FINAL

SERES Engineering & Services, LLC
ARCADIS
a joint venture

Final
Barrier Wall Performance Monitoring Work Plan

Shepley's Hill Landfill
Former Fort Devens Army Installation
Devens, Massachusetts

Contract No. W912WJ-19-D-0014

Contract Delivery Order No. W912WJ-20-F-0022

May 9, 2022

FINAL

Barrier Wall Performance Monitoring Work Plan

Shepley's Hill Landfill
Former Fort Devens Army Installation
Devens, Massachusetts

May 9, 2022

Prepared By:
SERES-Arcadis 8(a) JV 2, LLC
669 Marina Drive, Suite B-7
Charleston, South Carolina 29492
Tel 843 216 8531

Prepared For:
U.S. Army Corps of Engineers
New England District

CERTIFICATION

I hereby certify that the enclosed Work Plan, shown and marked in this submittal, is that proposed to be incorporated with Contract Number W912WJ-19-D-0014. This document was prepared in accordance with the U.S. Army Corps of Engineers Scope of Work and is hereby submitted for Government approval.

Reviewed By:

Andy Vitolins, PG
Project Manager

Heather Levesque

Heather Levesque, PMP
Deputy Project Manager

Received By:

REDDY.PENELOPE.W.1502105066
Digitally signed by REDDY.PENELOPE.W.1502105066
Date: 2022.05.10 07:55:29 -04'00'

Penelope Reddy
U.S. Army Corps of Engineers Project Manager

Contents

Acronyms and Abbreviations iv

1 Introduction 1

- **1.1 Site Background** 1
- **1.2 Site Geology/Hydrogeology** 2
- **1.3 Site Geochemistry and Arsenic Distribution** 4
- **1.4 Document Organization** 5

2 Data Quality Objectives 7

- **2.1 Step 1 – State the Problem** 7
- **2.2 Step 2 – Identify the Decision (Project Goals)** 7
- **2.3 Step 3 – Identify Inputs to the Decision** 8
- **2.4 Step 4 – Define the Study Boundaries** 10
  - **2.4.1 Groundwater Upgradient of the Barrier Wall** 10
  - **2.4.2 Groundwater and Soil Between the Barrier Wall and Red Cove** 10
  - **2.4.3 Aquatic Environment of the Red Cove Area of Plow Shop Pond** 11
  - **2.4.4 Aquatic Environment of Plow Shop Pond Outside of Red Cove** 11
- **2.5 Step 5 – Develop a Decision Rule** 11
- **2.6 Step 6 – Specify Limits on Decision Errors** 12
- **2.7 Step 7 – Optimize the Design for Obtaining Data** 13
  - **2.7.1 Field Approach** 13
  - **2.7.2 Analytical Approach** 14

3 Detailed Plan for Obtaining Data 16

- **3.1 Field Measurements** 16
  - **3.1.1 Aquatic Location Field Measurements** 16
  - **3.1.2 Groundwater Location Field Measurements** 16
  - **3.1.3 Surface Water Flow** 17
  - **3.1.4 Aquatic Hydraulic Head Measurement** 17
- **3.2 Surface Water Sampling** 18
  - **3.2.1 Surface Water Sample Collection** 18
  - **3.2.2 Surface Water Sample Analysis** 18
- **3.3 Soil Sampling** 19
  - **3.3.1 Soil Sample Collection** 20

3.3.2 Soil Sample Analysis 20
3.4 Sediment Sampling 21
3.4.1 Sediment Sample Collection 21
3.4.2 Sediment Sample Analysis 21
3.5 Porewater Sampling 23
3.5.1 Porewater Sample Collection 23
3.5.2 Porewater Sample Analysis 24
3.6 Groundwater Sampling 25
3.6.1 Groundwater Sample Collection 25
3.6.2 Groundwater Sample Analysis 25
4 Barrier Wall Performance Monitoring Report 27
4.1 Summary of Field Activities and Data Reporting 27
4.2 Data Use Objective Goal 1: Description of Current Conditions 27
4.3 Data Use Objective Goal 2: Assessment of Arsenic Flux 27
4.4 Data Use Objective Goal 3: Assessment of Arsenic Sources in Plow Shop Pond 29
4.5 Data Use Objective Goal 4: Long-Term Monitoring Recommendations 30
5 Schedule 31
6 References 32

Tables

Table 1 Project Goals and Data Use Objectives (in text)
Table 2 Summary of Decision Inputs (in text)
Table 3 Summary of Proposed Barrier Wall Performance Monitoring (attached)
Table 4 Summary of Decision Rules (in text)
Table 5 Summary of Decision Errors (in text)
Table 6 Surface Water Sample Summary and Proposed Analytical Methods (in text)
Table 7 Soil Sample Summary and Proposed Analytical Methods (in text)
Table 8 Sediment Sample Summary and Proposed Analytical Methods (in text)
Table 9 Porewater Sample Summary and Proposed Analytical Methods (in text)
Table 10 Groundwater Sample Summary and Proposed Analytical Methods (in text)
Table 11 Anticipated Project Schedule (in text)

Figures

Figure 1 **Site Location**

Figure 2 **Generalized Conceptual Site Model Cross-Section AA’ (in text)**

Figure 3 **Proposed Sediment, Surface Water, Porewater, and Groundwater Locations**

Appendix

Appendix A. **Quality Assurance Project Plan Addendum**

Appendix B. **Comments and Responses**

Acronyms and Abbreviations

ASTM	ASTM International
bss	below sediment surface
CSM	conceptual site model
DO	dissolved oxygen
DOC	dissolved organic carbon
DQO	data quality objective
DUO	data use objective
g/year	gram per year
g/year/m^2	gram per year per square meter
KGS	KOMAN Government Solutions, LLC
LTMMP	long-term monitoring and maintenance plan
MassDEP	Massachusetts Department of Environmental Protection
mg/kg	milligram per kilogram
Monitoring Report	Barrier Wall Performance Monitoring Report
MS	matrix spike
MSD	matrix spike duplicate
NTCRA	non-time-critical removal action
ORD	Office of Research and Development
ORP	oxidation-reduction potential
PRG	preliminary remediation goal
PSP	Plow Shop Pond
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
SEP	sequential extraction procedure
SERES-Arcadis JV	SERES-Arcadis 8(a) Joint Venture 2, LLC
SHL	Shepley’s Hill Landfill
Site	former Fort Devens Army Installation in Devens, Massachusetts
SWI	sediment-water interface
TOC	total organic carbon

TSS	total suspended solids
USEPA	United States Environmental Protection Agency
USGS	United States Geological Survey
Work Plan	Barrier Wall Performance Monitoring Work Plan

1 Introduction

On behalf of the U.S. Army Corps of Engineers, SERES-Arcadis 8(a) Joint Venture 2, LLC (SERES-Arcadis JV) prepared this Barrier Wall Performance Monitoring Work Plan (Work Plan) for the Shepley's Hill Landfill (SHL), located within the former Fort Devens Army Installation in Devens, Massachusetts (Site; Figure 1). SERES-Arcadis JV prepared this Work Plan under Contract No. W912WJ-19-D-0014.

In 2012 and 2013, non-time-critical removal actions (NTCRAs) were conducted to install an 850-foot-long hydraulic barrier wall along the eastern boundary of SHL, between the SHL and Plow Shop Pond (PSP), and subsequently excavate contaminated Red Cove sediments from PSP (United States Environmental Protection Agency [USEPA] 2020b). The objectives of the NTCRAs were to address confirmed impacts and minimize continued landfill discharge to PSP, and remove the accumulation of arsenic in Red Cove sediments. As confirmed in the Record of Decision for Plow Shop Pond (AOC 72) – Red Cove and Former Railroad Round House (SA-71), installation of the groundwater barrier wall between SHL and Red Cove along with sediment removal actions successfully mitigated potential risks associated with PSP sediments (Sovereign 2015a).

On June 22 and December 7, 2020, the Massachusetts Department of Environmental Protection (MassDEP) and the USEPA, respectively, provided comments on the Barrier Wall Analysis provided in Appendix G of the Draft 2019 Annual Long-Term Monitoring and Maintenance Report for the SHL (KOMAN Government Solutions, LLC [KGS] 2020a). The comments stated that the evaluations of barrier wall performance were insufficient because they were based on estimates of arsenic flux and indicated that the barrier wall performance evaluation will need to be based on direct measurements of porewater and other media. In addition, to evaluate barrier wall performance and verify continued attainment of the Record of Decision (U.S. Army Environmental Center 1995) remedial action objective for PSP, the USEPA recommended the performance of a minimum of two rounds of media sampling (sediment and porewater) and toxicity testing prior to the 2025 five-year review. Accordingly, the U.S. Army issued a Scope of Work in February 2021 to SERES-Arcadis JV to develop a plan to conduct the appropriate analyses to evaluate arsenic flux through the barrier wall and to PSP (USACE. 2021). The results from this work will then be incorporated into the revised long-term monitoring and maintenance plan (LTMMP) for SHL along with any other changes required as part of the Phase I and Phase II work or monitoring program. This Work Plan is being submitted in accordance with the Scope of Work and describes the sampling activities to be completed to collect the additional data requested by the USEPA and MassDEP. This Work Plan satisfies the USEPA's request for a draft work plan for porewater and sediment sampling in the Red Cove area of PSP, as included in the 2020 five-year review report (KGS. 2020b). Although sediment toxicity testing was recommended, collection of these data is not needed to assess the effectiveness of the barrier wall installation for the reduction of arsenic flux. Therefore, collection and testing of sediment for benthic toxicity are not included in this Work Plan.

1.1 Site Background

The U.S. Army operated SHL for more than 70 years (at least as early as 1917 until 1992; KGS 2020b). Known waste within SHL includes demolition debris, asbestos, sanitary wastes, glass, and incinerator ash. Contaminants of concern include arsenic and volatile organic compounds. Since closure of SHL in the 1990s, several remedial efforts have been implemented to address arsenic impact, migration, and exposure at the SHL (KGS 2020a), as listed below:

- Landfill capping
- Groundwater monitoring
- Installation and operation of a groundwater extraction and treatment system
- Barrier wall installation between SHL and the Red Cove area of PSP
- Excavation of contaminated Red Cove sediments following the Barrier Wall installation
- Land use controls in SHL and the North Impact Area.

As pertains to this Work Plan and listed in the fourth bullet (above), in 2012, a low permeable barrier wall was installed along the eastern limit of the landfill and to the west of the Red Cove area of PSP. The barrier wall extends from the ground surface through native sandy glacial deposits and glacial till, and to the bedrock surface. The boundaries and length of the barrier wall were based on the identified areas of impacted sediment in Red Cove, groundwater concentrations along the eastern edge of the SHL, and particle track analysis as predicted by the SHL groundwater model. The barrier wall was designed to intercept and divert groundwater flowing in the overburden soils away from Red Cove. It consists of an 850-foot long soil-bentonite barrier measuring approximately 2.5-feet wide, with an effective hydraulic conductivity of 1 x 10-7 centimeters per second.

1.2 Site Geology/Hydrogeology

SHL is located within a bedrock valley, and the overburden deposits beneath SHL consist of glacially deposited, well-graded to poorly graded sands with silts and gravel. A discontinuous layer of till is present at the base of the sands directly overlying bedrock. The hydrostratigraphic unit within the overburden comprises primarily medium and fine to medium sands with little variability.

In general, groundwater in the southern portion of SHL flows to the northeast (prior to installation of the barrier wall, toward PSP), groundwater in the northern portion of SHL flows to the north, and groundwater to the northeast of SHL flows to the northwest. Groundwater in the deep overburden at the northern extent of SHL (in the Nearfield Area) is extracted as part of the arsenic treatment plant operations. The depth to groundwater in the barrier wall area in 2020 ranged from approximately 19 to 28 feet below ground surface.

Prior to installation of the barrier wall, groundwater in the area flowed to the northeast toward Red Cove and PSP. Arsenic flux to Red Cove prior to the installation of the barrier wall was documented in several reports, notably the Arsenic Fate, Transport and Stability Study (USEPA 2008), Remedial Investigation for AOC 72, Plow Shop Pond – Draft (AMEC 2010), and various USEPA presentations.

Key Hydrogeology Takeaways

- Following installation of the barrier wall, groundwater flow toward Red Cove area and PSP was reduced
- The pre-barrier wall arsenic flux to Red Cove for 2011 was estimated as 39.4 grams per year per square meter (g/year/m^2). Following installation of the barrier wall, arsenic flux to Red Cove was estimated as 1.8 to 9.1 g/year/m^2.
- In 2020, post-barrier wall arsenic flux to Red Cove was estimated as 1.3 g/year/m^2.

Following installation of the barrier wall, groundwater flow toward Red Cove and PSP was reduced. As presented in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a), groundwater elevation contours in the barrier wall area showed some mounding of groundwater upgradient of the wall (to the west) compared to the area between the wall and Red Cove (to the east). This is expected based on the low hydraulic conductivity of materials used for construction of the barrier wall. The groundwater elevation contours also show groundwater moving to the north along the upgradient (western) side of the barrier wall, which effectively reduces the amount of groundwater flow from SHL to PSP. The Long-Term Monitoring and Maintenance Plan Update (Sovereign 2015b) and subsequent Addendum to the Long-Term Monitoring and Maintenance Plan (KGS 2018) include the monitoring required to evaluate the hydraulic performance of the barrier wall.

Mass flux of arsenic has, in the past, been estimated over segments of the surficial aquifer between SHL and PSP as well as mass flux via groundwater discharge to Red Cove within PSP:

- **Arsenic flux across the Barrier Wall**: Mass flux of arsenic across the barrier wall and to Red Cove has, pre- and post-barrier installation, been estimated over segments of the surficial aquifer by calculating groundwater flow across the barrier wall and average upgradient dissolved arsenic concentrations, as presented in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a). Mass discharge of arsenic in the overburden across the barrier wall was calculated from Darcy velocity using 3PE vector analysis results and 2020 dissolved arsenic concentration data. This method conservatively assumed that groundwater flows from west to east across the wall, rather than to the northwest, as the 3PE analysis indicates. Based on this analysis, arsenic flux (q*C) across the wall taking into consideration the full cross-sectional areas of flow in 2020 ranged from 0.6 gram per year (g/year) in fall to 2.4 g/year in spring. This flux is approximately 3 orders of magnitude less than the estimated pre-wall flux calculated at 686 g/year across the same aquifer cross section (Appendix E in SERES-Arcadis JV 2021a).
- **Arsenic flux to Red Cove:** Mass discharge of arsenic to Red Cove was previously calculated by the USEPA Office of Research and Development (ORD) for pre- and post-barrier wall conditions (Ford et al. 2018). The pre-barrier wall arsenic flux in 2011 was 39.4 g/year/m^2. Following installation of the barrier wall, arsenic flux was estimated as 1.8 to 9.1 g/year/m^2. Using the same methodology, post-barrier wall arsenic flux to Red Cove was estimated in 2020 as 1.3 g/year/m^2 (SERES-Arcadis JV 2021a). The 2020 flux estimate is consistent with the previous post-barrier wall flux estimated by the ORD.

The 3PE analysis used to assess barrier wall performance in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a) indicated that the barrier wall is effectively reducing groundwater flow from SHL to Red Cove and PSP.

PSP is the furthest downstream pond in a series of six ponds that discharge to Nonacoicus Brook. While the majority of water in PSP originates from the upstream ponds, there is local contribution from groundwater seepage. As presented in the Remedial Investigation for AOC 72, Plow Shop Pond – Draft (AMEC 2010), low flow, measured as the 7-day, 10-year low flow at the outlet of PSP, was estimated as 2.6 cubic feet per second. Bathymetry and sediment thickness were measured in 1999 by the U.S. Geological Survey (Mercadante et al. 1999). While these measurements predate excavation in the Red Cove area of PSP, they provide good general information on depths and sediment thickness. At the time of the survey, the deepest part of PSP was 8 feet, near the center, and the greatest sediment thickness was 18 feet along the western side of the pond, east of Red Cove. Within Red Cove, pre-excavation water depths were less than 2.5 feet and sediment thickness was less than 8 feet.

1.3 Site Geochemistry and Arsenic Distribution

Sources of dissolved arsenic in groundwater at SHL include anthropogenic (landfill waste) and geogenic materials. Groundwater within the SHL footprint and immediately downgradient contain high concentrations of dissolved metals consistent with what would be expected below a landfill with an impermeable cap. Specifically, water quality is indicative of active reductive dissolution of iron and manganese, resulting in the dissolution of co-occurring arsenic. At landfill sites, reductive dissolution of iron minerals is common, as is the concomitant dissolution of arsenic that is co-precipitated with the iron minerals (USGS 2004).

Key Geochemistry Takeaways

- No sediment or porewater data were collected after the Red Cove sediment removal action.
- Arsenic can cycle between the sediment bed and PSP.

Groundwater at SHL generally has circumneutral pH and relatively low concentrations of anions, such as phosphate, that can compete with arsenic for sorption to precipitated iron minerals (such as iron oxyhydroxides). Therefore, mobilization, fate, and transport of arsenic in site groundwater are primarily dependent on oxidation-reduction conditions (SERES-Arcadis JV 2021b). Precipitation of iron oxyhydroxides and the co-precipitation of arsenic is expected to occur where the water (groundwater or surface water) transitions from reduced to oxic conditions. These conditions are commonly present in the hyporheic zone at the groundwater-surface water interface. Additionally, precipitation of iron oxyhydroxides (and co-precipitation of arsenic) can be biologically mediated by iron-oxidizing bacteria that occur both in groundwater and surface water.

A generalized Conceptual Site Model (CSM) cross-section extended from SHL and across PSP depicting the major hydraulic features controlling the mobility and distribution of arsenic in the soils, sediment, and water is presented below in Figure 2. The location of the CSM cross-section is presented on Figure 1. Groundwater from SHL flows northeast toward the barrier wall, where most is diverted to the north toward the extraction wells. A minor component can seep through the wall which, along with groundwater contribution from the underlying bedrock, results in lateral flow from the area around the barrier wall to PSP. As discussed in Section 1.2, the flux has been greatly reduced after construction of the barrier wall.

Arsenic had previously co-precipitated with iron in the surficial sand aquifer between the barrier wall and PSP. The precipitated iron is principally in the form of amorphous or weakly mineralized oxyhydroxides and well mineralized goethite and hematite. The amorphous oxyhydroxides are not stable compared to the more mineralized forms and will cycle between precipitation and dissolution along with any co-associated arsenic. This cycle of precipitation and dissolution of the oxyhydroxides along with on-going contribution from the underlying bedrock is the dominant source of the dissolved arsenic in groundwater between the barrier wall and PSP.

A portion of the iron and dissolved arsenic reaches the sediment-water interface for PSP. Flow within the Red Cove area in PSP is very restricted, and once in PSP, the iron and co-associated arsenic cycles between the sediment in the hyporheic zone and water column. Factors, including biologically mediated precipitation and dissolution can enhance the cycling. The poor flushing and continued input of iron and arsenic in the groundwater results in an accumulation of the iron oxyhydroxides and associated elevated arsenic in the Red Cove area surface water and sediment.

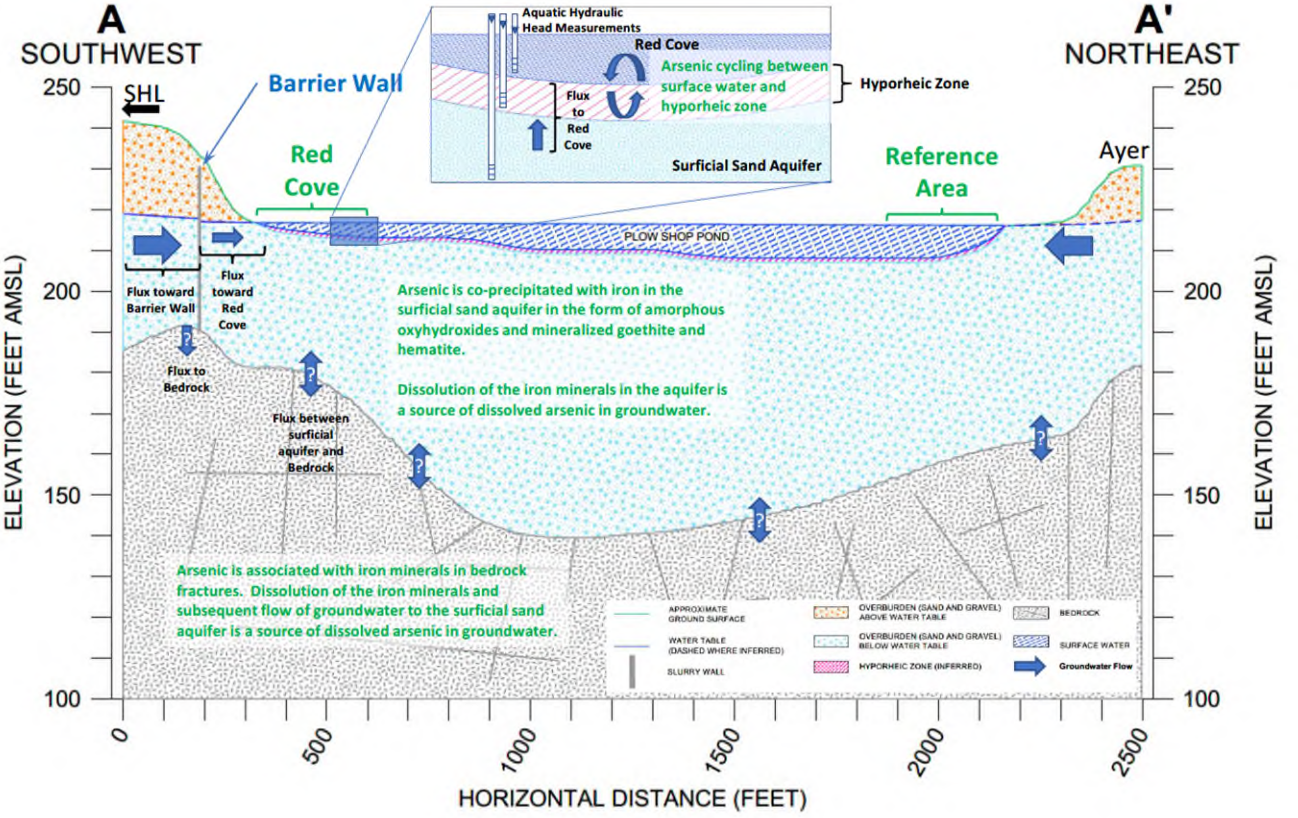


Figure 2: Generalized Conceptual Site Model Cross-Section AA'

Findings from the ORD investigations in 2008 and 2009 provide much of the basis for the current USEPA conceptual site model for arsenic fate, transport, and stability in the Red Cove area of PSP. The ORD investigations were expanded in 2010, as discussed in the Remedial Investigation for AOC 72, Plow Shop Pond – Draft (AMEC 2010). These investigations were completed prior to sediment excavation in the Red Cove area and construction of the barrier wall that provides hydraulic separation between SHL and PSP. The methods used and results from these investigations have served as a guide in development of this Work Plan to allow for direct comparison of the performance data to be collected under this work plan with historical data. Where appropriate, the methods used, such as high-resolution core sampling and push-probe porewater sampling, will be implemented as part of the Work Plan to provide continuity with previous results. Where necessary, additional methods, target locations, and analytes will be incorporated to address data gaps identified in the previous investigations.

1.4 Document Organization

This Work Plan was developed using the seven-step data quality objectives (DQOs) process outlined in the Data Quality Objectives Process for Hazardous Waste Site Investigations, EPA QA/G-4HW (USEPA 2000) The seven steps are presented in Section 2 and the detailed plan for the collection and analysis of data is presented in

Section 3. Discussion of the evaluation and reporting of data is presented in Section 4. Section 5 contains the project schedule and Section 6 list the references used in the Work Plan. Appendix A contains the Uniform Federal Policy - Quality Assurance Project Plan (QAPP) Addendum (QAPP Addendum), which provides details on the sampling and analysis procedures for implementation of the Work Plan along with the chemical DQOs, field data-gathering methods, analytical methods and measurements, quality assurance/quality control (QA/QC) protocols necessary to achieve the DQOs, and data assessment procedures for the evaluation and the identification of any data limitations. The QAPP Addendum will facilitate the generation of data with acceptable precision, accuracy, representativeness, comparability, and completeness.

2 Data Quality Objectives

2.1 Step 1 – State the Problem

Problem Summary

- Arsenic has been detected in surface water at concentrations greater than criteria.
- Additional data are needed to determine current conditions and arsenic flux to the Red Cove area of PSP.

A barrier wall was installed in SHL to reduce arsenic flux to the Red Cove area of PSP through groundwater. Monitoring before and after wall installation demonstrates that arsenic flux has been reduced, but the estimated reduction is based on indirect measurements of arsenic flux from measured arsenic concentration data in groundwater and calculated groundwater flow. Assessments of barrier wall effectiveness based on historical geochemical characterization have been inconclusive for a variety of reasons including:

- The reduced groundwater velocities between the barrier wall and Red Cove have significantly reduced the rate of porewater flushing, and residual groundwater that derived from SHL prior to the barrier wall construction may still be present between the barrier wall and PSP.
- Downgradient of the barrier wall, iron and co-occurring arsenic that precipitated in the aquifer is still present as amorphous ferric oxyhydroxides and more mineralized forms (goethite and hematite). Rate of dissolution of the oxyhydroxides and the co-occurring arsenic will depend on 1) rate of change in the groundwater geochemical composition (major ions, pH, and oxidation-reduction potential [ORP]); 2) groundwater flow rates; and 3) the mineral form of the iron precipitates.
- Biogeochemical cycling of arsenic in PSP between the sediment and sediment porewater and suspended iron flocculates in the water column. Inadequate water circulation and flushing in Red Cove and the influence of natural organic matter from algae blooms and die-off and other potential sources such as iron oxidizing bacteria, if present would be likely causes of this biogeochemical arsenic cycling.

Additionally, arsenic concentrations in surface water collected after installation of the barrier wall and the excavation of sediments from Red Cove have exceeded the chronic Ambient Water Quality Criteria of 150 ug/L (USEPA 2002). As described above, at the request of the USEPA and MassDEP, direct measurements of porewater, sediment, and other matrices are needed to support the estimates of flux from SHL to the Red Cove area of PSP.

2.2 Step 2 – Identify the Decision (Project Goals)

In accordance with the USEPA (2020b), this Work Plan includes the collection of porewater and sediment samples to evaluate the effectiveness of the barrier wall at reducing arsenic flux to PSP. Additionally, this Work Plan includes the collection of groundwater, surface water, and sediment geotechnical data to assist in the evaluation. As previously mentioned, although sediment toxicity testing was recommended, collection of these data is not needed to assess the effectiveness of the barrier wall installation for the reduction of arsenic flux. Therefore, collection and testing of sediment for benthic toxicity are not included in this Work Plan.

As discussed in Section 1.2, previous study and analysis has been conducted to define the hydraulic conditions at and near the barrier wall. As such, this study will focus more on the cycle and movement of arsenic in the system than on the movement of groundwater upgradient of PSP. Existing evaluations of groundwater and arsenic flux will be used during the interpretation of data collected as part of this Work Plan. The previous groundwater flux estimates will be augmented with flux measurements collected across the hyporheic zone in Red Cove.

The main goals of this Work Plan are presented in Table 1, below.

Table 1 Project Goals and Data Use Objectives

Goal / DUO #	Goal	Decision Question	DUO
1	Define current conditions in Red Cove	Is the arsenic concentration in surface sediment greater than 270 mg/kg (PRG from NTCRA)?	Document the current levels of arsenic in Red Cove sediment, porewater, and surface water.
2	Quantify arsenic flux from SHL to the Red Cove area of PSP	Is there arsenic flux from SHL to PSP and if so, how much?	Develop a flux estimate for arsenic from the upgradient area of SHL to the downgradient area and from the area between the barrier wall and Red Cove to the Red Cove area of PSP. (Refer to Figure 1)
3	Identify the ongoing source of arsenic present in PSP	Is arsenic in Red Cove surface water a result of arsenic cycling within the aquatic system (i.e., the hyporheic zone) and if not, what other sources are contributing?	Understand the form, mobility, and stability of iron and co-occurring arsenic in groundwater, porewater, surface water, soil and sediment at SHL and in the Red Cove area of PSP.
4	Develop recommendations to monitor Red Cove for the attainment of remedial action objectives through time, if needed.	Based on the results of DUOs 1 through 3, what locations, media, and sample frequency are needed for long-term monitoring of Red Cove?	Define the geographic extent of groundwater influence from SHL to PSP in the post barrier wall condition to refine the Red Cove monitoring area.

Notes:
DUO = data use objective
mg/kg = milligram per kilogram
PRG = preliminary remediation goal

2.3 Step 3 – Identify Inputs to the Decision

The collection of temporal and spatial synoptic samples in groundwater, porewater, surface water, soil and sediment will define current conditions in Red Cove and allow for a comprehensive understanding of the geochemical cycle of arsenic in the system. These data will also be used to better define the zone of influence of SHL on the Red Cove area of PSP and will be used to compare arsenic concentrations between areas influenced/not influenced by SHL. The target area for the samples, methods that will be used, and information collected will be sufficiently compatible with previous investigations in the Red Cove area to allow for pre- and post-wall installation comparisons of arsenic and associated constituents. A summary of the decision inputs is presented in Table 2, below.

Table 2. Summary of Decision Inputs

Data Input	**Goal or DUO**				**Data Collection Method**	**Laboratory Analysis/ Field Parameter**
	1	**2**	**3**	**4**		
Aquatic field measurements	X		X	X	Probing rod and multiparameter probe	Water/sediment depth DO, pH conductivity, ORP, turbidity, temperature
Groundwater field measurements		X	X	X	Measuring tape and multiparameter probe	Depth to water DO, pH, conductivity, ORP, turbidity, temperature
Surface water flow measurements	X	X			Probing rod and electromagnetic velocity meter	Width, depth, and velocity
Aquatic hydraulic head measurements	X	X			Paired piezometer	Elevations at water surface, 2 and 10 feet bss
Surface water samples	X	X	X	X	Peristaltic pump or grab sampler	Major cations (includes arsenic/iron) and anions, DOC, TDS, TSS
Soil samples		X	X		Direct Push Rig	Metals (As, Fe, Mg, Mn, Al, Ca), TOC, pH, total sulfur, grain size, and SEP.
Sediment samples	X	X	X	X	Manual cores via Lexan or aluminum tubes with vibracore	Metals and TOC with a subset for: pH, sulfur, grain size, and SEP
Geotechnical sediment samples	X	X			Shelby tube	Hydraulic conductivity
Porewater samples	X	X	X	X	PushPoint™ samplers	Arsenic, iron, TSS with a subset for: major cations and anions, DOC, TDS, and ammonia
Groundwater samples		X	X	X	Peristaltic pump or grab sampler (e.g., bailer)	Major cations (includes arsenic/iron) and anions, DOC, TDS, TSS, and ammonia with a subset for: carbon dioxide gas and iron-oxidizing bacteria

Notes:
bss = below sediment surface

DO = dissolved oxygen
DOC = dissolved organic carbon
ORP = oxidation-reduction potential
SEP = sequential extraction procedure
TOC = total organic carbon
TSS = total suspended solids
Major cations = arsenic, sodium, iron, magnesium, manganese, barium, potassium, aluminum, and calcium (total and dissolved)
Major anions = sulfate, alkalinity, and chloride

2.4 Step 4 – Define the Study Boundaries

To achieve the study goals outlined in Section 2.2, the investigation area has been divided into four distinct areas as shown on Figure 1 and the CSM (Figure 2). These geographies include:

- Groundwater upgradient of the barrier wall
- Groundwater and soil between the barrier wall and Red Cove
- Aquatic environment of the Red Cove area of PSP
- Aquatic environment of PSP outside of Red Cove

These areas are described below. The proposed locations near the barrier wall and Red Cove include 10 groundwater sampling locations (4 existing wells upgradient of the barrier wall and 6 existing wells between the barrier wall and Red Cove), three soil location between the barrier wall and Red Cove, and porewater, sediment, and aquatic sampling locations (20 in red cove). Also, eight reference locations are proposed in the PSP area for aquatic environment monitoring. These are shown on Figure 3 and presented in Table 3 (included at the end of the document).

2.4.1 Groundwater Upgradient of the Barrier Wall

This area represents one potential source area for arsenic detected in the Red Cove area. Four existing groundwater monitoring wells will be sampled in the upgradient area. The sampling locations were selected to provide representative groundwater characteristics between SHL and the barrier wall. Groundwater levels from these wells will be compared to previous measurements to determine if the water levels are within the range expected.

2.4.2 Groundwater and Soil Between the Barrier Wall and Red Cove

This area represents the transitional zone between SHL and PSP. Six existing groundwater monitoring wells will be sampled and three soil borings (adjacent to existing wells) in the area, as shown in Figure 3. The sampling locations were selected to provide representative groundwater and soil characteristics between the barrier wall and PSP. Groundwater levels from these wells will be used to confirm that water levels are within the expected range based on previous measurements.

2.4.3 Aquatic Environment of the Red Cove Area of Plow Shop Pond

This area of concern is the primary receiving area for groundwater from SHL prior to installation of the barrier wall and represents the initial extent for long-term monitoring locations for remedy effectiveness. A total of 20 porewater, sediment, and aquatic sample locations (identified as location RC-01 to RC-20) are targeted for the Red Cove area to provide an adequate number of locations to evaluate spatial distribution in the data and to allow for the area to be subdivided for statistical comparison to the reference locations or other areas within the Red Cove (please see Section 2.4.4 for details on reference locations). In addition to geochemical samples, vertical flow through the hyporheic zone will be measured using clustered piezometers (in clusters of two) installed at 10 locations co-incident with undisturbed samples collected for vertical hydraulic conductivity analysis within Red Cove. The sample locations in Red Cove will be arranged in a grid-like pattern, radiating from the shoreline, and spaced approximately 100 feet apart. These sample locations are identified in Figure 3 and Table 3.

2.4.4 Aquatic Environment of Plow Shop Pond Outside of Red Cove

This area represents a reference or comparison area for aquatic data collected from the Red Cove area. An initial review of the data indicates that within PSP, the northern cove is likely outside the hydraulic influence of SHL. A total of eight porewater, sediment, and aquatic sample locations (identified as PS-01 to PS-08) are established as reference locations in this northern area of PSP. Similar to the Red Cove sample locations, these locations are arranged in a grid-like pattern, spaced approximately 100 feet apart. These locations are identified in Figure 3 and Table 3.

2.5 Step 5 – Develop a Decision Rule

A summary of the decision rules is presented in Table 4, below.

Table 4 Summary of Decision Rules

Goal / DUO #	Goal	Decision Question	Decision Rule and Metric
1	Define current conditions in Red Cove	Is the arsenic concentration in surface sediment greater than 270 mg/kg (PRG from NTCRA)?	If no, no further action is required for arsenic. If yes, additional study/monitoring may be required and would be conducted under the revised LTMMP.
2	Quantify arsenic flux from SHL to the Red Cove area of PSP	Is there arsenic flux from SHL to PSP and if so, how much?	If no, no further action is required. If yes, and a larger than expected flux is encountered or if the results are determined to be inconclusive, additional study/monitoring may be required to quantify the level of flux through/around the barrier wall. Additional study/monitoring would be conducted under the revised LTMMP.

Goal / DUO #	Goal	Decision Question	Decision Rule and Metric
3	Identify the ongoing source of arsenic present in PSP	Is arsenic in Red Cove surface water a result of arsenic cycling within the aquatic system (i.e., the hyporheic zone) and if not, what other sources are contributing?	If no, additional study/monitoring may be required to identify the other sources. If yes, is there evidence that concentrations are stable or decreasing with time? If sufficient evidence for stable or decreasing trends is not present, then additional study/monitoring may be required and would be conducted under the revised LTMMP.
4	Develop recommendations to monitor Red Cove for the attainment of remedial action objectives through time, if needed	Based on results of DUOs 1 through 3, what locations, media, and sample frequency are needed for long-term monitoring of Red Cove?	Locations and frequencies will be proposed to monitor arsenic trends in PSP under the revised LTMMP. These data will be used to answer study questions above or demonstrate stable or decreasing arsenic concentrations in Red Cove over time.

2.6 Step 6 – Specify Limits on Decision Errors

The potential decision errors associated with each decision question and proposed limits for those errors are presented in Table 5 below:

Table 5 Summary of Decision Errors

Goal / DUO #	Decision Question	Potential Decision Error	Limits for error
1	Is the arsenic concentration in surface sediment greater than 270 mg/kg (PRG from NTCRA)?	Concentration used for comparison are biased low.	A multiple line-of-evidence statistical approach will be used to establish representativeness of the samples and potential error in the results.
2	Is there arsenic flux from SHL to PSP and if so, how much?	Estimated arsenic flux in Red Cove cannot be allocated between SHL, cycling in the PSP system or other sources	Collection of robust analytical data from the 4 geographies described in Section 2.4 will allow for appropriate comparisons to be made.

Goal / DUO #	Decision Question	Potential Decision Error	Limits for error
3	Is arsenic in Red Cove surface water a result of arsenic cycling within the aquatic system (i.e., the hyporheic zone) and if not, what other sources are contributing?	Insufficient data exist to quantify amount of arsenic cycling in the PSP	Collection of groundwater, porewater, sediment, and surface water will allow for appropriate comparisons to be made.
4	Based on results of DUOs 1 through 3, what locations, media, and sample frequency are needed for long-term monitoring of Red Cove?	Long term monitoring recommendations are insufficient to monitor attainment of RAOs	Sample sizes for long term monitoring will be determined based on the statistical distribution of data collected.

The data acquisition program looks at all steps of the process (i.e., design of the sampling approach, field collection practices, laboratory analysis, data validation, and data evaluation) to ensure the program is designed to accomplish the data goals and objectives. Quality control (QC) samples will be collected to assess the quality performance criteria. Field duplicates will be collected at a frequency of one sample per 10 parent samples (10%); Matrix spike (MS)/matrix spike duplicate (MSD) samples will be collected at a frequency of one each per 20 parent samples (5%). Additional detail on the data performance and acceptance criteria is provided in the QAPP Addendum (Appendix A).

2.7 Step 7 – Optimize the Design for Obtaining Data

To achieve the study objective discussed in Section 2.2, a comprehensive plan was developed to collect and evaluate groundwater, sediment, porewater, and surface water. The general approach for data collection and analysis is presented below, and details are presented in Section 3.

