppm
	M Int		1000 ppm	Al, Ca, Fe, K, Mg, Na	50 mL	3 mL HNO_3	100 mL	500 ppm
ICV Intermediate	Hg ICV Int	CGHG1-1	1000 ppm	Hg	1.0 mL	1 mL HNO_3	100 mL	10 ppm

Table G:
REAGENTS

Reagent/Stock Std	Used In	Purity	Vendor	Order #
Hydrochloric Acid, (36.5-38.0% HCL)	Std. And Prep	Trace Metals Grade	MG Scientific	9530-33
Nitric Acid (69.0-70.0% HNO_3)	Std. And Prep	Trace Metals Grade	MG Scientific	9598-34
Nanopure or Milli-Q Water	all	18 Meg Ohms	On-Site	On-Site
Hydrogen Peroxide (30% H_2O_2)	Prep	Trace Metals Grade	Fischer Scientific	H325-4

Table H:
CALIBRATION LEVELS

Element	Cal Blank	Calibration Standard 2 and RLVS as applicable	Calibration Standard 3 and RLVS as applicable	Calibration Standard 4	Calibration Standard 5	Calibration Standard 6
Aluminum	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Antimony	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Arsenic	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Barium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Beryllium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Boron	0 ppb		5 ppb	50 ppb	250 ppb	500 ppb
Cadmium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Calcium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Chromium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Cobalt	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Copper	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Iron	0 ppb	250 ppb	500ppb	2,500 ppb	12,500 ppb	25,000 ppb
Lithium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Lead	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Magnesium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Manganese	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Mercury	0 ppb	0.2 ppb	0.5 ppb	1.0 ppb	10 ppb	25 ppb
Molybdenum	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Nickel	0 ppb	1 ppb	5	50 ppb	250 ppb	500 ppb
Phosphorus	0 ppb	50 ppb	250 ppb	2500 ppb	12500 ppb	25000 ppb
Potassium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Selenium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Silicon	0 ppb	50 ppb	250 ppb	2500 ppb	12500 ppb	25000 ppb
Silver	0 ppb	0.5 ppb	2.5 ppb	25 ppb	125 ppb	250 ppb
Sodium	0 ppb	250 ppb	500 ppb	2,500 ppb	12,500 ppb	25,000 ppb
Strontium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Thallium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Tin	0 ppb		5 ppb	50 ppb	250 ppb	500 ppb
Titanium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Uranium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Vanadium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Zinc	0 ppb		5 ppb	50 ppb	250 ppb	500 ppb
Zirconium	0 ppb	1 ppb	5 ppb	50 ppb	250 ppb	500 ppb
Internal Standards$_1$	50 ppb each	50 ppb each	50 ppb each	50 ppb each	50 ppb each	50 ppb each

Note: Calibration levels may change if noted. All solutions in 10% HNO_3 and 5% HCl.

1 = The Internal Standard Concentration is a result of a 4X Dilution via a T adapter in the delivery lines to the nebulizer.

Table I:
INTERNAL STANDARDS
(Multiple Listing Indicates Interpolation Between the Surrounding Internal Standards)

ISTD	Analytes
^{45}Sc-CCT	Li, Be, B
^{45}Sc-KED ^{72}Ge	Na – Zn
^{72}Ge ^{89}Y	As – Sr
^{89}Y ^{115}In	Mo – Cd
^{115}In ^{159}Tb	Sn – Ba
^{159}Tb ^{209}Bi	Hg – Pb
^{209}Bi	U

Table J:
EQUATIONS

Analyte	Recommended Elemental Interference Equations
^{43}Ca [2]	-0.00125 * ^{88}Sr
^{45}Sc-KED	-0.06099* ^{31}P
^{45}Sc-CCT	-0.06099* ^{31}P -0.09699* ^{28}Si
^{51}V [1]	-2.0995 * ^{53}ClO
^{53}ClO	–0.11400 * ^{52}Cr
^{52}Cr [1]	–0.00032 * ^{35}Cl
^{55}Mn	-0.00125*^{54}Fe
^{54}Fe	–0.02841 * ^{52}Cr
^{60}Ni	–0.00020 * ^{43}Ca
^{72}Ge	–0.00050 * ^{54}Fe
^{78}Se	–0.03065 * ^{83}Kr
^{111}Cd	–0.00055 * ^{95}Mo
^{115}In	–0.01416 * ^{118}Sn
^{201}Hg	-0.00055 * ^{184}W
^{208}Pb	1.00000 * ^{206}Pb + 1.00000 * ^{207}Pb

1 – The equation may require periodic adjustment based on the KED add gas and tuning parameters. The mean value in counts per second (cps) for the calibration blank **MUST** be > 0 cps (ideally, all replicates should be > 0 cps). The normal operating range in cps is 0-1000cps for the calibration blank and must be inspected by the analyst.
2 – Both the ICSAB and the LCS sample are used in the evaluation of this equation. Ca is affected by doubly charged strontium and can vary from day to day plasma conditions. The ICSAB will not fully identify doubly charged conditions based on the ratio of Ca to Sr, whereas the LCS sample for waters and soils have sufficiently large Sr concentrations compared to Ca and will assist in identification of adjustment requirements for the Ca interference equation.

Table K:
INTEGRATION TIMES

Mass	Element	Resolution Mode	Dwell Time Per Mass (mSec)	Mass	Element	Resolution Mode	Dwell Time Per Mass (mSec)
7	Li	Standard	10				
9	Be	Standard	10	72	Ge	Standard	10
10	B	Standard	10	75	As	Standard	50
23	Na	High	5	78	Se	Standard	50
25	Mg	Standard	10	83	Kr	Standard	50
27	Al	Standard	10	89	Y	Standard	10
28	Si	Standard	10	95	Mo	Standard	10
31	P	Standard	10	107	Ag	Standard	10
34	S	High	10	111	Cd	Standard	10
35	Cl	High	10	115	In	Standard	10
39	K	High	10	118	Sn	Standard	10
43	Ca	Standard	10	121	Sb	Standard	10
45	Sc-KED	Standard	10	137	Ba	Standard	10
45	Sc-CCT	High	10	159	Tb	Standard	10
47	Ti	Standard	10	184	W	Standard	10
51	V	Standard	10	201	Hg	Standard	10
52	Cr	Standard	10	205	Tl	Standard	10
54	Fe	Standard	10	206	Pb	Standard	10
55	Mn	Standard	10	207	Pb	Standard	10
59	Co	Standard	10	208	Pb	Standard	10
60	Ni	Standard	10	209	Bi	Standard	10
63	Cu	Standard	10	238	U	Standard	10
66	Zn	Standard	10				

Table L:
INSTRUMENT QC LIMITS

Element	ICV (ppb)	CCV (ppb)	ICV/CCV Limits	ICSA (ppb)	ICSAB (ppb)	ICSAB Limits	RLVS (ppb)	RLVS Limits
Aluminum	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Antimony	110	100	90%-110%		100	80%-120%	1	70-130%
Arsenic	110	100	90%-110%		100	80%-120%	1	70-130%
Barium	110	100	90%-110%		100	80%-120%	1	70-130%
Beryllium	110	100	90%-110%		100	80%-120%	1	70-130%
Boron	110	100	90%-110%		100	80%-120%	5	70-130%
Cadmium	110	100	90%-110%		100	80%-120%	1	70-130%
Calcium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Chromium	110	100	90%-110%		100	80%-120%	1	70-130%
Cobalt	110	100	90%-110%		100	80%-120%	1	70-130%
Copper	110	100	90%-110%		100	80%-120%	1	70-130%
Iron	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Lithium	110	100	90%-110%		100	80%-120%	1	70-130%
Lead	110	100	90%-110%		100	80%-120%	1	70-130%
Magnesium	110	100	90%-110%	50,000	50,000	80%-120%	250	70-130%
Manganese	11000	10000	90%-110%		100	80%-120%	1	70-130%
Mercury	4	5	90%-110%		5	80%-120%	0.2	70-130%
Molybdenum	110	100	90%-110%	1,000	1,100	80%-120%	1	70-130%
Nickel	110	100	90%-110%		100	80%-120%	1	70-130%
Phosphorus	5500	5000	90%-110%	50000	55000	80%-120%	50	70-130%
Potassium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Selenium	110	100	90%-110%		100	80%-120%	1	70-130%
Silicon	5500	5000	90%-110%		5000	80%-120%	25	70-130%
Silver	55	50	90%-110%		50	80%-120%	0.5	70-130%
Sodium	11000	10000	90%-110%	50,000	50,000	80%-120%	250	70-130%
Strontium	110	100	90%-110%		100	80%-120%	1	70-130%
Thallium	110	100	90%-110%		100	80%-120%	1	70-130%
Tin	110	100	90%-110%		100	80%-120%	5	70-130%
Titanium	110	100	90%-110%	1,000	1,100	80%-120%	1	70-130%
Uranium	110	100	90%-110%		100	80%-120%	1	70-130%
Vanadium	110	100	90%-110%		100	80%-120%	1	70-130%
Zinc	110	100	90%-110%		100	80%-120%	5	70-130%
Zirconium	110	100	90%-110%		100	80%-120%	1	70-130%

Table M:
AQUEOUS BATCH QC
ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	Method Blank (RL in ppb)	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	250	5,000	80-120	5,000	75-125	5,000	80-120
Antimony	1	500	80-120	500	75-125	500	80-120
Arsenic	1	500	80-120	500	75-125	500	80-120
Barium	1	500	80-120	500	75-125	500	80-120
Beryllium	1	500	80-120	500	75-125	500	80-120
Boron	20	500	80-120	500	75-125	500	80-120
Cadmium	1	500	80-120	500	75-125	500	80-120
Calcium	250	5,000	80-120	5,000	75-125	5,000	80-120
Chromium	1	500	80-120	500	75-125	500	80-120
Cobalt	1	500	80-120	500	75-125	500	80-120
Copper	1	500	80-120	500	75-125	500	80-120
Iron	250	5,000	80-120	5,000	75-125	5,000	80-120
Lithium	1	500	80-120	500	75-125	500	80-120
Lead	1	500	80-120	500	75-125	500	80-120
Magnesium	250	5,000	80-120	5,000	75-125	5,000	80-120
Manganese	1	500	80-120	500	75-125	500	80-120
Mercury	0.2	5	80-120	5	75-125	5	80-120
Molybdenum	1	500	80-120	500	75-125	500	80-120
Nickel	1	500	80-120	500	75-125	500	80-120
Phosphorus	50	500	80-120	500	75-125	500	80-120
Potassium	250	5,000	80-120	5,000	75-125	5,000	80-120
Selenium	1	500	80-120	500	75-125	500	80-120
Silicon	50	2500	80-120	2500	75-125	2500	80-120
Silver	0.5	250	80-120	250	75-125	250	80-120
Sodium	250	5,000	80-120	5,000	75-125	5,000	80-120
Strontium	1	500	80-120	500	75-125	500	80-120
Thallium	1	500	80-120	500	75-125	500	80-120
Tin	10	500	80-120	500	75-125	500	80-120
Titanium	1	500	80-120	500	75-125	500	80-120
Uranium	1	500	80-120	500	75-125	500	80-120
Vanadium	1	500	80-120	500	75-125	500	80-120
Zinc	510	500	80-120	500	75-125	500	80-120
Zirconium	1	500	80-120	500	75-125	500	80-120

Please see the QA manager for the most current MDLs.

Table N:
SOLIDS BATCH QC
ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	Method Blank (RL in ppb)	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	250	5000	80-120	5000	75-125	5000	80-120
Antimony	1	500	80-120	500	75-125	500	80-120
Arsenic	1	500	80-120	500	75-125	500	80-120
Barium	1	500	80-120	500	75-125	500	80-120
Beryllium	1	500	80-120	500	75-125	500	80-120
Boron	20	500	80-120	500	75-125	500	80-120
Cadmium	1	500	80-120	500	75-125	500	80-120
Calcium	250	5000	80-120	5000	75-125	5000	80-120
Chromium	1	500	80-120	500	75-125	500	80-120
Cobalt	1	500	80-120	500	75-125	500	80-120
Copper	1	500	80-120	500	75-125	500	80-120
Iron	250	5000	80-120	5000	75-125	5000	80-120
Lithium	1	500	80-120	500	75-125	500	80-120
Lead	1	500	80-120	500	75-125	500	80-120
Magnesium	250	5000	80-120	5000	75-125	5000	80-120
Manganese	1	500	80-120	500	75-125	500	80-120
Mercury	0.2	5	80-120	5	75-125	5	80-120
Molybdenum	1	500	80-120	500	75-125	500	80-120
Nickel	1	500	80-120	500	75-125	500	80-120
Phosphorus	50	500	80-120	500	75-125	500	80-120
Potassium	250	5000	80-120	5000	75-125	5000	80-120
Selenium	1	500	80-120	500	75-125	500	80-120
Silicon	50	2500	80-120	2500	75-125	2500	80-120
Silver	0.5	250	80-120	250	75-125	250	80-120
Sodium	250	5000	80-120	5000	75-125	5000	80-120
Strontium	1	500	80-120	500	75-125	500	80-120
Thallium	1	500	80-120	500	75-125	500	80-120
Tin	40	500	80-120	500	75-125	500	80-120
Titanium	1	500	80-120	500	75-125	500	80-120
Uranium	1	500	80-120	500	75-125	500	80-120
Vanadium	1	500	80-120	500	75-125	500	80-120
Zinc	20	500	80-120	500	75-125	500	80-120
Zirconium	1	500	80-120	500	75-125	500	80-120

Please see the QA manager for the most current MDLs.

Table O:
BIOTA BATCH QC ON INSTRUMENT
CONCENTRATIONS AND LIMITS

Element	Method Blank (RL in ppb)	LCS/ LCSD (ppb)	LCS/LCSD Limits (%)	MS/MSD Amount (ppb)	MS/MSD Limits (%)	PS Amount (ppb)	PS Limits (%)
Aluminum	250	10,000	80-120	10,000	75-125	10,000	80-120
Antimony	1	200	80-120	200	75-125	200	80-120
Arsenic	1	200	80-120	200	75-125	200	80-120
Barium	1	200	80-120	200	75-125	200	80-120
Beryllium	1	200	80-120	200	75-125	200	80-120
Boron	20	200	80-120	200	75-125	200	80-120
Cadmium	1	200	80-120	200	75-125	200	80-120
Calcium	1000	10,000	80-120	10,000	75-125	10,000	80-120
Chromium	1	200	80-120	200	75-125	200	80-120
Cobalt	1	200	80-120	200	75-125	200	80-120
Copper	5	200	80-120	200	75-125	200	80-120
Iron	250	10,000	80-120	10,000	75-125	10,000	80-120
Lithium	1	500	80-120	500	75-125	500	80-120
Lead	1	200	80-120	200	75-125	200	80-120
Magnesium	1000	10,000	80-120	10,000	75-125	10,000	80-120
Manganese	5	200	80-120	200	75-125	200	80-120
Mercury	0.2	5	80-120	5	75-125	5	80-120
Molybdenum	10	200	80-120	200	75-125	200	80-120
Nickel	1	200	80-120	200	75-125	200	80-120
Phosphorus	50	200	80-120	200	75-125	200	80-120
Potassium	1000	40,000	80-120	40,000	75-125	40,000	80-120
Selenium	2	200	80-120	200	75-125	200	80-120
Silicon	50	1000	80-120	1000	75-125	1000	80-120
Silver	0.5	100	80-120	100	75-125	100	80-120
Sodium	1000	10,000	80-120	10,000	75-125	10,000	80-120
Strontium	1	200	80-120	200	75-125	200	80-120
Thallium	1	200	80-120	200	75-125	200	80-120
Tin	75	200	80-120	200	75-125	200	80-120
Titanium	5	200	80-120	200	75-125	200	80-120
Uranium	1	200	80-120	200	75-125	200	80-120
Vanadium	2	200	80-120	200	75-125	200	80-120
Zinc	20	200	80-120	200	75-125	200	80-120
Zirconium	1	200	80-120	200	75-125	200	80-120

Note: Biota spikes are fortified for K as a result of its natural presence in the Chicken blank.
Please see the QA manager for the most current MDLs.

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STANDARD OPERATING PROCEDURE

DETERMINATION OF METALS BY INDUCTIVELY COUPLED PLASMA (ICP) SPECTROSCOPY BY 6010B

REFERENCE METHODS: SW-846 METHOD 6010B WITH 3010A AND 3050B

LOCAL SOP NUMBER:	S-GB-M-014-Rev.03
EFFECTIVE DATE:	Date of Final Signature
SUPERSEDES:	S-GB-M-014-Rev.02
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APPROVAL

Nils Melberg, Laboratory General Manager — Date: 12/19/12

Kate Grams, Laboratory Quality Manager — Date: 12/7/2012

Chad Rusch, Department Manager — Date: 12/07/2012

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION **PAGE**

1. PURPOSE **3**

2. SUMMARY OF METHOD **3**

3. SCOPE AND APPLICATION **3**

4. INTERFERENCES **5**

5. SAFETY **5**

6. DEFINITIONS **6**

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING **7**

8. EQUIPMENT AND SUPPLIES **7**

9. REAGENTS AND STANDARDS **9**

10. CALIBRATION **18**

11. PROCEDURES **21**

12. QUALITY CONTROL **26**

13. METHOD PERFORMANCE **28**

14. POLLUTION PREVENTION AND WASTE MANAGEMENT **29**

15. REFERENCES **29**

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.29

17. REVISIONS **30**

1. PURPOSE

This Standard Operating Procedure (SOP) documents the procedures used by PASI – Wisconsin to determine the concentration of specific metals in environmental water, wipe, paint chips, sludges, soils, and solid samples. The laboratory utilizes the ICP (Inductively Coupled Plasma – Optical Emission Spectrometer) and bases these documented procedures on those listed in EPA SW-846 Method 6010B. Sample preparation procedures are based on SW-846 Methods 3010A and 3050B.

2. SUMMARY OF METHOD

2.1 Samples are digested, excluding filtered groundwater, by heating with appropriate acids and oxidizing agents to solubilize the target elements. Portions of the digestates (or filtered, acidified groundwater samples) are pumped into a nebulizer to produce an aerosol. The aerosol is aspirated into the torch of Inductively Coupled argon Plasma Optical Emission Spectrometer (ICP-OES) where it is evaporated and decomposed into atoms and ions. The plasma energy causes the target atoms to become excited and, during relaxation, emit characteristic light in the visible and/or ultraviolet emissions. Each element in the sample emits photons at a discrete wavelength(s), which are specific to that element. The light emissions are separated into wavelength and order by passing through a prism and onto an Echelle grating. The signal is then read and quantified by a Charge Injection Device (CID). The intensities of the wavelengths are proportional to the quantity of the target elements that is determined through a comparison to a know concentration (a calibration curve). The signals received from the CID are digitized and relayed to the instrument computer as an analytical signal.

2.2 Background correction may be required to compensate for spectral interferences. Background is measured adjacent to analyte lines at a wavelength selected to be free of spectral interference and which reflects the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a correction would actually degrade the analytical result.

3. SCOPE AND APPLICATION

3.1 This procedure may be used to determine concentrations of trace metals in water, wipe, paint chips, sludges, soils, and solid samples. A list of applicable compounds and Practical Reporting Limits (PRL) for aqueous, solid and TCLP samples are shown in Table 3.1.

Table 3.1 – Elements and associated PRLs for water and leachates

	Element	Wavelength (nm)	PRL Aqueous (ug/L)	PRL Solid* (mg/kg)	PRL TCLP/SPLP/ASTM (mg/L)
Ag	Silver	328.0	10.0	1.0	0.250
Al	Aluminum	396.1	500	50.0	2.50
As	Arsenic	189.0	20	2.0	0.250
B	Boron	208.9	100	10.0	0.500
Ba	Barium	455.4	5.0	0.50	2.50
Be	Beryllium	313.0	4.0	0.40	0.250
Ca	Calcium	317.9	200	20	5.000
Cd	Cadmium	228.8	5.0	0.50	0.005
Co	Cobalt	228.6	5.0	0.50	0.250
Cr	Chromium	267.7	5.0	0.50	0.250
Cu	Copper	324.7	10	1.0	0.250
Fe	Iron	259.9	100	10.0	2.50
K	Potassium	766.4	200	20.0	5.000
Mg	Magnesium	279.0	200	20.0	5.000
Mn	Manganese	257.6	5.0	0.50	0.250
Mo	Molybdenum	202.0	20	2.0	0.250
Na	Sodium	589.5	1000	100.0	5.000
Ni	Nickel	231.6	10	1.0	0.250
Pb	Lead	220.3	7.5	1.0	0.0075
Sb	Antimony	206.8	20	2.0	0.250
Se	Selenium	196.0	20	2.0	0.250
Sn	Tin	189.9	50	5.0	0.250
Sr	Stronium	407.7	5.0	0.50	0.250
Ti	Titanium	334.9	5.0	0.50	0.250
Tl	Thallium	190.8	40	4.0	0.250
V	Vandaium	292.4	5.0	0.50	0.250
Zn	Zinc	206.2	40	4.0	0.250

* Based on standard weights/volumes (0.5g/50mL). Final RLs for individual samples may vary depending in actual weights/volumes and percent solids used. Soil include soils, sludges, paint chips, wipes, and solids. (Unit for wipes is in Total ug.)

Soils are reported on a dry weight basis.

3.2 This procedure is restricted to use by, or under the supervision of, analysts experienced in the digestion of samples for metals analysis and analysis of digestates by ICP. Each analyst must demonstrate the capability to generate acceptable results with this method to be considered qualified to report sample results.

3.3 This method cannot be substituted for other similar published methods where permit or regulatory compliance is required.

3.4 Current Method Detection Limits can be found in current LIMS or can be provided by the Quality Department.

4. INTERFERENCES

4.1 Spectral Interferences – Overlap of emission lines from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena and stray light can contribute to spectral interferences. These interferences can typically be minimized by careful selection of quantitation wavelengths, interelement corrections, and background correction.

4.2 Physical Interferences – Changes in sample viscosity, surface tension, or other effects associated with sample transport and nebulization can produce significant inaccuracies, especially in samples containing high concentrations of dissolved solids and acids. Dissolved solids may build up on the nebulizer tip, altering the sample flow rate and causing instrument drift. These effects can be minimized by sample dilution or use of a specially designed high-solids nebulizer. Use of internal standards helps in recognizing sample introduction issues.