2.7.1 Field Approach

The sampling approach includes collection of samples for analysis from 20 aquatic sample locations within Red Cove area of PSP, eight aquatic reference locations in the northeast portion of PSP, and ten existing groundwater monitoring well locations and three soil borings adjacent to Red Cove area of PSP. The 20 aquatic sample locations within Red Cove encompass the area previously sampled after completion of the removal action in 2013 (Figure 3). At each of the 20 aquatic locations, paired porewater and sediment measurements/samples will be collected. At a subset of aquatic sample locations, sediment core samples will be collected for evaluation of hydraulic conductivity using Shelby tubes. At the same aquatic sample locations as the sediment core samples, surface water measurements and samples will be collected. At locations where hydraulic conductivity is measured in Red Cove sediments, clustered piezometers will be installed at depths of 2 and 10 feet bss to measure vertical gradient across the hyporheic zone. Alternate locations will be attempted if refusal is encountered. Based on current well logs, there is no reason to assume refusal will happen. Up to two additional attempts within a 10-feet radius will take place if refusal is encountered. Groundwater samples will be collected at 10 monitoring wells (four upgradient and six between the barrier wall and Red Cove) to provide temporal and analytically comparable results to assist in the evaluation. At each well, depth to groundwater will be gauged, field parameters measured, and samples collected for laboratory analysis, as discussed in Section 3.4.

Eight aquatic reference locations will also be sampled within PSP, for a total of 28 aquatic sample locations. The refence locations are intended to be outside of the hydraulic influence of groundwater from SHL (Figure 3).

Groundwater level measurements from wells between the barrier wall and Red Cove, vertical gradient from the clustered piezometers constructed in Red Cove, and surface water level measurements from aquatic locations, will be evaluated to provide estimates of hydraulic head and gradients. These hydraulic head/gradient estimates will be combined with the hydraulic conductivity data and porewater analytical results to determine groundwater velocity and the associated arsenic flux into Red Cove.

The proposed sample locations are shown on Figure 3 and presented in Table 3 (included at the end of the document).

2.7.2 Analytical Approach

Based on previous studies, iron geochemistry is the principal controlling mechanism for dissolved arsenic occurrence in groundwater and surface water at SHL. As previously discussed, reducing conditions created by SHL and the landfill cap resulted in dissolution of iron minerals (amorphous iron oxyhydroxides and better crystalized forms, such as goethite and hematite). As the dissolved iron and co-occurring arsenic that was mobilized in reduced portions of groundwater migrate toward more oxic conditions, the iron will precipitate as amorphous iron oxyhydroxides. Arsenic in groundwater co-precipitates with the oxyhydroxides at a relatively constant proportion characteristic of the soil and groundwater geochemistry for the site. The amorphous oxyhydroxides are generally an intermediate form (metastable) and more soluble than mineralized iron oxides. With sufficient time, the amorphous oxyhydroxides will mineralize to a more stable form, such as goethite.

The relative proportions of the more soluble amorphous oxyhydroxides and more stable goethite can be observed from sequential extractions of iron by the SEP. The SEP extracts iron (and co-precipitated arsenic) from a soil sample through a series of solutions, ranging from less aggressive (soluble) to more aggressive (residual) methods. A common SEP sequence, from more soluble to less soluble, consists of:

water soluble/exchangeable > carbonate extractable > oxide extractable > sulfide/organic matter extractable > acid soluble > residual

Most of the iron and arsenic in the soluble extract are weakly sorbed to soil or organic matter. Most metastable forms, such as the amorphous oxyhydroxides, are extracted by the intermediate oxide procedure. The most stable forms (goethite and hematite) are extracted by the acid soluble/residual procedure.

Iron stability, and by association arsenic stability, is a critical consideration in understanding source, mobility, and fate of arsenic in groundwater, sediment, and surface water at PSP. Investigations, including SEP tests and x-ray diffraction and scanning electron microscopy mineral analyses, have been completed to assess iron minerology, principally on the underlying bedrock. These investigations have aided in identifying the relationship between arsenic and iron in soil and groundwater, principal forms of iron present in soil, conditions responsible for dissolution and precipitation of iron, and associated co-precipitation/dissolution of arsenic. The same types of information are important in understanding the form, mobility, and stability of iron and co-occurring arsenic in groundwater, porewater, surface water, and sediment in the Red Cove area of PSP.

In addition to characterizing the iron-arsenic geochemical cycle, analysis of the dominant contributing ions is equally important in evaluating geochemical stability and provenance of the water analyzed from groundwater, porewater in the hyporheic zone, and surface water. Along with major ions, previously identified indicator parameters (such as potential higher potassium and barium concentrations associated with groundwater flowing from SHL [USEPA 2008] or potential higher ammonia concentrations associated with past tannery operations from upstream of PSP [AMEC 2010]) will be analyzed in these samples. Methodology previously used by EPA to assess provenance, including use of molar ratio comparisons as well as other graphical methods (piper, stiff, and Pourbaix diagrams) will be used to aid this assessment.

3 Detailed Plan for Obtaining Data

This section describes the collection of temporal and spatial synoptic samples in groundwater, porewater, surface water, and sediment. The 28 proposed porewater, sediment, and aquatic sample locations along with the 10 groundwater sample locations are shown on Figure 3 and presented in Table 3 (included at the end of the document).

3.1 Field Measurements

Field measurements will be collected from each sample location as part of the field sampling program. This includes both the aquatic and groundwater sample locations described below.

3.1.1 Aquatic Location Field Measurements

Field measurements will be recorded from a boat (to minimize any potential disturbance to the sediment bed or water column) at each of the 28 aquatic sample locations (20 in Red Cove and eight reference locations) before sample collection. These measurements will include water surface elevation, water depth, and sediment depth (as determined by refusal of a metal metered rod using manual pressure). DO, conductivity, ORP, pH, turbidity, and temperature will be measured using a multiparameter probe at the following depths:

- Top Sample (see Section 3.2.2)
- Bottom Sample (2 to 6 inches from above sediment surface)
- Six to 12 inches bss
- Eighteen to 24 inches bss
- Thirty to 36 inches bss
- Forty-two to 48 inches bss.

The depth to water will not be measured within 48 hours of a preceding precipitation event to provide base measurements. The in-situ field measurements will be used in conjunction with other data collected to determine where groundwater flux into the Red Cove area of PSP from SHL may be highest. Water surface elevation can be measured through the establishment and daily reading of a staff gage or use of a differential global positioning system if the sampling vessel is so equipped. Water level measurements will be gauged with a water level meter tied to a benchmark elevation on land in order to determine the water surface elevation.

3.1.2 Groundwater Location Field Measurements

Field measurements will be recorded at 10 groundwater sample locations during well purging and again prior to sample collection. The groundwater levels will not be measured within 48 hours of a significant preceding precipitation event (i.e., greater than 1 inch in 24 hours) to provide base measurements. In addition to the 10 monitoring wells that will be sampled, other groundwater wells that are within the PSP watershed, as listed on Table 3, will be included in the synoptic groundwater gauging event for a total of 40 wells. Depth to water, DO, conductivity, ORP, pH, turbidity, and temperature will be measured using a multiparameter probe. As part of the low-flow sampling protocol, during the purging cycle depth to water will be measured periodically to ensure a drawdown of less than 0.1 meter.

3.1.3 Surface Water Flow

Instantaneous flow into and out of PSP will be measured two times (once at the commencement of field activities and once at the end of field activities). The flows will not be measured within 48 hours of a preceding precipitation event to provide base flow measurements. Flow from Grove Pond to PSP will be measured at the culvert east of the railroad causeway and flow from PSP to Nonacoicus Brook will be measured at the dam on the western edge of PSP. Flow will be calculated from velocity measured along a transect across the culvert and dam, and a bathymetric profile measured at the time the flow velocity is acquired.

The measured flows into and exiting PSP will provide supporting information for the water balance for PSP. Methodology that will be followed in collecting these measurements is described in the standard operating procedure for Surface Water Flow Measurement provided as Attachment 1 to the QAPP Addendum (Appendix A).

3.1.4 Aquatic Hydraulic Head Measurement

Water elevations will be measured from clustered piezometers at 10 locations within Red Cove, co-incident with Shelby tube samples collected for vertical hydraulic conductivity analysis (as shown in Figure 3). The elevations will not be measured within 48 hours of a preceding precipitation event to provide base flow measurements. These clustered piezometers, along with measured PSP stage, will provide a measured vertical gradient at each location, as shown on the CSM (Figure 2). The groundwater head, along with the measured hydraulic conductivity from the Shelby tubes, will be used to estimate groundwater flow through the hyporheic zone to Red Cove. The clustered piezometers will consist of two 0.75-inch ID pipes with 6-inch screen. At each location, one piezometer will be hand driven to a depth of 10 feet bss with a screen interval of 9.5 to 10 feet bss. A second piezometer will be hand driven to a depth of 2 feet bss with a screen interval of 1.5 to 2 feet bss. Up to two additional alternate locations will be attempted within a 10-feet radius if refusal is encountered. Based on current well logs, there is no reason to assume refusal will happen. Each piezometer will consist of 0.75-inch ID stainless steel drive pipe with 6-inch, 50-mesh (0.014-in opening) stainless steel screen. At the location with the greatest water depth, a polyvinyl chloride stilling pipe will be added to the well cluster to measure stage in PSP. All pipes within a cluster will be secured with “T” connectors to improve stability for the 2-foot piezometers and stilling pipe. Top of pipe elevations for all piezometers will be surveyed to North American Vertical Datum of 1988.

Synoptic water levels will be manually gauged in all piezometers from a boat and the stilling pipe during three events, each separated by a minimum of 4 weeks between events. Water levels will also be gauged in the monitoring wells listed under Section 3.1.2 (Groundwater Location Field Measurements). Water levels will be gauged to 0.01 foot.

3.1.5 Temporal Flux Variation

Pressure transducers will be installed in eight groundwater monitoring wells, eight piezometers in Red Cove, two piezometers in the PSP reference area, and the PSP surface water. The pressure transducers will be installed in these locations coincident with the first aquatic head measurements and will record water levels at an hourly interval through the duration of the field investigations. The hourly water level measurements will provide information on variations in hydraulic gradients in response to seasonal and precipitation changes. The selected locations are clustered wells and piezometers and will provide information on vertical gradients between:

- the surficial sand aquifer and bedrock and the surficial aquifer, and
- the surficial sand aquifer, hyporheic zone, and surface water.

Pressure transducers will be installed in the following areas as shown on Figure 3:

- Four of the well clusters (eight transducers) in Red Cove,
- One well cluster in the reference area in PSP,
- PSP stilling well
- Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (N2-P1/P2 and N3-P1/P2 well clusters) adjacent to Red Cove
- Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (PZ-12-06 and SHM-11-02 cluster; RSK-28 and SMH-11-07 cluster) upgradient of the barrier wall.

3.2 Surface Water Sampling

Surface water samples will be collected from a boat (to minimize any potential disturbance to the sediment bed or water column) and analyzed as described below. The surface water samples will not be collected within 48 hours of a preceding precipitation event.

3.2.1 Surface Water Sample Collection

Two surface water samples will be collected from 14 locations (10 in Red Cove and four reference locations) using a peristaltic pump or grab sampler at the water column depths specified below:

- Top sample (see text below)
- Bottom sample (2 to 6 inches from the bottom).

If total water depth is 2 feet or less, the top sample will be collected near surface (2 to 6 inches from the surface). If the water column is more than 2 feet, the top sample will be collected from the midpoint of the water column.

The analytical approach for surface water samples is presented in Section 3.2.2. The surface water chemistry data will be used in conjunction with the porewater and groundwater collected data to evaluate arsenic flux and geochemical cycle in the Red Cove area of PSP.

3.2.2 Surface Water Sample Analysis

Surface water samples will be collected from two depths at 14 locations (28 samples) and analyzed for:

- Major cations (arsenic, sodium, iron, magnesium, manganese, barium, potassium, aluminum, calcium), total and dissolved
- Major anions (sulfate, alkalinity, chloride)
- DOC
- TDS and TSS.

Samples from seven surface water locations (collocated with grain size samples as shown in Figure 3) will be analyzed for iron-oxidizing bacteria. At the seven locations, 2 samples will be collected, with one top and one bottom of water column sample at each location. Locations of surface water samples to be analyzed for iron-oxidizing bacteria were selected to provide a distribution representative of the varying geochemical characteristics within the SHL and PSP; proposed well locations will be adjusted based on floc observations made during well development. Because iron oxidizing bacteria require a minimum level of oxygen to convert iron from ferrous to ferric and given the lack of water circulation (i.e., stagnant water) in Red Cove, there is a reasonable likelihood that the dissolved oxygen levels at the groundwater-surface water interface are too low to support a large population of iron oxidizing bacteria. It is more likely that a higher population count is present closer to the water surface where the dissolved oxygen levels are higher. The sampling approach should confirm this assumption.

The proposed analytical methods and sample counts for surface water are presented in Table 6, below.

Table 6. Surface Water Sample Summary and Proposed Analytical Methods

Surface Water	**Analyses**		
Sample Depth	**DOC, TDS, TSS**	**Cations and Anions [1]**	**Iron-Oxidizing Bacteria**
Top sample	14	14	14
Bottom sample (2 to 6 inches from the bottom)	14	14	0
Field duplicates (10%)	3	3	2
MS/MSD (5%)	2	2	0
Total	33	33	16

Note:

[1] Major cations = arsenic, sodium, iron, magnesium, manganese, barium, potassium, aluminum, and calcium (total and dissolved); major anions = sulfate, alkalinity, and chloride.

3.3 Soil Sampling

Soil cores from the surficial sand aquifer will be collected adjacent to three well locations between the barrier wall and Red Cove (Figure 3) and analyzed as described below:

- Area with elevated (greater than 2x the GW-1 standard of 10 µg/L) dissolved arsenic in groundwater:
    - SHL-4 (3-13 ft bgs)
    - SHP-01-38A (1.5-6.5 ft bgs)
- Area with lower dissolved arsenic in groundwater:
    - SHL-19 (20-30 ft bgs)

3.3.1 Soil Sample Collection

Samples from the three locations will be collected as 5-foot composites from the ground surface to the top of bedrock using a Direct Push Rig (DPT refusal will be used as the indication that the depth of the top of bedrock has been reached.)

3.3.2 Soil Sample Analysis

All soil core samples will be analyzed for:

- Metals (arsenic, iron, magnesium, manganese, aluminum, calcium)
- TOC by Llyod Kahn
- pH
- Total sulfur
- Grain size with sieve and hydrometer (ASTM D7928).

The metals and organic carbon to be analyzed are target compounds (e.g., arsenic), compounds/constituents that arsenic co-precipitates with (iron and manganese) or strongly binds to (organic carbon), or constituents that influence the geochemistry, such as the pH and ion exchange, of the sediment (aluminum, calcium, and magnesium).Two samples from each location will be collected for SEP: one within the well screen interval and the other from the core above the contact with bedrock.

The proposed analytical methods and sample counts for soil samples are presented in Table 7, below.

Table 7. Soil Sample Summary and Proposed Analytical Methods

Soil	Analyses			
Sample Depth	**Metals [1]**	**TOC**	**General[2]**	**SEP**
0 to 5 feet	3	3	3	3
5 to 10 feet	3	3	3	
10 to 15 feet	3	3	3	
15 to 20 feet	3	3	3	
20 to 25 feet	3	3	3	
25 to 30 feet[4]	3	3	3	3
Field duplicates (10%)	1	1	1	1
MS/MSD (5%)	1	1	1	0
Total	~20	~20	~20	7

Notes:

[1] Soil metals = arsenic, iron, magnesium, manganese, aluminum, calcium.

[2] Soil general = pH, sulfur, and grain size.

[3] SEP will include: Cations (arsenic, iron, magnesium, manganese, aluminum, and calcium), Anions (sulfur), and DOC

[4] The soil cores will be collected until refusal (i.e., top of bedrock is reached). The assumption is this will occur around 30 feet based on nearby well logs. Thus, the total number of samples is approximate.

3.4 Sediment Sampling

Sediment samples will be collected from a boat (to minimize any potential disturbance to the sediment bed or water column) and analyzed as described below.

3.4.1 Sediment Sample Collection

Sediment cores for chemical analysis will be advanced from a boat manually or with a vibracore to a minimum depth of 4 feet bss at 28 locations (20 in Red Cove and eight reference locations). Sediment samples from these cores will be sectioned into five intervals: 0 to 6 inches, 6 to 12 inches, and 1-foot intervals thereafter. The bottom interval of samples will be adjusted based on sample recovery, field measurements taken before the collection of cores, or if distinct visual horizons are noted in the sediment core. Use of the cores to collect sediment samples instead of an Eckman sampler provides:

- Greater sample depth than is attainable with an Eckman sampler
- Substantially better vertical profile to collect high-resolution samples.

Use of cores to collect the sediment samples is necessary to meet the objectives of this study. The analytical approach for collected sediment samples is presented in Section 3.4.2.

At 10 Red Cove locations and one reference location, an additional sediment sample will be collected using a 2-inch-diameter, 36-inch-long Shelby tube advanced manually. These samples will be submitted to the laboratory to evaluate the vertical hydraulic conductivity of the sediment.

3.4.2 Sediment Sample Analysis

Five sediment samples will be collected from each of the 28 aquatic sample locations shown on Figure 3 (20 in Red Cove and eight reference locations). Each sample will be submitted for analyses that will be used to provide additional information on stability of the iron minerals (and by association the co-precipitated arsenic) and to assess other characteristics that influence iron and arsenic mobility. Sediment sample collection and analysis are summarized below:

- All sediment samples (up to five samples from 28 locations = 140 samples) will be analyzed for:
  - Metals (total arsenic, iron, magnesium, manganese, aluminum, and calcium)
  - TOC by Llyod Kahn.

  The metals and organic carbon to be analyzed are target compounds (e.g., arsenic), compounds/constituents that arsenic co-precipitates with (iron and manganese) or strongly binds to (organic carbon), or constituents that influence the geochemistry, such as the pH and ion exchange, of the sediment (aluminum, calcium, and magnesium). To provide a high-resolution profile through the hyporheic zone, samples will be collected at 6-inch intervals for the first 1 foot below the sediment-water interface (SWI) and 1-foot composites to a depth of 4 feet. Based on the high-resolution profiles collected from previous investigations in PSP, these samples are expected to span the hyporheic zone for PSP.

- Samples from three depths per location (0 to 6, 6 to 12, and 42 to 48 inches; total of 84 samples) will also be analyzed for the following:
  - pH
  - Sulfur
  - Grain size with sieve and hydrometer (ASTM D7928).

  Samples that straddle the hyporheic zone (0 to 12 and 36 to 48 inches below the SWI) will be analyzed for constituents that influence the geochemical reaction (pH and sulfur), and mineral form (sulfur). Of principal concern for these constituents are their presence/concentrations bounding the hypoheric zone. Less vertical variation is expected over the transition zone (12 to 36 inches) compared to the metals of interest, and only bounding samples will be collected. In addition to these constituents, grain size will be measured from the top and bottom samples to provide a comparison to the field classification that will be completed for each sample.

- Samples from two depths at 14 locations (10 in Red Cove and four reference locations at 6 to 12 and 42 to 48 inches; total of 28 samples) will be analyzed by SEP:
  - Water soluble/exchangeable
  - Carbonate
  - Oxide
  - Sulfide/organic
  - Acid soluble
  - Residual.

  Similar to pH and sulfur, samples that straddle the hyporheic zone will be analyzed for SEP. These samples will bracket the expected extremes in the transition and provide information on stability, mineralogy (iron sulfide versus amorphous iron oxyhydroxides, versus mineralized goethite and hematite), and arsenic co-association with the iron precipitates. The co-associations established from the samples collected at the vertical extents will be applied to analytical results from the high-resolution samples. The biological activity at the surface interval (0 to 6 inches below SWI) is expected to be too active and results too transitory to provide interpretable results from the SEP analysis, and the sample representing the upper interval of the hyporheic zone will be collected from 6 to 12 inches below SWI.

- Shelby tube samples will be collected from 10 locations in Red Cove for analysis of triaxial hydraulic conductivity via ASTM 5084 to determine vertical hydraulic conductivity of the sediment samples. These tests will be performed using the entire 4-foot length of the tube to maintain the undisturbed nature of the sample.

The proposed analytical methods and sample counts for sediment are presented in Table 8, below.

Table 8. Sediment Sample Summary and Proposed Analytical Methods

Sediment	Analyses				
Sample Depth	**Metals [1]**	**TOC**	**General [2]**	**SEP[3]**	**Triaxial Hydraulic Conductivity**
0 to 6 inches	28	28	28	0	10
6 to 12 inches	28	28	28	14	
12 to 24 inches	28	28	0	0	
24 to 36 inches	28	28	0	0	
42 to 48 inches	28	28	28	14	
Field duplicates (10%)	14	14	9	3	0
MS/MSD (5%)	7	7	5	0	0
Total	161	161	98	31	10

Notes:

[1] Sediment metals = total arsenic, iron, magnesium, manganese, aluminum, and calcium.

[2] Sediment general = pH, sulfur, and grain size.

[3] SEP will include: Cations (arsenic, iron, magnesium, manganese, aluminum, and calcium), Anions (sulfur), and DOC

3.5 Porewater Sampling

Porewater samples will be collected from a boat (to minimize any potential disturbance to the sediment bed or water column) and analyzed as described below. The porewater samples will not be collected within 48 hours of a preceding precipitation event.

3.5.1 Porewater Sample Collection

Consistent with previous data collection sampling programs, porewater samples will be collected using PushPoint™ samplers at 28 locations (20 in Red Cove and eight reference locations) in accordance with the procedure outlined by the Operating Procedure, Porewater Sampling (USEPA 2020a). Porewater samples will be collected at the following four depths:

- Six to 12 inches bss
- Eighteen to 24 inches bss
- Thirty to 36 inches bss
- Forty-two to 48 inches bss.

The analytical approach for porewater is presented in Section 3.5.2.

3.5.2 Porewater Sample Analysis

Porewater samples will be collected from four depths at 28 locations (112 samples) and analyzed for:

- Metals (total and dissolved): arsenic and iron
- TSS.

Samples from two depths per location (6 to 12 and 42 to 48 inches; total of 56 samples) will also be analyzed for:

- Major cations (arsenic, sodium, iron, magnesium, manganese, barium, potassium, aluminum, and calcium), total and dissolved
- Major anions (sulfate, alkalinity, chloride)
- DOC
- TDS and TSS
- Ammonia.

The major ions will provide important information on the geochemistry of the porewater for use in evaluating form, mobility, and potential to form various iron precipitates with an accumulation of co-associated arsenic across the hyporheic zone. Samples collected from the top and base of the hyporheic zone provide the range in concentration across this mixing zone between the surface water and groundwater. Because mixing within the hyporheic zone is highly transitory, dependent on factors such as season and precipitation, characterization of these constituents within the intermediate zones is of limited use and will not be collected.

Samples from two depths at four locations (three locations from PSP and one location from a reference location; total of eight samples) will also be analyzed for iron-oxidizing bacteria. The presence of iron-oxidizing bacteria is an important consideration in assessing precipitation mechanism for iron and co-precipitation of arsenic. Because the test for these bacteria is present-absent, and if present is likely widespread, only a limited number of samples is necessary.

The proposed analytical methods and sample counts for porewater are presented in Table 9, below.

Table 9. Porewater Sample Summary and Proposed Analytical Methods

Porewater	**Analyses**			
Sample Depth	**Metals [1] (total and dissolved), TSS**	**DOC, TDS**	**Cations and Anions [2]**	**Iron-Oxidizing Bacteria**
6 to 12 inches	28	28	28	4
18 to 24 inches	28	0	0	0
30 to 36 inches	28	0	0	0
42 to 48 inches	28	28	28	4

Porewater	**Analyses**			
Sample Depth	**Metals [1] (total and dissolved), TSS**	**DOC, TDS**	**Cations and Anions [2]**	**Iron-Oxidizing Bacteria**
Field duplicates (10%)	12	6	6	1
MS/MSD (5%)	6	3	3	0
Total	130	65	65	9

Notes:

[1] Porewater metals = arsenic and iron (total and dissolved).

[2] Major cations = arsenic, sodium, iron, magnesium, manganese, potassium, aluminum, and calcium (total and dissolved); major anions = sulfate, alkalinity, ammonia, and chloride.

3.6 Groundwater Sampling

Groundwater samples will be collected and analyzed as described below. The groundwater samples will not be measured within 48 hours of a significant preceding precipitation event (i.e., greater than 1 inch in 24 hours).

3.6.1 Groundwater Sample Collection

Groundwater samples will be collected from 10 existing monitoring wells (Figure 3 and Table 3). One synoptic round of water level measurements from all the wells listed in Table 3 (40 wells) will be recorded before the start of sample collection activities. Before sampling, the monitoring wells will be redeveloped, if needed by over-pumping to remove sediment and iron floc, if present. Sampling would not occur until a minimum 24 hours have passed after redevelopment. Groundwater samples will then be collecting using low-flow sampling procedures. After sample collection, and after the water levels have recovered within 95% of their static level as measured before initiating sampling, rising / falling head tests will be completed on all 10 wells. The results of the rising / falling head tests will provide estimated hydraulic conductivity for the wells that will be included in the groundwater flux evaluation.

The analytical suite for groundwater is presented in Section 3.6.2 and is generally the same as the suite proposed for porewater and surface water. Water level measurements and field measurements for groundwater will be coincident with measurements at the aquatic locations.

3.6.2 Groundwater Sample Analysis

The groundwater samples from all 10 wells will be analyzed for:

- Arsenic and iron (total and dissolved)
- Geochemical parameters that are important in controlling mobility of iron and arsenic in groundwater at the site:

- Remaining major cations (sodium, magnesium, manganese, barium, potassium, aluminum, and calcium), total and dissolved
- Major anions (sulfate, alkalinity, and chloride)
- DOC
- TDS and TSS
- Ammonia.

The groundwater samples from four wells (two upgradient locations and two locations between the barrier wall and Red Cove) will be analyzed for:

- Carbon dioxide gas
- Iron-oxidizing bacteria.

Analytical results will be used with previous sample results to:

- Assess changes over time (including before and after barrier wall construction) in groundwater characteristics, including iron, manganese, and arsenic; major ion composition; and potential indicator parameters such as barium, potassium, and ammonia.
- Evaluate relationships between arsenic (and iron) concentrations in groundwater upgradient of the hyporheic zone groundwater and sediment samples
- Characterize variability (vertical and lateral) major ion chemistry that controls iron and arsenic chemical speciation.

The proposed analytical methods and sample counts for groundwater are presented in Table 10, below.

Table 10. Groundwater Sample Summary and Proposed Analytical Methods

Groundwater	**Analyses**				
Sample Depth	**Arsenic (total and dissolved)[1]**	**Cations and Anions [1]**	**DOC, TDS, TSS**	**Iron-Oxidizing Bacteria**	**Carbon Dioxide Gas**
Existing wells	10	10	10	4	4
Field duplicates (10%)	1	1	1	1	1
MS/MSD (5%)	1	1	1	0	1
Total	12	12	12	5	6

Note:

[1] Major cations = arsenic, sodium, iron, magnesium, manganese, potassium, aluminum, calcium, (total and dissolved); major anions = sulfate, alkalinity, chloride.

4 Barrier Wall Performance Monitoring Report

Upon completion of the activities discussed in this Work Plan, a Barrier Wall Performance Monitoring Report (Monitoring Report) will be prepared to summarize the activities performed; present field measurements and observations and results of analytical testing; and provide estimates of arsenic flux to the Red Cove area of PSP from SHL. Following completion of the data evaluation, recommendations will be made to supplement the Long-Term Monitoring Work Plan (KGS 2018) to monitor conditions in Red Cove. A schedule showing the timeline for finalizing the Work Plan, the upcoming field activities, and reporting is included as Table 11 below in Section 5.

4.1 Summary of Field Activities and Data Reporting

The Monitoring Report will describe the field activities completed and document any deviations from this Work Plan. All field measurement and analytical data will be tabulated and presented in the Monitoring Report. Data interpretation will be presented in subsequent sections of the Monitoring Report.

4.2 Data Use Objective Goal 1: Description of Current Conditions

The groundwater, porewater, surface water, and sediment data will be evaluated to present current conditions in Red Cove and the adjacent areas in the upland and PSP. Sample coverage for Red Cove includes the 2013 post-excavation area. Results will be compared to:

- Upgradient groundwater trends
- The 2013 post-excavation arsenic concentrations
- Reference sediment in PSP.

Data evaluations will include the development of population statistics for comparison to applicable standards and project goals. Although there is no single statistical test to determine if a sampling of data is from a single statistical population, there are several lines of evidence that can be indicative of a single population. These lines of evidence include a single statistical distribution, a linear probability plot, a low coefficient of variation, and relatively few statistical outliers. Concentrations will be presented graphically to show the horizontal and vertical distribution in Red Cove and relational correlations will be developed between media (surface water, sediment, porewater, and groundwater).

4.3 Data Use Objective Goal 2: Assessment of Arsenic Flux

The evaluation of arsenic flux will incorporate:

- Existing hydraulic head data collected from wells upgradient of the barrier wall and between the barrier wall and Red Cove as part of the annual monitoring program to establish lateral hydraulic gradients.
- Groundwater levels measured in the clustered piezometers and PSP stage to establish the vertical hydraulic gradient across the hyporheic zone in Red Cove.

- Vertical hydraulic conductivity measured in undisturbed Shelby tube samples at the clustered piezometer locations.
- Arsenic analytical results.

These measurements will consider flux from several different areas as illustrated on the CSM Figure 2 (shown again below):

- Flux across the barrier wall
- Flux from the barrier wall to Red Cove
- Flux from the surficial sand aquifer, through the hyporheic zone, to surface water in Red Cove

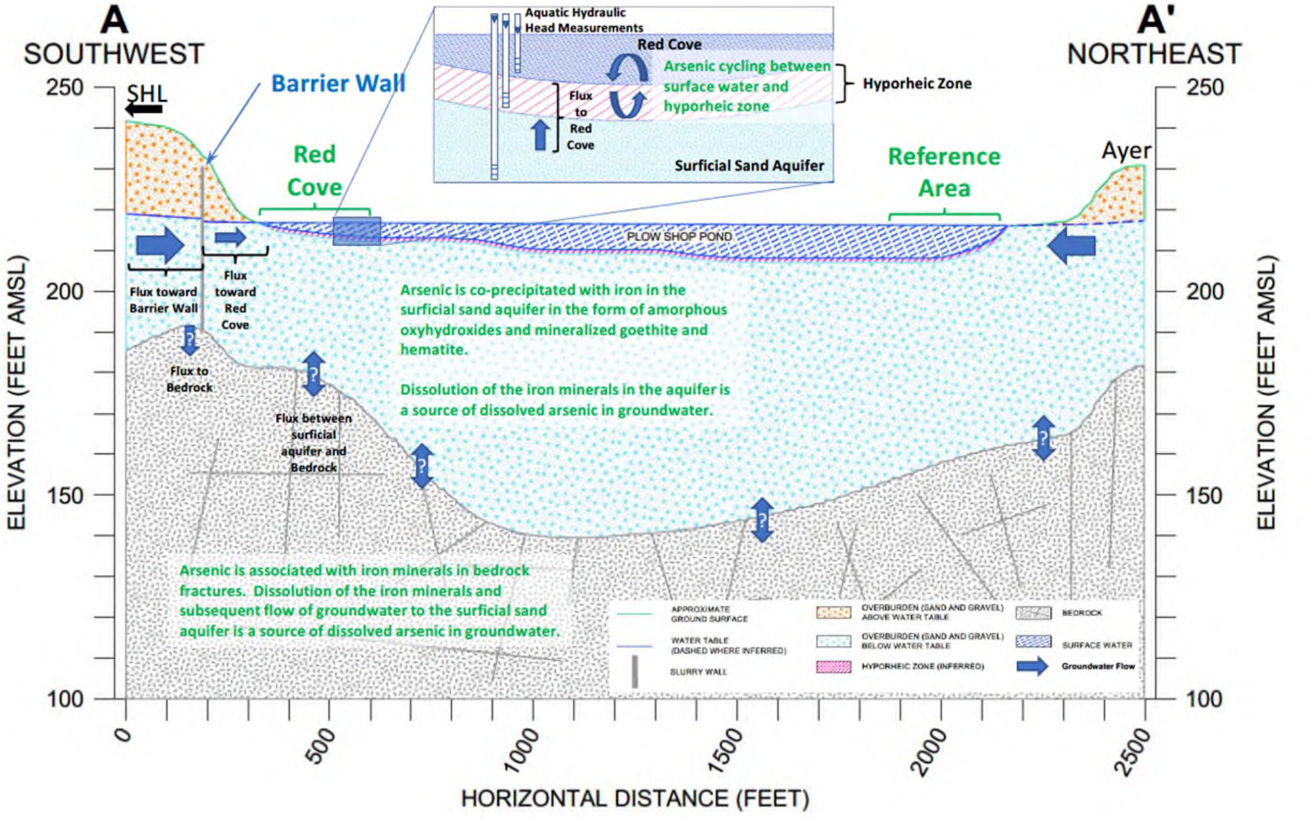


Figure 2: Generalized Conceptual Site Model Cross Section AA'

Flux across the barrier wall is calculated as part of the Annual Reports, and includes flux calculations upgradient of the barrier wall and between the barrier wall and Red Cove, and lateral flux from the barrier wall to Red Cove.

Flux from the surficial sand aquifer through the hyporheic zone to the surface water will be calculated from vertical gradients measured in the clustered piezometers in Red Cove, vertical hydraulic conductivity measured at the same locations, surface water stage in PSP, and arsenic analytical results from the associated porewater samples will be used to calculate arsenic flux through the hyporheic zone to Red Cove. The methodology used in the Annual Reports to calculate flux in the groundwater will be used to calculate vertical arsenic flux to Red Cove, except the vertical gradient will be based on the measurements at the individual clustered piezometers. The arsenic flux calculations through the barrier wall and to Red Cove will be compared to pre-barrier wall and previous post-barrier wall arsenic flux calculations.

4.4 Data Use Objective Goal 3: Assessment of Arsenic Sources in Plow Shop Pond

The geochemical assessment will be provided, focusing on the constituents controlling arsenic form and mobility in the groundwater, porewater, and surface water. A key question to be assessed is the source of the ongoing elevated arsenic levels in Red Cove surface water. This assessment will evaluate potential sources in:

- Upgradient groundwater
- Sediment and porewater in the hyporheic zone
- Suspended and dissolved-phase iron and arsenic cycling in the water column and sediment surface.

This assessment will build on past assessments and will include an evaluation of solid- and dissolved-phase iron and arsenic geochemistry and biogeochemistry in the upland, within the hyporheic zone at the groundwater-surface water interface, and in surface water. Components most critical to this assessment are the sediment, porewater, and surface water analytical results. In completing this assessment, the following information will be used:

1. The major ions and general chemistry (including field measurements) will provide information important in evaluating iron form and stability, and by extension the co-associated arsenic. Graphical methods (piper, stiff, and Pourbaix diagrams) along with molar ratio comparisons will be important tools in this assessment.
2. SEP analysis will be used to provide information on solubility of the various iron oxyhydroxide and sulfide minerals, and the co-occurrence of arsenic with the mineral forms as well as with natural organic matter. This information may be used to provide insight to the stability of these precipitates and mineral forms within the hyporheic zone, and, if necessary, provide supporting information to assessing dissolution rates.
3. Analysis for iron-oxidizing bacteria in the sediment and surface water to establish the potential role of biogenic formation of iron oxyhydroxides with co-occurring arsenic in the sediment and water column of Red Cove.
4. Re-evaluation of geochemical tracers previously used by ORD to identify leachate impacted groundwater from SHL prior to construction of the barrier wall. These geochemical tracers include barium, potassium, calcium, ammonia, sulfate, and chloride. These tracers will be combined with the major ion and general geochemistry results to assess provenance of groundwater and porewater between the barrier wall and Red Cove.

The combined results of the hydraulic and geochemical analyses will be used to determine provenance and disposition of arsenic in groundwater as it flows from upgradient groundwater, through the sediment in the hyporheic zone, seeps through the SWI and into the Red Cove surface water column, and cycles within the surface water and sediment of Red Cove.

4.5 Data Use Objective Goal 4: Long-Term Monitoring Recommendations

As discussed in Section 1, the USEPA recommended two rounds of sample data be collected to support evaluation of the remedy over the long term and support data decisions made during the five-year review process. The second round of data collection requested will be conducted as part of the long-term monitoring program and recommendations on the scope of the data collection will be incorporated as an addendum to the Long-Term Monitoring and Maintenance Plan, if needed (KGS 2018). The data collected will inform decision making and identification of follow-on sampling scope to be performed, if needed, to evaluate barrier wall performance.

5 Schedule

The anticipated project schedule is presented below in Table 11. The work plan review schedule follows the Primary Document review process outline in the Federal Facilities Agreement between the Army and the USEPA for Fort Devens.

Table 11. Anticipated Project Schedule

Task Name	Duration	Start	Finish
Army Submits Draft Work Plan to EPA/DEP	1 day	11/16/21	11/16/21
EPA/DEP Review of Draft Work Plan	45 days	11/17/21	1/3/22
Army Issues Response Letter to EPA/DEP Comments on Draft Work Plan	45 days	1/4/22	2/17/22
Army Submits Draft Final Work Plan to EPA/DEP	45 days	2/18/22	4/4/22
EPA/DEP Review of Draft Final Work Plan	16 days	4/5/22	4/20/22
Army Submits Final Work Plan to EPA/DEP	18 days	4/21/22	5/9/22
Field Work	Summer/Fall 2022		
Draft Barrier Wall Performance Monitoring Report	Winter 2022/2023		

6 References

AMEC. 2010. Remedial Investigation for AOC 72, Plow Shop Pond – Draft. Devens, Massachusetts. Prepared for U.S. Army Corps of Engineers New England District. March.

Acree, S., Ford R., Lien, B., Ross, Randall. 2018. Tools for Estimating Groundwater Contaminant Flux to Surface Water. USEPA Office of Research and Development, NARPM Webinar, September 5, 2018.

KGS. 2018. Addendum to the Long-Term Monitoring and Maintenance Plan, Shepley's Hill Landfill. Former Fort Devens Army Installation, Devens, Massachusetts. January 25.