4.3 Chemical Interferences – Molecular compound formation, ionization effects, and solute vaporization effects are typically not significant with ICP determinations. If observed, they can be minimized by careful selection of plasma and spectrometer operating parameters.

4.4 Memory Interferences – Sample deposition on the nebulizer tubing, spray chamber, and plasma torch can cause apparent sample carryover. Memory interferences can be minimized by flushing the system with rinse blanks between samples. If memory interference is suspected for a sample, the sample must be re-analyzed after a sufficient rinse period.

4.5 High Salt Concentrations – High salt concentrations in sample digestates can cause signal suppression and confuse interference tests. Use of internal standards helps in recognizing signal suppression.

5. SAFETY

5.1 Standards and Reagents

5.2 The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound shall be treated as a potential health hazard. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Special care shall be taken when handling the high-concentration acids and oxidizing reagents used for sample digestion. All digestions must be conducted in a properly functioning fume hood. The use of personal protective equipment (gloves, lab coats and safety glasses) is required.

5.3 Samples

5.3.1 Although sample check-in should be notified of any hazardous samples, samples shall always be considered as "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples.

5.4 Equipment

5.4.1 ICP Radio Frequency (RF) Generator – The RF generator used to induce the atomic plasma produces a high energy radio emission. The electrical requirements for this equipment are substantial. The RF generator must only be serviced by those trained specifically for service and repair of the instrument.

5.4.2 ICP RF Coil – The wound coil around the top of the torch produces a high energy, oscillating radio frequency field. The field is substantial and may interfere with surrounding electronics including implanted medical devices. Those individuals with such devices (i.e. pacemakers) must stay clear of this instrument while in operation.

6. DEFINITIONS

6.1 Refer to Glossary section of the Pace Quality Assurance Manual (QAM) for a comprehensive list of terms and definitions. In addition to those listed in the QAM, the following are additional terms found in this SOP.

6.2 Instrumental Detection Limit (IDL) – The concentration equivalent to a signal, due to the analyte, which is equal to the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day at the same wavelength.

6.3 Instrument Check Standard – A multi-element standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. This is the same as the Continuing Calibration Verification Standard (CCV) referred to in the calibration section of this SOP.

6.4 Spectral Interference Check Solution (SIC) – A solution of selected analytes of higher concentrations used to evaluate the procedural routine for correcting known inter-element spectral interferences. This is also known as the Inter-element Correction Solution (ICSA).

6.5 Linear Detection Range – Defined as the upper limit of quantitation for an analyte. The LDR is determined as the upper range limit of an observed signal that is no more than 10% below the level extrapolated from the lower standards.

6.6 Interference Check Sample (ICSAB) – A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.

7. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

7.1 Collection, Preservation, Storage and Hold Times

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil/ Solid/ Wipe/Paint Chip/Sludge	Pre-cleaned plastic or glass containers. Refer to Pace S-ALL-Q-021, Sub-Sampling SOP, for obtaining representative aliquots.	No Preservation	Refrigerated at 4+/- 2°C	Up to 6 months
Total Metals –Aqueous	Pre-cleaned plastic containers with HNO_3	Nitric Acid (HNO_3) to pH<2, preserved at time of collection	Room Temperature	Up to 6 months
Dissolved Metals – Aqueous(1)	Pre-cleaned plastic containers – Filter sample at time of collection through a 0.45µm membrane filter.	Nitric Acid (HNO_3) to pH<2, filtered then preserved at time of collection.	Room Temperature	Up to 6 months
TCLP/SPLP/ASTM(1)	Pre-cleaned plastic containers	-Filtered then preserved at time of digestion. -Refrigerate at 4±2 °C until preservation. -Nitric Acid (HNO_3) to pH<2	Refrigerate at 4±2 °C	From Field Collection to Leach Extraction: 180 Days. From Digestion to Determinative Analysis: 180 Days. (Total Elapsed Time: 360 Days).

(1) If filtration cannot be performed in the field, the sample must be taken in a pre-cleaned, unpreserved plastic container and transported to the lab as soon as possible. On arrival at the lab, the sample must be filtered and then preserved to a pH <2. The sample must sit for **24 hours** prior to preparation. This is to ensure that the sample does not have a buffering effect that raises the pH to above <2. The data is qualified if sample preservation fails to bring pH down to ≤2. (Note: TCLP Extracts are not required to sit for 24 hours prior to digestion after the addition of nitric acid.)

7.2 Preservative Check

The pH of samples must be verified to be pH<2 and documented in the Sample Receiving or Metals pH logbook prior to aliquoting for analysis. If a sample has a pH>2, additional preservative must be added to return the sample to <2. Acid volume shall not to exceed 2% of the container capacity. Once adjusted, the sample must be allowed to sit for 24 hours prior to preparation. If the sample is not able to maintain a preservation of a pH of<2, then it must be qualified as such.

8. EQUIPMENT AND SUPPLIES

8.1 Instrumentation

Equipment	Vendor	Model / Version	Description / Comments
ICAP System	Thermo Scientific	ICAP 6500	Serial Number: 20073913
Autosampler	Cetac	ASX-520	Serial Number: 080735A 520
Data System	iTEVA	2.0.039	
Reciculator	Neslab	ThermoFlex900	Serial Number: 107271037
Analytical Balance			

8.2 General Supplies

Item	Vendor (or equivalent)	Model / ID (or equivalent)	Catalog # (or equivalent)	Description
Argon – high purity grade	PraxAir			Bulk
Autosampler Vials	Fisher Scientific	15 mL Polypropylene Test Tubes	14-375-150	
Autosampler Pump Tubing	Analytical West		PT-2180PS	red/red 1.14mm ID – Carrier
			PT-2200SAS	1.30mm ID – Waste
			PT-2080PS-F	orange/orange 0.38mm ID – Internal Standard
Digestion Vials	Environmental Express		SC499	Pre-cleaned polypropylene 70-mL screw cap vials.
Watch Glass	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Heating Block	Environmental Express			Polypropylene, to fit 70-mL digestion vial
Mechanical Pipettes and Tips	Eppendorf			Or equivalent
Wash Bottle				
Filter paper	Whatman		541 Filter Paper	
pH Strips				
Y Connectors	Hewlett-Packard		G1820-65106	
Duo Torch / Duo Ceramic Cone	Thermo Fisher Scientific		8423-120-51241 / 84223-120-51261	
Spray Chamber/Spray Chamber adapter	Thermo Fisher Scientific		8423-120-51411 / 8423-120-51251	
2mm Center Tube	Thermo Fisher Scientific		8423-120-51371	
SC-FAST Probe	ESI		SC-5037-3995-150	7" Teflon/Carbon Probe 1.0mmID
SC-FAST Waste Line	ESI		SC-0323-0002-215	Waste Line
SC-FAST Valve	ESI	F7	SC-0599-1024	F7 Valve Head
	ESI		ES-2501-PPF2	
	ESI		ES-2501-PPMZ	
Peri-Pump	ESI			Fluoropolymer union, barbed
Nebulizer Line	ESI		SC-0317-0250-30	
Nuts and Ferrules	ESI		SC-0599-0116-K	For high flow 1/16" tubing
Nuts and Ferrules	ESI		SC-0599-0108-W	For high flow 1/8" tubing
Vacuum Line	ESI		SC-0321-32-215	For SC-FAST high flow valve
Sample Loop	ESI		SC-0318-15	1.5mL sample loop
Nebulizer	ESI		ES-2040	Micro Flow PFA-St nebulizer

8.3 Glassware

Glassware	Vendor / Item # / Description
Volumetric Flasks	Class A
Graduated Cylinders	Class A

9. REAGENTS AND STANDARDS

9.1 Reagents

Reagent	Concentration/ Description	Requirements/ Vendor/ Item #
De-ionized Nano-pure water (DI)	ASTM Type II, or equivalent (18mOhm or higher resistivity)	Nanopure Water
Nitric Acid (HNO_3)	Trace metals grade, Fisher Insta-analyzed, or equivalent	J.T Baker Cat #9598-34 or equivalent
Hydrochloric Acid (HCl)	Trace metals grade, Fisher Insta-analyzed, or equivalent	Fisher Cat # A508 or equivalent
Hydrogen Peroxide (H_2O_2)	30% certified reagent grade; trace metals grade if available. Stabilized solutions come with Sodium added.	Fisher Cat # H325-4 or equivalent
Acidified Nano-pure water	5mL Nitric Acid and 5mL Hydrochloric Acid per 100mL Nano-pure water	

9.2 Standards Definitions, Storage, and Sources

9.2.1 Definitions

Standard	Description	Comments
Initial Calibration Standard(s)	Standards prepared from single and/or multi-element standard(s) at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range for single point calibration.	Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier.
Initial Calibration Verification Standard	Standard prepared near the midpoint of the calibration range and is used to verify the accuracy of the calibration.	Must be prepared from a source independent of CCV and Calibration Solutions.
Continuing Calibration Verification Standard	Standards prepared from single and/or multi-element standards at appropriate acid concentrations. Solutions containing all analyte elements are prepared at a concentration near the mid-point of the calibration range. This standard verifies the accuracy of the calibration curve.	May be prepared from the same source as the calibrations standards
Low Level Check Standard (CRDL)	Must be performed if the lowest non-zero calibration point is not at or below the reporting limit. The recovery must be between 50 and 150% to report sample data. The recovery must be between 60 and 140% for samples reported to the State of MN.	NELAC requirement for single point calibrations, a client specific requirement for certain QAPPs
Inter-element correction solution (ICSA)	A solution prepared with high concentrations of interfering elements (Al, Ca, Fe, Mg, …). This is run daily at a minimum to verify the inter-element correction factors.	Al, Ca, Fe, Mg
Inter-element correction solution (ICSAB)	A solution prepared with high concentrations of interfering elements and low level concentrations of the other target elements. This is analyzed by the Green Bay Laboratory per client request, and for samples requiring QC Level 3 and 4 data packages, immediately following the ICSA solution.	Not a 6010B requirement but may be required on a client specific basis.
Internal Standard	A solution added to all standards, samples, spikes, control samples, and method blanks prior to analysis. This standard solution contains a non-target element (yttrium (Y)) at a concentration yielding an appropriate final concentration in the sample.	Y at 5ppm with Li. Added in-line to all samples and standards.
Single Element Standards	Stock standards purchased from venders containing one element. Used for checking IECs and may be used for checking linear ranges.	Must be 99.99% pure.
Spiking Standard	This solution contains all target analytes and should not be prepared from the same standards as the calibration standards.	

9.2.2 Storage Conditions

Standard Type	Description	Expiration	Storage
Stock Solutions	▪ Concentrated reference solution purchased directly from approved vendor	▪ Manufacturer's recommended expiration date for unopened standards.	▪ Manufacturer's recommended storage conditions ▪ When standard is opened, record all information in the standard logbook.
Intermediate and Working Standard Solutions	▪ Reference solutions prepared by dilutions of the stock solution	▪ Should be prepared as needed or at least every 6 months. The intermediate or working standard can not exceed the expiration date recommended by the manufacturer for each component used.	▪ Store at room temperature

9.3 Standard Sources

9.3.1 Standards are prepared from commercially available multi-compound stock solutions. The sources of the stock solutions, formulas for preparing dilutions and working standards, and concentrations for all compounds are presented in tables within section 9.3. All intermediate and working standards are prepared using acidified Nano-pure water and used immediately or stored for no longer than 6 months.

9.3.2 Analytical Working Standard Preparations - Depending on the volume of each solution needed, the standards are brought to volume in volumetric flasks with Nano-pure water unless otherwise noted.

Table 9.3.1 – Calibration Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	2 ml	10 mL HNO3/ 10 mL HCl	200 ml	1.0 ppm
	Ba	100 ppm				1.0 ppm
	Be	100 ppm				1.0 ppm
	Cd	100 ppm				1.0 ppm
	Co	100 ppm				1.0 ppm
	Cr	100 ppm				1.0 ppm
	Cu	100 ppm				1.0 ppm
	Mn	100 ppm				1.0 ppm
	Ni	100 ppm				1.0 ppm
	Pb	100 ppm				1.0 ppm
	Se	100 ppm				1.0 ppm
	Sr	100 ppm				1.0 ppm
	Tl	100 ppm				1.0 ppm
	V	100 ppm				1.0 ppm
	Zn	100 ppm				1.0 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	4 ml			2.0 ppm
	Mo	100 ppm				2.0 ppm
	Sb	100 ppm				2.0 ppm
	Sn	100 ppm				2.0 ppm
	Ti	100 ppm				2.0 ppm
	Ag	50 ppm				1.0 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	10 ml			50 ppm
	Ca	1000 ppm				50 ppm
	Fe	1000 ppm				50 ppm
	K	1000 ppm				50 ppm
	Mg	1000 ppm				50 ppm
	Na	1000 ppm				50 ppm

Table 9.3.2 – CCV Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
Spex #1 (Spex MixStd 1-100)	As	100 ppm	5.0 ml	50 mL HNO3/ 50 mL HCl	1000 ml	0.5 ppm
	Ba	100 ppm				0.5 ppm
	Be	100 ppm				0.5 ppm
	Cd	100 ppm				0.5 ppm
	Co	100 ppm				0.5 ppm
	Cr	100 ppm				0.5 ppm
	Cu	100 ppm				0.5 ppm
	Mn	100 ppm				0.5 ppm
	Ni	100 ppm				0.5 ppm
	Pb	100 ppm				0.5 ppm
	Se	100 ppm				0.5 ppm
	Sr	100 ppm				0.5 ppm
	Tl	100 ppm				0.5 ppm
	V	100 ppm				0.5 ppm
	Zn	100 ppm				0.5 ppm
Spex #2 (Spex MixStd2-100)	B	100 ppm	10.0 ml			1.0 ppm
	Mo	100 ppm				1.0 ppm
	Sb	100 ppm				1.0 ppm
	Sn	100 ppm				1.0 ppm
	Ti	100 ppm				1.0 ppm
	Ag	50 ppm				0.5 ppm
Spex #3 (Spex MixStd3-100)	Al	1000 ppm	25.0 ml			25 ppm
	Ca	1000 ppm				25 ppm
	Fe	1000 ppm				25 ppm
	K	1000 ppm				25 ppm
	Mg	1000 ppm				25 ppm
	Na	1000 ppm				25 ppm

Table 9.3.3 – ICV (Second Source) Working Standard Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)
IV #1 (PA-STD-1B)	As	200 ppm	0.5 ml	10 mL HNO3/ 10 mL HCL	200 ml	0.5 ppm
	Ba	200 ppm				0.5 ppm
	Be	200 ppm				0.5 ppm
	Cd	200 ppm				0.5 ppm
	Co	200 ppm				0.5 ppm
	Cr	200 ppm				0.5 ppm
	Cu	200 ppm				0.5 ppm
	Mn	200 ppm				0.5 ppm
	Ni	200 ppm				0.5 ppm
	Pb	200 ppm				0.5 ppm
	Se	200 ppm				0.5 ppm
	Sr	200 ppm				0.5 ppm
	Tl	200 ppm				0.5 ppm
	V	200 ppm				0.5 ppm
	Zn	200 ppm				0.5 ppm
IV #2 (PA-STD-2B	B	200 ppm	1.0 ml			1.0 ppm
	Mo	200 ppm				1.0 ppm
	Sb	200 ppm				1.0 ppm
	Sn	200 ppm				1.0 ppm
	Ti	200 ppm				1.0 ppm
	Ag	100 ppm				0.5 ppm
IV #3 (PA-STD-3B)	Al	2000 ppm	2.5 ml			25 ppm
	Ca	2000 ppm				25 ppm
	Fe	2000 ppm				25 ppm
	K	2000 ppm				25 ppm
	Mg	2000 ppm				25 ppm
	Na	2000 ppm				25 ppm

Table 9.3.4 – Spiking Standard (for LCS and MS/MSD) Dilutions and Concentrations

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppm)	Spike Concentration*
IV #1 (PA-STD-1B)	As	200 ppm	25 ml	12 mL HNO3	200 ml	50 ppm	500 ppb
	Ba	200 ppm				50 ppm	500 ppb
	Be	200 ppm				50 ppm	500 ppb
	Cd	200 ppm				50 ppm	500 ppb
	Co	200 ppm				50 ppm	500 ppb
	Cr	200 ppm				50 ppm	500 ppb
	Cu	200 ppm				50 ppm	500 ppb
	Mn	200 ppm				50 ppm	500 ppb
	Ni	200 ppm				50 ppm	500 ppb
	Pb	200 ppm				50 ppm	500 ppb
	Se	200 ppm				50 ppm	500 ppb
	Sr	200 ppm				50 ppm	500 ppb
	Tl	200 ppm				50 ppm	500 ppb
	V	200 ppm				50 ppm	500 ppb
	Zn	200 ppm				50 ppm	500 ppb
IV #2 (PA-STD-2B)	B	200 ppm	25 ml			50 ppm	500 ppb
	Mo	200 ppm				50 ppm	500 ppb
	Sb	200 ppm				50 ppm	500 ppb
	Sn	200 ppm				50 ppm	500 ppb
	Ti	200 ppm				50 ppm	500 ppb
	Ag	100 ppm				25 ppm	250 ppb
IV #3 (PA-STD-3B)	Al	2000 ppm	25 ml			500 ppm	5000 ppb
	Ca	2000 ppm				500 ppm	5000 ppb
	Fe	2000 ppm				500 ppm	5000 ppb
	K	2000 ppm				500 ppm	5000 ppb
	Mg	2000 ppm				500 ppm	5000 ppb
	Na	2000 ppm				500 ppm	5000 ppb

*** Use 1.0 ml of spiking std to 50 ml**

Table 9.3.5 – Low Level Check Standard (CRDL) Intermediate

Pace Custom Standard (Stock Standard)	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
	Ag	10 ppm				1,000 ppb
	Al	500 ppm				50,000 ppb
	As	20 ppm				2,000 ppb
	B	100 ppm				10,000 ppb
	Ba	5 ppm				500 ppb
	Be	4 ppm				400 ppb
	Ca	200 ppm				20,000 ppb
	Cd	5 ppm				500 ppb
	Co	5 ppm				500 ppb
	Cr	5 ppm				500 ppb
	Cu	10 ppm				1,000 ppb
	Fe	100 ppm				10,000 ppb
	K	1000 ppm				100,000 ppb
4400-100118CS01	Mg	200 ppm	10 ml	5 mL HNO3/ 5 mL HCl	100 ml	20,000 ppb
	Mn	5 ppm				500 ppb
	Mo	20 ppm				2,000 ppb
	Na	1000 ppm				100,000 ppb
	Ni	10 ppm				1,000 ppb
	Pb	7.5 ppm				750 ppb
	Sb	20 ppm				2,000 ppb
	Se	20 ppm				2,000 ppb
	Sn	50 ppm				5,000 ppb
	Sr	5 ppm				500 ppb
	Ti	5 ppm				500 ppb
	Tl	40 ppm				4,000 ppb
	V	5 ppm				500 ppb
	Zn	40 ppm				4,000 ppb

Table 9.3.5b – Low Level Check Standard (CRDL)

Low Level Check Standard Intermediate	Element Components	Stock Concentration (ppm)	Stock Volume Used (mL)*	Acid Used (mL)	Final Total Volume (mL)	Final Concentration in Standard (ppb)
CRDL Intermediate (In Table 9.3.5)	Ag	1,000 ppb	2 ml	10 mL HNO3/ 10 mL HCl	200 ml	10 ppb
	Al	50,000 ppb				500 ppb
	As	2,000 ppb				20 ppb
	B	10,000 ppb				100 ppb
	Ba	500 ppb				5 ppb
	Be	400 ppb				4 ppb
	Ca	20,000 ppb				200 ppb
	Cd	500 ppb				5 ppb
	Co	500 ppb				5 ppb
	Cr	500 ppb				5 ppb
	Cu	1,000 ppb				10 ppb
	Fe	10,000 ppb				100 ppb
	K	100,000 ppb				1000 ppb
	Mg	20,000 ppb				200 ppb
	Mn	500 ppb				5 ppb
	Mo	2,000 ppb				20 ppb
	Na	100,000 ppb				1000 ppb
	Ni	1,000 ppb				10 ppb
	Pb	750 ppb				7.5 ppb
	Sb	2,000 ppb				20 ppb
	Se	2,000 ppb				20 ppb
	Sn	5,000 ppb				50 ppb
	Sr	500 ppb				5 ppb
	Ti	500 ppb				5 ppb
	Tl	4,000 ppb				40 ppb
	V	500 ppb				5 ppb
	Zn	4,000 ppb				40 ppb

Table 9.3.6– Inter-element Correction Solution (ICSA) Working Dilutions and Concentrations

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	10	200	Al	500	10000
Single Element Ca	Ultra Scientific	ICP-120	10	200	Ca	500	10000
Single Element Mg	Ultra Scientific	ICP-112	10	200	Mg	500	10000
Single Element Fe	Ultra Scientific	ICP-126	4	200	Fe	200	10000

Table 9.3.6b– Inter-element Correction Solution (ICSAB) Working Dilutions and Concentrations – Include as needed.

Pace Standard #	Vendor	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (µg/mL)	Stock Concentration (µg/mL)
Single Element Al	Ultra Scientific	ICP-113	9.5	200	Al	475	10000
Single Element Ca	Ultra Scientific	ICP-120	9.5	200	Ca	475	10000
Single Element Mg	Ultra Scientific	ICP-112	9.5	200	Mg	475	10000
Single Element Fe	Ultra Scientific	ICP-126	3.5	200	Fe	175	10000
IV #1 (PA-STD-1B)	Inorganic Ventures	PA-STD-1B	0.5	200	As	0.5	200 ppm
					Ba	0.5	200 ppm
					Be	0.5	200 ppm
					Cd	0.5	200 ppm
					Co	0.5	200 ppm
					Cr	0.5	200 ppm
					Cu	0.5	200 ppm
					Mn	0.5	200 ppm
					Ni	0.5	200 ppm
					Pb	0.5	200 ppm
					Se	0.5	200 ppm
					Sr	0.5	200 ppm
					Tl	0.5	200 ppm
					V	0.5	200 ppm
					Zn	0.5	200 ppm
IV #2 (PA-STD-2B)	Inorganic Ventures	PA-STD-2B	1.0	200	B	1.0	200 ppm
					Mo	1.0	200 ppm
					Sb	1.0	200 ppm
					Sn	1.0	200 ppm
					Ti	1.0	200 ppm
					Ag	0.5	100 ppm
IV #3 (PA-STD-2B)	Inorganic Ventures	PA-STD-3B	2.5	200	Al	25	2000 ppm
					Ca	25	2000 ppm
					Fe	25	2000 ppm
					K	25	2000 ppm
					Mg	25	2000 ppm
					Na	25	2000 ppm
Single Element Ni	Ultra Scientific	ICP-028	0.1	200	Ni	1	1000 ppm

Table 9.3.7– Internal Standard Solution Working Dilutions and Concentrations

Pace Standard #	Stock Catalog Number	Stock Volume Used (mL)	Final Total Volume (mL)	Element Components	Final Concentration in Standard (mg/L)	Stock Concentration (mg/L)
IS	ICP69N-1 (Accustandard)	5	1000	Yttrium	5 mg/L	1000
	ICP-103-5 (Ultra Scientific)	100	1000	Lithium	1000 mg/L	10000

10. CALIBRATION

10.1 Profile

The iCAP 6500 ICP will automatically check a carbon reference line against ambient air conditions each time the plasma is ignited to maintain wavelength accuracy.