KGS. 2020a. Draft 2019 Annual Operations, Maintenance, and Monitoring Report, Shepley's Hill Landfill, Former Fort Devens Army Installation. May.

KGS. 2020b. Final Fifth 2020 Five-Year Review Report, Former Fort Devens Army Installation, BRAC Legacy Sites, Devens, Massachusetts. Prepared for the U.S. Army Corps of Engineers New England District. September.

Mercadante A.M., J.A. Colman, and M.L. Buursink. 1999. Map Showing Morphometry, Bathymetry, and Soft-Sediment Thickness of Plow Shop Pond and Grove Pond, Ayer, Massachusetts. USGS Water-Resources Investigation Report (WRI) 99-4241.

SERES-Arcadis JV. 2021a. Final 2020 Annual Operations, Maintenance, and Monitoring Report. Shepley's Hill Landfill, Former Fort Devens Army Installation, Devens, Massachusetts. Prepared for U.S. Army Corps of Engineers New England District. August.

SERES-Arcadis JV. 2021b. Final Phase I EPA SOW – Demonstrate Plume Capture, Technical Memorandum Phase I Subtask 1.g Delineate Capture Zone based on Hydraulic and Geochemical Data, SHL Former Fort Devens Army Installation. June.

Sovereign. 2015a. Record of Decision for Plow Shop Pond (AOC 72) – Red Cove and Former Railroad Round House (SA-71), Former Fort Devens Army Installation, Devens, Massachusetts. September.

Sovereign. 2015b. Long-Term Monitoring and Maintenance Plan Update. Shepley's Hill Landfill, Former Fort Devens Army Installation, Devens, Massachusetts. September.

U.S. Army Environmental Center. 1995. Record of Decision, Shepley's Hill Landfill Operable Unit, Fort Devens, Massachusetts. Fort Devens Feasibility Study for Group 1A Sites. September.

USACE. 2021. Addendum No. 002 to the Seed Task Order for Additional Work at Shepley's Hill Landfill. Performance Work Statement for Environmental Services BRAC Legacy Sites At Former Fort Devens, Devens, MA. February. IDIQ CONTRACT NO. W912WJ19D0014 (Seed) Task Order No. W912WJ20F0022 Modification.

USEPA. 2000. Data Quality Objectives Process for Hazardous Waste Site Investigations, EPA QA/G-4HW. EPA/600/R-00/007. January.

USEPA. 2002. National Recommended Water Quality Criteria 2002, EPA 822-R-02-047. November.

USEPA. 2008. Final Report Arsenic Fate, Transport and Stability Study – Groundwater, Surface Water, Soil and Sediment Investigation – Fort Devens Superfund Site, Devens, Massachusetts. U.S. Environmental Protection Agency Office of Research and Development. September 30.

USEPA. 2020a. Operating Procedure, Pore Water Sampling. ID: LSASDPROC-513-R4. May 13. Available online at: https://www.epa.gov/sites/default/files/2017-07/documents/pore_water_sampling513_af.r3.pdf.

USEPA. 2020b. Email from C. Keating (USEPA) to R. Herzig (MassDevelopment), P. Reddy (USACE), H. Levesque (Seres-ES), A. Vitolins (Arcadis), and D. Chaffin (DEP) Re: Recommendation to Develop/Implement Field Sampling Program to Evaluate Barrier Wall Performance and Verify Attainment of RAO for Plow Shop Pond SHL - 2020 Devens Five Year Review. July 20

USGS. 2004. Natural Remediation of Arsenic Contaminated Ground Water Associated With Landfill Leachate. Fact Sheet 2004-3057. May. .

Tables

Table 3
Summary of Proposed Barrier Wall Performance Monitoring
Barrier Wall Performance Monitoring Work Plan
Shepley's Hill Landfill, Former Fort Devens Army Installation
Devens, Massachusetts

Location	Area	Easting (feet)	Northing (feet)	Screen Interval (ft bgs)	Hydraulic Head Measurement	Sediment	Porewater	Surface Water	Groundwater	Transducer	Soil Core	Grain Size	Rationale for Sampling Location
RC-01	Red Cove	630,620	3,027,169	Not Applicable (NA)	Yes	4-foot core - 5 samples	4 samples	2 samples	No	2 at different depths (2 and 10 feet)	No	2 x 36 inch Shelby tube - 1 sample	Located within Red Cove area that was previously excavated
RC-02	Red Cove	630,694	3,027,258	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-03	Red Cove	630,736	3,027,161	NA	Yes	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-04	Red Cove	630,764	3,027,340	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-05	Red Cove	630,807	3,027,245	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	2 at different depths (2 and 10 feet)	No	2 x 36 inch Shelby tube - 1 sample	
RC-06	Red Cove	630,847	3,027,151	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-07	Red Cove	630,777	3,027,574	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-08	Red Cove	630,819	3,027,479	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-09	Red Cove	630,860	3,027,382	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	2 at different depths (2 and 10 feet)	No	2 x 36 inch Shelby tube - 1 sample	
RC-10	Red Cove	630,903	3,027,287	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-11	Red Cove	630,945	3,027,195	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-12	Red Cove	630,984	3,027,103	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-13	Red Cove	630,830	3,027,701	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	Located adjacent to the previously excavated area in Red Cove and serve as bounding points
RC-14	Red Cove	630,870	3,027,612	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-15	Red Cove	630,913	3,027,518	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-16	Red Cove	630,955	3,027,421	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-17	Red Cove	630,996	3,027,326	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-18	Red Cove	631,036	3,027,234	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
RC-19	Red Cove	631,078	3,027,141	NA	Yes	4-foot core - 5 samples	4 samples	No	No	No	No	No	
RC-20	Red Cove	631,116	3,027,052	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	
PS-01	Reference	631,714	3,028,505	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	Reference area located on opposite side of PSP upstream of Red Cove
PS-02	Reference	631,792	3,028,445	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	
PS-03	Reference	631,645	3,028,424	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
PS-04	Reference	631,723	3,028,363	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	
PS-05	Reference	631,804	3,028,299	NA	No	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
PS-06	Reference	631,584	3,028,349	NA	No	4-foot core - 5 samples	4 samples	No	No	2 at different depths (2 and 10 feet)	No	No	
PS-07	Reference	631,662	3,028,290	NA	Yes	4-foot core - 5 samples	4 samples	2 samples	No	No	No	2 x 36 inch Shelby tube - 1 sample	
PS-08	Reference	631,741	3,028,224	NA	No	4-foot core - 5 samples	4 samples	No	No	No	No	No	
PZ-12-02	Upgradient	630,468	3,027,384	24.0 - 34.0	Yes	No	No	No	1 sample	No	No	No	Upgradient of Barrier Wall, screened in the surficial sand aquifer
PZ-12-04	Upgradient	630,453	3,027,194	22.0 - 32.0	Yes	No	No	No	1 sample	No	No	No	
PZ-12-06	Upgradient	630,455	3,027,082	26.0 - 36.0	Yes	No	No	No	1 sample	Yes	No	No	
PZ-12-08	Upgradient	630,546	3,026,962	18.0 - 28.0	Yes	No	No	No	1 sample	No	No	No	
RSK-28	Upgradient	630,419	3,027,126	21.40 - 26.40	Yes	No	No	No	No	Yes	No	No	
SHM-11-02	Upgradient	630,458	3,027,076	39.0 - 49.0	Yes	No	No	No	No	Yes	No	No	Upgradient of Barrier Wall, screened in the bedrock
SHM-11-07	Upgradient	630,415	3,027,133	41.0 - 46.0	Yes	No	No	No	No	Yes	No	No	
N2-P2	Between Barrier Wall and Red Cove	630,659	3,027,311	4.0 - 9.0	Yes	No	No	No	1 sample	Yes	No	No	Upgradient of Red Cove located between Barrier Wall and Red Cove
N3-P2	Between Barrier Wall and Red Cove	630,778	3,027,130	4.0 - 9.0*	Yes	No	No	No	1 sample	Yes	No	No	
SHP-01-37X	Between Barrier Wall and Red Cove	630,697	3,027,498	1.0 - 6.0	Yes	No	No	No	1 sample	No	No	No	
SHL-4	Between Barrier Wall and Red Cove	630,576	3,027,057	3.0 - 13.0	Yes	No	No	No	1 sample	No	Yes	No	
SHL-19	Between Barrier Wall and Red Cove	630,665	3,026,946	20.0 - 30.0	Yes	No	No	No	1 sample	No	Yes	No	
SHP-01-38A	Between Barrier Wall and Red Cove	630,546	3,027,171	1.5 - 6.5	Yes	No	No	No	1 sample	No	Yes	No	
N2-P1	Between Barrier Wall and Red Cove	630,659	3,027,311	35.0 - 40.0	Yes	No	No	No	No	Yes	No	No	
N3-P1	Between Barrier Wall and Red Cove	630,778	3,027,130	33.0 - 35.0*	Yes	No	No	No	No	Yes	No	No	

Table 3
Summary of Proposed Barrier Wall Performance Monitoring
Barrier Wall Performance Monitoring Work Plan
Shepley's Hill Landfill, Former Fort Devens Army Installation
Devens, Massachusetts

Location	Area	Easting (feet)	Northing (feet)	Screen Interval (ft bgs)	Hydraulic Head Measurement	Sediment	Porewater	Surface Water	Groundwater	Transducer	Soil Core	Grain Size	Rationale for Sampling Location
N1-P3	Water Level Only Location	630,723	3,027,868	12.0 - 17.0	Yes	No	No	No	No	No	No	No	Located in the area potentially upgradient or was previously upgradient (prior to construction of the Barrier Wall) of Plow Shop Pond for the purpose of providing synoptic groundwater elevations
PZ-12-01	Water Level Only Location	630,488	3,027,384	24.0 - 34.0	Yes	No	No	No	No	No	No	No	
PZ-12-03	Water Level Only Location	630,474	3,027,193	22.0 - 32.0	Yes	No	No	No	No	No	No	No	
PZ-12-05	Water Level Only Location	630,479	3,027,087	26.0 - 36.0	Yes	No	No	No	No	No	No	No	
PZ-12-07	Water Level Only Location	630,568	3,026,972	18.0 - 28.0	Yes	No	No	No	No	No	No	No	
PZ-12-09	Water Level Only Location	630,741	3,026,801	22.0 - 32.0	Yes	No	No	No	No	No	No	No	
PZ-12-10	Water Level Only Location	630,724	3,026,778	22.0 - 32.0	Yes	No	No	No	No	No	No	No	
SHL-3	Water Level Only Location	630,911	3,026,705	24.0 - 34.0	Yes	No	No	No	No	No	No	No	
SHL-7	Water Level Only Location	631,333	3,026,278	11.0 - 21.0	Yes	No	No	No	No	No	No	No	
SHL-10	Water Level Only Location	630,877	3,026,868	24.0 - 39.0	Yes	No	No	No	No	No	No	No	
SHL-11	Water Level Only Location	630,496	3,027,316	12.0 - 27.0	Yes	No	No	No	No	No	No	No	
SHL-18	Water Level Only Location	631,186	3,026,475	16.0 - 26.0	Yes	No	No	No	No	No	No	No	
SHL-20	Water Level Only Location	630,463	3,027,330	39.0 - 49.0	Yes	No	No	No	No	No	No	No	
SHM-11-06	Water Level Only Location	630,411	3,027,590	25.0 - 35.0	Yes	No	No	No	No	No	No	No	
SHM-93-18B	Water Level Only Location	631,180	3,026,453	78.5 - 88.5	Yes	No	No	No	No	No	No	No	
RSK-7	Water Level Only Location	630,636	3,027,270	5.45 - 10.45	Yes	No	No	No	No	No	No	No	
RSK-15	Water Level Only Location	630,585	3,027,062	9.85 - 14.85	Yes	No	No	No	No	No	No	No	
RSK-19	Water Level Only Location	630,560	3,027,220	4.98 - 9.98	Yes	No	No	No	No	No	No	No	
RSK-27	Water Level Only Location	630,422	3,027,049	21.22 - 26.22	Yes	No	No	No	No	No	No	No	
RSK-32	Water Level Only Location	630,425	3,027,211	21.04 - 26.04	Yes	No	No	No	No	No	No	No	
RSK-35	Water Level Only Location	630,438	3,027,522	22.65 - 26.65	Yes	No	No	No	No	No	No	No	
RSK-37	Water Level Only Location	630,730	3,027,115	33.91 - 38.91	Yes	No	No	No	No	No	No	No	
SHP-01-36X	Water Level Only Location	630,738	3,027,689	3.0 - 8.0	Yes	No	No	No	No	No	No	No	
SHP-05-43	Water Level Only Location	630,533	3,027,747	50.5 - 60.5	Yes	No	No	No	No	No	No	No	
SHP-05-44	Water Level Only Location	630,588	3,027,588	51.0 - 61.0	Yes	No	No	No	No	No	No	No	
Total Number of Samples:	**--------**	**28 Aquatic Locations**	**10 Groundwater Locations**	**--------**		**140 Samples**	**112 Samples**	**28 Samples**	**10 Samples**	**16 Transducers**	**3 Soil Locations**	**14 Samples**	
Sampling Intervals:						0 to 6 inches bss 6 to 12 inches bss 12 to 24 inches bss 24 to 36 inches bss 36 to 48 inches bss	6 to 12 inches bss 18 to 24 incjes bss 30 to 36 inches bss 42 to 48 inches bss	Top (See Section 3.2.1) Bottom (2 to 6 inches above sediment surface)		1.5 to 2 ft bss 9.5 to 10 ft bss	Surface to bedrock refusal at 5-ft intervals.		

Notes:

* Estimated value derived from Supplemental Groundwater Investigation (Harding ESE 2003).

PS-Plow Show Pond Reference Samples RC- Red Cove Primary Samples

Figures

Imagery Source: ESRI, ArcGIS Online, World Imagery

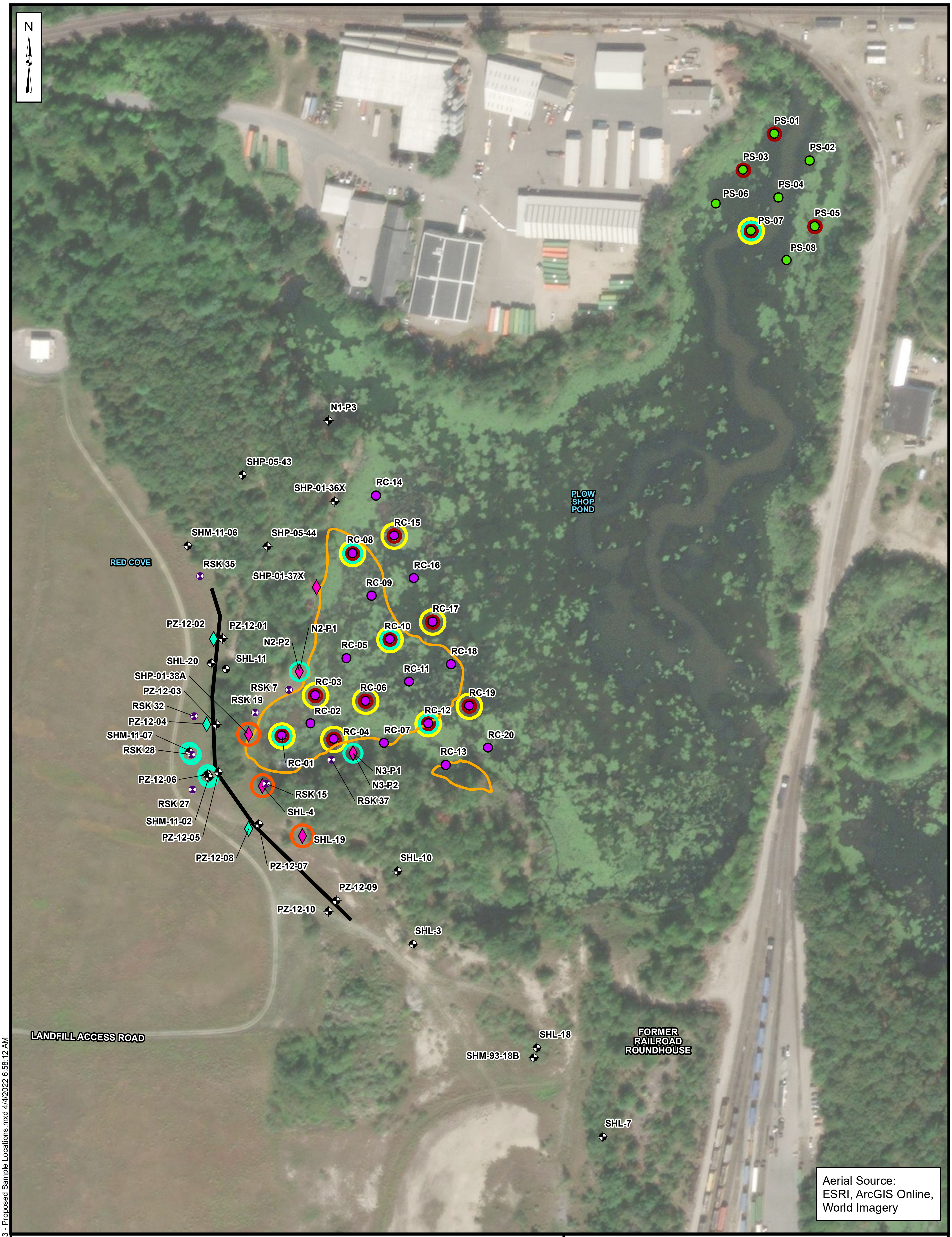
BARRIER WALL PERFORMANCE MONITORING WORK PLAN
SHEPLEY'S HILL LANDFILL
FORMER FORT DEVENS ARMY INSTALLATION
DEVENS, MASSACHUSETTS

Site Location

SERES Engineering & Services, LLC
ARCADIS
a joint venture

FIGURE
1

City: Div/Group: Created By: Last Saved By: PENASACK
Project (Project #30048392)
T:_ENV\Devens_RFTA\Seed_Task_Order\MXDs\SHL\Barrier Wall WP\Figure 1 - Site Location.mxd 2/9/2022 11:51:35 AM



Aerial Source:
ESRI, ArcGIS Online,
World Imagery

Legend

- ▬ Barrier Wall
- ▬ Approximate 2013 Excavation Area

Water Level Only Locations

- ⊕ Overburden Monitoring Well/Piezometer
- ⊗ Monitoring Well

Proposed Sampling Locations

- ○ Hydraulic Conductivity and Aquatic Head Measurement Location
- ○ Pressure Transducer
- ○ Soil Core Sample
- ◎ Grain Size and Surface Water Location
- ● Red Cove (RC) Location
- ● Plow Shop Pond (PS) Reference Location
- ◆ Well Between Barrier Wall and Red Cove
- ◆ Upgradient Well

0 100 200
Feet

Barrier Wall Performance Monitoring Work Plan
Shepley's Hill Landfill
Former Fort Devens Army Installation
Devens, Massachusetts

Proposed Sediment, Surface Water, Porewater, and Groundwater Locations

SERES Engineering & Services, LLC
ARCADIS
a joint venture

Figure
3

City: Div/Group: Created By: Last Saved By: PENASACK
Project (Project #30048392)
T:_ENV\Devens_RFTA\Seed_Task_Order\MXDs\SHL\Barrier Wall WP\Figure 3 - Proposed Sample Locations.mxd 4/4/2022 6:58:12 AM

Appendix A

Quality Assurance Project Plan Addendum

VERSION 0

FINAL ADDENDUM

UNIFORM FEDERAL POLICY FOR QUALITY ASSURANCE PROJECT PLAN - ANNUAL LONG-TERM MONITORING AND MAINTENANCE PROGRAM

BARRIER WALL PERFORMANCE MONITORING

FORMER FORT DEVENS ARMY INSTALLATION
DEVENS, MASSACHUSETTS

MAY 2022

Contract No. W912WJ-19-D-0014
Contract Delivery Order No. W912WJ-20-F-0022

Prepared For:

U.S. ARMY CORPS OF ENGINEERS, NEW ENGLAND DISTRICT
696 Virginia Road
Concord, Massachusetts 01742

Prepared By:

SERES-ARCADIS JV, LLC
669 Marina Drive, Suite B-7
Charleston, South Carolina 29492

Introduction

SERES-Arcadis 8(a) Joint Venture 2, LLC[1] (SERES-Arcadis JV) prepared this Addendum Uniform Federal Policy for Quality Assurance Project Plan – Annual Long-Term Monitoring and Maintenance Program (QAPP Addendum) under Contract No. W912WJ-19-D-0014, Contract Delivery Order No. W912WJ-20-F-0022. This QAPP Addendum is directed by and used in conjunction with the Quality Assurance Project Plan, Annual Long-Term Monitoring and Maintenance Program (LTMMP QAPP; SERES-Arcadis JV 2020) at the former Fort Devens Army Installation, located within Devens, Massachusetts (Site). This QAPP Addendum provides detailed information on the execution of the field program for sediment, surface water, porewater, and groundwater sampling at or near the barrier wall between Shepley's Hill Landfill (SHL) and Plow Shop Pond (PSP), located on Site.

All samples collected will be sent to the Eurofins TestAmerica Lancaster laboratory for analysis. As described in the Barrier Wall Performance Monitoring Work Plan (SERES-Arcadis JV 2021), at the request of the United States Environmental Protection Agency (USEPA) and Massachusetts Department of Environmental Protection (MassDEP), direct measurements of porewater, sediment, and other matrices are needed to support the estimates of arsenic flux from SHL to the Red Cove area of PSP. Environmental investigation at the Site is governed by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); therefore, results of the barrier wall evaluation will be submitted to state and federal regulatory agencies for review as part of the CERCLA process.

Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP; Intergovernmental Data Quality Task Force 2005) worksheets are developed to systematically document the planning process, sampling rationale, sampling protocols, quality assurance (QA)/quality control (QC) procedures, and other relevant components. Only the LTMMP QAPP (SERES-Arcadis JV 2020) worksheets modified for the barrier wall investigation are included in this QAPP Addendum. The analytical data from this investigation will be uploaded and validated in the Former Fort Devens electronic data management system (EDMS) according to the procedures specified in the LTMMP QAPP. Worksheet 2 provides cross-references to the location of CERCLA-required content within this QAPP Addendum or within the LTMMP QAPP. The LTMMP QAPP and this QAPP Addendum were developed with the understanding that unanticipated conditions may dictate a change in the plan as currently written.

Staff who will be participating in project and field efforts are required to read this QAPP Addendum and understand the objectives of the work to be performed, as well as the procedures to be used to conduct the field investigation, perform laboratory analyses, and evaluate and report the data that are collected. In addition, key personnel are responsible for mentoring assigned staff in aspects of this QAPP Addendum that potentially impact their assigned work to ensure project activities are executed in accordance with this QAPP Addendum.

The goals for the barrier wall performance monitoring are to:

1. Define Current conditions in Red Cove.
2. Quantify arsenic flux from SHL to the Red Cove area of PSP.
3. Identify the ongoing source of arsenic present in PSP.
4. Develop recommendations to monitor Red Cove for the attainment of remedial action objectives through time, if needed.

The objectives of this QAPP Addendum are to generate project data that are technically valid, legally defensible, and useful in meeting the project goals, as well as to integrate the technical and QC

---

[1] The SERES-Arcadis JV is composed of protégé firm SERES Engineering & Services, LLC, and its mentor Arcadis U.S., Inc.

requirements for future investigation activities. The technical approach for the sampling investigation is designed to achieve the data quality objectives (DQOs) described in Section 2 of the Work Plan and listed in Worksheet #11.

The following attachments are provided to supplement the information presented in this QAPP Addendum:

Attachment 1	Field Sampling Standard Operating Procedures
Attachment 2	Laboratory Standard Operating Procedures
Attachment 3	Laboratory Certifications

TABLE OF CONTENTS

INTRODUCTION..I

QAPP WORKSHEETS #1 AND #2: TITLE AND APPROVAL PAGE................................ 1

QAPP WORKSHEET #3: DISTRIBUTION LIST FOR FORT DEVENS 6

QAPP WORKSHEET #4: PERSONNEL SIGN-OFF SHEET.. 7

QAPP WORKSHEET #10: CONCEPTUAL SITE MODEL.. 8

QAPP WORKSHEET #11: DATA QUALITY OBJECTIVES 12

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA 13

QAPP WORKSHEET #12A: MEASUREMENT PERFORMANCE CRITERIA (CARBON DIOXIDE).. 14

QAPP WORKSHEET #12B: MEASUREMENT PERFORMANCE CRITERIA (INORGANIC IONS BY IC [CL, SO_4] IN WATER).. 16

QAPP WORKSHEET #12C: MEASUREMENT PERFORMANCE CRITERIA (ALKALINITY) 17

QAPP WORKSHEET #12D: MEASUREMENT PERFORMANCE CRITERIA (TOC) .. 18

QAPP WORKSHEET #12E: MEASUREMENT PERFORMANCE CRITERIA (DOC) .. 19

QAPP WORKSHEET #12F: MEASUREMENT PERFORMANCE CRITERIA (METALS – ICP-MS) 20

QAPP WORKSHEET #12G: MEASUREMENT PERFORMANCE CRITERIA (PH)..... 21

QAPP WORKSHEET #12H: MEASUREMENT PERFORMANCE CRITERIA (GRAIN SIZE) 22

QAPP WORKSHEET #12I: MEASUREMENT PERFORMANCE CRITERIA (TSS)..... 23

QAPP WORKSHEET #12J: MEASUREMENT PERFORMANCE CRITERIA (TDS).... 24

QAPP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS...... 25

QAPP WORKSHEETS #14 AND 16: PROJECT TASKS AND SCHEDULE 26

QAPP WORKSHEET #15: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS 27

WORKSHEET #15-1: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS IN GROUNDWATER) 29

WORKSHEET #15-2: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN GROUNDWATER) 30

WORKSHEET #15-3: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN GROUNDWATER) 31

WORKSHEET #15-4: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN GROUNDWATER) 32

WORKSHEET #15-5: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DISSOLVED GASES IN GROUNDWATER) 33

WORKSHEET #15-6: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS (ICP-MS) IN SURFACE WATER) 34

WORKSHEET #15-7: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN SURFACE WATER) 35

WORKSHEET #15-8: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN SURFACE WATER) 36

WORKSHEET #15-9: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN SURFACE WATER) 37

WORKSHEET #15-10: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS ICP-MS IN SOIL/SEDIMENT) 38

WORKSHEET #15-11: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TOC IN SOIL/SEDIMENT) 39

WORKSHEET #15-12: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS IN POREWATER) 40

WORKSHEET #15-13: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN POREWATER) 41

WORKSHEET #15-14: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN POREWATER) 42

WORKSHEET #15-15: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN POREWATER) ... 43

WORKSHEET #15-16: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TOC AND GRAIN SIZE IN SOIL/SEDIMENT) 44

WORKSHEET #15-17 REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TSS AND TDS IN WATER) 45

QAPP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE 46

QAPP WORKSHEET #18: SAMPLE LOCATIONS AND METHODS 53

QAPP WORKSHEETS #19 AND #30: SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES 55

QAPP WORKSHEET #20: FIELD QC SAMPLE SUMMARY 59

QAPP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES 62

QAPP WORKSHEET #23: ANALYTICAL STANDARD OPERATING PROCEDURES64

QAPP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION 72

WORKSHEET #24-1: ANALYTICAL INSTRUMENT CALIBRATION – GC CARBON DIOXIDE 72

WORKSHEET #24-2: ANALYTICAL INSTRUMENT CALIBRATION – ANIONS 74

WORKSHEET #24-3: ANALYTICAL INSTRUMENT CALIBRATION − PH ELECTRODE: ACIDITY, ALKALINITY 75

WORKSHEET #24-4: ANALYTICAL INSTRUMENT CALIBRATION – TOC 76

WORKSHEET #24-5: ANALYTICAL INSTRUMENT CALIBRATION – PERCENT MOISTURE 79

WORKSHEET #24-6: ANALYTICAL INSTRUMENT CALIBRATION – METALS – SOIL/SEDIMENT 80

WORKSHEET #24-7: ANALYTICAL INSTRUMENT CALIBRATION – METALS – WATER 83

WORKSHEET #24-8: ANALYTICAL INSTRUMENT CALIBRATION – SAMPLE PREPARATION 86

WORKSHEET #24-9: ANALYTICAL INSTRUMENT CALIBRATION – ANALYTICAL BALANCE 90

WORKSHEET #24-10: ANALYTICAL INSTRUMENT CALIBRATION – TOTAL SUSPENDED SOLIDS 91

WORKSHEET #24-11: ANALYTICAL INSTRUMENT CALIBRATION – TOTAL DISSOLVED SOLIDS 92

QAPP WORKSHEET #28: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION 93

WORKSHEET #28-1: LABORATORY QC SAMPLES (RSK-175) 93

WORKSHEET #28-2: LABORATORY QC SAMPLES TABLE (WET CHEMISTRY – INORGANIC IONS BY IC (CL, SO_4) 95

WORKSHEET #28-3: LABORATORY QC SAMPLES (ALKALINITY) 97

WORKSHEET #28-4: LABORATORY QC SAMPLES (ANIONS/9056)........ 99

WORKSHEET #28-5: LABORATORY QC SAMPLES (TOC)........ 101

WORKSHEET #28-6: LABORATORY QC SAMPLES (DOC) 102

WORKSHEET #28-7: LABORATORY QC SAMPLES (PERCENT MOISTURE) 103

WORKSHEET #28-8: LABORATORY QC SAMPLES (METALS-SOIL/SEDIMENT) 104

WORKSHEET #28-9: LABORATORY QC SAMPLES (METALS-WATER)........ 106

WORKSHEET #28-10: LABORATORY QC SAMPLES (METALS-HG-WATER) 108

WORKSHEET #28-11: LABORATORY QC SAMPLES (METALS-HG-SOIL/SEDIMENT) 110

WORKSHEET #28-12: LABORATORY QC SAMPLES (PH)........ 112

WORKSHEET #28-13: LABORATORY QC SAMPLES (GRAIN SIZE)........ 113

WORKSHEET #28-14: LABORATORY QC SAMPLES (TSS) 114

WORKSHEET #28-15: LABORATORY QC SAMPLES (TDS) 116

REFERENCES........ 118

ATTACHMENTS

Attachment 1	Field Sampling Standard Operating Procedures
Attachment 2	Laboratory Standard Operating Procedures
Attachment 3	Laboratory Certifications

Acronyms and Abbreviations

µg/L	microgram per liter
µg/kg	microgram per kilogram
--	??
>	greater than
<	less than
≤	less than or equal to
°C	degree Celsius
%RSD	percent relative standard deviation
AASHTO	American Association of State Highway and Transportation Officials
Al	aluminum
AOC	area of contamination
Army	U.S. Army
ASTM	ASTM International
Br	bromine
BRAC	Base Realignment and Closure
bss	below sediment surface
Ca	calcium
$CaCO_3$	calcium carbonate
CAS	Chemical Abstracts Service
CCB	continuing calibration blank
CCV	continuing calibration verification
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
Cl	chlorine
CSM	conceptual site model
DCL	Fort Devens Consolidation Landfill
DL	detection limit
DO	dissolved oxygen
DOC	dissolved organic carbon
DoD	U.S. Department of Defense
DQE	data quality evaluation
DQI	data quality indicator
DQO	data quality objective
EDMS	electronic data management system
ELLE	Eurofins Lancaster Laboratories Environmental, LLC
F	flourine
Fe	iron
FID	flame ionization detector
GC	gas chromatography
g/kg	gram per kilogram
HGL	HydroGeoLogic, Inc.
HNO_3	nitric acid
IC	ion chromatography
ICAL	initial calibration
ICB	initial calibration blank
ICP	inductively coupled plasma
ICS	interference check sample
ICV	initial calibration verification

LIST OF ACRONYMS AND ABBREVIATIONS (continued)

ISO	International Organization for Standardization
K	potassium
KGS	Koman Government Solutions, LLC
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
LTM	long-term monitoring
LTMMP QAPP	Quality Assurance Project Plan, Annual Long-Term Monitoring and Maintenance Program
LTMMP	Long-Term Monitoring and Maintenance Plan
MassDEP	Massachusetts Department of Environmental Protection
MassDevelopment	Massachusetts Development and Finance Agency
MCL	maximum contaminant level
MDL	method detection limit
Mg	magnesium
mg	milligram
mg C	tonne of Carbon
mg/L	milligram per liter
mL	milliliter
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
Na	sodium
NA	not applicable
ng/L	nanogram per liter
NO_2	nitrogen dioxide
NO_3	nitrate
ORP	oxidation-reduction potential
PAL	project action level
PE	Professional Engineer
PG	Professional Geologist
PM	Project Manager
PO_4	phosphate
ppm	part per million
PSP	Plow Shop Pond
QA	quality assurance
QAPP Addendum	Draft Addendum Uniform Federal Policy for Quality Assurance Project Plan – Annual Long-Term Monitoring and Maintenance Program
QC	quality control
QSM	Quality Systems Manual
r^2	coefficient of determination
RL	reporting limit
RPD	relative percent difference
RSD	relative standard deviation
S	sulfur
SEP	sequential extraction procedure
SERES-Arcadis JV	SERES-Arcadis 8(a) Joint Venture 2, LLC
SHL	Shepley’s Hill Landfill
Si	silicon
Site	former Fort Devens Army Installation in Devens, Massachusetts

SL	screening level
SO_4	sulfate
SOP	standard operating procedure
Sovereign	Sovereign Consulting, Inc.
TDS	total dissolved solids
TOC	total organic carbon
TSS	total suspended solids
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
USACE	U.S. Army Corps of Engineers
USEPA	United States Environmental Protection Agency
VOC	volatile organic compound

QAPP WORKSHEETS #1 AND #2: TITLE AND APPROVAL PAGE

This worksheet identifies the principal points of contact for all organizations having decision authority in the project and documents their commitment to implement the UFP-QAPP (Intergovernmental Data Quality Task Force 2005). Signatories usually include the Project Manager (PM) and QA Manager, and individuals with approval or oversight authority. Signatures indicate that officials have reviewed the UFP-QAPP and concur with the implementation as written. It is the lead organization's responsibility to make sure all signatures are in place before work begins.

1. Project Identifying Information:
    a. Site name/project name: Former Fort Devens Army Installation, Barrier Wall Performance Monitoring
    b. Site location: Devens, Massachusetts
    c. Contract/work assignment number: SERES-Arcadis JV, Contract No. W912WJ-19D-0014, Contract Delivery Order No. W912WJ-20-F-0022, Environmental Services and Remedial Action Operations for BRAC Legacy Sites – Former Fort Devens, Devens, MA
    d. Lead Organization: USEPA Region 1 and U.S. Army Corps of Engineers (USACE)
    e. Geographical Corps District: New England District
2. Department of Defense Organization(s):
    a. U.S. Army (Army), Base Realignment and Closure (BRAC)
        i. Name: Thomas Lineer
        ii. Title: BRAC Environmental Coordinator
    b. USACE, New England District (CENAE)
        i. Name: Penelope Reddy
        ii. Title: USACE PM
        iii. Signature: ______________________________ Date: ______________

3. Contractor: SERES-Arcadis JV
    a. SERES-Arcadis JV PM
        i. Name: Andy Vitolins, Professional Geologist (PG)
        ii. Title: SERES-Arcadis JV PM
        iii. Signature: ______________________________ Date: 05/09/2022
    b. SERES-Arcadis JV Corporate QA Manager
        i. Name: John Nocera, Professional Engineer (PE)
        ii. Corporate Quality Manager
        iii. Signature: John J Nocera ______________________________ Date: 05/09/2022
4. Federal Regulatory Agency: USEPA Region I
5. State/Territory Regulatory Agency: MassDEP
6. Other Stakeholders:
    a. Massachusetts Development and Finance Agency (MassDevelopment)
    b. Restoration Advisory Board
    c. Local residents and businesses
7. Plans and reports from previous investigations, presented in the following table.

Plans and Reports from Previous Investigations Relevant to this Project	1995	Record of Decision, Shepley's Hill Landfill Operable Unit, Fort Devens, Massachusetts. Fort Devens Feasibility Study for Group 1A Sites	U.S. Army Environmental Center
	2008	Final Report Arsenic Fate, Transport and Stability Study – Groundwater, Surface Water, Soil and Sediment Investigation – Fort Devens Superfund Site, Devens, Massachusetts.	USEPA
	2010	Remedial Investigation for AOC 72, Plow Shop Pond – Draft. Devens, Massachusetts.	AMEC
	2015	Record of Decision for Plow Shop Pond (AOC 72) – Red Cove and Former Railroad Round House (SA-71), Former Fort Devens Army Installation, Devens, Massachusetts	Sovereign Consulting Inc. (Sovereign)
	2015	Long Term Monitoring and Maintenance Plan Update – Shepley's Hill Landfill	Sovereign
	2018	Addendum to the Long-Term Monitoring and Maintenance Plan, Shepley's Hill Landfill. Former Fort Devens Army Installation, Devens, Massachusetts.	Koman Government Solutions, LLC (KGS)
	2020	Draft 2019 Annual Operations, Maintenance, and Monitoring Report, Shepley's Hill Landfill, Former Fort Devens Army Installation	KGS
	2020	Final Fifth 2020 Five-Year Review Report, Former Fort Devens Army Installation, BRAC Legacy Sites, Devens, Massachusetts.	KGS
	2021	Final 2020 Annual Operations, Maintenance, and Monitoring Report. Shepley's Hill Landfill, Former Fort Devens Army Installation, Devens, Massachusetts.	SERES-Arcadis JV

Required UFP-QAPP (Intergovernmental Data Quality Task Force 2005) elements and information that are not included in the LTMMP QAPP with elements specific to the barrier wall investigation are indicated in the table below with their location in this QAPP Addendum. Required UFP-QAPP elements that are included in the LTMMP QAPP without need for modification for the barrier wall investigation are indicated as not applicable (NA).