10.2 Daily Initial Calibration

Allow the instrument 30 minutes to warm up after igniting the torch. The ICP must be calibrated each time it is set up for analysis or every 24 hours according to the manufacturer's instructions. Calibration requires analysis of a Calibration Blank and at least one level of calibration solution. Calibration standards can be made and used for 6 months unless a standard used to make them expires earlier. It would then expire on that date. Instrument standardization date and time must be recorded in the raw data. See Table 9.3.1 for details on the preparation of the Initial Calibration Standard.

10.3 Calibration Verification

10.3.1 Initial Calibration Verification (ICV) / Second Source

To assess the accuracy relative to the purity of the calibration standards, a standard from a secondary source must be analyzed and the results obtained must be assessed relative to the known true value. This step is referred to as **Secondary Source Verification** or, alternatively as **Initial Calibration Verification**. This secondary source must be from an alternative vendor or, in the event an alternative vendor is not available, from a different lot from the same vendor. See the most current version of SOP S-GB-Q-026 *Standard and Reagent Management and Traceability* for more information of standards. This standard must be analyzed immediately after the calibration and before any batch QC or client samples. The accuracy of the standard is assessed as a percent difference from the true value according to the following equation:

$$\% \text{ Difference} = [\text{Result}_{ICV} - \text{TrueValue}_{ICV}] / \text{TrueValue}_{ICV} * 100$$

See Table 9.3.3 for details on the preparation of this standard. See Table 10.2 for control criteria

10.3.2 Reporting Limit Verification Standard (CRDL)

With every Initial Calibration, a standard corresponding to the practical reporting limit (PRL) must also be analyzed and meet established acceptance criteria.

Note: If a multi-point calibration is being run on the ICP, results detected below the lowest level calibration standard analyzed can only be reported if a PRL standard is also run and verified with that ICAL. If a PRL standard is not analyzed with that ICAL, results detected below the lowest calibration standard run for the multi-point calibration may not be reported. If the lowest calibration point corresponds to the PRL, this requirement is not applicable. The limits for the CRDL are +/- 50% of the true concentration, for work from MN the limits are +/-40%. See Table 9.3.5 for details on the preparation of this standard.

10.3.3 ICSA (Interference Check Standard) Verification

With every Initial Calibration, the inter-element check solution standard must be run to verify the inter-element correction factors. At a minimum, the ICSA standard must be analyzed at the beginning of the run, following the initial calibration and evaluated against the criteria in Table 10.2. If client specific requirements dictate that the ICSA must be analyzed at both the beginning and end of the analysis, the ICSA may be analyzed immediately after the completion of those specific samples and is not required to be placed at the very end of the sequence. If client specific requirements dictate that the ICSAB be run, the ICSAB must be analyzed immediately following the ICSA and be held to the criteria in Table 10.2.

10.3.4 Continuing Calibration Verification (CCV)

As part of the analytical process, the instrumentation must be checked periodically to determine if the response has changed significantly since the initial calibration was established. This verification process is known as **Continuing Calibration Verification**. The validity of the initial calibration is checked after every 10 samples and at the end of an analytical sequence by analyzing a midpoint calibration standard (CCV).

The values obtained from the analysis of the CCV are compared to the true values and a percent difference calculated according to the formula in Section 10.2.1. The percent difference must meet the method specified criteria in Table 10.2 for the analysis to proceed.

Table 10.2 – Calibration Acceptance and Verification Criteria

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression for elements requiring multiple calibration levels	Correlation coefficient ≥ 0.995	If not met, remake standards and recalibrate. One point calibration is standard for the Wisconsin laboratory.
Initial Calibration Verification Standard (Second Source)	Immediately after each initial calibration	≤±10% of true value The % RSD between replicate integrations of an ICV std must be <5% (min of 2 integrations).	If 4 or more replicates are used for analysis the RSD must be ≤ 3%
Calibration Blank	Immediately after each initial calibration (ICB), every 10 samples (CCB), and at the end of the analytical sequence	≤ PRL	If criteria are not met, follow the corrective actions in 10.3.2. If the concentration in the blank is outside the control limit and <1/10 the concentration in the sample, the sample result may be reported.
CRDL Standard	Once per ICAL, immediately after verification	Each element tested must be at least between 50-150% of the PRL value. If reporting for the state of MN, limits of 60-140% are applied.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.4 *Pace Metals 3P Team determined it is a best practice to use PRL limits of at least 50-150%. Limits may be more strict if required by client.
ICSA	Once per ICAL, immediately after verification	Results for non-interference elements = ND ± 2x RL Recovery of interfering elements must be 80-120% of true value	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.5
ICSAB	Once per ICAL, immediately after ICSA if requested by the client.	Results for non-interference elements = True Value ± 20%.	If recoveries are not acceptable, stop analysis and follow corrective actions in 10.3.5
Continuing Calibration Verification	After every 10 samples and at the end of analytical sequence	≤±10% of true value The % RSD between replicate integrations of a CCV std must be <5% (min of 2 integrations).	If criteria not met, follow corrective actions in 10.3.6

10.4 Calibration Corrective Actions

10.4.1 Calibration Linearity Problems

10.4.1.1 Reanalyze the original calibration standards to determine instrument consistency.

10.4.1.2 Prepare and analyze a new calibration standard to determine preparation consistency / standard integrity.

10.4.1.3 Perform instrument maintenance, document in instrument log book

10.4.1.4 Recalibrate instrument according to procedures in Section 9.

10.4.2 Calibration Blank Problems

10.4.2.1 Reanalyze the original CB standard to determine instrument consistency.

10.4.2.2 Prepare and analyze a new CB standard to determine preparation consistency / standard integrity. Note: If you run two CB, only one may be used for reporting purposes.

10.4.2.3 Perform instrument maintenance, document in instrument log book

10.4.2.4 Recalibrate instrument according to procedures in Section 10.

10.4.2.5 Reanalyze any samples not bracketed by passing CBs.

10.4.2.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CB that exceeded criteria may be reported if 10X concentration in the blank is less than the reported result.
Samples analyzed with a bracketing CB that exceeded criteria may be reported if the sample result is a non-detect.

10.4.3 Initial Calibration Verification (Secondary Source) Problems

10.4.3.1 Reanalyze the original ICV standard to determine instrument consistency.

10.4.3.2 Prepare and analyze a new ICV standard to determine preparation consistency / standard integrity.

10.4.3.3 Perform instrument maintenance, document in instrument log book

10.4.3.4 Recalibrate instrument according to procedures in Section 10.

10.4.3.5 Reanalyze any samples not bracketed by passing ICVs.

10.4.3.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

10.4.4 Exceptions: Samples analyzed with a bracketing ICV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

10.4.5 CRDL Standard Problems

10.4.5.11 Check CAL0 – Elevated counts resulting from contamination may cause the CRDL to fail low

10.4. 5.2 Reanalyze the original CRDL standard to determine instrument consistency.

10.4.5.3 Prepare and analyze a new CRDL standard to determine preparation consistency / standard integrity.

10.4.5.4 Perform instrument maintenance, document in instrument log book

10.4.5.5 Recalibrate instrument according to procedures in Section 10.

10.4.4.5 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with CRDL that is outside criteria may be footnoted or the CRDL for the reported sample adjusted.

10.4.6 ICSA/ICSAB Standard Problems

10.4.5.1 Reanalyze the original **ICSA/ICSAB** standard to determine instrument consistency.
10.4.5.2 Prepare and analyze a new **ICSA/ICSAB** standard to determine preparation consistency / standard integrity.
10.4.5.3 Adjust IEC and rerun **ICSA/ICSAB.** To update IECs, follow the instruction below.
10.4.5.4 The Interelement Correction Factors (IEC's) are used to correct for false signals (positive and negative) produced when peaks overlap in the spectra.
10.4.5.5 To set up Initial IEC, run single element standards with only background correction points in place. The standards are run at the linear range.

The following calculation is done:

$$\frac{\text{Element result(ug/L)}}{\text{Interfering element result(ug/L)}} = \text{IEC}$$

10.4.5.6 This ratio is entered in the IEC table in the method file. Type the ratio under the k1 header in the appropriate row. Enter a negative before the ratio if it is a negative interference.
10.4.5.6.1 Perform instrument maintenance, document in instrument log book
10.4.5.6.2 Recalibrate instrument according to procedures in Section 9.

10.4.7 Continuing Verification Problems

10.4.6.1 Reanalyze the original CCV standard to determine instrument consistency.
10.4.6.2 Prepare and analyze a new CCV standard to determine preparation consistency / standard integrity.
10.4.6.3 Perform instrument maintenance, document in instrument log book
10.4.6.4 Recalibrate instrument according to procedures in Section 10.
10.4.6.5 Reanalyze any samples not bracketed by passing CCVs.
10.4.6.6 If samples were analyzed in spite of verification failures, note the following exceptions for addressing those results. Deviations from this requirement must be noted on the run log with a thorough explanation for the deviation from policy.

Exceptions: Samples analyzed with a bracketing CCV that exceeded criteria due to an increase in response may be reported if the target analytes were not detected in the samples.

11. PROCEDURES

11.1 Aqueous Sample Preparation for Total or Dissolved Metals by 3010A – Hot Block Digestion

11.1.1 Groundwater samples for dissolved metals may be analyzed without digestion

unless there is visible sediment. Samples contains visible sediment must be digested. All other water samples must be digested.

11.1.2 Aqueous samples for total or dissolved metals (if visual precipitation) may be prepared according to EPA 3010A. Samples for dissolved metals must be filtered prior to preservation. Once preserved, samples should be allowed to sit for 24 hours prior to preparation. TCLP/SPLP/ASTM extracts do not require to sit for 24 hours prior to digestion.

11.1.3 Verify that the hood is turned on. Turn on the Hot Block and set the temperature so that samples on the block maintain a temperature of 95 +/- 5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample temperature in the sample preparation logbook. The well position should be rotated on a per batch basis to ensure that the block temperature is verified across the block.

11.1.4 Place a 50mL well-mixed aliquot of each sample in digestion vials. Place 50 mL of Nano-pure water into digestion vials for each MB and LCS (one per 20 samples). Prepare the MS and MSD by placing 50mL aliquots of selected sample from the batch into digestion vials. One MS and one duplicate or MSD must be prepared for each analytical batch of 20 samples or less. Additionally, one MS must be prepared for each TCLP/SPLP/ASTM sample matrix digested in a digestion batch.