Worksheet #	**Required Information**	**Crosswalk to Related Information**
A. Project Management and Objectives		
Documentation		
1	Title and Approval Page	Page 1
2	UFP-QAPP Identifying Information	Page 1
3	Distribution List	Page 5
4	Project Personnel Sign-Off Sheet	Page 6
Project Organization		
5	Project Organizational Chart	Page 6
6	Communication Pathways	NA
7	Personnel Responsibilities and Qualifications Table	NA
8	Special Personnel Training Requirements Table	NA
Project Planning/Problem Definition		
9	Project Scoping Session Participants Sheet	NA
10	Conceptual Site Model	Page 7
11	Data Quality Objectives	Page 9
12	Measurement Performance Criteria Table	Page 11
13	Secondary Data Criteria and Limitations	Page 20
14	Project Tasks and Schedule	Page 21
15	Project Action Limits and Laboratory-Specific Detection/Quantitation Limits	Page 22
16	Project Tasks and Schedule	Page 21
B. Measurement/Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	Page 41
18	Sample Locations and Methods	Page 44
19	Sample Containers, Preservation, and Hold Times	Page 45
20	Field QC Sample Summary	Page 48
21	Project Sampling SOP References	Page 50
22	Field Equipment Calibration, Maintenance, Testing, and Inspection	Page 51

Worksheet #	**Required Information**	**Crosswalk to Related Information**
Analytical Tasks		
23	Analytical Standard Operating Procedures	Page 52
24	Analytical Instrument Calibration	Page 54
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	NA
Sample Collection		
26	Sample Handling System, Documentation Collection, Tracking, Archiving and Disposal, and Custody SOPs Sample Handling Flow Diagram	NA
27	Sample Custody Requirements	NA
Quality Control Samples		
28	Analytical Quality Control and Corrective Action	Page 69
Data Management Tasks		
29	Project Documents and Records Table	NA
30	Analytical Services Table	NA
C. Assessment Oversight		
31	Planned Project Assessments Table	NA
32	Assessment Findings and Corrective Action Responses Table	NA
33	QA Management Reports Table	NA
D. Data Review		
34	Verification (Step I) Process Table	NA
35	Validation (Steps IIa and IIb) Process Table	NA
36	Validation (Steps IIa and IIb) Summary Table	NA
37	Data Usability Assessment Table	NA

Note:

SOP = standard operating procedure

QAPP WORKSHEET #3: DISTRIBUTION LIST FOR FORT DEVENS

QAPP Addendum Recipients	Title	Organization	Telephone Number	E-mail Address
Alex Lo	Program Manager	SERES-Arcadis JV	(843) 388-7804	ahlo@seres-es.com
Andy Vitolins	PM	SERES-Arcadis JV	(518) 461-3145	Andy Vitolins
John Nocera	Corporate QC Manager	SERES-Arcadis JV	(251) 405-4560	john.nocera@arcadis.com
Grey Coppi	Health and Safety Manager	SERES-Arcadis JV	(732) 661-3851	grey.coppi@arcadis.com
Ian Martz	Field Operations Lead	SERES-Arcadis JV	(978) 322-4526	ian.martz@arcadis.com
Michael Shivell	Task Lead	SERES-Arcadis JV	(585) 662-4031	michael.shivell@arcadis.com
Jennifer Singer	Project Chemist	SERES-Arcadis JV	(716) 667-6664	jennifer.singer@arcadis.com
Nathan Mullens	Contract PM	SERES-Arcadis JV	(843) 478-0366	nrmullens@seres-es.com
Melissa McDermott	Laboratory PM	Eurofins TestAmerica – Lancaster	(717) 556-7359	melissa.mcdermott@eurofinset.com
Pei Geng	Data Validation PM	Laboratory Data Consultants, Inc.	(760) 827-1110	pgeng@lab-data.com
Penelope Reddy	PM	USACE	(978) 318-8160	penelope.reddy@usace.army.mil
Yixian Zhang	Project Chemist	USACE	(978) 318-8730	yixian.zhang@usace.army.mil
Thomas Lineer	BRAC Program Manager	Army	(703).371-6785	thomas.a.lineer.civ@army.mil
Carol Keating	Remedial PM	USEPA Region I	(617) 918-1393	keating.carol@epa.gov
David Chaffin	Federal Sites Program	MassDEP	(617) 348-4005	david.chaffin@state.ma.us
Robert Lim	Remedial PM	USEPA Region I	(617) 918-1392	lim.robert@epa.gov

QAPP WORKSHEET #4: PERSONNEL SIGN-OFF SHEET

Signatures for the SERES-Arcadis JV personnel responsible for completion of the work associated with this QAPP Addendum are provided below. Confirmation of QAPP Addendum receipt will be requested from Eurofins TestAmerica Lancaster once the document is final.

ORGANIZATION: SERES-Arcadis JV

Name	Project Title/Role	Telephone Number	Signature/Date[2]
Alex Lo, PE	Program Manager		Signature on file
Andy Vitolins, PG	PM		Signature on file
John Nocera, PE, BCEE	Corporate Quality Manager		Signature on file
Shawn Atkinson	Federal Contracts Manager		Signature on file
Grey Coppi, CSP, CIH	Health and Safety Officer		Signature on file
Jennifer Singer	Project Chemist		Signature on file
Whitney Plasket, PE (Maine)	Senior Environmental Engineer		Signature on file
Mike Shivell, PE (New York)	Task Manager		Signature on file
Britt McMillan	Geologist/Hydrogeologist		Signature on file
Mike Hay	Geochemist		Signature on file

Note:
[1] Signatures indicate that personnel have read and agree to implement this QAPP Addendum as written (signatures required for final submittal only).

ORGANIZATION: Eurofins TestAmerica (primary laboratory)

Name	Project Title/Role	Education/Experience	Signature/Date[1]
Melissa McDermott	Laboratory PM	Representative for project laboratory	Signature on file
Kim Chamberlain	Laboratory QA Manager	Representative for project laboratory	Signature on file

Note:
[1] Signatures indicate personnel have read and agree to implement this QAPP Addendum as written (signatures required for final submittal only).

ORGANIZATION: Laboratory Data Consultants, Inc. (Data Validator)

Name	Project Title/Role	Education/Experience	Signature/Date[1]
Pei Geng	Data Validation PM	Representative for project data validation.	Signature on file

Note:
[1] Signatures indicate personnel have read and agree to implement this QAPP Addendum as written (signatures required for final submittal only).

QAPP WORKSHEET #10: CONCEPTUAL SITE MODEL

10.1 Site Description and History

SHL encompasses approximately 84 acres in the northeast corner of the main post of the Site. The landfill is bordered to the northeast by PSP, to the west by Shepley’s Hill, to the south by recent commercial development, and to the east by land formerly containing a railroad roundhouse. Nonacoicus Brook, which drains PSP, is located north of the landfill in the North Impact Area. Figure 1 in the Barrier Wall Performance Monitoring Work Plan shows the SHL site location.

10.2 Site Constituents of Concern

Contaminants of concern include arsenic and volatile organic compounds (VOCs). Since closure of SHL in the 1990s, several remedial efforts have been implemented to address arsenic impact, migration, and exposure at SHL (KGS 2020), as listed below:

- Landfill capping
- Groundwater monitoring
- Installation and operation of a groundwater extraction and treatment system
- Barrier wall installation between SHL and Red Cove area of PSP
- Excavation of contaminated Red Cove sediments following the Barrier Wall installation
- Land use controls in SHL and the North Impact Area.

10.3 Key Physical Aspects of the Site

10.3.1 Site Groundwater Hydrology

In general, groundwater in the southern portion of SHL flows to the northeast (prior to installation of the barrier wall, toward PSP), groundwater in the northern portion of SHL flows to the north, and groundwater to northeast of SHL flows to the northwest. The depth to groundwater in the barrier wall area in 2020 ranged from approximately 19 to 28 feet below ground surface. Section 1.2 in the Barrier Wall Performance Monitoring Work Plan contains more details on the hydrology at SHL.

10.3.2 Site Geology

SHL is located within a bedrock valley, and the overburden deposits beneath the landfill consist of glacially deposited, well-graded to poorly graded sands with silts and gravel. A discontinuous layer of till is present at the base of the sands, directly overlying bedrock. The hydrostratigraphic unit within the overburden comprises primarily medium and fine-medium sands with little variability.

10.4 Primary Release Mechanism/Fate and Transport Consideration

Prior to the installation of the barrier wall, groundwater in the area flowed to the northeast toward Red Cove and PSP. Arsenic flux to Red Cove prior to the installation of the barrier wall was documented in several reports, notably the Arsenic Fate, Transport and Stability Study (USEPA 2008), Remedial Investigation for AOC 72, Plow Shop Pond – Draft (AMEC 2010), and various USEPA presentations. Following installation of the barrier wall, groundwater flow toward Red Cove and PSP was reduced. As presented in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a), groundwater elevation contours in the barrier wall area showed some mounding of groundwater upgradient of the wall (to the west) compared to downgradient of the wall (to the east). This is expected based on the low hydraulic conductivity of materials used for construction of the barrier wall. The groundwater elevation contours also show groundwater moving to the north along the upgradient (western) side of the barrier wall, which effectively reduces the amount of groundwater flow from SHL to PSP. The Long-Term Monitoring and Maintenance Plan Update (Sovereign 2015b) and subsequent Addendum to the Long-Term Monitoring and Maintenance Plan (KGS 2018) include the monitoring required to evaluate the hydraulic performance of the barrier wall.

Mass flux of arsenic has, in the past, been estimated over segments of the surficial aquifer between SHL and PSP as well as mass flux to Red Cove within PSP:

- **Arsenic flux across the Barrier Wall**: Mass flux of arsenic across the barrier wall and to Red Cove has, pre- and post-barrier installation, been estimated over segments of the surficial aquifer by calculating groundwater flow across the barrier wall and average upgradient dissolved arsenic concentrations, as presented in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a). Mass discharge of arsenic in the overburden across the barrier wall was calculated from Darcy velocity using 3PE vector analysis results and 2020 dissolved arsenic concentration data. This method conservatively assumed that groundwater flows from west to east across the wall, rather than to the northwest, as the 3PE analysis indicates. Based on this analysis, arsenic flux (q*C) across the wall taking into consideration the full cross-sectional areas of flow in 2020 ranged from 0.6 gram per year (g/year) in fall to 2.4 g/year in spring. This flux is approximately 3 orders of magnitude less than the estimated pre-wall flux calculated at 686 g/year across the same aquifer cross section (Appendix E in SERES-Arcadis JV 2021a).
- **Arsenic flux to Red Cove:** Mass discharge of arsenic to Red Cove was previously calculated by the USEPA Office of Research and Development (ORD) for pre- and post-barrier wall conditions (Ford et al. 2018). The pre-barrier wall arsenic flux for 2011 was 39.4 g/year/m^2. Following installation of the barrier wall, arsenic flux was estimated as 1.8 to 9.1 g/year/m^2. Using the same methodology, post-barrier wall arsenic flux to Red Cove was estimated in 2020 as 1.3 g/year/m^2. The 2020 flux estimate is consistent with the previous post-barrier wall flux estimated by the ORD.

The 3PE analysis used to assess barrier wall performance in Appendix E of the 2020 Annual Operations, Maintenance, and Monitoring Report (SERES-Arcadis JV 2021a) indicated that the barrier wall is effectively reducing groundwater flow from SHL to Red Cove and PSP.

PSP is the furthest downstream pond in a series of six ponds that discharge to Nonacoicus Brook. While the majority of water in PSP originates from the upstream ponds, there is local contribution from groundwater seepage. As presented in the Remedial Investigation for AOC 72, Plow Shop Pond – Draft (AMEC 2010), low flow, measured as the 7-day, 10-year low flow at the outlet of PSP, was estimated as 2.6 cubic feet per second. Bathymetry and sediment thickness were measured in 1999 by the U.S. Geological Survey (Mercadante et al. 1999). While these measurements predate excavation in the Red Cove area of PSP, they provide good general information on depths and sediment thickness. At the time of the survey, the deepest part of PSP was 8 feet, near the center, and the greatest sediment thickness was 18 feet along the western side of the pond, east of Red Cove. Within Red Cove, pre-excavation water depths were less than 2.5 feet and sediment thickness was less than 8 feet.

10.5 Data Gaps

As described in the Introduction to the Barrier Wall Performance Monitoring Work Plan, on June 22 and December 7, 2020, MassDEP and the USEPA, respectively, provided comments on the Barrier Wall Analysis provided in Appendix G of the Draft 2019 Annual Long-Term Monitoring and Maintenance Report for the SHL (KGS 2020). The comments stated that the evaluations of barrier wall performance were insufficient because they were based on estimates of arsenic flux and indicated that the barrier wall performance evaluation will need to be based on direct measurements of porewater and other media. In addition, to evaluate barrier wall performance and verify continued attainment of the Record of Decision (U.S. Army Environmental Center 1995) remedial action objective for PSP, the USEPA recommended the performance of a minimum of two rounds of media sampling (sediment and porewater) and toxicity testing prior to the 2025 five-year review. Accordingly, the U.S. Army issued a Scope of Work in February 2021 to SERES-Arcadis JV to develop a plan to conduct the appropriate analyses to evaluate arsenic flux through the barrier wall and to PSP (USACE 2021). The results from this work will then be incorporated into the revised long-term monitoring and maintenance plan (LTMMP) for SHL along with any other changes required as part of the Phase I and Phase II work or monitoring program. The Work Plan and this accompanying QAPP Addendum are being submitted in accordance with the Scope of Work and describe the sampling activities to be completed to collect the additional data requested by the USEPA and MassDEP.

The project goals/data gaps to be addressed are described below along with the data use objectives (DUOs) for the project:

Table 1 Project Goals and Data Use Objectives

Goal / DUO #	Goal	Decision Question	DUO
1	Define current conditions in Red Cove	Is the arsenic concentration in surface sediment greater than 270 mg/kg (PRG from NTCRA)?	Document the current levels of arsenic in Red Cove sediment, porewater, and surface water.
2	Quantify arsenic flux from SHL to the Red Cove area of PSP	Is there arsenic flux from SHL to PSP and if so, how much?	Develop a flux estimate for arsenic from the upgradient area of SHL to the downgradient area and from the area between the barrier wall and Red Cove to the Red Cove area of PSP.

Goal / DUO #	Goal	Decision Question	DUO
3	Identify the ongoing source of arsenic present in PSP	Is arsenic in Red Cove surface water a result of arsenic cycling within the aquatic system (i.e., the hyporheic zone) and if not, what other sources are contributing?	Understand the form, mobility, and stability of iron and co-occurring arsenic in groundwater, porewater, surface water, soil, and sediment at SHL and in the Red Cove area of PSP.
4	Develop recommendations to monitor Red Cove for the attainment of remedial action objectives through time, if needed.	Based on the results of DUOs 1 through 3, what locations, media, and sample frequency are needed for long-term monitoring of Red Cove?	Define the geographic extent of groundwater influence from SHL to PSP in the post barrier wall condition to refine the Red Cove monitoring area.

Notes:
DUO = data use objective
mg/kg = milligram per kilogram
PRG = preliminary remediation goal
NTCRA=non-time-critical removal action

QAPP WORKSHEET #11: DATA QUALITY OBJECTIVES

DQOs define the type, quantity, and quality of data that are needed to answer project-specific questions and support project-specific decisions:

DQO	Problem Statement	Project Goals	Information Inputs	Study Boundaries	Decision Rules	Performance Criteria	Plan for Obtaining Data
Explanation	*Define the problem that necessitates this study*	*Identify study questions*	*Identify data and information needed to answer study questions*	*Specify the spatial and temporal boundaries.*	*Define the conditions under which the data will be utilized.*	*Specify the limits on decision errors*	*Design an effective data collection strategy based on previous steps*
SHL, Red Cove area od PSP, and PSP	The problem statement is described in detail in Section 2.1 of the Work Plan. As a summary, arsenic has been detected in surface water at concentrations greater than criteria. Additional data are needed to determine current conditions and arsenic flux to the Red Cove area of PSP.	The project goals and DUOs are defined in Section 2.2. of the Work Plan and summarized above in **Worksheet #10**, Section 10.5.	The summary of decision inputs is described in Section 2.3 of the Work Plan and in **Worksheet #17**. The sampling locations and required analysis are presented in **Worksheet #18**. A complete listing of the target analytes is provided in **Worksheet #15**.	The investigation area has been divided into four distinct areas, as described in Section 2.4 of the Work Plan: • Groundwater upgradient of the barrier wall • Groundwater and soil between the barrier wall and Red Cove • Aquatic environment of the Red Cove area of PSP • Aquatic environment of PSP outside of Red Cove	The decision rules for the project are described in Section 2.5 of the Work Plan. All samples will be collected and analyzed in accordance with the QAPP Addendum SOP documents to ensure that subsequent decisions are made based on valid data.	The limits of decision errors for the project are described in Section 2.6 of the Work Plan Project-specific MPCs, which are the criteria that collected data must meet to satisfy the DQOs, are presented in **Worksheet #12**.	The plan for obtaining the data necessary to meet the goals of the Work Plan is detailed Section 3 of the Work Plan and in **Worksheet #17** of the QAPP Addendum.

QAPP WORKSHEET #12: MEASUREMENT PERFORMANCE CRITERIA

Measurement performance criteria (MPC) for field QC sampling results are used to evaluate project data quality indicators (DQIs) such as accuracy/bias and precision. The following tables define different laboratory and field blanks that will be used as QC measures to evaluate the site-specific DQIs. MPC are provided below by matrix and analytical group in Worksheets #12A through #12G.

QAPP WORKSHEET #12A: MEASUREMENT PERFORMANCE CRITERIA (CARBON DIOXIDE)

Matrix: Water
Concentration Level: Low
Analytical Method: CO2 RSK175/ WI9644

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision	Field duplicates	RPD ≤20%
Accuracy/bias	MS/MSD	Lab statistical recoveries and RPD ≤20%
Accuracy/bias	LCS	Lab statistical recoveries and RPD ≤20%
Accuracy/field contamination	Field blank	< LOQ
Accuracy/lab contamination	Method blank	< LOQ
Accuracy/transport contamination	Trip blank	< LOQ
Accuracy/holding time	Reported sample data	14 days
Completeness	Reported sample data	As determined by the project
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

≤ = less than or equal to
LCS = laboratory control sample
MSD = matrix spike duplicate
PAL = project action level
RPD = relative percent difference
LOQ = limit of quantitation
LOD = limit of detection

DL = detection limit

QAPP WORKSHEET #12B: MEASUREMENT PERFORMANCE CRITERIA (INORGANIC IONS BY IC [CL, SO_4] IN WATER)

Matrix: Water
Concentration Level: Low
Analytical Method: Wet Chemistry – Inorganic Ions by IC (Cl, SO_4)

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/bias	LCS/MS	Laboratory limits
Accuracy/laboratory contamination	Method blanks	No analytes detected <MDL or >1/10 the amount measure in any sample.
Precision	Lab duplicate	Laboratory statistical limits
Precision	Field duplicate	RPD ≤ 30%
Accuracy/field contamination	Field blank	< LOQ
Completeness	Reported sample data	As determined by the project
Bias/holding time	Reported sample data	SO_4, Cl ≤28 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

\> = greater than
< = less than
Cl = chlorine
IC = ion chromatography
MDL = method detection limit
MS = matrix spike
SO_4 = sulfate

QAPP WORKSHEET #12C: MEASUREMENT PERFORMANCE CRITERIA (ALKALINITY)

Matrix: Water
Concentration Level: Low
Analytical Method: Alkalinity

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/bias	LCS/MS	Laboratory statistical window
Accuracy/laboratory contamination	Method blank	No analytes detected >LOQ or >1/10 the amount measured in any sample
Precision	Lab duplicate	Laboratory statistical RPD
Precision	Field duplicate	RPD ≤ 30%
Accuracy/field contamination	Field blank	< LOQ
Completeness	Reported sample data	As determined by the project
Bias/holding time	Reported sample data	≤14 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Note:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

QAPP WORKSHEET #12D: MEASUREMENT PERFORMANCE CRITERIA (TOC)

Matrix: Soil / Sediment
Concentration Level: Low
Analytical Method: TOC

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/bias	LCS/MS	Laboratory statistical window
Accuracy/laboratory contamination	Method blank	No analytes detected >LOQ or >1/10 the amount measured in any sample
Precision	Lab duplicate	Laboratory statistical RPD
Precision	Field duplicate	RPD ≤ 30%
Accuracy/field contamination	Field blank	<LOQ
Completeness	Reported sample data	As determined by the project
Bias/holding time	Reported sample data	≤14 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

TOC = total organic carbon

QAPP WORKSHEET #12E: MEASUREMENT PERFORMANCE CRITERIA (DOC)

Matrix: Water
Concentration Level: Low
Analytical Method: DOC

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/bias/precision	LCS/MS	Laboratory statistical limits and RPD
Accuracy/laboratory contamination	Method blank	No analytes detected >LOQ or >1/10 the amount measured in any sample
Precision	Lab duplicate	Laboratory statistical RPD
Precision	Field duplicate	RPD ≤ 30%
Accuracy/field contamination	Field blank	< LOQ
Completeness	Reported sample data	As determined by the project
Bias/holding time	Reported sample data	≤28 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

DOC = dissolved organic carbon

QAPP WORKSHEET #12F: MEASUREMENT PERFORMANCE CRITERIA (METALS – ICP-MS)

Matrix: Soil / Sediment and water
Concentration Level: Low
Analytical Method: Metals – ICP-MS

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/bias/precision	LCS/MS and their duplicates	Recovery limits per Quality Systems Manual, Version 5.3 (DoD 2019a). For elements not in the Quality Systems Manual, use ±25% for MS and ±20% for LCS; RPD ≤20%
Accuracy/laboratory contamination	Method blank	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample
Precision	Lab duplicate	RPD ≤20%
Precision	Field duplicate	RPD ≤ 30%
Accuracy/field contamination	Field blank	< LOQ
Completeness	Reported sample data	As determined by the project
Bias/holding time	Reported sample data	≤ 6 months
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

ICP = inductively coupled plasma

QAPP WORKSHEET #12G: MEASUREMENT PERFORMANCE CRITERIA (PH)

Matrix: Soil / Sediment

Concentration Level: --

Analytical Method: Metals – pH

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision	Duplicates	Laboratory statistical RPD
Accuracy/Bias	LCS	Laboratory statistical window
Bias/Holding Time	Reported Sample Data	NA
Completeness	Reported Sample Data	As determined by project

Notes:

1. The above table complies with the requirements of Quality Systems Manual, Version 5.3 (DoD 2019a).

ICP = inductively coupled plasma

QAPP WORKSHEET #12H: MEASUREMENT PERFORMANCE CRITERIA (GRAIN SIZE)

Matrix: Solid
Concentration Level: Low
Analytical Method: Metals – Grain Size

DQIs	QC Sample of Measurement Performance Activity	MPC
Precision	Lab Duplicate	Laboratory statistical RPD
Precision	Field Duplicate	RPD as set by project
Completeness	Reported Sample Data	As determined by the project
Bias/Holding Time	Reported Sample Data	NA

QAPP WORKSHEET #12I: MEASUREMENT PERFORMANCE CRITERIA (TSS)

Matrix: Water
Concentration Level: Low
Analytical Method: Metals – Wet Chemistry – Total Suspended Solids (TSS)

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/Bias	Laboratory Control Spike	Laboratory statistical windows
Accuracy/Laboratory Contamination	Method Blank	No analytes detected > LOQ or >1/10 the amount measured in any sample
Precision	Lab Duplicate	Method specified RPD
Precision	Field Duplicate	RPD as set by project
Accuracy/Field Contamination	Field Blank	No detected target compounds
Completeness	Reported Sample Data	As determined by the project
Bias/Holding Time	Reported Sample Data	≤ 7 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

QAPP WORKSHEET #12J: MEASUREMENT PERFORMANCE CRITERIA (TDS)

Matrix: Water
Concentration Level: Low
Analytical Method: Metals – Wet Chemistry – Total Dissolved Solids (TDS)

DQIs	QC Sample of Measurement Performance Activity	MPC
Accuracy/Bias	Laboratory Control Spike	Laboratory statistical windows
Accuracy/Laboratory Contamination	Method Blank	No analytes detected > LOQ or >1/10 the amount measured in any sample
Precision	Lab Duplicate	Method specified RPD
Precision	Field Duplicate	RPD as set by project
Accuracy/Field Contamination	Field Blank	No detected target compounds
Completeness	Reported Sample Data	As determined by the project
Bias/Holding Time	Reported Sample Data	≤ 7 days
Sensitivity	LOQs, LODs, DLs	LOQs ≤PALs

QAPP WORKSHEET #13: SECONDARY DATA CRITERIA AND LIMITATIONS

Secondary Data	Data Source (originating organization, report title, and date)	Data Generator(s) (originating organization, data types, data generation/ collection dates)	How Data Will Be Used	Limitations on Data Use
Annual LTM Program Reports	Annual reports for individual AOCs: AOC 32/ and 43A, AOC 43G, AOC 57, AOC 69W, DCL, Sudbury Annex, SHL, and AOC 50.	Historical groundwater, surface water, sump water, and leachate sampling summary data for respective AOCs: 2000 through current.	Compared to historical results to identify spatial and temporal trends at respective AOCs.	None, except as identified for individual data points in the associated DQE.
LTMMP	Current LTMMPs for all AOCs.	LTMMP groundwater sampling summary and analytical data.	Compared to current results to identify spatial and temporal trends at respective AOCs	None
DCL Permit Compliance	Industrial Pretreatment Self-Monitoring Reports	Landfill leachate data: October 2006 through current.	Identify changes in discharge concentrations	None, except as identified for individual data points in the associated DQE

Notes:
DCL = Fort Devens Consolidation Landfill
DQE = data quality evaluation
LTMMP = Long-Term Monitoring and Maintenance Plan

QAPP WORKSHEETS #14 AND 16: PROJECT TASKS AND SCHEDULE

(UFP-QAPP Manual Section 2.8.2)
(USEPA 2106-G-05 Section 2.2.4)

The project schedule, including the specific task/activity and person or group responsible for execution is presented in the following table. The complete schedule for this project timeline, including planning, document preparation, field activities, report preparation, and meeting support, is provided as an attachment in the Final QAPP Addendum. Note that the schedule is subject to updates and revisions based on field conditions.

Activity	Responsible Party	Deliverable(s)	Deliverable Due Date
DFW 1: Pre-Mobilization Activities			
Final QAPP Addendum	Andy Vitolins SERES-Arcadis JV PM	Draft and Draft Final QAPP Addendum submittals	One week after resolution of Draft Final QAPP Addendum comments.
DFW 2: Mobilization/Site Preparation			
Mobilization	Ian Martz SERES-Arcadis JV Field Supervisor	Field notes, DQCR	Weekly submittals due Friday of the week following performance of the activity.
DFW 3: Environmental Data Collection			
Field Activities	Monique De Jesus SERES-Arcadis JV Task Manager	Data package, including data validation summary	Twelve weeks after completion of field activities.
DFW 4: Final Report			
Draft Report	Andy Vitolins SERES-Arcadis JV PM	Draft Report	Approx. one year after completion of field activities.
Draft Final/Final Report	Andy Vitolins SERES-Arcadis JV PM	Draft Final/Final Report	45 days after receipt of Draft/Draft Final comment resolution.

QAPP WORKSHEET #15: PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/QUANTITATION LIMITS

The following Worksheet #15 tables identify the PALs and provide a comparison of the PALs to analytical laboratory detection limits (i.e., limit of detections [LODs] and LOQs) for groundwater, surface water, sediment, and porewater per analytical method. The objective is for the laboratory to achieve LOQs low enough to measure analytes at concentrations less than the PALs to obtain a dataset of known quality and sufficient sensitivity to meet project DQOs.

The laboratory reference limits are provided by the selected laboratory: Eurofins TestAmerica, located in Lancaster, Pennsylvania.

The analytical laboratory reference limits presented in Worksheet #15 tables are as follows:

- *LOD*. The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a nondetect (ND) of a specific analyte in a specific matrix with a specific method at 99% confidence.
- *LOQ*. The lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias.

The worksheets listed below provide laboratory detection limits and evaluation of constituents in groundwater, surface water, sediment, and porewater:

- Worksheet #15-1: Metals in Groundwater
- Worksheet #15-2: Anions in Groundwater
- Worksheet #15-3: Alkalinity in Groundwater
- Worksheet #15-4: DOC in Groundwater
- Worksheet #15-5: Dissolved Gases in Groundwater
- Worksheet #15-6: Metals in Surface water
- Worksheet #15-7: Anions in Surface water
- Worksheet #15-8: Alkalinity in Surface water
- Worksheet #15-9: DOC in Surface water
- Worksheet #15-10: Metals ICP in Soil / Sediment
- Worksheet #15-11: TOC in Soil / Sediment
- Worksheet #15-12: Percent Moisture in Sediment
- Worksheet #15-13: Metals in Porewater
- Worksheet #15-14: Anions in Porewater
- Worksheet #15-15: Alkalinity in Porewater

- Worksheet #15-16: DOC in Porewater
- Worksheet #15-17: TOC and Grain Size in Soil / Sediment.
- Worksheet #15-18: Total Suspended Solids and Total Dissolved Solids in Surface water, Porewater, and Groundwater.

WORKSHEET #15-1: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS IN GROUNDWATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (µg/L)		
		MassDEP GW-1 MCL[a] (µg/L)	USEPA Site-Specific SL[b] µg/L	LOQ	LOD	DL
Analytical Group: Metals 6020B_DOD5						
Arsenic	7440-38-2	10	0.01	2.00	1.60	0.680
Aluminum	7429-90-5	50	20	35	30	19.7
Barium	7440-39-3	2,000	2.0 E+03	2.00	1.60	0.746
Calcium	7440-70-2	--	--	125	120	73.6
Iron	7439-89-6	30	14	50	40	22.8
Magnesium	7439-95-4	--	--	50	25	10.4
Manganese	7439-96-5	30	0.43	2.00	1.60	0.634
Potassium	7440-09-7	--	--	200	160	107
Sodium	7440-23-5	20,000	--	200	160	50

Notes:

[a] MassDEP maximum contaminant level (MCL) in drinking water (GW-1).

[b] Site-specific groundwater screening levels (SLs) for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

-- = Not Available
µg/L = microgram per liter
CAS = Chemical Abstracts Service
ng/L = nanogram per liter

WORKSHEET #15-2: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN GROUNDWATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (mg/L)		
		MassDEP GW-1 MCL[a] (mg/L)	USEPA Site-Specific SL[b] (mg/L)	LOQ	LOD	DL
Analytical Group: Anions 300 ORGFM_28D						
Sulfate	14808-79-8	250	--	1.00	0.900	0.300
Chloride	16887-00-6	--	--	0.400	0.300	0.200

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-3: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN GROUNDWATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (mg/L)		
		MassDEP GW-1 MCL[a] (mg/L)	USEPA Site-Specific SL[b] (mg/L)	LOQ	LOD	DL
Analytical Group: Alkalinity 2320B						
Total alkalinity as $CaCO_3$ to pH 4.5	STL00171	--	--	8.00	6.00	2.60

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

$CaCO_3$ = calcium carbonate

WORKSHEET #15-4: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN GROUNDWATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (mg/L)		
		MassDEP GW-1 MCL[a] (mg/L)	USEPA Site-Specific SL[b] (mg/L)	LOQ	LOD	DL
Analytical Group: DOC 415.1_Diss						
DOC	7440-44-0	--	--	1.00	0.900	0.500

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-5: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DISSOLVED GASES IN GROUNDWATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (µg/L)		
		MassDEP GW-1 MCL[a] (µg/L)	USEPA Site-Specific SL[b] (µg/L)	LOQ	LOD	DL
Analytical Group: Dissolved Gases RSK_175_CO2_D5						
Carbon dioxide	124-38-9	--	--	12000	8000	2600

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-6: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS (ICP-MS) IN SURFACE WATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (µg/L)		
		MassDEP GW-1 MCL [a] (µg/L)	USEPA Site-Specific SL[b] (µg/L)	LOQ	LOD	DL
Analytical Group: Metals 6020B_DOD5						
Aluminum	7429-90-5	50	2.4E+04	35.0	30.0	19.7
Arsenic	7440-38-2	10	1.0E+01	2.00	1.60	0.680
Barium	7440-39-3	2,000	2.0 E+03	2.00	1.60	0.746
Calcium	7440-70-2	--	--	125	120	73.6
Iron	7439-89-6	300	1.4E+04	50	40	22.8
Magnesium	7439-95-4	--	--	50	25	10.4
Manganese	7439-96-5	300	--	2.00	1.60	0.634
Potassium	7440-09-7	--	--	200	160	107
Sodium	7440-23-5	20,000	--	200	160	50

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-7: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN SURFACE WATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (µg/L)		
		MassDEP GW-1 MCL[a] (µg/L)	USEPA Site-Specific SL[b] (µg/L)	LOQ	LOD	DL
Analytical Group: Anions 300 ORGFM 28D						
Sulfate	14808-79-8	2.5 E+5	--	1.00	0.900	0.300
Chloride	16887-00-6	2.5E+5	--	0.400	0.300	0.200

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-8: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN SURFACE WATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (µg/L)		
		MassDEP GW-1 MCL[a] (ng/L)	USEPA Site-Specific SL[b] (ng/L)	LOQ	LOD	DL
Analytical Group: Alkalinity 2320B						
Total alkalinity as $CaCO_3$ to pH 4.5	STL00171	--	--	8.00	6.00	2.60

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-9: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN SURFACE WATER)

(UFP-QAPP Manual Section 2.6.2.3) / (USEPA 2106-G-05 Section 2.2.6)

Analyte	CAS #	PAL		Laboratory Achievable DLs (mg/L)		
		MassDEP GW-1 MCL[a] (ng/L)	USEPA Site-Specific SL[b] (ng/L)	LOQ	LOD	DL
Analytical Group: DOC 415.1_Diss						
DOC	7440-44-0	--	--	1.00	0.900	0.500

Notes:

[a] MassDEP MCL in drinking water (GW-1).

[b] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-10: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS ICP-MS IN SOIL/SEDIMENT)

Analyte	CAS #	PAL	Laboratory Achievable DLs (mg/kg)		
		USEPA Site-Specific SL[a] (mg/kg)	LOQ	LOD	DL
Analytical Group: Metals (ICP-MS) 6020B_DOD5					
Aluminum	7429-90-5	3.0E+04	10.0	8.00	4.37
Arsenic	7440-38-2	1.5E-03	0.220	0.160	0.0669
Calcium	7440-70-2	--	25.0	16.0	7.49
Iron	7439-89-6	3.5E+02	10.0	10.0	3.75
Magnesium	7439-95-4	--	5.00	5.00	1.57
Manganese	7439-96-5	1.9E+03	0.200	0.160	0.106

Note:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-11: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TOC IN SOIL/SEDIMENT)

Analyte	CAS #	PAL	Laboratory Achievable DLs (mg/kg)		
		USEPA Site-Specific SL[a] (µg/kg)	LOQ	LOD	DL
Analytical Group: TOC SM5310B					
TOC	7440-44-0	--	300	200	100

Notes:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

µg/kg = microgram per kilogram

WORKSHEET #15-12: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (METALS IN POREWATER)

Analyte	CAS #	PAL	Laboratory Achievable DLs (µg/L)		
		USEPA Site-Specific SL[a] (µg/L)	LOQ	LOD	DL
Analytical Group: Metals (ICP-MS) 6020B_DOD5					
Aluminum	7429-90-5	2.0E+04	35	30	19.7
Arsenic	7440-38-2	1.5E-03	2.00	1.60	0.680
Barium	7440-39-3	2.0 E+03	2.00	1.60	0.746
Calcium	7440-70-2	--	125	120	73.6
Iron	7439-89-6	3.5E+02	50	40	22.8
Magnesium	7439-95-4	--	50	25	10.4
Manganese	7439-96-5	--	2.00	1.60	0.634
Potassium	7440-09-7	--	200	160	107
Sodium	7440-23-5	--	200	160	50

Note:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-13: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ANIONS IN POREWATER)

Analyte	CAS #	PAL	Laboratory Achievable DLs (mg/L)		
		USEPA Site-Specific SL[a] (µg/L)	LOQ	LOD	DL
Analytical Group: Anions, Ion Chromatography 300_ORGFM_28D					
Sulfate	14808-79-8	--	1.00	0.900	0.300
Chloride	16887-00-6	--	0.400	0.300	0.200

Note:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-14: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (ALKALINITY IN POREWATER)

Analyte	CAS #	PAL	Laboratory Achievable DLs (mg/L)		
		USEPA Site-Specific SL[a] (µg/L)	LOQ	LOD	DL
Analytical Group: Alkalinity 2320B					
Total alkalinity as $CaCO_3$ to pH 4.5	STL00171	--	8.00	6.00	2.60

Note:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-15: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (DOC IN POREWATER)

Analyte	CAS #	PAL	Laboratory Achievable DLs (mg/L)		
		USEPA Site-Specific SL[a] (µg/L)	LOQ	LOD	DL
Analytical Group: DOC 415.1_Diss					
DOC	7440-44-0	--	1.00	0.900	0.500

Note:

[a] Site-specific groundwater SLs for residential child scenario was revised based on May 2021 USEPA (USEPA 2021).

WORKSHEET #15-16: REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TOC AND GRAIN SIZE IN SOIL/SEDIMENT)

Laboratory-Specific Reporting Limits[a] (g/kg)												
Method and Analyte Description	**CAS #**	**Cleanup Goals (mg/L)[1]**	**LOQ**	**DL**	**LOD**	**Units**	**LCS - Low**	**LCS-High**	**LCS - RPD %**	**MS-Low**	**MS-High**	**MS-RPD %**
9060A TOC	7440-44-0	--	4.0	0.902	2.0	g/kg	46	130	20	46	130	20
Grain size	NA	--	--	0.5	1.0	% passing	--	--	--	--	--	--

Notes:

[a] Eurofins TestAmerica laboratory-specific limits for TOC for sediment.

g/kg = gram per kilogram
RL = reporting limit

WORKSHEET #15-17 REFERENCE LIMITS AND EVALUATION – EUROFINS TESTAMERICA, LANCASTER, PENNSYLVANIA (TSS AND TDS IN WATER)

Laboratory-Specific Reporting Limits[a] (mg/L)												
Method and Analyte Description	**CAS #**	**Cleanup Goals (mg/L)[1]**	**LOQ**	**DL**	**LOD**	**Units**	**LCS-Low**	**LCS-High**	**LCS-RPD %**	**MS-Low**	**MS-High**	**MS-RPD %**
Total Suspended Solids (TSS)	STL00161	--	3.00	1.00	2.50	mg/L	89	105	20	--	--	--
Total Dissolved Solids (TDS)	STL00242	--	30.0	12.0	25.0	mg/L	72	127	23	72	127	23

Notes:

mg/L = milligram per liter
RL = reporting limit

QAPP WORKSHEET #17: SAMPLING DESIGN AND RATIONALE

The primary goals of the field activities are to define current conditions in Red Cove, to quantify arsenic flux from SHL to the Red Cove area of PSP, to identify the ongoing source of arsenic present in PSP, and if needed, to develop recommendations to monitor Red Cove for the attainment of remedial action objectives through time.

17.1 Overall Investigation Approach Rationale

This worksheet describes the design for data collection and documents Step 7 of the DQO process. For each field task, this section summarizes the data gap, rationale for data collection, and approach for filling the data gap. The collection of temporal and spatial synoptic samples in groundwater, porewater, surface water, soil, and sediment will define current conditions in Red Cove and allow for a comprehensive understanding of the geochemical cycle of arsenic in the system. These data will also be used to better define the zone of influence of SHL on the Red Cove area of PSP and will be used to compare arsenic concentrations between areas influenced/not influenced by SHL. The target area for the samples, methods that will be used, and information collected will be sufficiently compatible with previous investigations in the Red Cove area to allow for pre- and post-wall installation comparisons of arsenic and associated constituents. Figure 2 in the Barrier Wall Performance Monitoring Work Plan shows the 28 proposed porewater, sediment, and aquatic sample locations along with the eight groundwater sample locations.

In addition to the SOPs identified in the LTMMP QAPP (SERES-Arcadis JV 2020), sampling will be conducted in accordance with the following SOP, provided in Attachment 1 (see also Worksheet #21):

- **SOP-003:** SERES-Arcadis TGI – Vibracore Sediment Collection (Rev #1, 30 March 2021)

17.2 Field Measurements

17.2.1 Aquatic Location Field Measurements

Field measurements will be collected and recorded from a boat at each of the 28 aquatic sample locations (20 in Red Cove and eight reference locations) before primary sample collection. These measurements will include water surface elevation, water depth, and sediment depth (as determined by refusal of a metal metered rod using manual pressure). Dissolved oxygen (DO), conductivity, oxidation-reduction potential (ORP), pH, turbidity, and temperature will be measured using a multiparameter probe at the following depths:

- Top Sample (see Section 3.2.2 of the Work Plan)
- Bottom Sample (2 to 6 inches from above sediment surface)
- Six to 12 inches below sediment surface (bss)
- Eighteen to 24 inches bss

- Thirty to 36 inches bss
- Forty-two to 48 inches bss.

The depth to water will not be measured within 48 hours of a preceding precipitation event to provide base measurements. The in-situ field measurements will be used in conjunction with other data collected to determine where groundwater flux into the Red Cove area of PSP from SHL may be highest. Water surface elevation can be measured through the establishment and daily reading of a staff gage or use of a differential global positioning system if the sampling vessel is so equipped. Water level measurements will be gauged with a water level meter tied to a benchmark elevation on land in order to determine the water surface elevation.

17.2.2 Groundwater Location Field Measurements

Field measurements will be recorded at each of the 10 groundwater sample locations during well purging and again prior to sample collection. The groundwater levels will not be measured within 48 hours of a significant preceding precipitation event (i.e., greater than 1 inch in 24 hours) to provide base measurements. These measurements will include depth to water, DO, conductivity, ORP, pH, turbidity, and temperature and will be measured using a multiparameter probe. As part of the low flow sampling protocol, during the purging cycle, depth to water will be measured periodically to insure a drawdown of less than 0.1 meters.

17.2.3 Surface Water Flow

Instantaneous flow into and out of PSP will be measured two times (once at the commencement of field activities and once at the end of field activities. The flows will not be measured within 48 hours of a preceding precipitation event to provide base flow measurements. Flow from Grove Pond to PSP will be measured at the culvert east of the railroad causeway and flow from PSP to Nonacoicus Brook will be measured at the dam on the western edge of the pond. Flow will be calculated from velocity measured along a transect across the culvert and dam, and a bathymetric profile measured at the time the flow velocity is acquired. The measured flows into and exiting PSP will provide supporting information for the water balance for PSP.

17.2.4 Aquatic Hydraulic Head Measurement

Water elevations will be measured from clustered piezometers at 10 locations within Red Cove, co-incident with Shelby tube samples collected for vertical hydraulic conductivity analysis. The elevations will not be measured within 48 hours of a preceding precipitation event to provide base flow measurements. These clustered piezometers, along with measured PSP stage will provide a measured vertical gradient at each location. The groundwater head, along with the measured hydraulic conductivity from the Shelby tubes, will be used to estimate groundwater flow through the hyporheic zone to Red Cove. The clustered piezometers will consist of two 0.75-inch ID pipes with 6-inch screen. At each location, one piezometer will be hand driven to a depth of 10-feet bss with a screen interval of 9.5 to 10 feet bss. A second piezometer will be hand driven to a depth of 2-feet bss with a screen interval of 1.5 to 2 feet bss. Up to two additional alternate locations will be attempted within a 10-feet radius if refusal is encountered. Based on current well logs, there is no reason to assume refusal will happen. Each piezometer will consist of 0.75-inch ID stainless steel drive pipe with 6-inch 50 mesh (0.014-in opening) stainless steel screen. At the location with the greatest water depth, a PVC stilling pipe will be added to the well cluster to measure stage in PSP. All pipes within a cluster will be secured with “T” connectors to improve stability for the 2-foot piezometers and stilling pipe. Top of pipe elevations for all piezometers and stilling pipes will be surveyed to North American Vertical Datum of 1988.

Synoptic water levels will be manually gauged in the in all piezometers from a boat and the stilling pipe during three events, each separated by a minimum of 4-weeks between events. Water levels will be gauged to 0.01-foot.

17.2.5 Temporal Flux Variation

Pressure transducers will be installed in eight groundwater monitoring wells, eight piezometers in Red Cove, two piezometers in the PSP reference area, and the PSP surface water. The pressure transducers will be installed in these locations coincident with the first aquatic head measurements and will record water levels at an hourly interval through the duration of the field investigations. The hourly water level measurements will provide information on variations in hydraulic gradients in response to seasonal and precipitation changes. The selected locations are clustered wells and piezometers and will provide information on vertical gradients between:

- the surficial sand aquifer and bedrock and the surficial aquifer, and
- the surficial sand aquifer, hyporheic zone, and surface water.

Pressure transducers will be installed in the following areas as shown on Figure 3:

- Four of the well clusters (eight transducers) in Red Cove,
- One well cluster in the reference area in PSP,
- PSP stilling well
- Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (N2-P1/P2 and N3-P1/P2 well clusters) adjacent to Red Cove
- Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (PZ-12-06 and SHM-11-02 cluster; RSK-28 and SMH-11-07 cluster) upgradient of the barrier wall.

17.3 Surface Water Sampling

Surface water samples will be collected from up to 14 locations (10 in Red Cove and four reference locations) using a peristaltic pump or grab sampler at the water column depths specified below:

- Top sample (see text below)
- Bottom sample (2 to 6 inches from the bottom).

If total water depth is 2 feet or less, the top sample will be collected near surface (2 to 6 inches from the surface). If the water column is more than 2 feet, the top sample will be collected from the midpoint of the water column. The surface water samples will be analyzed for metals, dissolved oxygen carbon (DOC), TDS, TSS, major cations and anions, and iron-oxidizing bacteria. Field tasks associated with the surface water investigation are presented in Table 17-1, below.

Table 17-1. Surface Water Investigation Tasks

Surface Water Investigation Task	Supporting Document
Surface water sampling	**SOP-004:** SERES-Arcadis TGI – SERES-Arcadis TGI – Surface Water Sample Collection (Rev #2, 07 May 2020), provided in Attachment 1
Measurement of water quality parameters	**SOP-006**: SERES-Arcadis TGI - In-Situ and Ex-Situ Water Quality Parameters (Rev #0, October 2018), provided in Attachment 1

Surface water samples will be collected from a boat (to minimize any potential disturbance to the sediment bed or water column). Surface water samples will be analyzed by Eurofins TestAmerica in Lancaster, Pennsylvania. Samples will be shipped on ice and handled with chain-of-custody documentation. Water quality parameters will be collected from each sample location and will include DO, ORP, specific conductance, temperature, turbidity, and pH. Details on the surface water sampling can also be found in Section 3.2 of the Work Plan.

17.4 Soil Sampling

Soil cores from the surficial sand aquifer will be collected adjacent to three well locations between the barrier wall and Red Cove (Figure 3) and analyzed as described below:

- Area with elevated (greater than 2x the GW-1 standard of 10 µg/L) dissolved arsenic in groundwater:
  - SHL-4 (3-13 ft bgs)
  - SHP-01-38A (1.5-6.5 ft bgs)
- Area with lower dissolved arsenic in groundwater:
  - SHL-19 (20-30 ft bgs)

Samples from the three locations will be collected as 5-foot composites from the ground surface to the top of bedrock using a Direct Push Rig (DPT refusal will be used as the indication that the depth of the top of bedrock has been reached.) Soil samples will be analyzed for metals, TOC, pH, total sulfur, and grain size. Sequential extraction procedure (SEP) will be conducted on a selected number of samples. Details on the sediment sampling can also be found in Section 3.3 of the Work Plan.

Table 17-2. Soil Investigation Tasks

Soil Investigation Task	Supporting Document
Soil sampling	**SOP-001:** SERES-Arcadis TGI – Soil Drilling and Sample Collection Rev#1, provided in Attachment 1

17.5 Sediment Sampling

Five sediment samples will be collected from each of the 28 aquatic sample locations shown on Figure 3 (20 in Red Cove and eight reference locations). Sediment cores for chemical analysis will be advanced from a boat manually or with a vibracore to a minimum depth of 4 feet bss. Sediment samples from these cores will be sectioned into five intervals: 0 to 6 inches, 6 to 12 inches, and 1-foot intervals thereafter. The bottom interval of samples will be adjusted based on sample recovery, field measurements taken before the collection of cores, or if distinct visual horizons are noted in the sediment core. The field tasks associated with the sediment investigation are presented in Table 17-2, below. Sediment coring is used instead of the Eckman sampler employed in earlier sampling programs to allow for the collection of sediment deeper than the surficial interval. Additionally, the surficial interval of 0 to 6 inches is being targeted; an Eckman sampler typically collects samples from only the upper 3 to 4 inches and the actual sediment collection depth cannot be ascertained.

At one-half of the locations, one additional sediment sample will be collected using a 2-inch-diameter, 36-inch-long Shelby tube advanced manually. The tubes will be capped for transport. These samples will be submitted to the laboratory to evaluate the vertical hydraulic conductivity of the sediment. These tests will be performed using the entire length of the tube to maintain the undisturbed nature of the sample.

All sediment samples will be analyzed for metals and total organic carbon. Some samples will be analyzed for pH, sulfur, SEP, and grain size. Details on the sediment sampling can also be found in Section 3.4 of the Work Plan.

Table 17-3. Sediment Investigation Tasks

Sediment Investigation Task	Supporting Document
Sediment sampling	**SOP-003:** SERES-Arcadis TGI – Vibracore Sediment Collection (Rev#1, 30 March 2021), provided in Attachment 1

17.5 Porewater Sampling

Consistent with previous data collection sampling programs, porewater samples will be collected from up to 28 locations using PushPoint™ samplers (20 in Red Cove and eight reference locations) and analyzed for metals, major cations and anions, DOC, TSS, TDS, ammonia, and iron-oxidizing bacteria. The field tasks associated with the porewater investigation are presented in Table 17-3, below. Porewater samples will be collected at the following four depths:

- Six to 12 inches bss
- Eighteen to 24 inches bss
- Thirty to 36 inches bss
- Forty-two to 48 inches bss

Details on the porewater sampling can also be found in Section 3.5 of the Work Plan.

Table 17-4. Porewater Investigation Tasks

Surface Water Investigation Task	Supporting Document
Porewater sampling	**SOP-007:** Porewater Sampling, provided in Attachment 1
Measurement of water quality parameters	**SOP-006**: SERES-Arcadis TGI - In-Situ and Ex-Situ Water Quality Parameters (Rev #0, October 2018), provided in Attachment 1

17.6 Groundwater Sampling

Prior to the collection of groundwater samples, synoptic water levels will be collected according to their respective LTMMPs. Groundwater from wells will be purged and sampled in accordance with the Low Stress (Low-Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (USEPA Region 1 2017). Samples to be submitted for dissolved metals analysis will be field-filtered using a 0.45-micron filter. Static depth to groundwater measurements will be measured in accordance with established procedures. In addition, the pump intake depth will be recorded on field logs to provide information on pumping depth relative to the observed water table. The sampling pump intake depth will be set at the mid-point of the saturated screened interval for all wells with screen lengths of 10 feet or less. Water quality parameters DO, ORP, specific conductance, temperature, pH, and turbidity will be measured in accordance with established procedures. Turbidity samples will be collected from a T-valve placed on the sample tubing prior to the flow-through cell and measured with a stand-alone meter.

Groundwater sampling is proposed at 10 existing monitoring wells. These samples will be collected during the same sampling event as the aquatic samples from PSP. The analytical suite for groundwater is generally the same as the suite proposed for porewater and surface water, and includes total and dissolved arsenic and iron, total and dissolved major cations (sodium, magnesium, manganese, barium, potassium, aluminum, and calcium), major anions (sulfate, chloride, and alkalinity), DOC, TDS, TSS, and ammonia. Samples from four wells will also be analyzed for CO2 and iron-oxidizing bacteria. Water level measurements and field measurements for groundwater will be consistent with measurements taken at the aquatic locations. Details on the groundwater sampling can also be found in Section 3.6 of the Work Plan.

QAPP WORKSHEET #18: SAMPLE LOCATIONS AND METHODS

Worksheet #17 describes the planned sampling events for the site barrier wall performance evaluation. Sample locations and analyses for the evaluation are presented in the table below.

Sample Location	Matrix	Sample Depth /Screening Interval (feet below ground surface)	Sample ID [a]	Analytes														Sample Type	Sampling SOP Reference	Rationale for Sample Location
				Total Metals	Dissolved Metals	TOC	Major Cations	Major Anions	DOC	Iron-Oxidizing Bacteria	SEP	Triaxial Hydraulic Conductivity	TSS	TDS	pH	Grain Size	CO2 Gas			
Ten existing monitoring wells	Groundwater	Groundwater samples will be collected in accordance with the methodologies outlined in the LTMMP.	MW-#-XX (DDMMYY)	10	10		10	10	10	4			10	10			4	Discrete	Low Stress (Low-Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (USEPA Region 1 2017).	See Work Plan, Section 3.6
Red Cove and reference locations	Porewater	Using PushPoint™ samplers porewater samples will be collected at 6 to 12, 18 to 24, 30 to 36, and 42 to 48 inches bss.	PW-#-XX	112	112		56	56	56	8			112	56				Discrete	Porewater Sampling SOP	See Work Plan, Section 3.5
Between barrier wall and Red Cove	Soil	Soil cores from three locations as 5-foot composites using DPT	SS-#-XX	18		18					6				18	18		Discrete	SERES-Arcadis TGI - Soil Drilling and Sample Collection Rev#1	See Work Plan, Section 3.3
Red Cove and reference locations	Sediment	Vibracore – minimum depth of 4 feet bss. Five intervals: 0 to 6 inches, 6 to 12 inches, and 1-foot	SB-#-XX	140		140					28	10			84	84		Discrete	SERES-Arcadis TGI – Vibracore Sediment Collection (Rev#1, 30 March 2021), provided in Attachment 1	See Work Plan, Section 3.4

Sample Location	Matrix	Sample Depth /Screening Interval (feet below ground surface)	Sample ID [a]	Analytes														Sample Type	Sampling SOP Reference	Rationale for Sample Location
				Total Metals	Dissolved Metals	TOC	Major Cations	Major Anions	DOC	Iron-Oxidizing Bacteria	SEP	Triaxial Hydraulic Conductivity	TSS	TDS	pH	Grain Size	CO2 Gas			
		intervals after that.																		
Red Cove and reference locations	Surface Water	Top sample (near surface or midpoint of water column) Near bottom in the water column (2 to 6 inches from the bottom).	SW-#-XX				28	28	28	14			28	28				Discrete	SERES-Arcadis TGI – Surface Water Sample Collection, (Rev #2, 07 May 2020), provided in Attachment 1	See Work Plan, Section 3.2

Notes:

[a] X = Represents sample depth to be determined. # = Represents Sample Number.

QAPP WORKSHEETS #19 AND #30: SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

Worksheets #19 and #30 summarize the analytical methods/matrix, including the required sample volume, containers, preservation, and holding time requirements. The laboratory analytical SOPs are listed in Worksheet #23 and provided in Attachment 2. Laboratory certifications can be found in Attachment 3.

Primary Analytical Laboratory Eurofins TestAmerica Lancaster 2425 New Holland Pike Lancaster, PA 17601 (717) 823-7123	PM: Melissa McDermott Email: Melissa.McDermott@eurofinset.com Certifications: DoD Environmental Laboratory Accreditation Program Sample Delivery Method: FedEx overnight
Eurofins TestAmerica Knoxville 5815 Middlebrook Pike Knoxville, TN 37921 (865) 291 - 3006	PM: Henry Ryan Email.: WilliamR.Henry@eurofinset.com Certifications: DoD Environmental Laboratory Accreditation Program Sample Delivery Method: FedEx overnight
Atlantic Testing Kassey A. Northrop 6431 US Highway 11 Canton, NY (315) 386-4578	PM: Kassey A. Northrop Email: knorthrop@atlantictesting.com Accreditation: American Association of State Highway and Transportation Officials Accredited Sample Delivery Method: FedEx overnight

Matrix	**Analytical Group**	**Laboratory**	**Method/SOP**	**Containers** (number, size, and type)	**Sample Volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time**[1] (preparation / analysis)
Water	CO_2	Eurofins TestAmerica Lancaster	RSK0175; WI9644	2 x 40 mL glass vials, no headspace	40 mL	Cool, 6°C, no headspace	14 days
Water	chloride, sulfate	Eurofins TestAmerica Lancaster	EPA 300.0 or SW-846 9056; WI11626	50 ml plastic vial	50 mL	Cool, 6°C	SO_4, Cl ≤28 days,
Water	Alkalinity	Eurofins TestAmerica Lancaster	SM 2320B-2011 or EPA 310.1; WI11475	250 mL plastic or glass bottle	250 mL	Cool < 6°C	14 days
Water	DOC	Eurofins TestAmerica Lancaster	SM 5310C/EPA 415.1; WI11637	2 x 40 mL amber glass vial	40 mL	Cool, 6°C, H_3PO_4 to pH <2	28 days
Soil/Sediment	TOC	Eurofins TestAmerica Lancaster	SM 5310B/SW-946 9060A; WI11627	4 or 8 ounce glass jar	20 grams	Cool, 6°C	28 days
Sediment	Moisture	Eurofins TestAmerica Lancaster	SM 2540 G-1997 %Moisture Calc; WI10697	50 gram plastic or glass jar	5 grams	Cool, 6°C	7 days
Soil/Sediment	Metals	Eurofins TestAmerica Lancaster	SW-846 6020A/B WI11933	4 or 8 ounce glass jar	100 grams	None, 2 to 6°C	6 months
Water	Metals	Eurofins TestAmerica Lancaster	SW-846 6020A/B WI11933 SW-846 7470A/ WI7965	250 mL plastic	250 mL	HNO_3, 2 to 6°C	6 months

Matrix	**Analytical Group**	**Laboratory**	**Method/SOP**	**Containers** (number, size, and type)	**Sample Volume** (units)	**Preservation Requirements** (chemical, temperature, light protected)	**Maximum Holding Time**[1] (preparation / analysis)
Soil/Sediment	pH/corrosivity	Eurofins TestAmerica Lancaster	SW-846 9045C/D/ WI11518	4 or 8 ounce glass jar	50 g	Cool, 6°C	As soon as possible after receipt in laboratory
Solid	Grain Size	Eurofins TestAmerica Lancaster	ASTM D422;WI11514	16 ounce glass jar, this must be a dedicated bottle for grain size only	500 g	Cool, 6°C	NA
Water	TSS	Eurofins TestAmerica Lancaster	SM 2540 D-1997;WI11604	1500 mL plastic or glass bottle	1000 ml	Cool, 6°C	7 days
Water	TDS	Eurofins TestAmerica Lancaster	SM2540 C-1997;WI11598	500 mL plastic or glass bottle	230 ml	Cool, 6°C	7 days
Water	Iron-oxidizing bacteria	Eurofins TestAmerica Lancaster	Biological Activity Reaction Test	50 ml plastic vial	50 ml	Cool, 2 to 6°C	21 days
Soil/Sediment	SEP	Eurofins TestAmerica Knoxville	Eurofins TestAmerica Knoxville SEP Summary 081319 (proprietary SOP)	NA	NA	NA	NA
Sediment	Triaxial Permeability – Undisturbed	Atlantic Testing	ASTM D5084	Shelby tube	NA	NA	NA

Notes:

[1] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

°C = degree Celsius
HNO_3 = nitric acid
mL = milliliter
TOC = total organic carbon
SEP = sequential extraction procedure
DOC = dissolved organic carbon
TSS =total suspended solids
TDS = total dissolved solids

QAPP WORKSHEET #20: FIELD QC SAMPLE SUMMARY

The table below presents the types of samples to be collected and analyzed and shows the relationship between the number of field samples and associated QC samples for each combination of analyte/analytical group and matrix. Sample locations are presented in table included in Worksheet #18; the table below lists the frequency of field QC sample collection.

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Equipment Blanks	Trip Blanks	Other
Surface Water	DOC[7]	See Worksheet #18	10 percent (%)	5%	5%	As needed	0	NA
Surface Water	Cations and anions[6]	See Worksheet #18	10%	5%	5%	As needed	0	NA
Surface Water	Iron-oxidizing bacteria	See Worksheet #18	10%	NA	NA	As needed	0	NA
Soil/Sediment	Metals[1] (total arsenic, iron, magnesium, manganese, aluminum, calcium)	See Worksheet #18	10%	5%	5%	As needed	0	NA
Soil/Sediment	TOC[3]	See Worksheet #18	10%	5%	5%	As needed	0	NA
Soil/Sediment	General[2]	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Equipment Blanks	Trip Blanks	Other
Soil/Sediment	SEP[4]	See Worksheet #18	10%	NA	NA	As needed	0	NA
Sediment	Triaxial hydraulic conductivity	See Worksheet #18	10%	NA	NA	As needed	0	NA
Porewater	Metals[5] (total and dissolved)	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Porewater	DOC[7]	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Porewater	Cations and anions[6]	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Porewater	Iron-oxidizing bacteria	See Worksheet #18	10%	N/A	NA	As needed	0	NA
Groundwater	Arsenic (total and dissolved)	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Groundwater	Cations and anions[6]	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Groundwater	DOC[7]	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Groundwater	Iron-oxidizing bacteria	See Worksheet #18	10%	NA	NA	As needed	0	NA

Matrix	Analyte/Analytical Group	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Equipment Blanks	Trip Blanks	Other
Groundwater	Carbon dioxide gas	See Worksheet #18	10%	5%	5% (duplicate)	As needed	0	NA
Surface Water, Groundwater, Porewater	TSS	See Worksheet#18	10%	5%	5% (duplicate)	As needed	0	NA
Surface Water, Groundwater, Porewater	TDS	See Worksheet#18	10%	5%	5% (duplicate)	As needed	0	NA

Notes:

1. Sediment metals = total arsenic, iron, magnesium, manganese, aluminum, calcium
2. Sediment general = pH, sulfur, grain size
3. TOC = total organic carbon
4. SEP = sequential extraction procedure
5. Porewater metals = arsenic and iron (total and dissolved)
6. Major cations = arsenic, sodium, iron, magnesium, manganese, potassium, aluminum, barium, and calcium, (total and dissolved); major anions = sulfate, alkalinity, and chloride.
7. DOC = dissolved organic carbon
8. TSS =total suspended solids
9. TDS = total dissolved solids
10. NA=Not Applicable

QAPP WORKSHEET #21: PROJECT SAMPLING SOP REFERENCES

The field sops associated with the project sampling (including, but not limited to, sample collection and sample handling and custody) are listed in the table below. Copies of the field sops are provided in Attachment 1.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)
SOP-001	Soil Drilling and Sample Collection Rev#1	JV	Soil sampling equipment	N
SOP-002	Surface Sediment Sampling with Grab Samplers	JV	Sediment sampling equipment	N
SOP-003	Vibracore Sediment Collection	JV	Vibracore sampler	N
SOP-004	Surface Water Sample Collection	JV	NA	N
SOP-005	Surface Water Flow Measurements	JV	NA	N
SOP-006	In-Situ and Ex-Situ Water Quality Parameters	JV	Water quality parameter probe	N
SOP-007	Porewater Sampling	USEPA	NA	N

QAPP WORKSHEET #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION

Field sampling equipment will be leased from a reputable equipment leasing supplier. All equipment will be received in good working order from the supplier. The field equipment and instruments expected to be used during the sampling events discussed in this QAPP Addendum may include:

- Water level indicators
- Water quality meters (YSI or similar)
- Peristaltic pumps
- Photoionization detector
- Flame ionization detector (FID)

Additional equipment may be needed depending on field conditions. Manufacturer’s instructions will be followed when using field equipment. The calibration, maintenance, testing, and/or inspection requirements are discussed in the field SOPs provided Attachment 1.

QAPP WORKSHEET #23: ANALYTICAL STANDARD OPERATING PROCEDURES

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
INORGANIC ANALYSES								
WI9644	Carbon Dioxide in Water Using Headspace Sampling Techniques and GC-TCD, Method RSK-175 or 8015, Rev 12, effective 12/2/19	8/25/2021	12	Definitive	CO_2	GC	Eurofins Lancaster Laboratories Environmental, LLC (ELLE)	N
WI11626	Determination of Inorganic Anions by Ion Chromatography in Waters and Soil by EPA 300.0, SW 846 9056, and SW 846 9056A, Rev 23, effective 4/30/2021	8/25/2021	23	Definitive	Water and solid	IC	ELLE	N
WI11475	Multi-Parameters in Solids and Waters by Man-Tech Multi-Parameter	8/25/2021	15	Definitive	Water	Man-Tech Multi-	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	System Rev 15; effective 04/30/2021					Parameter System		
QA-SOP11880	Balance, Syringe, Pipette, and Labware Verification, Rev 11, effective 08/31/2020	8/25/2021	11	NA	Maintenance	Balance	ELLE	N
WI11519	pH Probes and Meters, Rev 14, effective 12/28/2020	8/25/2021	14	NA	Water and solid	pH meter	ELLE	N
WI11637	Total Organic Carbon, Dissolved Organic Carbon, and Inorganic Carbon by SM 5310C or EPA 415.1 in Waters, Rev 17, effective 7/6/21	8/25/2021	17	Definitive	Water	TOC analyzer	ELLE	N
WI11627	Total Organic Carbon and Total Carbon in Solids and Sludges by	8/25/2021	17.1	Definitive	Solid	TOC Analyzer	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Combustion by SM 5310B, EPA 415.1, SW-846 9060/9060A, Lloyd Kahn, Rev 17.1, effective 7/6/21							
WI10697	% Moisture Calculation and % Solids Calculation (Gravimetric) by SM 2540 G-1997 and SM 2540 G-2011, Rev 19; effective 5/14/21	8/25/2021	19	Definitive	Solid	Analytical Balance/over 103-105 degrees	ELLE	N
WI7965	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B (aqueous, solid, tissue), and EPA 200.8 (aqueous), Rev 11, effective 11/12/2020	11/12/2020	11	Definitive	Solid, liquid, tissues Metals	ICP/MS	ELLE	N
WI8636	Sample Prep of Sediments,	5/11/2021	26	N/A	Solid and tissues	N/A	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS, Rev, 26, effective 05/11/2021				Inorganic Preparation SW-846 3050B			
WI11948	Preparation of Solids by EPA 7471A or B for Mercury Analysis, Rev 21, effective 05/13/2021	5/13/21	21	N/A	Solid, tissue, and oil Inorganic Preparation SW-846 7471A & SW-846 7471B	N/A	ELLE	N
WI8636	Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS, Rev, 26, effective 05/11/2021	8/25/2021	26	NA	Solid and tissues inorganic preparation SW-846 3050B	NA	ELLE	N
WI11941	Sample Preparation of Wastewater and Leachates for Analysis of Total Metals by Inductively Coupled Plasma	8/25/2021	13	NA	Liquid inorganic preparation SW-846 3010A	NA	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Atomic Emission Spectrometry, Rev 13, effective 12/14/2020							
WI11924	Digestion of Aqueous Samples by SW-846 Method 7470A, Rev 22, effective 11/05/2020	11/5/2020	22	N/A	Liquid Inorganic Preparation SW-846 7470A	N/A	ELLE	N
WI11937	Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) Rev 12, effective 01/19/2015	1/19/2015	12	N/A	Liquid Inorganic Preparation SW-846 3020A or SW-846 3010A	N/A	ELLE	N
WI11518	pH by EPA 9045C, 9045D and Corrosivity by SW-846 Chap 7 of Solids, Soils, and	11/3/21	13	Definitive	Solid	pH meter	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
	Solvents using Electrometic Methods, Rev 13, effective 07/08/2019							
WI11514	Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D-422-63, Rev 10, effective 1/24/19	11/.4/21	10	Definitive	Soil/Sediment	Hydrometer	ELLE	N
SOP11880	Balances, Syringe, Pipette Verification, Rev 10, effective 5/20/19	11/4/21	10	N/A	Maintenance	Balance	ELLE	N
WI11604	Total Suspended Solids (TSS)-Gravimetric by SM 2540 D-2011 and SM 2540 D-1997, Rev 11, effective 12/24/18	11/4/21	11	Definitive	Water	NA	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WI11598	Total Dissolved Solids (TDS)(Gravimetric) by SM 2540 C-2011, SM 2540 C-1997 or EPA 160.1 in Waters and Wastewaters, Rev 16, effective 4/8/19	11/4/21	16	Definitive	Water	NA	ELLE	N
SOP11880	9015389...LOM-SOP-LAB-235 Balances, Syringe, Pipette Verification, Rev 10, effective 5/20/19	11/4/21	10	NA	Maintenance	Balance	ELLE	N
EM-BT-S-8076	Biological activity Reaction Test	11/02/2020	5	NA	Water	Pipettes UV Light	ELLE	N
001[2]	Eurofins TestAmerica Knoxville SEP Detailed Summary 081319	11/4/21	NA	NA	Soil/Sediment	NA	ELLE	N

Laboratory SOP Number	Title, Revision Date, and/or Number[1]	Date	Revision	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)

Notes:

[1] SOPs are reviewed/revised on an annual schedule. The current version will be followed at the time of sample receipt.

[2] The Eurofins TestAmerica SEP SOP is proprietary. A procedure summary of the analysis was provided and can be found in Attachment 2 – Laboratory SOPs.

GC = gas chromatography

NA = Not Applicable

QAPP WORKSHEET #24: ANALYTICAL INSTRUMENT CALIBRATION

To confirm that the analytical methods and selected instrumentation meet project requirements, each instrument will be calibrated according to procedures detailed in method-specific SOPs (Worksheet #23 and Attachment 2). These worksheets summarize calibration requirements for each instrument. Analytical SOPs are reviewed and/or revised annually. The current version will be followed at the time of sample receipt.

WORKSHEET #24-1: ANALYTICAL INSTRUMENT CALIBRATION – GC CARBON DIOXIDE

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria[1]**	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference[2]**
GC Carbon Dioxide	Initial calibration with a minimum 5 points. Ranging from 10,800 to 360,000 mg/L	After continuing calibration fails	%RSD for ICAL ≤20% (average), linear r^2 ≥0.99	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI9644
GC Carbon Dioxide	MDL Standard	After each initial calibration	All compounds must be detected	Repeat ICAL procedure prior to analyzing samples. Repeat maintenance if needed.	ELLE Analyst	WI9644
GC Carbon Dioxide	ICV Standard	After each initial calibration	Target compounds +/- 20% of the nominal concentration and within established retention time windows	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.	ELLE Analyst	WI9644
GC Carbon Dioxide	LOD Standard	Quarterly	All compounds must be detected	Reprep and reanalyze LOD.	ELLE Analyst	WI9644
GC Carbon Dioxide	CCV Standard	Prior to sample analysis, after every 10 field samples, and at the end of the sequence.	Target compounds +/- 20% of the nominal concentration and within established retention time windows	Reanalyze the CCV in duplicate. If both pass, continue with analysis. If CCV recoveries are elevated, associated samples that are ND can	ELLE Analyst	WI9644

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
				be reported. All others since last acceptable CCV must be reanalyzed.		

Notes:

[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.

[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

%RSD = percent relative standard deviation
CCV = continuing calibration verification
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
ICAL = initial calibration
ICV = initial calibration verification
r^2 = coefficient of determination

WORKSHEET #24-2: ANALYTICAL INSTRUMENT CALIBRATION – ANIONS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
IC Anions 300.0	Initial calibration with a minimum of 5 points with a concentration span of 15x or 30x depending on the analyte	Every 60 days or when CCV fails	r >0.995; Level 1 standard must recover ≥50% of the true value	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI11626
IC Anions 300.0	ICB	After each initial calibration	No analytes detected > MDL	Correct problem and reanalyze the ICB. Recalibrate if needed.	ELLE Analyst	WI11626
IC Anions 300.0	ICV	After each initial calibration	Within +/- 10% of the nominal concentration	If ICV fails again do system maintenance and recalibrate.	ELLE Analyst	WI11626
IC Anions 300.0	LOD	Quarterly	Detect the compound	Reanalyze the LOD	ELLE Analyst	WI11626
IC Anions 300.0	CCV	Every 10 injections	Within +/- 10% of the nominal concentration	Recalibrate; reanalyze affected samples	ELLE Analyst	WI11626
IC Anions 300.0	CCB	Every 10 injections	No analytes detected > MDL	Recalibrate; reanalyze affected samples	ELLE Analyst	WI11626
IC Anions 300.0	Initial calibration with a minimum of 5 points with a concentration span of 15x or 30x depending on the analyte	Every 60 days or when CCV fails	r >0.995; Level 1 standard must recover ≥50% of the true value	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	

Notes:

[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.

[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

CCB = continuing calibration blank
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
ICB = initial calibration blank

WORKSHEET #24-3: ANALYTICAL INSTRUMENT CALIBRATION − PH ELECTRODE: ACIDITY, ALKALINITY

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
pH electrode	Calibration using at least 3 points, sloped with pH 4, 7, and 10 buffers	Daily	Percent slope between 92 and 102%	Correct the problem and recalibrate	ELLE Analyst	WI11518, WI11475
pH electrode	CCV standard	After each calibration, every 10 samples, and end of batch	±10% difference	Correct the problem, recalibrate, and reanalyze affected samples	ELLE Analyst	WI11518, WI11475

Notes:

[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.

[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

ELLE = Eurofins Lancaster Laboratories Environmental, LLC

WORKSHEET #24-4: ANALYTICAL INSTRUMENT CALIBRATION – TOC

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
TOC analyzer: TOC, DOC, and TIC in water	Initial calibration with a minimum 6 points ranging from 1.0 to 100 ppm	Monthly or after continuing calibration fails	$r^2 \geq 0.995$	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC, DOC, and TIC in water	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC, DOC, and TIC in water	ICB standard	After each initial calibration	No analytes detected > LOQ	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC, DOC, and TIC in water	Total inorganic check standard	Daily	Within +/- 20% of the nominal concentration	All affected samples are reanalyzed	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC, DOC, and TIC in water	CCV standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	Within +/- 10% of the nominal concentration	All affected samples are reanalyzed	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC, DOC, and TIC in water	CCB standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	No analytes detected > LOQ	All affected samples are reanalyzed	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**[1]	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference**[2]
TOC analyzer: TOC, DOC, and TIC in water	Initial calibration with a minimum 6 points ranging from 1.0 ppm to 100 ppm	Monthly or after continuing calibration fails	$r^2 \geq 0.995$	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI11637 (5310C); WI10038 (9060A)
TOC analyzer: TOC (9060A; 5310B)	Initial calibration with a minimum 4 points ranging from 0.3 to 6 mg C	Monthly or after continuing calibration fails	$r^2 \geq 0.995$	Perform more aggressive instrument maintenance and recalibrate	ELLE Analyst	WI11627
TOC analyzer: TOC (9060A; 5310B)	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD	ELLE Analyst	
TOC analyzer: TOC (9060A; 5310B)	ICV standard	After each initial calibration	Within +/- 10% of the nominal concentration	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.	ELLE Analyst	
TOC analyzer: TOC (9060A; 5310B)	MDL standard	Yearly	All compounds must be detected	Repeat ICAL procedure prior to analyzing samples. Repeat maintenance if needed.	ELLE Analyst	
TOC analyzer: TOC (9060A; 5310B)	CCV standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	Within +/- 10% of the nominal concentration	All affected samples are reanalyzed	ELLE Analyst	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
TOC analyzer: TOC (9060A; 5310B)	CCB standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	No analytes detected > LOQ	All affected samples are reanalyzed	ELLE Analyst	WI11627

Notes:
[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

ELLE = Eurofins Lancaster Laboratories Environmental, LLC
mg C = tonne of Carbon
ppm = part per million

WORKSHEET #24-5: ANALYTICAL INSTRUMENT CALIBRATION – PERCENT MOISTURE

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
Percent moisture	ICAL curve – minimum 5-point calibration	Daily	+/- 0.5 gram	Perform balance internal calibration, tag out of operation, and contact outside support	ELLE Analyst	WI10697

Notes:

[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.