11.1.5 Spike the LCS, MS, and MSD with 1.0mL of the spiking solution.

11.1.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial.

Digestion Program for all Aqueous Samples, EPA Method 3010A

Steps	Reagents[1]	Actions
1	5.0mL concentrated HNO_3, 2.5 mL HCl	Heat at 95 +/- 5°C °C for 4 hours
2		Remove digestates from Hot Block and allow to cool in the hood.
3		Adjust the final volume of each vial to 50 mL by diluting with 18 MOhm Nano-Pure Nano-pure water. Place on screw cap and shake thoroughly.
4		Centrifuge, let settle, or filter sample only if there is concern that insoluble materials may clog the nebulizer. Filtering is liable to cause sample contamination unless the filter and filtering apparatus are thoroughly cleaned and pre-rinsed with dilute HNO_3. If filtration is required, filter all QC samples as well.

1. Acid ratios must be maintained to match method. Those specified are based on a 50mL sample volume

Note: If a sample is allowed to go to dryness, low recoveries will result. Should this occur, discard the samples and re-prep.

11.1.7 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.2 Soil/ Solid/Sludge/Paint Chip/Wipe Sample Preparation by 3050B

11.2.1 Soil, sludges, and solids are prepared according to Method 3050B. Wipes and Paint Chips are also prepared using 3050B.

11.2.2 Verify that the hood is turned on. Turn on the Hot Block and set the temperature to 95±5°C. Before placing samples in the block to digest, verify that the sample well temperature is correct and adjust as required. Record the sample well ID and temperature in the sample logbook. This well measurement should be rotated on a per batch basis to ensure that the block temperature is consistent across the block.

11.2.3 To prepare Paint Chips for digestion, transfer paint chips into labeled drying tins. Dry samples at 103-105 degrees Celsius for 12-24 hours. Remove samples from oven and desiccate for 1 hour. Paint chips requirement that an LSC/LCSD be prepared per batch.

11.2.4 If possible, mix each sample thoroughly to homogenize. If homogenization is not feasible, remove the top layer of the sample from the container and discard. Remove a sample aliquot in the range of 0.5 – 0.599 g for analysis (0.5g dry sample). Weigh the aliquot to the nearest 0.01g and transfer to a digestion vial. Refer to Pace S-ALL-Q-021, *Sub-Sampling* SOP, for more details. For paint chips, Weigh out 0.5 gram of sample into a digestion tube. If the sample available is less than 0.5 gram, use the entire sample. For Wipes, digest the WHOLE wipe in the digestion vial. For sludges with % solids >10% and <50%, weigh out 2.5-5 grams.

11.2.5 Prepare the MB and LCS by adding 2.0 mL of Nano-pure water into digestion vials containing 0.500-0.504g of Teflon chips. The weight is documented as 0.500g so as to not bias the results. Prepare the duplicate, MS and MSD by placing aliquots of a selected sample from the batch into digestion vials as described for environmental samples. Spike the LCS, MS, and MSD with 1.0 mL spiking solution.

11.2.6 Carefully add the digestion reagents to each sample, MB, LCS, MS, and MSD vial and heat them with the Hot Block at 95±5°C according to the following digestion program.

11.2.7 MS/MSDs are not performed on paint chips, because the sample concentration is frequently excessively high and dwarfs any spike. As a result an LCSD is performed for paint chip batches...

Digestion Program for Soil/ Solid samples by method 3050B

Steps	Reagents[1]	Actions
1	Nano-pure water	Add Appoximately 2.0 mLs of water to all vessels in the prep batch.
2	10.0 mL 1:1 HNO3	Cover with watch glass and heat without boiling for 2 hours. Remove from heat and cool.
3	2.5 mL 30% H_2O_2	Cover with watch glass and heat on Hot Block for 15 minutes or until effervescence subsides. Warming will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Remove and allow to cool.
4	2.5mL concentrated HCl	Reflux on Hot Block until reaction is complete (no brown fumes) without boiling. Allow samples to cool.
5		Dilute sample to 50-mL final volume with 18 MOhm Nano-Pure water. Place screw cap on vials, shake, allow sample to settle, centrifuge at 2000-25000 rpm for 10 minutes, OR and filter if necessary.

1. Acid ratios must be maintained to match method. Those specified are based on a 0.5g sample amount
2. In effect, this produces the 1:1 nitric acid (required by method 3050) in the actual digestion cup.

11.2.8 Record the lot numbers of all reagents and materials in the digestion logbook. This includes the lot numbers of the digestion tubes, filters or filter paper and any additional materials in contact with the sample matrices.

11.3 ICP System Preparation

11.3.1 Preventative Maintenance

Inspect the sample introduction system including the nebulizer, torch, injector tube and up-take tubing for salt deposits, dirt and debris that could restrict solution flow and affect instrument performance. Document all maintenance in ICP maintenance logbook.

See attachment I for troubleshooting guide.

11.3.2 Operating Parameters

Set up and configure the ICP according to manufacturer's operating instructions using operating parameters shown in the following table.

Table 11.3.1 - Operating Parameter for ICP Systems

Operating Parameter	ICAP 6500 20073913
Argon Flow	80 L/min
Nebulizer Pressure	0.55 L/min
RF Output	1175 W
Pump Speed	15 rpm
Aux Gas Flow	0.5 L/min
Coolant Gas Flow	15 l/min

11.3.3 Calibrate ICP according to Section 10.

11.4 Sample Analysis

11.4.1 Batch Sequence

If using an autosampler, enter an autosampler sequence into the ICP data system per manufacturer's instructions. Calibration blanks, standards, initial checks, and interlement correction standards shall be run before analysis of environmental samples. All samples, including calibration, checks, and other standards, can also be run manually.

Table 11.4.1 – Example analytical sequence after calibration of a blank and a standard.

Autosampler Position	Sample Description	Autosampler Position	Sample Description
1	ICV	27	Sample 15
2	ICB	28	Sample 16
3	CRDL	29	CCV
4	ICSA*	30	CCB
5	CCV	31	Sample 17
6	CCB	32	Sample 18
7	MB1	33	Sample 19
8	LCS1	34	Sample 20
9	Sample 1	35	MB2
10	Sample 2	36	LCS2
11	Sample 3	37	Sample 21
12	Sample 3 Matrix Spike	38	Sample 22
13	Sample 3 Matrix Spike Duplicate	39	Sample 23
14	Sample 4	40	Sample 24
15	Sample 5	41	CCV
16	Sample 6	42	CCB
17	CCV	43	Sample 25
18	CCB	44	Sample 26
19	Sample 7	45	Sample 27
20	Sample 8	46	Sample 28
21	Sample 9	47	Sample 28 Matrix Spike
22	Sample 10	48	Sample 28 Matrix Spike Duplicate
23	Sample 11	49	Sample 29
24	Sample 12	50	Sample 30
25	Sample 13	51	CCV
26	Sample 14	52	CCB

* ICSAB will follow the ICSA if needed/requested by client.

11.4.2 Load Autosampler

11.6.1.1 Load the autosampler tray according to the vial position on the sequence table.

11.5 **Sample Analysis**

11.5.1 Analyze all standards, quality control samples, and environmental samples.

11.6 Data Reduction

11.6.1 **Quantitative Analysis** – The instrument produces results in ug/L. If the initial sample aliquot and final digestate volumes are the same, the ICP data system will calculate the concentration of each element directly. If the initial and final volumes are different, the actual values are posted into the LIMS system and calculations are performed in the LIMS.

11.6.1.1 Water Samples – Since initial sample aliquot and final digestate volumes are the same, the iCAP analyzer data system will calculate the concentration directly. No further calculations are necessary unless the sample was diluted.

11.6.1.2 Soil/Solid/Paint Chip/Sludge Samples -

Final Result (mg/kg dry weight corrected) =

$$\frac{\text{Raw data result (ug/L) * Final Volume (L) * Dilution Factor}}{\text{Sample weight (g) * Dry weight for soil/solid (decimal form)}}$$

11.6.1.3 Wipes

Final Result (ug/wipe) =

$$\frac{\text{Raw data result (ug/L) * Final Volume (L) * Dilution Factor}}{\text{1 wipe}}$$

11.6.2 Samples with analyte concentrations that exceed 90% of the upper linear range must be diluted and re-analyzed.

11.6.3 Any sample in which any element value is below the instrument detection limit or PRL must be reported as less than the PRL (or ND) for that specific element. Do not report data below the element PRL concentration unless it is qualified as an "estimated" result with a "J" or "B" data qualifier.

11.6.4 Metals Analytical Review and Reporting System (ADAMS) or LimsLink is used to transfer data from the instrument to the LIMS system.

12. QUALITY CONTROL

12.1 Refer to the most current version of the Pace Quality Manual Appendix I Quality Control Calculations and SOP: S-GB-Q-009 *Common Laboratory Calculations and Statistical Evaluation of Data* for equations and calculation details.

12.2 Instrument Quality Control

12.3 Refer to Table 11.2 for initial and continuing calibration criteria and corrective actions. This includes ICV, CCV, CB, PRL, and ICSA standard checks.

12.4 Batch Quality Control

Table 12.2 – Batch Quality Control Criteria

QA Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Nano-pure water	One per 20 samples	1) Target analytes must be less than the RL.	1) Re-analyze blank to confirm failure. 2) Qualify results and / or re-extract associated samples. **Exceptions:** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects no qualification is needed... 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Nano-pure water and Spiking solution (see table 10.3.3 for elements)	One per 20 samples	Laboratory Calculated Limits or default limits of 80-120% Should not exceed 80-120%	1) Reanalyze the LCS to verify failure 2) If LCS passes, review samples for potential problems 3) If problem persists, check spike solution 4) Re-digest and re-analyze samples where possible **Exceptions:** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Matrix Spike (MS)	Environmental Sample and Spiking solution (see table 10.3.3 for elements)	One per 20 samples. For leached samples one per sample matrix (soil and wood chips would each require a separate matrix spike).	Laboratory Calculated Limits or default limits of 75-125% Should not exceed 75-125%	1) If LCS and MBs are acceptable, the MS may be reported with appropriate footnote indicating matrix interferences
MSD / Duplicate	MS Duplicate OR (alternative) Sample Dup	A duplicate sample OR MS/MSD pair must be analyzed per batch of 20 samples.	Laboratory Derived Limits for RPD or default limits of 20%. Recovery criteria should not exceed 75-125%.	1) Results not meeting criteria must be footnoted on client report.

12.5 Dilution Test – A dilution test is recommended but not required for method 6010B. A dilution test is run only at client request.

12.5.1 Perform a 5X dilution on one sample per preparation batch. Diluted and undiluted sample results should agree within 10% of each other if the sample contains analyte concentrations at least 50 times the IDL. If agreement between diluted and undiluted samples meeting the concentration requirements is >10%, then matrix effect should be suspected and samples footnoted appropriately.

12.6 Post-digestion Spike Test or Analyte Addition – Post digestion spikes are recommended but not required for method 6010B. A post spike is run only at client request.

12.6.1 Spike one digestate from a preparation batch to achieve the same analyte addition level as found in the LCS. The acceptable recovery range is 75-125%. If outside this range, matrix effects should be suspected and samples annotated.