[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

ELLE = Eurofins Lancaster Laboratories Environmental, LLC

WORKSHEET #24-6: ANALYTICAL INSTRUMENT CALIBRATION – METALS – SOIL/SEDIMENT

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
CVAA: 7471B (sediment)	Initial Calibration consists of a Blank and 5 points ranging from 0.2 ppb to 5 ppb	Each new run	Correlation coefficient (r) >0.995	Recalibrate, perform instrument maintenance if calibration cannot conform to criteria, recalibrate	ELLE Analyst	WI7965
	ICV	After each calibration	±10% of true value	Reanalyze		
	ICB	Immediately after the ICV	No analytes > LOD	Reanalyze		
	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD		
	CCV	Immediately after the CRA and every 10 samples	±10% of true value	Reanalyze		
	CCB	Immediately after the CCV and at a frequency of every 10 samples	No analytes > LOD	Reanalyze		
	Low level Check (CRA)	Immediately after the ICB	± 20% True Value	Reanalyze		
ICP/MS: 6020B	Tuning	Daily	No AMU diff. of >0.1 P.W. ≥0.64 and ≥0.66; %RSD <5 for masses used for tuning	Perform mass calibration for AMU. Adjust mass calibration for P.W.	ELLE Analyst	WI11933
	Initial Calibration consists of Blank and 1 point: 0 and 10,000 ppb for Al, Ca, Fe, Mg, K, Na; 0 and	Each new run	Passing ICV and ICB	Recalibrate, perform instrument maintenance if calibration cannot conform to criteria, recalibrate		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
	1,000 ppb for As, Ba, Cr, Co, Cu, Mn, Ni, Ti, V, Zn; 0 and 100 ppb for Sb, Be, Cd, Pb. Mo, Se, Ag, Sr, Tl, Sn					
	ICV	After each calibration	±10% of true value	Reanalyze		
	ICB	Immediately after the ICV	No analytes > LOD	Reanalyze		
	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD		
	CCV	Immediately after the ICSAB and every 10 samples	±10% of true value	Reanalyze		
	CCB	Immediately after the CCV and every 10 samples	No analytes > LOD	Reanalyze		
	Interference Check Sample-A	At the beginning of each run immediately following the LLC	Absolute value of concentration for all non-spiked project analytes <1/2 LOQ	Recalibrate		
	Interference Check Sample-AB	At the beginning of each run immediately following the LLC	± 20% of the true value for each analyte	Recalibrate		
	Low Level Check (LLC)	Beginning of each sequence and before the	± 20% True Value	Reanalyze the sample		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
		interference check samples				
	Linear Dynamic Range (LDR) Check	Quarterly	±10% of true value	Samples > 90% of the linear range must be reanalyzed as a dilution		

Notes:
1This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
2SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

Al = aluminum
Ca = calcium
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
Fe = iron
Mg = magnesium
K = potassium
LLC = low-level check
Na = sodium
S = sulfur
Si = silicon
RSD = relative standard deviation

WORKSHEET #24-7: ANALYTICAL INSTRUMENT CALIBRATION – METALS – WATER

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
CVAA:7470A (water)	Initial Calibration consists of a Blank and 5 points ranging from 0.2 ppb to 5 ppb	Each new run	Correlation coefficient (r) >0.995	Recalibrate, perform instrument maintenance if calibration cannot conform to criteria, recalibrate	ELLE Analyst	WI7965
	ICV	After each calibration	±10% of true value	Reanalyze		
	ICB	Immediately after the ICV	No analytes > LOD	Reanalyze		
	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD		
	CCV	Immediately after the CRA and every 10 samples	±10% of true value	Reanalyze		
	CCB	Immediately after the CCV and at a frequency of every 10 samples	No analytes > LOD	Reanalyze		
	Low level Check (CRA)	Immediately after the ICB	± 20% True Value	Reanalyze		
ICP/MS: 6020A/B	Tuning	Daily	No AMU diff. of >0.1 P.W. ≥0.64 and ≥0.66; %RSD <5 for masses used for tuning	Perform mass calibration for AMU. Adjust mass calibration for P.W.	ELLE Analyst	WI11933

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
	Initial Calibration consists of Blank and 1 point: 0 and 10,000 ppb for Al, Ca, Fe, Mg, K, Na; 0 and 1,000 ppb for As, Ba, Cr, Co, Cu, Mn, Ni, Ti, V, Zn; 0 and 100 ppb for Sb, Be, Cd, Pb. Mo, Se, Ag, Sr, Tl, Sn	Each new run	Passing ICV and ICB	Recalibrate, perform instrument maintenance if calibration cannot conform to criteria, recalibrate		
	ICV	After each calibration	±10% of true value	Reanalyze		
	ICB	Immediately after the ICV	No analytes > LOD	Reanalyze		
	LOD	Quarterly	Analytes detected	Reprep and reanalyze LOD		
	CCV	Immediately after the ICSAB and every 10 samples	±10% of true value	Reanalyze		
	CCB	Immediately after the CCV and every 10 samples	No analytes > LOD	Reanalyze		
	Interference Check Sample-A	At the beginning of each run immediately following the LLC	Absolute value of concentration for all non-spiked project analytes <1/2 LOQ	Recalibrate		
	Interference Check Sample-AB	At the beginning of each run immediately following the LLC	± 20% of the true value for each analyte	Recalibrate		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
	Low Level Check (LLC)	Beginning of each sequence and before the interference check samples	± 20% True Value	Reanalyze the sample		
	Linear Dynamic Range (LDR) Check	Quarterly	±10% of true value	Samples > 90% of the linear range must be reanalyzed as a dilution		

Notes:
1 This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
2 SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

Al = aluminum
Ca = calcium
Fe = iron
Mg = magnesium
K = potassium
LLC = low-level check
Na = sodium
S = sulfur
Si = silicon
RSD = relative standard deviation

WORKSHEET #24-8: ANALYTICAL INSTRUMENT CALIBRATION – SAMPLE PREPARATION

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
HP5890, HP6890, or Agilent 7890 GC with FID	Injection port maintenance; column; FID maintenance	As needed	ICAL or CCV passes method criteria	Perform maintenance again; recalibrate if necessary.	ELLE Analyst	WI9689
Analytical balance	Assure the balance is in a vibration-free area, is level, and the interior housing is clean.	Each day of use	The reading must be ±0.1% or ±0.5 mg, whichever is greater.	Verify cleanliness of weights. Remove balance from service and place a call to service firm. Management must evaluate data generated since last acceptable reading to determine any potential impacts to data quality.	ELLE Analyst	SOP11880
Analytical balance	Annual calibration and maintenance	Annual	As per vendor's specifications in compliance with ISO certification	As per vendor's specifications in compliance with ISO certification.	Professional calibration vendor (ISO 17025 certified)	SOP11880
IC	As needed replacement of components	As needed maintenance/ calibration checks every 10 injections	90 to 110% for calibration checks (95 to 105% for Method 218.6)	Recalibration	ELLE Analyst	WI11625

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria[1]**	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference[2]**
pH/ISE Meter	Calibration checks	As needed maintenance/ calibration checks every 10 injections	90 to 110% for calibration checks	Recalibration	ELLE Analyst	WI11518, WI11519
Analytical balance	Verification with ASTM-certified weights	Each day of use	The reading must be ±0.1% or ±0.5mg, whichever is greater.	Verify cleanliness of weights. Remove balance from service and place a call to service firm. Management must evaluate data generated since last acceptable reading to determine any potential impacts to data quality .	ELLE Analyst	QA-SOP-11880, add SOP reference for the test you are using
Analytical balance	Annual calibration and maintenance	Annual	As per vendor's specifications in compliance with ISO certification	As per vendor's specifications in compliance with ISO certification	Professional calibration vendor (ISO 17025 certified)	QA-SOP-11880, add SOP reference for the test you are using

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria**[1]	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference**[2]
TOC analyzer	Calibration checks	As needed maintenance/ calibration checks every 10 injections	90 to 110% for calibration checks	Recalibration	ELLE Analyst	WI11637 (5310C waters); WI10038 (9060A waters); WI11627 (soil)
Analytical balance	Verification with ASTM-certified weights	Each day of use	The reading must be ±0.1% or ±0.5 mg, whichever is greater.	Verify cleanliness of weights. Remove balance from service and place a call to service firm. Management must evaluate data generated since last acceptable reading to determine any potential impacts to data quality.	ELLE Analyst	SOP11880
Analytical balance	Annual calibration and maintenance	Annual	As per vendor's specifications in compliance with ISO certification	As per vendor's specifications in compliance with ISO certification	Professional calibration vendor (ISO 17025 certified)	SOP11880

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria[1]**	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference[2]**
Thermo 6000 Series ICP-atomic emission spectroscopy	Calibration checks	As needed maintenance/ calibration checks every 10 injections	90 to 110% for the calibration checks	Recalibration	ELLE Analyst	WI11931

Notes:

[1] This table presents the acceptance criteria. Refer to the method SOP for specific or more information.

[2] SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.

ASTM = ASTM International
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
ISO = International Organization for Standardization

WORKSHEET #24-9: ANALYTICAL INSTRUMENT CALIBRATION – ANALYTICAL BALANCE

Instrument	**Calibration Procedure**	**Frequency of Calibration**	**Acceptance Criteria[1]**	**Corrective Action**	**Person Responsible for Corrective Action**	**SOP Reference[2]**
Analytical balance	Annual calibration and maintenance	Annual	As per vendor's specifications in compliance with ISO certification	As per vendor's specifications in compliance with ISO certification	ELLE Analyst	SOP11880

Notes:
1This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
2SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
ISO = International Organization for Standardization

WORKSHEET #24-10: ANALYTICAL INSTRUMENT CALIBRATION – TOTAL SUSPENDED SOLIDS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
TSS	NA	NA	NA	NA	ELLE Analyst	WI11604

Notes:
1This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
2SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
NA = Not Applicable
TSS = Total Suspended Solids

WORKSHEET #24-11: ANALYTICAL INSTRUMENT CALIBRATION – TOTAL DISSOLVED SOLIDS

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria[1]	Corrective Action	Person Responsible for Corrective Action	SOP Reference[2]
TDS	NA	NA	NA	NA	ELLE Analyst	WI11598

Notes:
1This table presents the acceptance criteria. Refer to the method SOP for specific or more information.
2SOPs are reviewed/revised annually. The current version will be followed at the time of sample receipt.
ELLE = Eurofins Lancaster Laboratories Environmental, LLC
TDS = Total Dissolved Solids

QAPP WORKSHEET #28: ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

(UFP-QAPP Manual Section 3.4)
(USEPA 2106-G-05 Section 2.3.5)

This worksheet includes information for each sampling technique, analytical method/SOP, matrix, and analytical group.

WORKSHEET #28-1: LABORATORY QC SAMPLES (RSK-175)

Matrix	Water
Analytical Group	Carbon Dioxide
Analytical Method / SOP Reference	RSK-175/WI9644

QC Sample	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
Method blanks	1 per prep batch of up to 20 samples	No analytes detected > ½ LOQ or >$^{1}/_{10}$ the amount measured in any sample	Reanalyze to confirm detections. If detects confirm, re-extract samples that are not ND or not >10x the blank value	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > ½ LOQ or >$^{1}/_{10}$ the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 (DoD 2019a). Laboratory statistical limits for compounds not in QSM	Flag outliers	ELLE Analyst	Accuracy/bias/precision	Results within acceptance limits

		5.3 (DoD 2019a); RPD ≤30%				
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 (DoD 2019a). Laboratory statistical limits for compounds not in QSM 5.3 (DoD 2019a); RPD ≤30%	Reanalyze LCS and associated samples. Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-extracted.	ELLE Analyst	Accuracy/bias/precision	Results within acceptance limits

ELLE =
LCSD = laboratory control sample duplicate
QSM = Quality Systems Manual

WORKSHEET #28-2: LABORATORY QC SAMPLES TABLE (WET CHEMISTRY – INORGANIC IONS BY IC (CL, SO_4)

Matrix	Water
Analytical Group	Wet Chemistry – Inorganic Ions by IC (Cl, SO_4)
Analytical Method /SOP Reference	EPA 300.0/9056; WI11626

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blanks	1 per prep batch of up to 20 samples	No analytes detected >MDL or $<{}^{1}/_{10}$ the amount measure in any sample.	Reanalyze to confirm detections. If detects confirm, re-extract samples that are not ND or not >10x the blank value	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > ½ LOQ or $>{}^{1}/_{10}$ the amount measured in any sample
MS	1 per 10 samples	Method limits	Flag outliers	ELLE Analyst	Accuracy/bias	Results within acceptance limits
LCS	1 per prep batch of up to 20 samples	Method limits	Reanalyze LCS and associated samples. Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-extracted.	ELLE Analyst	Accuracy/bias	Results within acceptance limits

Matrix	Water
Analytical Group	Wet Chemistry – Inorganic Ions by IC (Cl, SO_4)
Analytical Method /SOP Reference	EPA 300.0/9056; WI11626

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Duplicate	1 per 10 samples	Laboratory statistical	Flag outliers	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-3: LABORATORY QC SAMPLES (ALKALINITY)

Matrix	Water	
Analytical Group	Alkalinity	
Analytical Method/SOP Reference		SM 2320B-1997 or EPA 310.1; WI11475

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1 per prep batch of up to 20 samples	No analytes detected > LOQ or >$^{1}/_{10}$ the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, reanalyze samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > LOQ or >$^{1}/_{10}$ the amount measured in any sample
MS	1 per 20 samples	Laboratory statistical window	Flag outliers	ELLE Analyst	Accuracy/bias	Results within acceptance limits
LCS	1 per prep batch of up to 20 samples	Laboratory statistical window	Reanalyze LCS and associated samples.	ELLE Analyst	Accuracy/bias	Results within acceptance limits

Matrix	Water	
Analytical Group	Alkalinity	
Analytical Method/SOP Reference		SM 2320B-1997 or EPA 310.1; WI11475

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
			Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-analyzed.			
Duplicate	1 per 10 samples	Laboratory statistical RPD	Flag data	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-4: LABORATORY QC SAMPLES (ANIONS/9056)

Matrix	Water
Analytical Group	Anions by IC
Analytical Method/SOP Reference	EPA 9056A SA-GE-115

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1/preparatory batch (20 samples)	No target compounds >½ LOQ	Correct problem then re-prep and analyze method blank and all samples processed with the contaminated blank. Report results if sample results >10x blank result or sample results ND.	Analyst/Section Supervisor	Contamination	No target compounds >½ LOQ
LCS	1/preparatory batch (20 samples)	QSM (DoD 2019a) limits	Reanalyze LCS once. If acceptable, report. If LCS has high bias and samples are ND, report with case narrative comment. If LCS has low bias, evaluate and re-prep and reanalyze the LCS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available.	Analyst/Section Supervisor	Accuracy/bias	QSM (DoD 2019a) control limits
MS/MSD	1/preparatory batch (20 samples)	<u>Recovery</u>: QSM (DoD 2019a) limits <u>RPD</u>: $\leq$15%	If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error. For specific analyte(s) in parent sample, apply	Analyst/Section Supervisor	Accuracy/bias/ precision	QSM (DoD 2019a) control limits

Matrix	Water
Analytical Group	Anions by IC
Analytical Method/SOP Reference	EPA 9056A SA-GE-115

QC Sample	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
			J-flag if acceptance criteria are not met. Explain in the case narrative.			
Duplicate	1/preparatory batch (20 samples) if no MSD	RPD between duplicates ≤15%	For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. Data will be evaluated to determine the source of the difference.	Analyst/Section Supervisor	Precision	RPD ≤15%

WORKSHEET #28-5: LABORATORY QC SAMPLES (TOC)

Matrix	Soil/Sediment
Analytical Group	TOC
Analytical Method/ SOP Reference[2]	SM 5310B/SW-946 9060; WI11627

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blanks	1 per prep batch of up to 20 samples	No analytes detected > LOQ or >$^{1}/_{10}$ the amount measured in any sample or $^{1}/_{10}$ the regulatory limit, whichever is greater	Reanalyze blank to confirm detections. If detects confirm, re-prep samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > LOQ or >$^{1}/_{10}$ the amount measured in any sample or $^{1}/_{10}$ the regulatory limit, whichever is greater
MS	1 per 10 samples	Laboratory statistical limits	Flag outliers	ELLE Analyst	Accuracy/bias	Results within acceptance limits
LCS	1 per prep batch of up to 20 samples	Laboratory statistical limits	Correct problem, reprepare and reanalyze the LCS and all sample associated	ELLE Analyst	Accuracy/bias	Results within acceptance limits
Duplicate	1 per 10 samples	Laboratory statistical RPD	Flag data	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-6: LABORATORY QC SAMPLES (DOC)

Matrix	Water
Analytical Group	DOC
Analytical Method/SOP Reference	SM5310C/415.1; WI11637

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blanks	1 per prep batch of up to 20 samples	No analytes detected > MDL or >$^1/_{10}$ the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, re-prep samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > MDL or >$^1/_{10}$ the amount measured in any sample
MS	1 per 10 samples	Laboratory statistical limits	Flag outliers	ELLE Analyst	Accuracy/bias	Results within acceptance limits
LCS	1 per prep batch of up to 20 samples	Laboratory statistical limits	Correct problem, reprepare and reanalyze the LCS and all sample associated	ELLE Analyst	Accuracy/bias	Results within acceptance limits
Duplicate	1 per 10 samples	Laboratory statistical RPD	Flag data	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-7: LABORATORY QC SAMPLES (PERCENT MOISTURE)

Matrix	Soil/Sediment
Analytical Group	Percent Moisture
Analytical Method/SOP Reference	SM 2540 G-1997 %Moisture Calc; WI10697

QC Sample	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
LCS	1 per prep batch of up to 20 samples	Laboratory statistical windows	Correct problem, reprepare and reanalyze the LCS and all sample associated	ELLE Analyst	Accuracy/bias	Results within acceptance limits
Duplicate	1 per 10 samples	Laboratory statistical RPD	No corrective action, matrix related	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-8: LABORATORY QC SAMPLES (METALS-SOIL/SEDIMENT)

Matrix	Soil/Sediment
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020/A/B/WI11933

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1 per prep batch of up to 20 samples	No analytes detected > ½ RL or >$^{1}/_{20}$ the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/laboratory contamination	No analytes detected > ½ RL or >$^{1}/_{20}$ the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 (DoD 2019a) ±25% for elements not in QSM 5.3; RPD ≤20%	Analyze post-digestion spike and serial dilution	ELLE Analyst	Accuracy/bias/precision	Results within acceptance limits
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 (DoD 2019a) ±20% for elements not in QSM 5.3; RPD ≤20%	Analytes in the LCS that fail high and are ND in the samples can be reported. All others are redigested and reanalyzed.	ELLE Analyst	Accuracy/bias/precision	Results within acceptance limits

Matrix	Soil/Sediment					
Duplicate	1 per prep batch of up to 20 samples	RPD must be ≤20%	Flag data.	ELLE Analyst	Precision	Results within acceptance limits
Serial dilutions	Must be prepared with each background sample, evaluated only when analyte concentrations are >50x the LOQ	The percent difference must be ≤10%	Flag data.	ELLE Analyst	Precision	Results within acceptance limits
Post-digestion spike	Prepare with each background sample	± 20% true value	No specific action needed unless required by the project. Post-digestion spike is reported in data package.	ELLE Analyst	Accuracy/bias	Results within acceptance criteria
Internal standard	Every sample and QC	Must be 50 to 130% of the calibration blank	Reanalyze at a dilution.	ELLE Analyst	Precision	Results within acceptance criteria

WORKSHEET #28-9: LABORATORY QC SAMPLES (METALS-WATER)

Matrix	Water
Analytical Group	Metals
Analytical Method/SOP Reference	SW-846 6020A/B/WI11933

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1 per prep batch of up to 20 samples	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Laboratory Contamination	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 ±25% for elements not in QSM 5.3; RPD ≤20%	Analyze post digestion spike and serial dilution	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3 ±20% for elements not in QSM 5.3; RPD ≤20%	Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-digested and reanalyzed.	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits

Matrix	Water					
Duplicate	1 per prep batch of up to 20 samples	RPD must be ≤20%	Flag data	ELLE Analyst	Precision	Results within acceptance limits
Serial Dilutions	Must be prepared with each background sample, evaluated only when analyte concentrations are >50x the LOQ	The percent difference must be ≤10%	Flag data	ELLE Analyst	Precision	Results within acceptance limits
Post Digestion Spike	Prepare with each background sample	± 20% True Value	No specific action needed unless required by the project. PDS is reported in data package	ELLE Analyst	Accuracy/Bias	Results within acceptance criteria

WORKSHEET #28-10: LABORATORY QC SAMPLES (METALS-HG-WATER)

Matrix	Water
Analytical Group	Metals-Hg
Analytical Method/SOP Reference	SW-846 7470A /WI7965

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method Blank	1 per prep batch of up to 20 samples	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Laboratory Contamination	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Flag outliers	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-digested and reanalyzed.	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits

Matrix	Water					
Duplicate	1 per prep batch of up to 20 samples	RPD must be ≤20%	Flag data	ELLE Analyst	Precision	Results within acceptance limits
Method of Standard Addition (MSA)	When dilution fails or project requires it	NA	NA	ELLE Analyst	NA	Document in data package

WORKSHEET #28-11: LABORATORY QC SAMPLES (METALS-HG-SOIL/SEDIMENT)

Matrix	Soil/Sediment
Analytical Group	Metals-Hg
Analytical Method/SOP Reference	SW-846 7471A/B / WI7965

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
Method Blank	1 per prep batch of up to 20 samples	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Laboratory Contamination	No analytes detected > 1/2 RL or >1/20 the amount measured in any sample
MS/MSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Flag outliers	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits
LCS/LCSD	1 per prep batch of up to 20 samples	Recovery limits per QSM 5.3; RPD ≤20%	Analytes in the LCS that fail high and are ND in the samples can be reported. All others are re-digested and reanalyzed.	ELLE Analyst	Accuracy/Bias/Precision	Results within acceptance limits

Matrix	Soil/Sediment					
Duplicate	1 per prep batch of up to 20 samples	RPD must be ≤20%	Flag data	ELLE Analyst	Precision	Results within acceptance limits
Method of Standard Addition (MSA)	When dilution fails or project requires it	± 20% True Value	None, unless required by project	ELLE Analyst	Accuracy/Bias	Results within acceptance criteria. Report PDS in data package

WORKSHEET #28-12: LABORATORY QC SAMPLES (PH)

Matrix	Soil/Sediment
Analytical Group	pH
Analytical Method/SOP Reference	SW-846 6020B/WI11931

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
LCS	1 per batch	Laboratory statistical window	Reanalyze blank to confirm detections. If detects confirm, redigest samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Bias	Results within acceptance limits
Duplicate	1 per 10 samples	Laboratory statistical RPD	No corrective action, matrix related	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-13: LABORATORY QC SAMPLES (GRAIN SIZE)

Matrix	Solid
Analytical Group	Grain Size
Analytical Method/SOP Reference	ASTMD422; WI11514

QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**DQI**	**MPC**
Duplicate (when duplicate volume is provided)	1 per batch	Laboratory statistical RPD	No corrective action, matrix related	ELLE Analyst	Analytical Precision	Results within acceptance limits

WORKSHEET #28-14: LABORATORY QC SAMPLES (TSS)

Matrix	Water
Analytical Group	TSS
Analytical Method/SOP Reference	SM2540D-1997;WI11604

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1 per prep batch of up to 20 samples	No analytes detected > LOQ or >1/10 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, reprep samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Laboratory Contamination	No analytes detected > LOQ or >1/10 the amount measured in any sample
LCS	1 per prep batch of up to 20 samples	Laboratory statistical windows	Correct problem, reprepare	ELLE Analyst	Accuracy/Bias	Results within acceptance limits

Matrix	Water					
			and reanalyze the LCS and all sample associated			
Duplicate	1 per 10 samples	Method RPD	Flag data	ELLE Analyst	Precision	Results within acceptance limits

WORKSHEET #28-15: LABORATORY QC SAMPLES (TDS)

Matrix	Water
Analytical Group	TDS
Analytical Method/SOP Reference	SM2540C-1997;WI11598

QC Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	MPC
Method blank	1 per prep batch of up to 20 samples	No analytes detected > LOQ or >1/10 the amount measured in any sample	Reanalyze blank to confirm detections. If detects confirm, reprep samples that are not ND or not >10x the blank value.	ELLE Analyst	Accuracy/Laboratory Contamination	No analytes detected > LOQ or >1/10 the amount measured in any sample
LCS	1 per prep batch of up to 20 samples	Laboratory statistical windows	Correct problem, reprepare and reanalyze	ELLE Analyst	Accuracy/Bias	Results within acceptance limits

Matrix	Water

			the LCS and all sample associated			
Matrix Spike	1 per 20 samples	Laboratory statistical windows	Flag outliers	ELLE Analyst	Matrix Spike	1 per 20 samples
Duplicate	1 per 10 samples	Method RPD	Flag data	ELLE Analyst	Precision	Results within acceptance limits

REFERENCES

AMEC. 2010. Remedial Investigation for AOC 72, Plow Shop Pond – Draft. Devens, Massachusetts. Prepared for U.S. Army Corps of Engineers New England District. March.

DoD. 2019a. Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5.3. DoD Environmental Data Quality Workgroup. May.

DoD. 2019b. General Data Validation Guidelines. November.

KGS. 2018. 2018 Addendum to the Long-Term Monitoring and Maintenance Plan – Shepley's Hill Landfill. January.

KGS. 2020a. Draft 2019 Annual Operations, Maintenance, and Monitoring Report, Shepley's Hill Landfill, Former Fort Devens Army Installation. May.

KGS. 2020b. Final Fifth 2020 Five-Year Review Report, Former Fort Devens Army Installation, BRAC Legacy Sites, Devens, Massachusetts. Prepared for the U.S. Army Corps of Engineers New England District. September.

Intergovernmental Data Quality Task Force. 2005. Uniform Federal Policy for Quality Assurance Project Plans. March.

SERES-Arcadis JV. 2020. Quality Assurance Project Plan, Annual Long-Term Monitoring and Maintenance Program, Former Fort Devens Army Installation, Devens, Massachusetts. Prepared for U.S. Army Corps of Engineers New England District. October.

SERES-Arcadis JV. 2021. Final 2020 Annual Operations, Maintenance, and Monitoring Report. Shepley's Hill Landfill, Former Fort Devens Army Installation, Devens, Massachusetts. Prepared for U.S. Army Corps of Engineers New England District. August.

Sovereign. 2015a. Record of Decision for Plow Shop Pond (AOC 72) – Red Cove and Former Railroad Round House (SA-71), Former Fort Devens Army Installation, Devens, Massachusetts. September.

Sovereign. 2015b. Long-Term Monitoring and Maintenance Plan Update. Shepley's Hill Landfill, Former Fort Devens Army Installation, Devens, Massachusetts. September.

USACE. 2021. Addendum No. 002 to the Seed Task Order for Additional Work at Shepley's Hill Landfill. Performance Work Statement for Environmental Services BRAC Legacy Sites At Former Fort Devens, Devens, MA. February. IDIQ CONTRACT NO. W912WJ19D0014 (Seed) Task Order No. W912WJ20F0022 Modification.

U.S. Army Environmental Center. 1995. Record of Decision, Shepley's Hill Landfill Operable Unit, Fort Devens, Massachusetts. Fort Devens Feasibility Study for Group 1A Sites. September.

USEPA. 2008. Final Report Arsenic Fate, Transport and Stability Study – Groundwater, Surface Water, Soil and Sediment Investigation – Fort Devens Superfund Site, Devens, Massachusetts. U.S. Environmental Protection Agency Office of Research and Development. September 30.

USEPA. 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. January.

USEPA Region I. 2017. Low Stress (Low-Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells. September.

USEPA. 2020. Email from C. Keating (USEPA) to R. Herzig (MassDevelopment), P. Reddy (USACE), H. Levesque (Seres-ES), A. Vitolins (Arcadis), and D. Chaffin (DEP) Re: Recommendation to Develop/Implement Field Sampling Program to Evaluate Barrier Wall Performance and Verify Attainment of RAO for Plow Shop Pond SHL - 2020 Devens Five Year Review. July 20.

ATTACHMENT 1

FIELD SAMPLING STANDARD OPERATING PROCEDURES

TGI – SOIL DRILLING AND SAMPLE COLLECTION

Rev #: 1

Rev Date: May 12, 2020

VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 11, 2018	All	Updated and re-written as a TGI	Marc Killingstad
1	May 12, 2020	None	Review – no changes necessary	Marc Killingstad

APPROVAL SIGNATURES

Prepared by: 10/11/2018

Christopher Keen Date:

Technical Expert Reviewed by: 05/12/2020

Marc Killingstad (Technical Expert) Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM’s responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project’s scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) describes general drilling procedures and the methods to be used to field screen and collect soil samples for laboratory analysis in unconsolidated sediments. For soil description procedures, please refer to the *TGI - Soil Description*. For monitoring well installation in granular aquifers, please refer to the *TGI - Monitoring Well Installation*.

Overburden (unconsolidated sediments) drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and hand tools may also be used. Drilling within consolidated materials such as fractured bedrock is commonly performed using water-rotary (coring or tri-cone roller bit), air rotary or rotasonic methods. For guidance when drilling in consolidated materials (i.e., bedrock), please refer to the *TGI – Bedrock Core Collection and Description*.

The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

Field screening of soil samples is commonly performed using a photoionization detector (PID) and/or a flame ionization detector (FID). These instruments are used to measure relative concentrations of volatile organic compounds (VOCs) for the selection of samples for further laboratory or field analysis. Field screening for dense non-aqueous phase liquids (DNAPL) may be performed using hydrophobic dye (Oil Red O or Sudan IV), which is pertinent at chlorinated solvent sites.

Collection of soil samples for laboratory analysis may be performed using a variety of techniques including grab samples and composite or homogenized samples. Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample. Samples for VOC analysis will not be homogenized or composited and are collected as discrete grab samples.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools).

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or state/federal regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, Arcadis field personnel will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan (FIP), Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Personnel responsible for overseeing drilling operations will have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

Arcadis personnel directing, supervising, or leading soil sampling activities will have a minimum of 1 year of previous environmental soil sampling experience. Field employees with less than 6 months of experience will be accompanied by a supervisor (as described above) to ensure that proper sample collection techniques are employed.

Additionally, the Arcadis field team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

4 EQUIPMENT LIST

The following materials will be available, as required, during soil boring drilling, field screening, and sampling activities:

- Site-specific HASP and health and safety documents identified in the HASP
- Field Implementation Plan (FIP)/work plan that includes site map with proposed boring locations, field sampling plan (with corresponding depths, sample analyses, sample volume required, and sample holding time), and previous boring logs (as available)
- Appropriate personal protective equipment (PPE), as specified in the HASP
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Photoionization detector (PID), flame ionization detector (FID) or other air monitoring equipment, as needed, in accordance with the HASP
- Drilling equipment required by *ASTM D1586*, when performing split-spoon sampling
- Disposable plastic liners, when drilling with direct-push equipment
- Appropriate soil sampling equipment (e.g., stainless steel spatulas/spoons/bowls, knife)
- Stainless steel hand auger and stainless-steel spade if using manual methods
- Indelible ink pens
- Engineer’s ruler or survey rod
- Sealable plastic bags (e.g., Ziploc®)
- Air-tight sample containers and 8-oz. glass Mason jars or driller’s jars
- Aluminum foil
- Plastic sheeting (e.g., Weatherall Visqueen)
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels)
- Appropriate sample blanks (trip blank supplied by the laboratory), as specified in the FSP
- Soil sample containers and labels (supplied by the laboratory) appropriate for the analytical method(s) with preservative, as needed (parameter-specific)
- Appropriate transport containers (coolers) with ice and appropriate labeling, packing, and shipping materials;
- Appropriate soil boring log (**Attachment 1**)
- Chain-of-custody forms
- Field notebook.

- Digital camera (or smart phone with camera)
- Drums or other containers appropriate for soil and decontamination water, as specified by the site investigation-derived waste (IDW) management plan, and appropriate drum labels

5 CAUTIONS

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See appropriate guidance for proper utility clearance protocol. Work will be performed in accordance with the Arcadis *Utility Location and Clearance Health and Safety Standard* and the *Utilities and Structures Checklist* will be completed before beginning any intrusive work.

Prior to beginning field work, the project technical team will ensure that all field logistics (e.g., access issues, health and safety issues, communication network, schedules, etc.) and task objectives are clearly understood by all team members. An internal call with the project technical team to review the FIP/work plan scope and objectives is strongly recommended prior to mobilization to ensure that the field work will be effectively and efficiently executed.

Some regulatory agencies have specific requirements regarding borehole abandonment and grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling plan.

If DNAPL is known or expected to exist at the site, refer to the project specific documents (e.g., DNAPL Contingency Plan) for additional details regarding drilling to reduce the potential for inadvertent DNAPL remobilization.

Similarly, if light non-aqueous phase liquid (LNAPL) is known or expected to be present as “perched” layers above the water table, refer to the DNAPL Contingency Plan. Follow the general provisions and concepts in the DNAPL contingency plan during drilling above the water table at known or expected LNAPL sites.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Water used for drilling, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply will be considered.

Specifications of materials used for backfilling the borehole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPL is likely to be present or in groundwater with high salinity. In these situations, neat cement grout is preferred.

Store and/or stage empty and full sample containers and coolers out of direct sunlight. Be careful not to over-tighten lids with Teflon® liners or septa. Over-tightening can impair the integrity of the seal and can cause the glass to shatter and create a risk for hand injuries.

NOTE: *Field logs and some forms are considered to be legal documents. All field logs and forms will therefore be filled out in indelible ink. Do not use permanent marker or felt-tipped pens for labels on* *sample container or sample coolers. Permanent markers could introduce volatile constituents into the samples.*

NOTE: *An Arcadis employee that is appropriately trained at the correct level of internal hazardous materials/DOT (Department of Transportation) shipping must complete an Arcadis shipping determination to address applicable DOT and IATA (International Air Transport Association) shipping requirements. Review the applicable Arcadis procedures and guidance instructions for sample packaging and labeling. Prior to using air transportation, confirm air shipment is acceptable under DOT and IATA regulations.*

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Working outside at sites with suspected contamination may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include biological hazards (e.g., stinging insects, ticks in long grass/weeds, etc.), and potentially the use of sharp cutting tools (scissors, knife). Only use non-toxic peppermint oil spray for stinging insect nests. Review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives and use appropriate hand protection.

If thunder or lighting is present, discontinue drilling and sampling until 30 minutes have passed after the last occurrence of thunder or lighting.

7 PROCEDURE

The procedures for drilling and the methods to be used to field screen and collect soil samples for laboratory analysis are presented below:

DRILLING PROCEDURES

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area.
2. Advance boring to designated depth:
    a. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent)
    b. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent)

    c. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
    d. A common sampling method that produces high-quality soil samples with relatively little soil disturbance is described in *ASTM D1586 – Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM D1586).
        i. Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary
    e. Rotasonic drilling produces soil cores that, for the most part, are relatively undisturbed, but note that when drilling in consolidated or finer-grained sediment the vibratory action during core barrel advancement may create secondary fractures or breaks
    f. Dual-rotary removes cuttings by compressed air or water/mud and allow only a general assessment of geology

3. Describe each soil sample as outlined in the appropriate project records (refer to the description procedures outlined in the *TGI - Soil Description*)
    a. Record descriptions on the soil boring log (**Attachment 1**) and/or field notebook.
    b. When possible photo document the samples (e.g., soil cores, split-spoons)
    c. During soil boring advancement, document all drilling events in field notebook, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments) and work stoppages
    d. Blow counts will not be available if rotasonic, dual-rotary, or direct-push methods are used; however, if standard penetration testing is required during rotasonic drilling, an automatic drop hammer may be used in conjunction with the method to switch from core barrel advancement to standard penetration testing
4. The drilling contractor will be responsible for obtaining accurate and representative samples, informing the supervising Arcadis geologist of changes in drilling pressure, and keeping a separate general log of soils encountered, including blow counts
    a. The term “samples” means soil materials from particular depth intervals, whether or not portions of these materials are submitted for laboratory analyses
    b. Records will also be kept of occurrences of premature refusal due to boulders or construction materials that may have been used as fill
    c. Where a boring cannot be advanced to the desired depth, the boring will be abandoned, and an additional boring will be advanced at an adjacent location to obtain the required sample
    d. Where it is desirable to avoid leaving vertical connections between depth intervals (e.g., if DNAPL or perched LNAPL are known or expected to exist at the site), the borehole will be sealed using cement and/or bentonite (see **Section 5** above)

    e. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location

Direct-Push Method

The direct-push drilling method may also be used to complete soil borings. Examples of this technique include Geoprobe®, Diedrich Environmental Soil Probe (ESP) System, or AMS PowerProbe. Environmental probe systems typically use a hydraulically operated percussion hammer.

Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff to medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual tube liner for sampling soil (dual tube sampling system).

The outside diameter (OD) of the outer casing ranges from 2.25 to 6 inches and the OD of the inner sampling tube diameter ranges from 1.4 to 4.5 inches. The outer casing isolates overlying soil and permits the unit to continue to probe at depth. The dual tube sampling system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, a single rod system may be used that does not provide a cased boring and which does not allow for tremie-grouting from the bottom up.

The known or expected site conditions (e.g., presence of NAPL) will be evaluated when selecting the type of direct-push sampling system to be employed.

Direct-push drilling can generally achieve target depths 100 feet or less and the achievable depth is based on the site geology.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Advance soil boring to designated depth.
    a. Collect soil samples at appropriate interval as specified in in the FIP/work plan (or equivalent)
    b. Collect, document, and store samples for laboratory analysis as specified in in the FIP/work plan (or equivalent)
    c. Decontaminate equipment between samples in accordance with in the FIP/work plan (or equivalent)
    d. Samples will be collected using dedicated, disposable, plastic liners
3. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

Manual Methods

Manual methods may also be used to complete shallow soil borings. Examples of this technique include using a spade, spoon, scoop, hand auger, or slide hammer. Manual methods are typically used to collect surface soil samples (0 to 6 inches) or to complete soil borings/collect soil samples from a depth of 5 feet or less.

1. Find/identify boring location, establish work zone, and set up sampling equipment decontamination area
2. Clear the ground surface of brush, root mat, grass, leaves, or other debris
3. Use a spade, spoon, scoop, hand auger, or slide hammer to collect a sample of the required depth interval
4. Use an engineer's ruler or survey rod to verify that the sample is collected to the correct depth and record the top and bottom depths from the ground surface
5. To collect samples below the surface interval, remove the surface interval first; then collect the deeper interval
    a. To prevent the hole from collapsing, it may be necessary to remove a wider section from the surface or use cut polyvinyl chloride (PVC) pipe to maintain the opening
    b. Collect soil samples at appropriate interval as specified in the FIP/work plan (or equivalent)
    c. Collect, document, and store samples for laboratory analysis as specified in the FIP/work plan (or equivalent)
    d. Decontaminate equipment between samples in accordance with the FIP/work plan (or equivalent)
6. Describe samples in accordance with the procedures outlined in **Step 3** under ***Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods*** above (refer to the description procedures outlined in the *TGI - Soil Description*)

FIELD SCREENING PROCEDURES

PID and FID Screening

Soils are typically field screened with a PID or FID for a relative measure of the total VOCs at sites where VOCs are known or suspected to exist. The PID employs a UV lamp to measure VOCs and the ionization energy (IE) of the site constituents need to be considered when selecting the type of lamp (e.g., 10.6 eV, 11.7 eV) that will be used. In general, any compound with an IE lower than that of the lamp photons can be measured. The FID has a wide linear range and responds to almost all VOCs. Field screening is performed using one (or both) of the following two methods:

1. Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a gloved hand. Such readings will be obtained at several locations along the length of the sample.
2. A portion of the collected soil is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature. After warming, the cover is removed, the foil is pierced with the PID or FID probe, and a reading is obtained.

Initial PID readings will be recorded on the soil boring log (**Attachment 1**) and/or in the field notebook. The soil sample will be separated from the slough material (if any) by using disposable gloves and a pre-cleaned stainless-steel spoon.

For the second method, a representative portion of the sample will be placed in a pre-cleaned air-tight 8-ounce container (as quickly as possible to avoid loss of VOCs), filling the container half full to allow for the accumulation of vapors above the soil. An aluminum foil seal will be placed between the glass and metal cap and the cap will be screwed on tightly. Unless the screening will be performed immediately after the sample is placed in the container, the sample containers will be stored in a cooler chilled to approximately 4ºC until screening can be performed.

The headspace of the 8-ounce container will be measured using a PID or FID as follows:

1. Samples will be taken to a warm work space and allowed to equilibrate to room temperature for at least one hour.
2. Prior to measuring the soil vapor headspace concentration, the 8-ounce container will be shaken.
3. The headspace of the sample will then be measured directly from the 8-ounce container by piercing the aluminum foil seal with the probe of the PID or FID and measuring the relative concentration of VOCs in the headspace of the soil sample. The initial (peak) reading must be recorded.

The PID or FID must be calibrated according to the manufacturer's specifications at a minimum frequency of once per day prior to collecting PID or FID readings. The PID will be calibrated to a benzene-related compound (isobutylene) while the FID will be calibrated to methane.

The time, date, and calibration procedure must be clearly documented in the field notebook and/or the calibration log book.

If at any time the PID or FID results appear erratic or inconsistent with field observations, then the instrument will be recalibrated.

If calibration is difficult to achieve, then the PID’s lamp will be checked for dirt or moisture and cleaned, or technical assistance will be required. Maintenance and calibration records will be kept as part of the field quality assurance program.

NAPL Screening

To screen for the potential presence of non-aqueous phase liquid (NAPL) in soil, drilling procedures must allow for high-quality porous media samples to be taken. Split-spoon samplers or direct-push samplers will be collected continuously ahead of the auger, drill casing/rods, or probe rods.

Upon opening each split-spoon sampler or direct-push plastic liner sleeve, the soil will immediately be evaluated for the presence of visible NAPL. If NAPL is immediately visible in the sample, its depth will be noted.

Additionally, the soil will be screened for the presence of organic vapors using a PID or FID. During screening, the soil will be split open using a clean spatula or knife and the PID or FID probe will be placed in the opening and covered with a gloved hand (**Method 1** above). Such readings will be obtained along the entire length of the sample. Alternatively, **Method 2** for PID/FID screening (outlined above) may also be performed. If the PID or FID examination reveals the presence of organic vapors above 100 parts per million (ppm), the sample will undergo further detailed evaluation for visible NAPL.

The assessment for NAPL will include the following tests/observations:

- Evaluation for Visible NAPL Sheen or Free-Phase NAPL in Soil Sampler
    - NAPL sheen will be a colorful iridescent appearance on the soil sample
    - NAPL may also appear as droplets or continuous accumulations of liquid with a color typically ranging from yellow to brown to black, depending on the type of NAPL
    - Creosote DNAPL (associated with wood-treating sites) and coal tar DNAPL (associated with manufactured gas plant [MGP] sites) are typically black and have a characteristic, pungent odor
    - Pure chlorinated solvents may be colorless in the absence of hydrophobic dye. Solvents mixed with oils may appear brown
    - Particular care will be taken to fully describe any sheens observed, staining, discoloration, droplets (blebs), or NAPL saturation
- Soil-Water Pan Test
    - A portion of the selected soil interval with the highest PID or FID reading above 100 ppm will be placed in a disposable polyethylene dish along with a small volume of potable or distilled water
    - The dish will be gently tilted back and forth to mix the soil and water, and the surface of the water will be viewed in natural light to observe the development of a sheen, if any
    - A small quantity of Oil Red O or Sudan IV hydrophobic dye powder will be added, and the soil and dye will be manually mixed for approximately 30 to 60 seconds and smeared in the dish to create a paste-like consistency
    - A positive test result will be indicated by a sheen on the surface of the water and/or a bright red color imparted to the soil following mixing with dye
- Soil-Water Shake Test
    - A small quantity of soil (up to 15 cc) will be placed in a clear, colorless, jar containing an equal volume of potable or distilled water (40-mL vials are well suited to this purpose, but not required)
    - After the soil settles into the water, the surface of the water will be evaluated for a visible sheen under natural light
    - The jar will be closed and gently shaken for approximately 10 to 20 seconds
    - Again, the surface of the water will be evaluated for a visible sheen or a temporary layer of foam
    - A small quantity (approximately 0.5 to 1 cc) of Oil Red O or Sudan IV powder will be placed in the jar
    - The sheen layer, if present, will be evaluated for a reaction to the dye (change to bright red color)
    - The jar will be closed and gently shaken for approximately 10 to 20 seconds
    - The contents in the closed jar will be examined under natural light for visible bright red dyed liquid inside the jar

- A positive test result will be indicated by the presence of a visible sheen or foam on the surface of water, a reaction between the dye and the sheen layer upon first addition of the dye powder, a bright red coating on the inside of the vial (particularly above the water line), or red-dyed droplets within the soil

NOTE: *If NAPL is obviously present upon opening the soil sampler or evaluating the soil sample within the split-spoon sampler or direct-push liner sleeve, it is not necessary to perform a soil-water pan test or soil-water shake test. In addition, it is not necessary to perform both a soil-water pan test and a soil-water shake test; either test method is acceptable. The pan test may be preferred in some circumstances because the presence of a sheen may be easier to see on a wider surface.*

NOTE: *When using hydrophobic dye in the tests above, color will be assessed outdoors under natural light during the period between sunrise and sunset, regardless of the degree of cloud cover. The hydrophobic dye Safety Data Sheets (SDS) will be incorporated into the HASP and reviewed prior to use and the dyes will be carefully handled and disposed in accordance with regulations*.

SOIL SAMPLE COLLECTION FOR LABORATORY PROCEDURES

If not specifically identified in the FIP, soil samples will be selected for laboratory analysis based on:

1. Their position in relation to identified source areas
2. The visual presence of source residues (e.g., NAPL)
3. The relative levels of total VOCs based on field screening measurements
4. The judgment of the field coordinator

Samples designated for laboratory analysis will be placed in the appropriate containers.

Sample containers for VOC analysis will be filled first immediately following soil core retrieval to reduce loss of VOCs.

If samples will be collected for other analytical parameters, a sufficient amount of the remaining soil will then be homogenized as described below and sample containers will be filled for other parameters.

VOC samples will be collected as discrete samples using a small diameter core sampler (e.g., En Core® Sampler, Terra Core™ Sampler).

The En Core® Sampler is a disposable volumetric sampling device that collects, stores and delivers soil samples without in-field chemical preservation. The En Core® Sampler requires the use of a reusable T-handle.

The Terra Core™ Sampler is a one-time use transfer tool, designed to collect soil samples and transfer them to the appropriate containers for in-field chemical preservation (e.g., methanol).

The small diameter core sampler will be used according to the manufacturer's instructions (e.g., En Novative Technologies). Some regulatory agencies have specific requirements regarding VOC sample collection. Determine whether the oversight agency has specific requirements prior to commencing sampling and collect samples at appropriate interval as specified in the FIP/work plan (or equivalent). Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample.

NOTE: *Samples for VOC analysis will NOT be homogenized or composited and will be collected as discrete samples as described above*.

The procedure for mixing samples is provided below.

1. Mix the materials in a stainless steel (or appropriate non-reactive material) bowl using a stainless-steel spoon (or disposable equivalents)
    a. When dealing with large sample quantities, use disposable plastic sheeting and a shovel or trowel
    b. NOTE: *When preparing samples for metals analyses, do not use disposable aluminum (or metal tools or trays other than stainless steel), as it may influence the analytical results*
2. Flatten the pile by pressing the top without further mixing
3. Divide the circular pile by into equal quarters by dividing out two diameters at right angles
4. Mix each quarter individually using appropriate non-reactive bowls, spoons and/or sheeting
5. Mix two quarters (as described above) to form halves, then mix the two halves to form a composite or homogenized sample
6. Place composite or homogenized sample into specified containers
7. Remaining material will be disposed of in accordance with project requirements and applicable regulations
8. Sample containers will be labeled with sample identification number, date, and time of collection and placed on ice in a cooler (target 4° Celsius)
9. Samples selected for laboratory analysis will be documented (chain-of-custody forms), handled, packed, and shipped in accordance with the procedures outlined in the FIP/work plan (or equivalent).

8 WASTE MANAGEMENT

Investigative-Derived Waste (IDW) generated during drilling activities, including soil and excess drilling fluids (if used), decontamination liquids, and disposable materials (plastic sheeting, PPE, etc.) will be stored on site in appropriately labeled containers (disposable materials will be contained separately) and disposed of properly. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log book.

9 DATA RECORDING AND MANAGEMENT

Management of the original documents from the field will be completed in accordance with the site-specific QAPP.

In general, drilling activities will be documented on appropriate field/log forms as well as in a proper field notebook. All field data will be recorded in indelible ink. Field forms, logs/notes (including daily field and calibration logs), digital records, and chain-of-custody records will be maintained by the field team lead.

Initial field logs and chain-of-custody records will be transmitted to the Arcadis Certified Project Manager (CPM) and Technical Lead at the end of each day unless otherwise directed by the CPM. The field team leader retains copies of the field documentation.

Additionally, all documents (and photographs) will be scanned and electronically filed in the appropriate project directory for easy access. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of drilling activities, soil descriptions, soil boring information, and quantities of materials used.

In addition, the locations of soil borings will be documented photographically and in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

10 QUALITY ASSURANCE

Quality assurance procedures shall be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate TGI.

Field-derived quality assurance blanks will be collected as specified in the FIP/work plan and/or site-specific QAPP, depending on the project quality objectives. Typically, field rinse blanks (equipment blanks) will be collected when non-dedicated equipment (e.g., split-spoon sampler, stainless steel spoon) is used during soil sampling. Field rinse blanks will be used to confirm that decontamination procedures are sufficient and samples are representative of site conditions. Trip blanks for VOCs, which aid in the detection of contaminants from other media, sources, or the container itself, will be kept with the coolers and the sample containers throughout the sampling activities and during transport to the laboratory.

Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.

11 REFERENCES

ASTM D1586 - *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*. ASTM International. West Conshohocken, Pennsylvania.

12 ATTACHMENTS

Attachment 1. Soil Boring Log Form

ATTACHMENT 1

Soil Boring Log Form

ARCADIS Design & Consultancy for natural and built assets

SOIL BORING LOG

Boring/Well ____________ Project ____________ Page ____ of ____

Site Location ____________ Drilling Started ____________

Total Depth Drilled ________ Feet Hole Diameter ________ inches Drilling Completed ____________

Type of Sample or Coring Device ____________ Length and Diameter of Coring Device ____________ Sampling Interval ____________ feet

Drilling Method ____________ Drilling Fluid Used ____________

Drilling Contractor ____________ Driller ____________

Prepared By ____________ Helper ____________

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				**Udden-Wentworth Description:** principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, additional comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

ARCADIS Design & Consultancy for natural and built assets

SOIL BORING LOG (CONT'D)

Boring/Well__________ Prepared By __________ Page ____ of ____

Core Recovery (feet)	PID Reading (ppm)	Sample Depth (ft bgs)	MUD		SAND					GRAVEL				Udden-Wentworth Description: principal components, (angularity, plasticity, dilatency); minor components, (angularity, plasticity, dilatency); sorting, moisture content, consistency/density, color, addtl. Comments
			clay	silt	very fine	fine	medium	coarse	very coarse	granular	pebble	cobble	boulder	

ARCADIS
Design & Consultancy
for natural and
built assets

ARCADIS Design & Consultancy for natural and built assets

TGI – SURFACE SEDIMENT SAMPLING WITH GRAB SAMPLERS

Rev. #: 0

Rev Date: October 12, 2018

VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 12, 2018	All	Initial conversion to TGI format	Shannon Dunn

APPROVAL SIGNATURES

Prepared by: October 12, 2018

Jessica Geurts Date:

Technical Expert Reviewed by: Shannon Dunn October 12, 2018

Shannon Dunn - Technical Expert Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guidance Instruction (TGI) sets forth the field procedures for collection of surface sediment via Lexan tubing and grab samples using a hand-held dredge.

3 PERSONNEL QUALIFICATIONS

All field personnel must have the appropriate training required as described in the project Health and Safety Plan (HASP). Field sampling personnel will be versed in the relevant TGIs and will possess the skills and experience necessary to successfully complete the desired field work.

4 EQUIPMENT LIST

The following materials will be available, as required, during sediment sampling:

- personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP)

- sampling vessel equipped with health and safety equipment specified in the HASP; that has been inspected; is capable of supporting the work to be performed; and accommodates all workers, equipment, and samples. If the grab sampler is a full size ponar, or equivalent, the sampling vessel will have a davit, or equivalent, and winch from which to operate the grab sampler
- high-precision differential global positioning system (DGPS) with sub-meter accuracy pre-loaded with sample location target coordinates
- Navigation chart(s) for on-water activities (if available and as applicable);
- power grab, Van Veen, or Ponar sampler mounted on boat
- Lexan tubes (4-inch diameter) and plastic caps;
- weighted lead-line with accuracy of ±0.01 foot
- tape measure with accuracy of ±0.01 foot
- siphon tubing, peristaltic pump, and/or turkey baster
- Stainless-steel bowl or disposable aluminum pans;
- Stainless-steel spoons and/or spatulas;
- Steel core driver (i.e., slide hammer);
- calibrated rod for sediment depth measurement
- Polypropylene twine/rope
- Hacksaw, sawzall, or other cutting tool to cut core barrels into smaller sections, if needed
- calibrated photoionization detector (PID) and calibration gas
- zip-top plastic bags
- appropriate sample containers, labels, and forms
- camera
- dry erase board with marker
- indelible ink/rite in the rain pens
- decontamination supplies (see the TGI for Field Equipment Decontamination) including bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels
- field notebook, logging tools (see the TGI for Soil Description), field logs, and black ball point pen, and/or electronic data gathering environment (EDGE) tool
- sample packing and shipping materials (see the TGI for Chain-of-Custody and DOT HazMat sample handling, packing, and shipping handbook)
- Stainless steel, PVC or HDPE yard stick for measuring water depth.

5 CAUTIONS

Cores will be maintained vertically to minimize the potential for migration of porewater contaminants within the core when the core is horizontal. Decanting of overlying water in the core can result in suspension of surface sediment and the loss of that sediment.

6 HEALTH AND SAFETY CONSIDERATIONS

Health and safety hazards are discussed in the HASP.

7 PROCEDURE

Grab Sampler Procedure

The following procedure will be used to collect surface sediment with a grab sampler:

1. If publicly available data on surface water elevation is available, record these data. If publicly available data are not available, measure and record the surface water elevation at a surveyed reference location of known elevation where surface water conditions are equivalent to surface water at the sample location at 30-minute intervals throughout all on-water sampling activities.
2. Maneuver the sampling vessel to the target sample location and secure the vessel in place.
3. Decontaminate the grab sampler in accordance with the TGI for Field Equipment Decontamination. Inspect the grab sampler to ensure that bucket is able to fully close and seal and that top panels are secured in place.
4. Measure and record the sample location coordinates. Measure and record the water depth at the sample location immediately prior to each deployment of the sampler. Record the date and time, and wave/water conditions at the sample location at the time of each deployment of the sampler.
5. Lower the sampler through the water column until the sediment is reached.
6. Once the sampler is on the bottom, close the sampler jaws.
7. Using the winch, pull the sampler upward out of the sediments. Lift the sampler on board the vessel.
    a. Observe the water surface during grab deployment for evidence of sheen. Record observations.
8. Immediately open the sampler top panels. Assess and document the acceptability of the sample:
    a. Jaws are completely closed
    b. Sampler achieved target penetration depth and did not over penetrate. Over penetration is indicated by sediment pressed against the top panels or sediment on top of the sampler.
    c. Sampler sealed and retains standing surface water over the recovered sediment

If the sample is determined to meet the acceptability criteria, keep the sample and continue with Section Processing Surface Sediment Samples, described below. If these acceptability criteria are not achieved, return to step 5.

Push Core Procedure

The following procedure will be used to collect surface sediment with a push core:

1. If publicly available data on surface water elevation is available, record these data. If publicly available data are not available, measure and record the surface water elevation at a surveyed reference location of known elevation where surface water conditions are equivalent to surface water at the sample location at 30-minute intervals throughout all on-water sampling activities.

2. Maneuver the sampling vessel to the target sample location and secure the vessel in place.

3. Measure and record the sample location coordinates. Measure and record the water depth at the sample location immediately prior to each deployment of the sampler. Record the date and time, and wave/water conditions at the sample location at the time of each deployment of the sampler.

4. At each sample location, lower a section of Lexan tube until it reaches the top of sediment.

5. Push the Lexan tube by hand 6 inches into the sediment or until refusal is encountered. Measure the depth of sediment.

6. Seal the top of core tube with a cap to retain capillary pressure within barrel. Slowly pull the tube from the sediment. Before the bottom of the coring tube breaks the water surface, place a cap over the bottom to prevent the loss of material from the core tube. Place the cap on the core tube by reaching down into the water. Keep the core tube vertical at all times.

7. Document penetration depth (i.e. depth Lexan tube went into sediment), appearance of sediment, and recovery of the sample (i.e. linear measurement of sediment sample in Lexan tube) to confirm acceptability of the sample. Acceptability will be based on project specific definition of surface sediment depth. If sample does not meet acceptability criteria, return to step 4.

8. Once an acceptable sample is collected, continue with Processing Surface Sediment Samples, described below.

Processing Surface Sediment Samples

The following procedure will be used to process surface sediment samples:

1. Siphon the standing surface water off the sample with tubing primed with site water. Be careful not to disturb the sediment-water interface.

2. Photo-document the sediment sample to provide reference for post-processing questions regarding descriptions of color/staining, general texture, recovery, and other characteristics. Photos of the core will include a view of a dry-erase board marked with the grab sample ID, date, and time. The photo will also include a view of a tape measure for scale.

3. Log sediment samples according to the amended procedure described in the TGI for Soil Description.

4. Homogenize samples in a decontaminated stainless-steel bowl or disposable aluminum pan with a stainless-steel mixing spoon until the sediment is of uniform color.

Fill pre-cleaned laboratory-supplied sample jars with sample, as specified in the QAPP for the analytical suite

5. Fill out label and chain-of-custody form.

6. Pack and store for transport to laboratory.

7. Handle samples as described in the Chain-of-Custody SOP and the DOT HazMat handbook for sample handling, packing, and shipping.

8 WASTE MANAGEMENT

Solid investigation-derived wastes (IDWs), such as excess sediment generated through grab sampling activities, will be collected into 55-gallon drums and stored on site pending disposal. Liquid IDWs, such as decant water from grab samplers and decontamination liquids, will be collected into 55-gallon drums pending treatment and/or disposal. Non-aqueous liquid wastes, if generated (e.g., hexane, non-aqueous phase liquid [NAPL]), will be segregated and stored in appropriately sized buckets pending disposal. PPE, soiled disposable items, and other trash will be stored in 55-gallon drums on site pending disposal.

IDW will be sampled as needed for disposal characterization. IDW will be stored on site pending treatment and/or disposal. IDW may be managed in conjunction with remedial activities. All IDW will be stored in a secure onsite location pending disposal. Follow the procedures in the IDW TGI.

9 DATA RECORDING AND MANAGEMENT

See the Quality Procedures related to field activity documentation and sample documentation.

10 QUALITY ASSURANCE

Sample quality will be achieved by complying with the procedures outlined in this TGI. Cross-contamination will be prevented by following the protocols described in the TGI for Field Equipment Decontamination. Field activities will be supervised by appropriate experienced field supervisors. Additional quality assurance information is presented in the project-specific QAPP.

11 REFERENCES

Not applicable.

TGI - SURFACE WATER SAMPLE COLLECTION

Rev: 1

Rev Date: May 8, 2020

VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	April 9, 2019	All	Original approved document	Jessica Geurts / Shannon Dunn
1	May 8, 2020	None	Periodic review – no changes	Shannon Dunn

APPROVAL SIGNATURES

Prepared by: 04/05/2019

Jessica Geurts Date:

Technical Expert Reviewed by: Shannon Dunn 05/08/2020

Shannon Dunn Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guideline Instruction (TGI) describes the collection of surface water samples using a grab method, discrete depth sampler or peristaltic pump. This TGI should be followed whenever collecting surface water samples.

This TGI may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this TGI will be approved in advance by the Project Manager.

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, Arcadis field personnel will be versed in the relevant TGIs and SOPs and will possess the skills and experience necessary to successfully complete the desired field work. The project Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

4 EQUIPMENT LIST

The following equipment list contains materials that may be needed in carrying out the procedures contained in this TGI. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- personal protective equipment (PPE) and other safety equipment, as required in the project Health and Safety Plan (HASP)
- project Quality Assurance Project Plan (QAPP)
- Sampling and Analysis Plan (SAP)
- indelible ink pens
- appropriate sample containers, labels, and forms
- decontamination supplies including bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern.
- sample packing and shipping materials
- water-quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell. Several brands may be used, including:
    - YSI 6-Series Multi-Parameter Instrument
    - Hydrolab Series 3 or Series 4a Multiprobe and Display
    - Horiba U-10 or U-22 Water Quality Monitoring System
- for grab sampling method: pole with polyethylene and/or stainless steel dipper, if applicable
- for discrete depth sampling method: discrete depth samplers (e.g., Kemmerer or Van Dorn samplers)
- for peristaltic pump sampling method: peristaltic pump with appropriate power source, Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being used. For peristaltic pumps, dedicated Tygon® tubing (or other type as specified by the manufacturer) will also be used through the pump apparatus.

5 CAUTIONS

If heavy precipitation occurs and no cover over the sampling area can be erected, sampling must be discontinued until adequate cover is provided. Rain water could contaminate surface water samples.

Do not use permanent marker or felt-tip pens for labels on sample container or sample coolers – use indelible ink. The permanent markers could introduce volatile constituents into the samples.

It may be necessary to field-filter some parameters (e.g., metals) prior to collection, depending on preservation, analytical method, and project quality objectives.

Store and/or stage empty and full sample containers and coolers out of direct sunlight.

Be careful not to over-tighten lids with Teflon liners or septa (e.g., 40 mL vials). Over tightening can cause the glass to shatter or impair the integrity of the Teflon seal.

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

The ability to safely access the surface water sampling locations should be verified prior to sampling.

6 HEALTH AND SAFETY CONSIDERATIONS

Field activities will be performed in accordance with a project-specific HASP, a copy of which will be present onsite during such activities.

Safety hazards associated with sampling surface water include fast-moving water, deep water, and steep slopes close to sampling sites. Extreme caution should be used when approaching sampling sites. Work will be performed in accordance with the project-specific HASP.

7 PROCEDURE

Sampling Method

Surface water samples will be collected from sampling locations sequentially from downstream to upstream to prevent cross-contamination associated with sediment disturbance. Surface water samples will be collected prior to sediment sample collection.

Grab Sample Collection

Personnel conducting surface water sampling using grab sample collection techniques should perform the following:

1. Collect appropriate equipment, cleaned and decontaminated.

2. Obtain appropriate sampling containers.

3. Mobilize to surface water sampling location in accordance with the work plan or SAP.

4. Collect sample by directly lowering the laboratory-supplied sample container into the water and allowing the bottle to partially fill with water. The sampler will hold the bottle immediately below the water surface and allows the bottle to fill with sample. Field personnel will handle only the portions of the sample containers that do not come in contact with the sample, to avoid contamination. Additionally, care will be taken to avoid exposing samples and sample containers to atmospheric inputs such as dirt or dust.

5. Measure water quality parameters.

6. Transfer surface water samples into laboratory-supplied sample containers to complete the scope described in the SAP. Avoid overfilling sample containers to prevent preservatives, if present, in sample container from being lost.

Sample Collection Using a Discrete Depth Sampler (e.g., Kemmerer or Van Dorn)

Personnel conducting surface water sampling using grab sample collection techniques should perform the following:

1. Collect appropriate equipment, cleaned and decontaminated.
2. Obtain appropriate sampling containers.
3. Mobilize to surface water sampling location in accordance with the work plan or SAP.
4. Carefully set the sampling device so that water is allowed to pass through the tube.
5. Lower the pre-set sampling device to the predetermined depth using marked rope or line attached to the device.
6. When at desired depth; send down the messenger, closing the device. Avoid disturbing the bottom.
7. Retrieve sampler and discharge the first 10-20 mL to clear any potential cross-contamination.
8. Measure water quality parameters
9. Transfer surface water samples into laboratory-supplied sample containers to complete the scope described in the SAP. Avoid overfilling sample containers to prevent preservatives, if present, in sample container from being lost.
10. Pack and store samples appropriately for transport to laboratory.

Sample Collection Using Peristaltic Pump

Personnel conducting surface water sampling using peristaltic pump collection techniques should perform the following:

1. Surface water will be collected using a peristaltic pump if flow is slow and conventional sampling procedures are impossible without collecting excess suspended sediment in the sample. Note any observations such as color or odors and determine the depth of water. Record the information in the field log book or field log forms.
2. Personnel should be aware that contact with peristaltic pump apparatus (e.g., control knobs) can serve as a source of metals contamination in dissolved metals analyses. Operation of pump controls should be conducted with gloves that do not come into contact with the sample or with materials that contact the sample.
3. Attach tubing to pump and configure tubing such that intake in positioned at the desired sample depth within the water column and discharge is into desired sample container.

4. Turn the pump on and adjust the flow rate as necessary to avoid splashing or overfilling.

5. Measure water quality parameters on a volume of sample that will not be shipped to the laboratory to avoid cross-contamination in the sample to be analyzed.

6. Collect surface water samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container.

   - If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to surface water sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell.
   - When the sample container is full, tightly screw on the cap.
   - Samples should be collected in the following order: VOCs, TOC, SVOCs, metals and cyanide, and others (or other order as defined in the Sampling and Analysis Plan (SAP)).

7. Pack and store samples appropriately for transport to laboratory.

8 WASTE MANAGEMENT

Liquid investigation-derived wastes (IDWs), such decontamination liquids or excess surface water, will be collected into 55-gallon drums and may be transferred into large-volume polyethylene tanks with secondary containment pending treatment and/or disposal.

Non-aqueous liquid wastes, if generated (e.g., hexane, non-aqueous phase liquid [NAPL]), will be segregated and stored in appropriately sized buckets with secondary containment pending disposal.

PPE, soiled disposable items, and other trash will be stored in 55-gallon drums and stored on site pending disposal.

IDWs will be collected and stored on site in United States Department of Transportation (DOT)-compliant 55-gallon drums and/or large-volume tanks with secondary containment. Fifty-five-gallon drums and tanks will be labeled with DOT-compliant labels with the following information: drum contents, generator contact information, and date container was filled. IDWs known to be hazardous will be segregated and stored separately from non-hazardous IDWs. Solid IDWs will be segregated and stored separately from liquid IDWs.

IDWs will be sampled as needed for disposal characterization and stored on site pending treatment and/or disposal. IDWs may be managed in conjunction with remedial activities.

All IDWs will be stored in a secure onsite location pending treatment and disposal and/or discharge.

9 DATA RECORDING AND MANAGEMENT

Record field data in field notebook and/or on field log sheets.

10 QUALITY ASSURANCE

Sample quality will be achieved by complying with the procedures outlined in this TGI. Cross-contamination will be prevented by following standard decontamination protocols. Field activities will be supervised by appropriate experienced field supervisors. Additional quality assurance information is presented in the project-specific Quality Assurance Project Plan.

ARCADIS Design & Consultancy
for natural and
built assets

TGI: VIBRACORE SEDIMENT COLLECTION

Rev: 1

Rev Date: March 29, 2021

Version Control

Issue	Revision No.	Date Issued	Page No.	Description	Reviewed By
	0	October 9, 2018	All	Initial update to current TGI format	Shannon Dunn
	1	March 29, 2021	All	Updated to comply with new branding and…	Shannon Dunn / Elizabeth Hover

1 Introduction

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, regulation-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 Scope and Application

The general procedures to be utilized in obtaining vibracore sediment samples are outlined below. Following collection, the sediment cores will be transferred to a processing area. Processing of the sediment core is covered in a separate TGI.

3 Personnel Qualifications

All field personnel must have the appropriate training required as described in the project Health and Safety Plan (HASP). Field sampling personnel will be versed in the relevant TGIs, SOPs, and QPs and will possess the skills and experience necessary to successfully complete the desired field work.

4 Equipment List

The following equipment will be required for use during collection procedures:

- Personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP).
- Sampling vessel equipped with health and safety equipment specified in the HASP which has been inspected; is capable of supporting the work to be performed; and accommodates all workers, equipment, and samples. The sampling vessel will have an A-frame, or equivalent, and winch from which to operate the vibracore.
- High-precision differential global positioning system (DGPS) with sub-meter accuracy pre-loaded with sample location target coordinates.
- Navigation chart(s) for on-water activities (if available and as applicable).
- Vibracore device and power source (i.e., battery or generator).
- Lexan, polycarbonate, stainless steel, and/or aluminum core tubes (3-inch or 4-inch diameter) and plastic caps.
- Aluminum or stainless-steel core catchers.
- Power drill with bit suitable for drilling into Lexan, polycarbonate, stainless steel, and/or aluminum.
- Hacksaw, sawzall, or other cutting tool to cut core barrels into smaller sections, if needed.
- Calibrated rod for sediment depth measurement.
- Weighted lead-line with accuracy of ±0.01 ft.
- Waterproof permanent marker.
- Tape measure with accuracy of ± 0.01 ft.
- Aluminum foil.
- Automatically retracting safety knife.
- Duct tape.
- Rope to lash cores during transport.
- Storage rack and ice to store and chill cores until processing.
- Camera.
- Dry-erase board with marker.
- Decontamination supplies (see the TGI for Field Equipment Decontamination) including bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels.
- Field notebook, logging tools (see the TGI for Soil Descriptions), field forms, and black ball point pen.

5 Cautions

Cores will be maintained vertically to minimize the potential for migration of porewater contaminants within the core when the core is horizontal. Decanting of overlying water in the core can result in suspension of surface sediment and the loss of that sediment.

6 Health and Safety Considerations

Health and safety hazards are discussed in the HASP.

7 Procedure

The following procedures will be used to collect sediment samples using vibracore device:

1. If publicly available data on surface water elevation is available, record these data. If publicly available data are not available, measure and record the surface water elevation at a surveyed reference location of known elevation where surface water conditions are equivalent to surface water at the sample location at 30-minute intervals throughout all on-water sampling activities.
2. Maneuver the sampling vessel to the target sample location and secure the vessel in place.
3. Mount a new/unused or decontaminated core tube (Lexan®, polycarbonate, stainless steel, or aluminum) fitted with a cutter head, and of suitable length based on the desired penetration depth, into the vibracore head. Typically, Lexan® tube is suitable in soft sediments and aluminum or stainless-steel tube is suitable in coarse sediments. Reusable outer aluminum core barrels can also facilitate sampling with Lexan® or polycarbonate in a variety of sediment conditions. The type of tube to use will also be based on chemical compatibility with the site contaminants.
4. Check to confirm a good seal is achieved between the vibracore head and the core tube and that check valves are functioning properly.
5. Measure and record the sample location coordinates. Measure and record the water depth at the sample location immediately prior to sample collection. Record the date and time, and wave/water conditions at the sample location at the time of sampling.
6. Lower the vibracore with the core tube attached vertically through the water column, tube end first, until the sediment surface is reached.
7. Vibrate the core into the sediment to the target penetration depth or to refusal. While the vibracore is being driven, monitor to ensure penetration is vertical into the sediments.
   a. If needed, use a frame or floatation device to align vibracore vertically.
   b. Visually monitor the winch line to confirm the line remains taught.
   c. Visually monitor the position of the winch line where it intersects the water’s surface. Abrupt movement of the winch line during vibracoring is an indication that the vibracore assembly has fallen over.
8. Measure and record the depth of core tube penetration into the sediments.
9. Using the winch, pull the vibracore upward out of the sediments. Lift the vibracore on board the vessel while maintaining the core in a vertical position.
   a. Observe the water surface during vibracoring and vibracore retrieval for evidence of sheen. Record observations.
   b. If the cutter head is equipped with a core catcher, monitor for loss of material during retrieval. These types of equipment are typically sufficient to retain sediment inside the core barrel.
   c. If no core catcher is used, place a cap over the bottom of the core tube before it breaks the water surface, thus releasing the hydrostatic pressure within the core tube. Immediately secure the cap in place with duct tape once the core is brought on board the vessel.
10. Remove the core tube from the vibracore head while keeping the core tube in vertical position.
11. Use a decontaminated or foil-covered tip tape measure to measure the recovered length of the sediment core.
    - Carefully insert a decontaminated tape measure from the top of the core tube until the sediment surface is contacted.
    - Measure the distance from the top of the core tube to the top of the sediment in the core tube.

- The distance between the top of the sediment in the core tube and the bottom of the core tube is the recovered core length.

12. Compare the recovered core length with the core penetration depth.
    - If the recovered core length is more than 75 percent of the penetration depth, keep the core and continue with Procedure Step 13.
    - If recovery is less than 75 percent, contact the project manager to evaluate if another attempt should be made.
13. Decant standing water from above sediment in core tube.
    a. Allow standing water to settle until minimal suspended sediment is visible.
    b. Use tubing placed at least 1 inch above top of sediment, siphon water into a 5-gallon bucket. Use care to avoid disturbing surface sediment or siphoning fine grained material from sediment surface. Or,
    c. Drill 0.25- or 0.5-inch hole approximately 1 inch above sediment surface. Position a 5-gallon bucket to collect the stream of water from the hole. Use care to avoid disturbing surface sediment or tilting core tube so that surface sediment spills into discharging stream.
14. Label the core tube with the following:
    a. Location ID
    b. Date and time of core recovery
    c. “up” arrow
15. Record the following for each vibracore attempt:
    - date
    - time of recovery
    - sample position
    - water depth (feet)
    - core penetration depth (feet)
    - core recovery length
    - observations made during vibracore attempt (e.g., sheens, distressed wildlife)
16. Place a second cap on top of the core tube. Secure the cap in place with duct tape.
17. Store the core vertically while on the vessel and transport to the processing area and until processing is initiated on the core.

8 Waste Management

Solid investigation-derived wastes (IDWs), such as excess sediment generated through vibracoring activities, will be handled according to the project IDW plan or IDW TGI.

9 Data Recording and Management

See Quality Procedures related to field activity and sampling documentation.

10 Quality Assurance

Sample quality will be achieved by complying with the procedures outlined in this TGI. Cross-contamination will be prevented by following the protocols described in the TGI for Field Equipment Decontamination. Field activities will be supervised by appropriate experienced field supervisors. Additional quality assurance information is presented in the project-specific QAPP.

11 References

Not Applicable.

Arcadis U.S., Inc.
630 Plaza Drive, Suite 200
Highlands Ranch
Colorado 80129
Phone: 720 344 3500
Fax: 720 344 3535
www.arcadis.com

TGI - SURFACE WATER SAMPLE COLLECTION

Rev: 1

Rev Date: May 8, 2020

VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	April 9, 2019	All	Original approved document	Jessica Geurts / Shannon Dunn
1	May 8, 2020	None	Periodic review – no changes	Shannon Dunn

APPROVAL SIGNATURES

Prepared by: 04/05/2019

Jessica Geurts Date:

Technical Expert Reviewed by: Shannon Dunn 05/08/2020

Shannon Dunn Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

This Technical Guideline Instruction (TGI) describes the collection of surface water samples using a grab method, discrete depth sampler or peristaltic pump. This TGI should be followed whenever collecting surface water samples.

This TGI may change depending upon field conditions, equipment limitations, or limitations imposed by the procedure. Substantive modification to this TGI will be approved in advance by the Project Manager.

3 PERSONNEL QUALIFICATIONS

Arcadis field personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, Arcadis field personnel will be versed in the relevant TGIs and SOPs and will possess the skills and experience necessary to successfully complete the desired field work. The project Health and Safety Plan (HASP) and other documents will identify any other training requirements such as site-specific safety training or access control requirements.

4 EQUIPMENT LIST

The following equipment list contains materials that may be needed in carrying out the procedures contained in this TGI. Not all equipment listed below may be necessary for a specific activity. Additional equipment may be required, pending field conditions.

- personal protective equipment (PPE) and other safety equipment, as required in the project Health and Safety Plan (HASP)
- project Quality Assurance Project Plan (QAPP)
- Sampling and Analysis Plan (SAP)
- indelible ink pens
- appropriate sample containers, labels, and forms
- decontamination supplies including bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern.
- sample packing and shipping materials
- water-quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell. Several brands may be used, including:
  - YSI 6-Series Multi-Parameter Instrument
  - Hydrolab Series 3 or Series 4a Multiprobe and Display
  - Horiba U-10 or U-22 Water Quality Monitoring System
- for grab sampling method: pole with polyethylene and/or stainless steel dipper, if applicable
- for discrete depth sampling method: discrete depth samplers (e.g., Kemmerer or Van Dorn samplers)
- for peristaltic pump sampling method: peristaltic pump with appropriate power source, Teflon® tubing or Teflon®-lined polyethylene tubing of an appropriate size for the pump being used. For peristaltic pumps, dedicated Tygon® tubing (or other type as specified by the manufacturer) will also be used through the pump apparatus.

5 CAUTIONS

If heavy precipitation occurs and no cover over the sampling area can be erected, sampling must be discontinued until adequate cover is provided. Rain water could contaminate surface water samples.