12.7 **Internal Standards** – Yttrium is used as an internal standard. Yttrium is not calibrated for. Precision of the Yttrium reps are evaluated using < or = 5% RSD. Accuracy of the Yttrium average count levels are evaluated using 50-150% recovery as compared to the calibration blank. If either of these criteria is not meet, the sample or quality control standard must be rerun, possibly at a dilution.

12.8 **Multiple Instrument Intregration RSD** – The RSD is required to be evaluated between multiple instrument integrations. The RSD must be less than or equal to 20% if the analyte concentration is greater than the reporting limit. If the RSD is greater than 20%, the laboratory must reanalyze the sample.

13. METHOD PERFORMANCE

13.1 Method Detection Limit (MDL) Study: An MDL study must be conducted annually per S-GB-Q-020, *Determination of LOD and LOQ* per instrument.

13.2 Demonstration of Capability (DOC): Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, *Orientation and Training Procedures.*

13.3 Linear Dynamic Range Study

13.3.1 It is a Pace best practice to perform this linear dynamic range study determination once, keep the data, and then annually or semi-annually verify with a single high point. If additional range is to be considered, the laboratory must perform the study as indicated below.

13.3.2 A linear dynamic range study must be conducted at least annually. The study is conducted for each element by analyzing increasing concentrations (at least 3 levels) until the results generated exceed ±10% difference from the true value. The highest concentration within the 10% criteria is the maximum of the linear range for that element.

13.4 **Instrument Detection Limits (IDLs)** in ug/L are estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs must be determined at least quarterly.

13.5 **Method Modifications**

Method modifications for EPA method 6010B are as follows:

- Modifications should be targeted to improve quality, efficiency or the cost effectiveness of the procedure.
- All major modifications to the procedure that may directly affect data quality must be thoroughly documented. A new demonstration of capability and equivalency must be performed and kept on record.
- Procedures identified as "Best Practices" by the PACE 3P Program will be incorporated into this document as minimum requirements for Pace laboratories.
- When insufficient volume for the method specified matrix spike/matrix spike duplicate (MS/MSD) is not provided by the client, a laboratory control spike duplicate will be analyzed to demonstrate precision criteria. Laboratory batches will be qualified with the appropriate "M5" data qualifier. When performing this analysis on paint chip samples, a MS/MSD will not be completed on the samples due to high levels of elements present in the native sample.

14. POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Procedures for handling waste generated during this analysis are addressed in S-GB-W-001, *Waste Handling and Management.*

14.2 In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (i.e. before a reagent expires)

14.3 The company wide Chemical Hygiene and Safety Manual contains additional information on pollution prevention.

15. REFERENCES

15.1 USEPA, SW-846, Method 6010B, "Inductively Coupled Plasma Atomic Emission Spectrometry", December 1996.

15.2 USEPA, SW-846, Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectrometry", December 1996.

15.3 USEPA, SW-846, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils", December 1996.

16. TABLES, DIAGRAMS, FLOWCHARTS, ATTACHMENTS, APPENDICES, ETC.

16.1 **Attachment I** - ICP Troubleshooting Guide

16.2 **Attachment II** – ICP Flowchart

17. REVISIONS

Document Number	Reason for Change	Date
S-ALL-GB-M-002-REV.00	Restructured document format to template. Added tables and other content to create an "SOP Template" in which local edits are allowed. Modified signature page to include department manager and SOP template number Updated SOP template number. Made editable material red Updated calibration section to reflect change for PRL requirements. Updated linear range to include best practice established by PaceMetals 3P team. Added separate sections for 3005 and 3010A digestion procedures. Updated procedures to match what is being performed in Wisconsin (noted in red) Added reference to Interelement Correction Factors and Instrument Detection Limits. Add a flowchart.	28Feb2007
S-ALL-GB-M-002-REV.01	Updated Section 2 to include iCAP Updated Table 3.1 to include iCAP 6500 wavelengths and current PRLs. Removed reference to Conifer2K in sections 3.4. In section 8, changed hours from preservation to preparation/analysis from 16 to 24. In section 9, changed instrument to iCAP 6500 from iCAP 61E Removed "(5% nitric and 5% hydrochloric acid)" from 10.3 Added Sr to tables 10.3.1, 10.3.2, 10.3.3, 10.3.4, 10.3.5, and 10.3.6b. Renamed Tables 10.3.5 to 10.3.7 to 10.36b to 10.3.7 to eliminate table number duplication. Removed instructions for As profile. Updated table 10.3.4 to use 1.0mL spike instead of 0.5 mL. Removed As Profile from 11.1. Added Carbon profile. Removed Se and Pb exception from IEC determination in 11.4.3.24	19Nov2007
S-ALL-GB-M-002-REV.02	Updated signature Page Deleted Section 7. Renumbered document. Section 9.2 – Updated Inter-element correction solution (ICSAB) description for the Green Bay Laboratory. Section 11.1.1 – Digestion criteria updated. Section 11.2.3 – Paint chips LCS/LCSD requirement added. Table 12.2 – Changed the Method Blank acceptance criteria from 20x to 10x. Section 13 – Updated SOP references.	08Dec2008
S-GB-M-014-Rev.00	Updated Signature Page Renumbered document following SOP: S-ALL-Q-003 *Document Numbering* Table 3.1: Updated Pb RL to 7.5 Section 9.2.1 and 10.2: Added that calibration standards can be used until manufacturer's expiration date Section 11: Changed amount of HNO3 to 5mL from 3mL during digestion.	28Apr2010
S-GB-M-014-Rev.01	Added TCLP Criteria to body of document Corrected Formatting throughout document. Corrected Header information to 6010C from 6010B. Table 3.1: Updated Reporting Limits to match current. Table 3.1, Section 11.6.1.3: Changed unit for Wipe to Total ug. Section 7.1: Added TCLP/SPLP/ASTM Preservation Requirement and Holding Times. Section 7.2: Preservation Check: Changed maximum volume of acid to add as 2% from 5%. Table 9.3.6: Updated Vendor and Catalog numbers. Section 11.6.4: Updated reference to ADAMS From MARRs.	28Jun2011

Document Number	Reason for Change	Date
S-GB-M-014-Rev.02	Table 3.1: Updated TCLP/SPLP/ASTM PRLs. Section 9 Tables: Updated formatting to merged cells when the same standard, acids, final volume, etc. are used for one complete standard. Section 9: Updated expiration dates of standards to 6 month or manufacturer expiration date, whichever is sooner. Added Table 9.3.5 CRDL Intermediate Standard. Section 13.4: Changed requirement for IDL Studies to quarterly from yearly. Section 13.6.4 and 13.6.5: Added method modifications. Throughout document: Updated instances of Reagent Water to Nano-Pure Water, updated RLVS to CRDL standard	30Sep2011
S-GB-M-014-Rev.03	Section 4.2: Updated language on physical interferences. Section 4.5: Updated high salt interferences language. Table 9.3.2: Updated volume of standards used. Table .9.3.5: Updated K concentration in stock and working std. Table 9.3.6: Updated Ag concentration in stock and working std. Section 11.2.4: Updated digestion procedures	07Dec2012

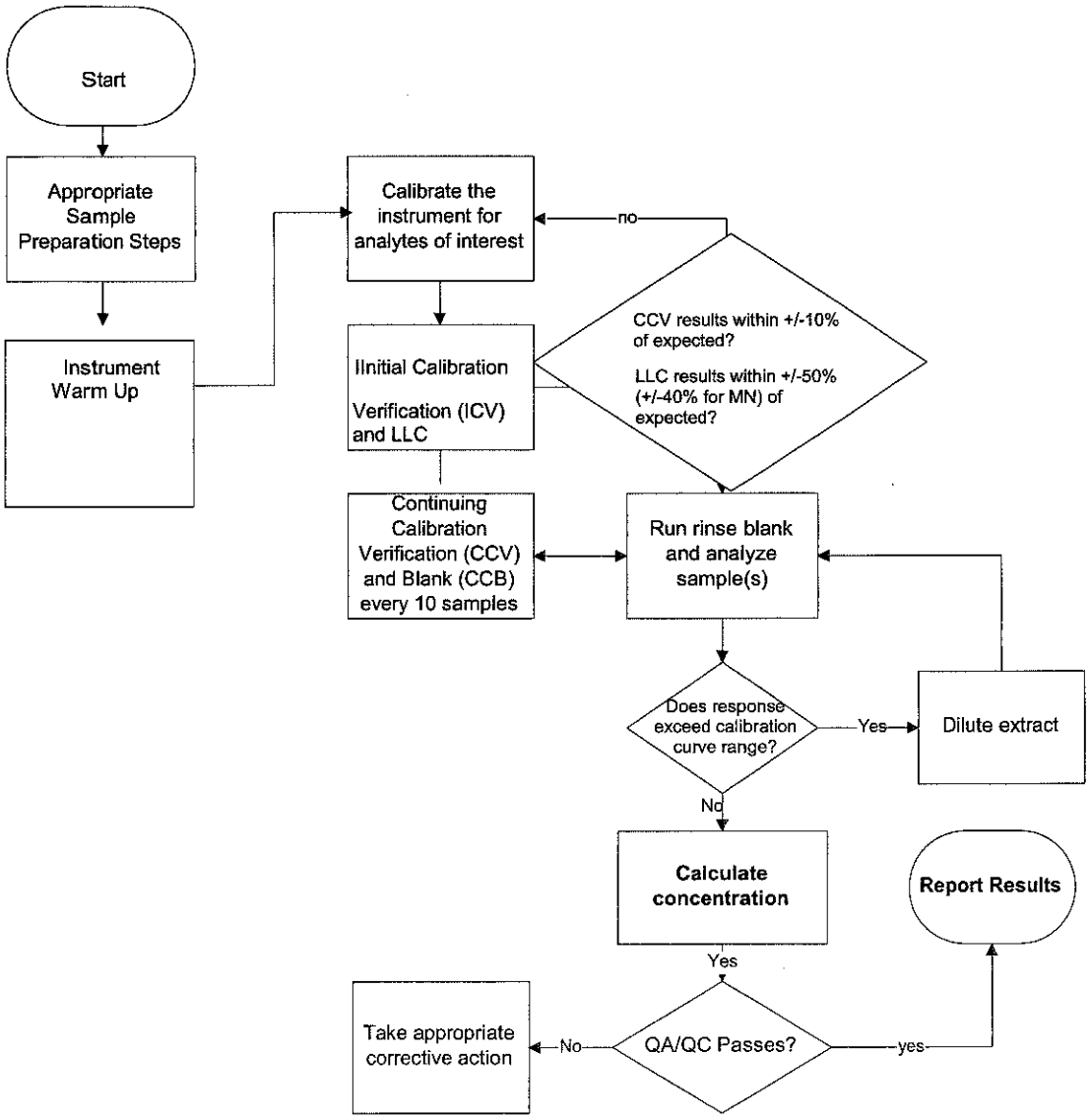
Attachment I

GENERAL ICP TROUBLESHOOTING GUIDE

Problem	Solution
Poor linearity in a dilution series of standards.	This may be as easy as not having spiked your standards correctly. Before you remake your standards, first examine the sample introduction area, making sure you're getting a consistent flow of sample through your pump tubing and that your connectors are not too tight. Then check your nebulizer and injector for visible build up. If build up is present, clean in a dilute nitric acid (HNO_3) solution for about 10-15 minutes. Also examine your torch, making sure it is also reasonably clean. Lastly, check the argon tank to determine that it is not running on empty. If everything is satisfactory, remake standards. If problems persist, try a second source of standards to determine if your initial set of standards has gone bad.
Bubbles are collecting in the back area of the nebulizer (glass type).	The reason why bubbles form in this back area is because the pump tubing is not inserted deeply enough into the cavity of the nebulizer. Cut the tip of the pump tubing to be inserted into the nebulizer at a roughly 45° angle, preferably with a razor blade to obtain a nice, clean cut. Dip the cut end into a wetting agent such as dilute Triton X solution, and start to insert into the nebulizer. Using moderate pressure, begin to work the pump tubing into the nebulizer while twisting the tubing. The twisting action causes the tubing to gradually decrease in diameter, making it easier to insert deeply into the cavity.
Hard instrument failure (software related). These are generally problems associated with the software communication with the instrument.	Exit out of the program. Shut down the PC that is linked to the instrument itself and restart. During the PC reboot process, locate the reset button(s) on the front and/or back of the instrument and depress them. Enter back into the program and continue your analysis. This generally will fix this type of problem, but if you encounter additional problems of this type, then a service call may be necessary for more advanced troubleshooting advice.
Peristaltic pump occasionally stutters or stops. This problem may be hard to notice since this is a gradual wearing of the belt that attaches to the pump motor and turns the rollers themselves.	Replace the belt if you have an available spare. If you do not have a replacement, a service call needs to be made. A service person will generally come out and install the belt but on occasion, based on your mechanical abilities, they may ship the part to you and have you perform the installation.

Attachment II

ICP Flowchart



Pace Analytical™

STANDARD OPERATING PROCEDURE

CATION EXCAHNGE CAPACITY OF SOILS

Reference Methods: SW-846, Method 9081

Local SOP Number:	S-KS-M-007-rev.5
Effective Date:	Date of Final Signature
Supersedes:	S-KS-M-007-rev.4

Approvals

Laboratory General Manager	Date: 6/14/13
Laboratory Quality Manager	Date: 6/13/13
Department Manager	Date: 6/13/13

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION PAGE

1. Purpose/Identification of Method 3
2. Summary of Method 3
3. Scope and Application 3
4. Applicable Matrices 3
5. Limits of Detection and Quantitation 3
6. Interferences 3
7. Sample Collection, Preservation, Shipment and Storage 3
8. Definitions 3
9. Equipment and Supplies 4
10. Reagents and Standards 4
11. Calibration and Standardization 4
12. Procedure 4
13. Quality Control 5
14. Data Analysis and Calculations 6
15. Data Assessment and Acceptance Criteria for Quality Control Measures 6
16. Corrective Actions for Out-of-Control Data 6
17. Contingencies for Handling Out-of-Control or Unacceptable Data 6
18. Method Performance 6
19. Method Modifications 6
20. Safety 6
21. Waste Management 7
22. Pollution Prevention 7
23. References 7
24. Tables, Diagrams, Flowcharts, and Validation Data 7
25. Revisions 8

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1. Purpose/Identification of Method

1.1. The purpose of this method is to provide a laboratory-specific procedure meeting the requirements in Method 9081 for the determination of the cation exchange capacity of soil samples.

2. Summary of Method

2.1. A soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the absorbed sodium with ammonium. The concentration of the displaced sodium is then determined by emission spectroscopy (ICP).

3. Scope and Application

3.1. Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. Parameters: This SOP measures the degree to which a soil can adsorb and exchange cations.

4. Applicable Matrices

4.1. This method is applicable to most soils, including calcareous and noncalcareous soils.

5. Limits of Detection and Quantitation

5.1. This method's reporting limit (LOQ) for cation exchange capacity (CEC) is 0.1 meq/100g. MDLs (LODs) are not applicable to this procedure.

6. Interferences

6.1. Method interferences may be caused by contamination in reagents, glassware, and other sample processing hardware. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

7. Sample Collection, Preservation, Shipment and Storage

Table 7.1 Sample Collection, Preservation, and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Soil	Glass jar, 4-ounce.	N/A	N/A	180 days

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

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9. Equipment and Supplies

Table 9.1 Equipment and Supplies

Supply	Vendor	Model / Version	Comments
Analytical balance	Mettler-Toledo	PB3002-S	or equivalent
Centrifuge	International Equipment Company	Clinical	rotor with 50-mL slots
Centrifuge tubes	Fisher	06-443-19	50-mL
Filter paper	Fisher	09-850-D	Whatman Grade 41, 12.5 cm dia.
Oblong bottles	Fisher	02-911-953	125-mL, HDPE
Shaker table	Eberbach	6010	reciprocating shaker
Volumetric flasks	Fisher	100-mL, 1- , 2-L	Class A

10. Reagents and Standards

Table 10.1 Reagents and Standards

Reagent/Standard	Concentration/ Description	Vendor/ Item #
Acetic acid, glacial	ACS Reagent Grade	Fisher / A38
Ammonium hydroxide	~14.8N, ACS Reagent Grade	Fisher / A669
Glass beads	KIMAX KG-33 glass	Environmental Express / GL13500
Isopropanol	HPLC Grade	Fisher / A451
Reagent water	ASTM Type II	SOP S-KS-Q-011 (latest revision)
Reference Soil	North American Proficiency Testing Program	Soil Science Society of America
Sodium acetate trihydrate	Crystalline, ACS Reagent Grade	Fisher / S209
Sodium hydroxide pellets	ACS Reagent Grade	Fisher / S318

10.1. Sodium acetate solution, 1.0N: Dissolve 136 g of sodium acetate in reagent water and dilute it to 1 liter. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the pH of the solution to 8.2.

10.2. Ammonium acetate solution, 1.0N: In a 2 liter vessel, dilute 114 mL of glacial acetic acid with reagent water to a volume of approximately 1 liter. Add 138 mL of concentrated ammonium hydroxide and add reagent water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution. Add more NH_4OH, as needed, to obtain a pH of 7. Dilute the solution to a volume of 2 liters with reagent water.

11. Calibration and Standardization

11.1. Calibrate the analytical balance as per SOP S-KS-Q-036, *Support Equipment* each day of use.

12. Procedure

12.1. Method Blank (MB): Weigh five grams of glass beads into a labeled, 50-mL centrifuge tube.

12.2. Laboratory Control Sample (LCS): Weigh five grams of Reference Soil into a labeled, 50-mL centrifuge tube.

12.3. Client Samples and Sample Duplicate: Weigh 5 grams of soil and transfer to the appropriately labeled, 50-mL centrifuge tube.

12.4. Add 33 mL of sodium acetate solution to each tube, cap. Place on a mechanical shaker for 5 minutes. Centrifuge the solution until the supernatant liquid is clear.

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12.5. Decant and dispose of the liquid. Loosen compacted soil from bottom of centrifuge tube.

12.6. Repeat the process of adding solution, shaking and centrifuging two more times.

12.7. Add 33 mL of isopropanol to each tube, cap. Place on a mechanical shaker for 5 minutes. Centrifuge the solution until the supernatant liquid is clear.

12.8. Decant and dispose the liquid. Loosen compacted soil from bottom of centrifuge tube.

12.9. Repeat the process of adding isopropanol, shaking and centrifuging two more times.

12.10. Add 33 mL of ammonium acetate solution and screw the cap on the tube. Place on a mechanical shaker for 5 minutes. Centrifuge the solution until the supernatant liquid is clear.

12.11. Filter the decanted washing into a 100-mL volumetric flask. Loosen compacted soil from bottom of centrifuge tube.

12.12. Repeat the process of adding solution, shaking and centrifuging two more times.

12.13. Dilute the combined washings to the 100-mL mark with the ammonium acetate solution and transfer to labeled, 125-mL HDPE oblong bottles.

12.14. Analyze washings for sodium by SOP S-KS-M-005, *Inductively Coupled Plasma Atomic Emission Spectroscopy*.

13. Quality Control

Table 13.1 Batch Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Glass beads	One per batch of up to 20 samples	Target analytes must be less than the reporting limit.	1) Re-analyze blank to confirm failure. (Repeat only once) 2) Qualify results and / or reprep associated samples. ***Exceptions:*** 1) If sample ND, report sample without qualification 2) If sample result >10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier indicating blank contamination. 3) If sample result <10x MB detects, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	Reference Soil	One per batch of up to 20 samples	60-140%	1) Analyze another LCS to confirm failure (Repeat only once) 2) If failure confirms, qualify results and / or reprep associated samples. 3) If failure confirms, perform system maintenance and/or recalibrate system ***Exceptions:*** 1) If LCS rec > QC limits and these compounds are non-detect in the associated samples, the sample data may be reported with appropriate data qualifiers.
Duplicate	Sample Dup	One per batch of up to 20 samples	Max RPD: 30%	1) Report results with an appropriate footnote.

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14. Data Analysis and Calculations

$$\text{CEC (meq/100g)} = \frac{([Na])(V)(100)}{(22.9)(W)(1000)}$$

where:

[Na] = Sodium concentration in washings, mg/mL
V = Volume of the washings, mL
W = Weight of sample, g.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See Table 13.1.

16. Corrective Actions for Out-of-Control Data

16.1. See Table 13.1.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. See Table 13.1.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files

18.2. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020, Training Procedures.

18.2.1. Analyze of four replicates of Reference Soil.

18.2.2. If the average recovery meets the matrix-specific LCS criteria and the RSD of the replicates is <20%, system performance is acceptable and analysis of samples may begin. If, however, RSD >20% or average recovery falls outside LCS criteria, system performance is unacceptable. In this event, correct the problem and repeat the test.

19. Method Modifications

19.1. Not applicable.

20. Safety

20.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

20.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and

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safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

20.3. **Mercury**: Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

21. Waste Management

21.1. Procedures for handling waste generated during this analysis are addressed in S-KS-S-002, Waste Handling.

21.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

22. Pollution Prevention

22.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

23. References

23.1. Pace Quality Assurance Manual - most current version.

23.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

23.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

23.4. EPA Test Methods for Evaluating Solid Waste. SW-846, Third Edition, 9/1986, Method 9081.

24. Tables, Diagrams, Flowcharts, and Validation Data

24.1. Not applicable.

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25. Revisions

Document Number	Reason for Change	Date
KS-I-4340-A	New	July 11, 2001
KS-I-4340-B	Grammatical. Removal of outdated information	June 3, 2004
KS-M-007-rev.2	Grammatical. Removal of outdated information	September 12, 2006
S-KS-M-007-rev.3	Overall conversion to template format. Changes required based on conversion are not explicitly noted unless change represents a significant policy change. Section 10 – Added reference sample. Section 13 – Added DOC procedure and control limit generation.	May 8, 2008
S-KS-M-007-rev.4	Section 7 – Revised Dir QST and Distribution. Table 9.1 – Revised instrument. Section 12 – Use boiling stones for blank. Section 15 – Changed Waste SOP reference. Section 16 – Revised references.	January 31, 2011
S-KS-M-007-rev.5	SOP – Updated to latest prescribed format. Table 7.1 – Changed storage temperature. Table 10.1 – Replaced boiling stones with glass beads. Section 12 – Replaced boiling stones with glass beads. Added "loosen soil after decanting". Table 13.1 – Revised acceptance criteria and corrective actions	June 13, 2013

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