Do not use permanent marker or felt-tip pens for labels on sample container or sample coolers – use indelible ink. The permanent markers could introduce volatile constituents into the samples.

It may be necessary to field-filter some parameters (e.g., metals) prior to collection, depending on preservation, analytical method, and project quality objectives.

Store and/or stage empty and full sample containers and coolers out of direct sunlight.

Be careful not to over-tighten lids with Teflon liners or septa (e.g., 40 mL vials). Over tightening can cause the glass to shatter or impair the integrity of the Teflon seal.

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

The ability to safely access the surface water sampling locations should be verified prior to sampling.

6 HEALTH AND SAFETY CONSIDERATIONS

Field activities will be performed in accordance with a project-specific HASP, a copy of which will be present onsite during such activities.

Safety hazards associated with sampling surface water include fast-moving water, deep water, and steep slopes close to sampling sites. Extreme caution should be used when approaching sampling sites. Work will be performed in accordance with the project-specific HASP.

7 PROCEDURE

Sampling Method

Surface water samples will be collected from sampling locations sequentially from downstream to upstream to prevent cross-contamination associated with sediment disturbance. Surface water samples will be collected prior to sediment sample collection.

Grab Sample Collection

Personnel conducting surface water sampling using grab sample collection techniques should perform the following:

1. Collect appropriate equipment, cleaned and decontaminated.

2. Obtain appropriate sampling containers.

3. Mobilize to surface water sampling location in accordance with the work plan or SAP.

4. Collect sample by directly lowering the laboratory-supplied sample container into the water and allowing the bottle to partially fill with water. The sampler will hold the bottle immediately below the water surface and allows the bottle to fill with sample. Field personnel will handle only the portions of the sample containers that do not come in contact with the sample, to avoid contamination. Additionally, care will be taken to avoid exposing samples and sample containers to atmospheric inputs such as dirt or dust.

5. Measure water quality parameters.

6. Transfer surface water samples into laboratory-supplied sample containers to complete the scope described in the SAP. Avoid overfilling sample containers to prevent preservatives, if present, in sample container from being lost.

Sample Collection Using a Discrete Depth Sampler (e.g., Kemmerer or Van Dorn)

Personnel conducting surface water sampling using grab sample collection techniques should perform the following:

1. Collect appropriate equipment, cleaned and decontaminated.
2. Obtain appropriate sampling containers.
3. Mobilize to surface water sampling location in accordance with the work plan or SAP.
4. Carefully set the sampling device so that water is allowed to pass through the tube.
5. Lower the pre-set sampling device to the predetermined depth using marked rope or line attached to the device.
6. When at desired depth; send down the messenger, closing the device. Avoid disturbing the bottom.
7. Retrieve sampler and discharge the first 10-20 mL to clear any potential cross-contamination.
8. Measure water quality parameters
9. Transfer surface water samples into laboratory-supplied sample containers to complete the scope described in the SAP. Avoid overfilling sample containers to prevent preservatives, if present, in sample container from being lost.
10. Pack and store samples appropriately for transport to laboratory.

Sample Collection Using Peristaltic Pump

Personnel conducting surface water sampling using peristaltic pump collection techniques should perform the following:

1. Surface water will be collected using a peristaltic pump if flow is slow and conventional sampling procedures are impossible without collecting excess suspended sediment in the sample. Note any observations such as color or odors and determine the depth of water. Record the information in the field log book or field log forms.
2. Personnel should be aware that contact with peristaltic pump apparatus (e.g., control knobs) can serve as a source of metals contamination in dissolved metals analyses. Operation of pump controls should be conducted with gloves that do not come into contact with the sample or with materials that contact the sample.
3. Attach tubing to pump and configure tubing such that intake in positioned at the desired sample depth within the water column and discharge is into desired sample container.

4. Turn the pump on and adjust the flow rate as necessary to avoid splashing or overfilling.

5. Measure water quality parameters on a volume of sample that will not be shipped to the laboratory to avoid cross-contamination in the sample to be analyzed.

6. Collect surface water samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container.

   - If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to surface water sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell.
   - When the sample container is full, tightly screw on the cap.
   - Samples should be collected in the following order: VOCs, TOC, SVOCs, metals and cyanide, and others (or other order as defined in the Sampling and Analysis Plan (SAP)).

7. Pack and store samples appropriately for transport to laboratory.

8 WASTE MANAGEMENT

Liquid investigation-derived wastes (IDWs), such decontamination liquids or excess surface water, will be collected into 55-gallon drums and may be transferred into large-volume polyethylene tanks with secondary containment pending treatment and/or disposal.

Non-aqueous liquid wastes, if generated (e.g., hexane, non-aqueous phase liquid [NAPL]), will be segregated and stored in appropriately sized buckets with secondary containment pending disposal.

PPE, soiled disposable items, and other trash will be stored in 55-gallon drums and stored on site pending disposal.

IDWs will be collected and stored on site in United States Department of Transportation (DOT)-compliant 55-gallon drums and/or large-volume tanks with secondary containment. Fifty-five-gallon drums and tanks will be labeled with DOT-compliant labels with the following information: drum contents, generator contact information, and date container was filled. IDWs known to be hazardous will be segregated and stored separately from non-hazardous IDWs. Solid IDWs will be segregated and stored separately from liquid IDWs.

IDWs will be sampled as needed for disposal characterization and stored on site pending treatment and/or disposal. IDWs may be managed in conjunction with remedial activities.

All IDWs will be stored in a secure onsite location pending treatment and disposal and/or discharge.

9 DATA RECORDING AND MANAGEMENT

Record field data in field notebook and/or on field log sheets.

10 QUALITY ASSURANCE

Sample quality will be achieved by complying with the procedures outlined in this TGI. Cross-contamination will be prevented by following standard decontamination protocols. Field activities will be supervised by appropriate experienced field supervisors. Additional quality assurance information is presented in the project-specific Quality Assurance Project Plan.

ARCADIS

Design & Consultancy
for natural and
built assets

F-5: SURFACE WATER FLOW MEASUREMENT

Rev: 1

Rev Date: June 23, 2016

APPROVAL SIGNATURES

Reviewed by: ____ Date: 6/23/2016
(Technical Expert)

1 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) describes the procedures to be utilized to determine surface water flow rates at designated locations. In general, these procedures include dividing the body of water into appropriate increments and determining the average velocity and cross-sectional area of each increment. The total flow rate at the measuring point is determined by summing the products of the cross-sectional area (in square feet) times the flow velocity (in feet per second) for each increment. Detailed procedures are provided below.

2 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, Arcadis field sampling personnel will be versed in the relevant SOPs and possess the skills and experience necessary to successfully complete the desired field work.

3 EQUIPMENT LIST

The following materials will be available, as required, during flow measurement activities:

- Health and safety equipment (as required by the Accident Prevention Plan)
- Boat with oars
- Anchor
- Surveyor’s rod
- Rope
- Duct tape
- 200-foot measuring tape
- Electromagnetic or ADV velocity meter
- Field notebook and pen

4 CAUTIONS

The selection of sampling equipment and methodology needs to be made based on the constituents of concern, sampling objectives, and site conditions.

5 HEALTH AND SAFETY CONSIDERATIONS

Working on or near the water presents variety of hazards. Please consult the project Accident Prevention Plan.

6 PROCEDURE

- Don personal protective equipment (as required by the HASP).
- Extend rope across the water.
- Measure the width of the water, then divide and mark into equally spaced measurement locations. For rivers/streams greater than 100 feet in width, the spacing should be 20 feet.
- Calibrate velocity meter as per manufacturer's specifications.
- Lower the surveyor's rod and measure and record the water depth to the nearest 0.1-foot at each measurement location.
- Velocities will be determined using the two-point method. Attach the velocity meter probe to the surveyor's rod, measure, and record the velocity in feet per second at depths equaling 0.2 and 0.8 times the total river depth at each measurement location. Average the two velocity measurements to obtain the average velocity for that vertical section.
- Record all measurements in field notebook.
- Calculate the river flow rate by multiplying the average velocity reading times the cross-sectional area of the 5-foot (or 10- or 20-foot) increment. The total flow rate is the sum of the velocity times the area for each increment.

$$Q_T = V_1 A_1 + V_2 A_2 + \ldots + V_n A_n$$

Where: Q_T = Total flow in cubic feet per second

$V_{1\text{-}n}$ = Average velocity for a vertical section (feet per second)

$A_{1\text{-}n}$ = Cross-sectional area of each increment (square feet)

7 WASTE MANAGEMENT

Disposable personnel protective equipment (such as gloves) and used supplies will be place appropriate disposal containers.

8 DATA RECORDING AND MANAGEMENT

All measurements and observations will be maintained in a field notebook or log. Upon project completion, field notebooks will be forwarded to the Project Manager for storage in the project files. Field staff should keep copies for their files. Field staff will forward copies to the Project Manager for quality assurance checks during project implementation at a frequency determined by the Project Manager.

9 QUALITY ASSURANCE

Samplers will forward copies of field notes to the Project Manager for quality assurance checks during project implementation daily or at a frequency determined by the Project Manager.

10 REFERENCES

Not applicable.

TGI - IN-SITU AND EX-SITU WATER QUALITY PARAMETERS

Rev: 0

Rev Date: October 16, 2018

VERSION CONTROL

Revision No	Revision Date	Page No(s)	Description	Reviewed by
0	October 16, 2018	All	Updated and re-written as TGI (in-situ and ex-situ adaptation)	Marc Killingstad

APPROVAL SIGNATURES

Prepared by: Chris Shepherd 10/16/2018

Chris Shepherd Date:

Technical Expert Reviewed by: 10/16/2018

Marc Killingstad (Technical Expert) Date:

1 INTRODUCTION

This document describes general and/or specific procedures, methods, actions, steps, and considerations to be used and observed by Arcadis staff when performing work, tasks, or actions under the scope and relevancy of this document. This document may describe expectations, requirements, guidance, recommendations, and/or instructions pertinent to the service, work task, or activity it covers.

It is the responsibility of the Arcadis Certified Project Manager (CPM) to provide this document to the persons conducting services that fall under the scope and purpose of this procedure, instruction, and/or guidance. The Arcadis CPM will also ensure that the persons conducting the work falling under this document are appropriately trained and familiar with its content. The persons conducting the work under this document are required to meet the minimum competency requirements outlined herein, and inquire to the CPM regarding any questions, misunderstanding, or discrepancy related to the work under this document.

This document is not considered to be all inclusive nor does it apply to any and all projects. It is the CPM's responsibility to determine the proper scope and personnel required for each project. There may be project- and/or client- and/or state-specific requirements that may be more or less stringent than what is described herein. The CPM is responsible for informing Arcadis and/or Subcontractor personnel of omissions and/or deviations from this document that may be required for the project. In turn, project staff are required to inform the CPM if or when there is a deviation or omission from work performed as compared to what is described herein.

In following this document to execute the scope of work for a project, it may be necessary for staff to make professional judgment decisions to meet the project's scope of work based upon site conditions, staffing expertise, state-specific requirements, health and safety concerns, etc. Staff are required to consult with the CPM when or if a deviation or omission from this document is required that has not already been previously approved by the CPM. Upon approval by the CPM, the staff can perform the deviation or omission as confirmed by the CPM.

2 SCOPE AND APPLICATION

Water quality parameters (e.g., turbidity, specific conductivity, pH, and temperature) of natural waters are usually measured in the field. The temperature, pH, specific conductivity, dissolved oxygen (DO), and oxidation reduction potential (ORP) and turbidity of surface water will be measured in-situ or ex-situ with a multiparameter water quality meter (i.e., multimeter).

Conductivity is the ability of a solution to pass an electric current. This current is carried by inorganic dissolved solids. The measurement is useful to quantify the chemical purity of the water relative to the amount of dissolved solids in a solution. Generally, temperature-compensated conductivity, termed specific conductivity, is measured by a water quality meter. Specific conductivity can also be used to estimate total dissolved solids (TDS) in solution (grams per liter or g/L) by multiplying the specific conductivity (measured in millisiemens per centimeter or mS/cm) by 0.65, which assumes that sodium chloride is a reasonable surrogate for the dissolved solids in solution.

Measuring the concentration of DO in water is an important component in evaluating the quality of natural as well as contaminated waters. The effects of wastes on rivers/streams, the suitability of water for fish and other organisms, as well as the effects of remediation efforts, can often be ascertained from the DO content. Dissolved oxygen levels generally range from 5 to 9 milligrams per liter (mg/L) when measured in water that is in equilibrium with air, depending on the temperature and barometric pressure; however, levels may be lower in a 'reducing' environment where anoxic conditions exist in water (e.g., gasoline plume undergoing bioremediation).

The measurement from an ORP sensor represents the net status of all the oxidation and reduction reactions in the sample being measured. Positive results indicate an oxidizing environment while negative results indicate a reducing environment. ORP is determined by measuring the potential of a chemically-inert electrode which is immersed in the solution. The sensing electrode potential is read relative to the reference electrode of the pH probe and the value is presented in millivolts (mV).

ORP meter results can indicate possible contamination and can be particularly useful if it is known that one component of the sample is primarily responsible for the observed value. For example, excess chlorine in a sample will result in a large positive ORP value while the presence of hydrogen sulfide will result in a large negative ORP value. Together with pH, temperature, and knowledge of the dominant species in a sample, ORP results help predict the oxidation state of ions in solution and whether certain reactions may take place.

Temperature readings will be taken at each water sampling location to assist understanding the water quality as well as compensating measurements. A thermometer is typically part of the multimeter. If not, a temperature probe or thermometer will be used.

The measurement of turbidity is useful in that it expresses the relative amount of suspended particles in the water column. Turbidity cannot be measured in-situ.

Refer to the following documents for procedures of groundwater sample collection:

> *TGI – Standard Groundwater Sampling for Monitoring Wells* (describes the methods to be used to collect groundwater samples using traditional purging and sampling techniques)
>
> *TGI - Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells* (describes low-flow purging and sampling techniques)
>
> *TGI – Passive Diffusion Bag Sampling, TGI – Groundwater Sampling with HydraSleeves*™, and *TGI - Bailer-Grab Groundwater Sampling* (describes no-purge/passive sampling techniques)

3 PERSONNEL QUALIFICATIONS

Arcadis field sampling personnel will have completed or are in the process of completing site-specific training as well as having current health and safety training as required by Arcadis, client, or regulations, such as 40-hour HAZWOPER training and/or OSHA HAZWOPER site supervisor training. Arcadis personnel will also have current training as identified in the site-specific Health and Safety Plan (HASP) which may include first aid, cardiopulmonary resuscitation (CPR), Blood Borne Pathogens (BBP) as needed. The HASP will also identify any access control requirements.

Prior to mobilizing to the field, the sampling team will review and be thoroughly familiar with relevant site-specific documents including but not limited to the task-specific work plan or field implementation plan

(FIP)/field sampling plan, Quality Assurance Project Plan (QAPP), HASP, historical information, and other relevant site documents.

Arcadis field sampling personnel will be knowledgeable in the relevant processes, procedures, and TGIs and possess the demonstrated required skills and experience necessary to successfully complete the desired field work. Additionally, the field sampling team will review and be thoroughly familiar with documentation provided by equipment manufacturers and become familiar with the operation of (i.e., hands-on experience) all equipment that will be used in the field prior to mobilization.

4 EQUIPMENT LIST

The following materials, as required, will be available during field measurement of water quality parameters:

- Site-specific HASP and health and safety documents identified in the HASP (e.g., job safety assessments [JSAs])
- Field Implementation Plan (FIP) that includes site map with sampling locations, well construction records (table or logs), sampling plan, and prior groundwater sampling records (if available)
- Field notebook, pen(s) (indelible ink) and/or smart device (smart phone or tablet)
- Field laptop / data manager
- Water Quality Measurement Log (**Attachment 1**)
- Water Quality Meter Calibration Log (**Attachment 2**)
- Appropriate personal protective equipment (PPE) (e.g., latex or nitrile gloves, safety glasses, etc.) as specified in the HASP
- Traffic cones, delineators, and caution tape as appropriate for securing the work area as specified in the Traffic Safety Plan (TSP)
- Well key(s)
- Photoionization detector (PID), flame ionization detector (FID) or other air monitoring equipment, as needed, in accordance with the HASP
- Dedicated plastic sheeting (e.g., Weatherall Visqueen) or other clean surface to prevent sampling equipment from coming in contact with the ground
- Electronic water-level indicator (e.g., Solinist Model 101) or oil/water interface probe with 0.01-foot accuracy (oil/water as appropriate, note that sampling will not be performed when sheen or light non-aqueous phase liquid [LNAPL] is present)
- Multiparameter (temperature/pH/specific conductivity/ORP/turbidity/DO) water quality meter or sonde with flow-through cell (as appropriate) plus reader and protective housing; for example:
    - YSI Professional Plus Multiparameter Instrument
    - YSI EX01 or EX02 Multiparameter Sonde

  - Horiba W-22 XD Multiparameter Instrument
  - Hydrolab DS5 Multiprobe and Display
- Operation manual(s) for multiparameter meter
- Water quality meter extension cable (as needed for in-situ readings of deepest well)
- Standard solutions for calibration
- Multimeter maintenance kit and extra DO membranes if using amperometric DO probe
- Extra batteries for the various instruments
- Tape measure and/or masking tape for in-situ readings
- Decontamination equipment (buckets, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels)

5 CAUTIONS

NOTE: *Proper operation and maintenance are very important for a functioning meter and accurate readings. Carefully follow manufacturer's instructions for operation (including down-hole use), maintenance, and calibration as each manufacturer's instructions will vary.*

Verify the meter is intended for in-situ measurements and that all necessary parts are available prior to field mobilization.

Check and record the expiration dates of calibration fluids. Verify that calibration readings are reasonable and correspond to previous calibration readings when available. Expired calibration fluids may result in erroneous results. If accurate measurements of TDS are required, site-specific calibration will be necessary in accordance with the manufacturer's specifications.

Turbidity meters will be stored and used in dry locations.

When taking measurements in the field, verify that readings make sense and compare to historical readings when possible.

6 HEALTH AND SAFETY CONSIDERATIONS

The HASP will be followed, as appropriate, to ensure the safety of field personnel.

Appropriate personal protective equipment (PPE) will be worn at all times in line with the task and the site-specific HASP. Also, at a minimum, wear latex gloves and safety glasses when working with calibration solutions.

Review all site-specific and procedural hazards as they are provided in the HASP, and review Job Safety Analysis (JSA) documents in the field each day prior to beginning work.

Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or non-aqueous phase liquid (NAPL) (e.g., oil). Other potential hazards include pressurized wells, stinging insects that may inhabit well heads, other biologic hazards (e.g. ticks in long grass/weeds around well head), and potentially the use of sharp cutting tools (scissors, knife)—open well caps slowly and keep face and body away to allow to vent any built-up pressure; only use non-toxic peppermint oil spray for stinging insect nests; review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives, and use appropriate hand protection.

If working at a site with hazardous levels of volatile organic compounds, headspace and/or breathing zone readings will be measured prior to working at/in the well, in accordance with the HASP.

7 PROCEDURE

The procedure is organized into the following sections:

A. Standard Procedures – calibration, operation, and maintenance procedures that apply to all methods and meters
B. Calibration – general calibration procedures for:
    a. Multimeter
    b. Turbidity meter
C. Operation – general measurement operations for:
    a. Ex-situ measurements
        i. Multimeter
        ii. Turbidity meter
    b. In-situ measurements (downhole multimeter only)
D. Maintenance
    a. Multimeter
    b. Turbidity meter

7.1 Standard Procedures

Calibration

- The meter will be calibrated following the manufacturer’s instructions as calibration steps vary by manufacturer.
- Calibration information will be recorded in the calibration log (Attachment 2) and the field notebook.
- Check batteries/power levels during calibration.
- More frequent calibration may be necessary in harsh conditions or per project plans.

Operation

- The meter will be operated following the manufacturer’s instructions.

- Mutimeters can be operated in-situ (down-hole, in water, etc.) or ex-situ (water is transferred to container for measurement).
- Two readings will be made after stabilization (minimum 2 minutes) and the average will be recorded in the field notebook or both measurements will be recorded in the water quality measurement log and/or logger.
- Additional details for pH, conductivity, temperature, dissolved oxygen, and turbidity operation are presented in section **7.3 Operation** below.

Maintenance

- The meter will be maintained according to the manufacturer's instructions.
- Maintenance information will be recorded in the field notebook or instrument log.
- Manufacturer recalibration or replacement probes may be required on a routine basis.
- A replacement meter and probes will be available onsite or ready for overnight shipment, as necessary.
- Additional details for pH, conductivity, temperature, dissolved oxygen, and turbidity maintenance are presented in section **7.4 Maintenance** below.
- More frequent maintenance (probe replacement, sensor reconditioning, etc.) may be necessary in harsh conditions or per project plans.

7.2 Calibration

Multimeter Calibration

1) Connect cables from meter to reader
2) Check probes and ensure they are clean
3) Switch on instrument and allow to warm-up
4) Check battery life and replace, if needed
5) Adjust date/time, if needed
6) Soak all probes in distilled or de-ionized water for at least 5 minutes (some probes may require longer soak times, see below and manufacturer instructions) and then shake off excess liquid.
    i. NOTE: *If the probes are recently reconditioned or have slight build-up, allow for longer soaking period.*
7) Navigate to calibration display/mode
8) **pH Calibration** – when in use, the pH meter will be calibrated daily, at a minimum.
    i. Connect electrode (if applicable) or remove protective cap from electrode
    ii. Rinse end of electrode in distilled/deionized water and shake off excess water

iii. Measure and record temperature of buffer solutions

iv. Immerse pH electrode in pH buffer 7.00, set the temperature to that of the buffer 7.00, and allow sufficient time for the electrode to stabilize

v. Adjust the calibration for the correct readout and temperature

vi. Confirm/press the calibration button

vii. Remove electrode from buffer and rinse with distilled/deionized water

viii. Immerse pH electrode in buffer 4.00, set the temperature control to that of the buffer 4.00, and allow sufficient time for the electrode to stabilize

ix. Adjust the calibration for the correct readout and temperature

x. Confirm/press the calibration button

xi. Rinse electrode with distilled/deionized water

xii. A pH 10 calibration will also be performed if high pH is anticipated, following manufacturers procedures

xiii. The pH meter is calibrated

9) **Conductivity Calibration** – when in use, the conductivity meter will be calibrated daily, at a minimum.

   i. Conductivity is generally measured in specific conductivity (temperature compensated), verify with the FIP/work plan

   ii. Soak the probe in distilled/deionized water for at least 30 minutes

   iii. Remove the probe from the water and fling out drops clinging inside

   iv. Immerse the probe to or beyond the vent holes in a beaker containing 1.413 mS/cm standard solution and gently agitate vertically to remove entrapped air

   v. Repeat Steps 3 and 4 at least once more

   vi. Press calibration button

10) **Temperature calibration** – temperature will be verified according to FIP/work plan, if applicable.

11) **Dissolved Oxygen (DO) calibration** – when in use, the DO meter will be calibrated daily using the air calibration method or less frequently if using an optical sensor (see below).

   i. Preparation

      1. Polarographic sensor – periodically recondition sensor, replace fluids, and Teflon® membrane, per manufacturer, and air bubbles should not be present

      2. Galvanic sensor – Periodically recondition sensor, replace fluids, and membrane, per manufacturer, and air bubbles should not be present

        3. Optical sensor – Per manufacturer, most will only require field checks and maintenance, however, weekly checks are recommended to verify the accuracy, at a minimum

    ii. Obtain a barometric pressure reading from a daily weather report or from the instrument, as required by instrument

    iii. Keep instrument upright and vent cap/cover while retaining a small amount of clean water (do not cover probes) for non-optical sensors only

    iv. Allow 5 to 15 minutes for optimum probe stabilization and polarization, for non-optical sensors (reading will range between 5 to 9 mg/L depending on temperature and barometric pressure)

    v. Press calibration button, if reading is more than 2% off of the standard

12) **Oxidation reduction potential (ORP) calibration** – when in use, the ORP meter will be calibrated daily, if required.

    i. Rinse the probe in distilled/deionized water

    ii. Remove the probe from the water and fling out drops clinging inside

    iii. Immerse the probe in the ORP solution. Gently agitate vertically to remove entrapped air

    iv. Repeat Steps 3 and 4 at least once more

    v. Press calibration button, if reading is more than 2% off of the standard

13) Adjust the instrument to READ or LOGGING mode, per instruction manual and project plans; the instrument is now ready for use

Turbidity Calibration (if required)

Turbidity meters are generally calibrated weekly, unless required otherwise. However, daily checks are recommended to verify the accuracy. The turbidity meter will be calibrated per steps below:

1) Switch on instrument and allow time to warm-up

2) Check battery life and replace, if needed

3) Turbidity sample tubes will always be washed prior to use, but not the calibration standard tubes, using a mild detergent to remove any dirt or finger prints

4) Dry the outside of the sample turbidity tubes with a clean, lint-free cloth or disposable wipe and allow the turbidity tubes to air-dry in an inverted position to prevent dust from entering the tube, as allowable (but, not the calibration standard tubes)—dirt or fingerprints can lead to inaccurate results

5) Wipe the 1 Nephelometric turbidity unit (NTU) standard and place in the chamber after aligning the arrows (manufacturer dependent)

6) Place the light shield over the turbidity standard and allow the meter to stabilize

7) Press the read and/or CAL button, per instructions

8) Repeat steps 3 thru 5 above
9) Adjust to READ mode and the instrument is now ready for use.

7.3 Operation

Water quality parameters can be measured ex-situ or in-situ in accordance with the FIP/work plan. The operating procedures for each method are described below. It is also possible to collect a depth-specific sample from a well or water body for ex-situ measurement.

Ex-situ Operation

Multimeter

1) Fill two 100-mL plastic disposable beakers or clean beakers with water from the sample
2) Insert the probe into the first beaker immersing all sensors and allow to stabilize (2 minutes at minimum)
3) Record readings
    i. Temperature – in degrees Celsius or Fahrenheit
    ii. pH – in standard units between 0 and 14, in 0.01 increments
    iii. Specific conductivity – in mS/cm or microsiemens per centimeter (uS/cm)
    iv. DO – in mg/L and percent (will typically read between 0 and 15 mg/L)
    v. ORP – in millivolts (mV; will typically read between -1,400 mV and + 1,400 mV), if required
4) Rinse probe off with distilled/deionized water
5) Repeat Steps 2 thru 4 for the other beaker
6) Log results on the water quality measurement log and/or in field notebook - the average will be the actual result
7) Rinse probe off with distilled/deionized water

NOTE: *If a flow through cell is used during low flow groundwater sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low Flow Groundwater Purging and Sampling.*

Turbidity Meter

1) Fill the two cleaned, manufacturer provided sample tubes with water from the sample
2) Wipe off and dry the outside of the sample turbidity tubes with a clean, lint-free cloth or disposable wipe
3) Insert the first sample tube and close the cover
4) Push the READ button
5) Record turbidity reading in NTUs (0 to 1,100 NTUs)

   i. If readings are over the instrument limit, a single 2X dilution can be run by mixing the sample with equal parts distilled or de-ionized water and gently mixing
   ii. The resulting value will need to be doubled (for example, if the diluted reading is 750 NTUs, the estimated turbidity would be 750 x 2 = 1,500 NTUs. Diluted samples will be qualified as estimated)

6) Repeat steps 2 thru 5 with the second sample tube
7) Log the results on the water quality measurement log and/or in field notebook - the average will be the actual result
8) Rinse sample tubes with distilled/deionized water

NOTE: *If a flow through cell is used during low flow groundwater sampling, a single reading can be obtained from the multimeter during each step of the wells purging in accordance with TGI - Low Flow Groundwater Purging and Sampling. The turbidity sample is recommended to be collected prior to passing through the flow through cell.*

In-situ Operation

1) Connect extension cable and protective housing to meter
2) Measure water level from reference point
3) Lay out plastic sheeting as needed to keep the multimeter clean
4) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with a mild soap and warm water
5) Organize work area to prevent dirt or objects from falling in the well
6) Measure and mark extension cable at planned measurement intervals
7) Slowly lower the probe into the well or water body to the desired measurement interval using caution to prevent rubbing of cable on the well and minimize water and sediment disturbance
8) Allow readings to stabilize (typically 1 to 3 minutes)
   i. For many instruments, the instrument may need to be slowly oscillated up and down a few inches to circulate water around the probes
9) Record readings
   i. Temperature – in degrees Celsius or Fahrenheit
   ii. pH – in standard units between 0 and 14, in 0.01 increments
   iii. Specific conductivity – in mS/cm or uS/cm
   iv. DO – in mg/L <u>and</u> percent (will typically read between 0 and 15 mg/L)
   v. ORP – in mV (will typically read between -1,400 mV and +1,400 mV), if required
10) Repeat steps 4 through 9 to complete targeted in-situ measurements

11) Log results on the water quality measurement log and/or in field notebook
12) Slowly retrieve the multimeter
13) Clean multimeter and cable per decontamination procedures and instruction manual and rinse with distilled/deionized water using caution, as most meters can only handle light washing with a mild soap and warm water

7.4 Maintenance

Multimeter

1) After use, the meter will be inspected and maintained according to the manufacturer's specifications and the inspection/maintenance activities will be recorded in the field notebook
2) Keep records of usage, maintenance, calibration, problems, and repairs
3) Recharge/replace batteries on a regular basis
4) Store meters or electrodes in protective casing when not in use
5) DO membranes will be stored moist, unless specified otherwise by manufacturer
6) Focused robe cleaning may be necessary in accordance with the operation manual and may be needed more frequently in harsh conditions
7) A replacement meter will be available onsite or ready for rapid delivery/shipment
8) Periodic manufacturer calibration may be necessary per operation manual and/or field plans

Turbidity Meter

1) Recharge battery on a regular basis
2) Store in protective casing when not in use
3) Keep records of usage, maintenance, calibration, problems, and repairs
4) After use, the meter will be inspected with results recorded in the field notebook
5) Keep sample tubes clean inside and out replacing them when they become scratched or etched and avoid handling the tubes in the region where the light beam enters them.
6) Clean lens periodically per instruction manual
7) Turbidity meter will be sent back to the manufacturer for service when needed

8 WASTE MANAGEMENT

Investigative-Derived Waste (IDW), including rinse water, excess sample water, spent calibration solutions, and disposable materials (plastic sheeting, PPE, etc.) generated during the procedures outlined in this TGI will be collected and stored on site in appropriately labeled containers (disposable materials will be contained separately) and disposed of properly. Containers must be labeled at the time of collection and will include date, location(s), site name, city, state, and description of matrix contained (e.g., soil, PPE). Waste will be managed in accordance with the *TGI – Investigation-Derived Waste Handling and Storage,* the procedures identified in the FIP or QAPP as well as state-, federal- or client-specific requirements. Be certain that waste containers are properly labeled and documented in the field log book.

PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures activities will be placed in plastic bags. These bags will be disposed of as general waste unless specified otherwise.

9 DATA RECORDING AND MANAGEMENT

Management of the original documents from the field will be completed in accordance with the site-specific QAPP. Records generated as a result of this TGI will be controlled and maintained in the project record files in accordance with project requirements.

In general, documentation of the following information is required:

- Calibration – calibration information will be recorded on a calibration form, field log, or electronic device per project plans
    - Meter manufacturer and model
    - Serial number
    - Calibration personnel
    - Calibration date/time
    - Standard value, initial and final reading
    - Observations, if applicable
- Readings - data will be recorded on a field log, sampling form, or electronic device per project plans
    - Instrument model
    - Measurement date/time
    - Field personnel
    - Weather
    - Measurement location and depth, if applicable
    - Value of readings and average reading, if applicable
    - Units of readings
    - In-situ vs ex-situ measurement method
    - Key observations

All records will be provided to the project manager and retained in the project files. Any maintenance needs will be communicated to the project manager promptly.

QUALITY ASSURANCE

Quality assurance procedures will be conducted in accordance with the Arcadis Quality Management System or the site-specific QAPP.

Refer to the QAPP or FIP/sampling plan/work plan for the frequency of calibrations.

10 REFERENCES

Not applicable.

11 ATTACHMENTS

Attachment 1 - Water Quality Measurement Log

Attachment 2 - Water Quality Meter Calibration Log

ATTACHMENT 1

Water Quality Measurement Log

WATER QUALITY MEASUREMENT LOG

Page______of _______

GENERAL INFORMATION

Date ______________________________
Project No ______________________________
Site Name ______________________________
Site Location ______________________________
Site/Well No. ______________________________
Weather ______________________________
Sample Personnel ______________________________
Sample ID ______________________________
Start reading time ______________________________
End reading time ______________________________

MEASUREMENT DATA

Measuring Point (MP) description ______________________________
Depth to Water (ft)/Time ______________________________
Well Screen Interval (ft) ______________________________
Casing Diameter (in) ______________________________
Meters calibrated (Y/N) ______________________________
Instrument model/serial no. ______________________________
Instrument model /serial no. 2 ______________________________
☐ In-situ ☐ Ex-situ reading
(see calibration log, if applicable)

Water Quality Parameters

Time	**Depth** (ft)	**pH** (S.U.)	**Spec. Cond.** (mS/cm or uS/cm)	**Temp** (°C or °F)	**DO** (mg/L)	(%)	**ORP** (mV)	**Turbidity** (NTU)	**Observations** (Odor, clarity, etc., if applicable)

REMARKS

____
____
____
____

ATTACHMENT 2

Water Quality Meter Calibration Log

ARCADIS Design & Consultancy for natural and built assets

WATER QUALITY METER CALIBRATION LOG

PROJECT NAME	
PROJECT NO.	
MODEL	
SERIAL #	
SAMPLER	
DATE	

TURBIDITY CALIBRATION

CAL. READING		CAL. READING		CAL. RANGE	TIME
(LOT #)		(LOT #)			
(EXP. DATE)		(EXP. DATE)			
PRE-CAL. / POST-CAL		PRE-CAL. / POST-CAL			
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	
/		/		☐ WITHIN RANGE	

AUTOCALIBRATION

AUTOCAL SOLUTION		CAL. READING	CAL. RANGE	TIME	CALIBRATION RANGES [1]	
(LOT #)						
(EXP. DATE)						
CALIBRATED PARAMETERS		PRE-CAL. / POST-CAL				
☐	pH	/	☐ WITHIN RANGE		pH	± 0.2 S.U.
☐	CONDUCTIVITY	/	☐ WITHIN RANGE		COND	± 1% OF CAL. STANDARD
☐	ORP	/	☐ WITHIN RANGE		ORP	± 25 mV
☐	DO	/	☐ WITHIN RANGE		DO	Atmospheric
☐	TURBIDITY	/	☐ WITHIN RANGE		TURB	± 5% OF CAL. STANDARD
☐		/	☐ WITHIN RANGE			
☐		/	☐ WITHIN RANGE			

(1) CALIBRATION RANGES ARE SPECIFIC TO THE MODEL OF THE WATER QUALITY METER

NOTES:

SIGNED DATE

CHECKED BY DATE

TGI – U.S. ENVIRONMENTAL PROTECTION AGENCY POREWATER SAMPLING

Rev #: 0

Rev Date: May 13, 2020

Region 4
U.S. Environmental Protection Agency
Laboratory Services and Applied Science Division
Athens, Georgia

Operating Procedure

Title: Pore Water Sampling	ID: LSASDPROC-513-R4
Issuing Authority: LSASD Field Branch Chief	
Effective Date: May 13, 2020	Review Due Date: May 13, 2024

Purpose

The purpose of this operating procedure is to describe the methods and considerations to be used when obtaining a pore water sample from soil or sediment.

Scope/Application

This document describes procedures generic to all pore water sampling methods to be used by field personnel when collecting and handling samples in the field. On the occasion that Laboratory Services and Applied Science Division (LSASD) personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a pore water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

TABLE OF CONTENTS

Purpose 1
Scope/Application 1
1 General Information 3
1.1 Documentation/Verification 3
1.2 General Precautions 3
1.2.1 Safety 3
1.2.2 Procedural Precautions 3
1.2.3 Records 4
2 Sampling Methodology 4
2.1 General 4
2.2 Collection Considerations 4
2.3 Summary of Procedure 4
2.4 Sampling Equipment 4
2.5 Pore Water Sampler Deployment Considerations 5
2.5 PushPoint™ Deployment Considerations 5
2.6 Pore Water Collection 6
2.6 PushPoint™ Basic Operation 6
2.6.1 Peristaltic Pump/Vacuum Jug Collection 6
2.6.2 Syringe 7
2.7 Quality Control 7
2.8 Specific Sampling Equipment Quality Assurance Techniques 8
3 Special Sampling Considerations 8
3.1 Volatile Organic Compounds (VOC) 8
3.2 Dissolved Metals Sample Collection 8
3.3 Special Precautions for Pore Water Sampling 9
3.4 Sample Handling and Preservation Requirements 9
Figure 1. Pore Water Sampling Device 10
Figure 2. Pore Water Sampler Deployment Using a Sampling Flange 11
Figure 3. Pore Water Sampler Deployment Using a Peristaltic Pump to Sample 12
References 13
Revision History 14

1 General Information

1.1 Documentation/Verification

This procedure was prepared by persons deemed technically competent by LSASD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the LSASD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.2 General Precautions

1.2.1 Safety

Proper safety precautions must be observed when collecting pore water samples. Refer to the LSASD Safety, Health and Environmental Management Program Procedures and Policy Manual (most recent version) and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.2.2 Procedural Precautions

The following precautions should be considered when collecting pore water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.

- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.

- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.

- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

- Documentation of field sampling is done in a bound logbook. Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.

- All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

1.2.3 Records

Information generated or obtained by LSASD personnel will be organized and accounted for in accordance with LSASD records management procedures found in LSASD Operating Procedure for Control of Records, LSASDPROC-002 (most recent version). Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation, in accordance with LSASD Operating Procedure for Logbooks, LSASDPROC-010 (most recent version), and LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005 (most recent version).

2 Sampling Methodology

2.1 General

The pore water sampling techniques and equipment described in this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in this section are followed, a representative sample of the pore water should be obtained.

2.2 Collection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (i.e., is not impounded). However, wading may disrupt bottom sediments causing biased results; therefore, the sampler should enter the area downstream of the sampling location and collect the sample facing upstream. If the stream is too deep to wade, the pore water sample may be collected from a platform such as a boat or by SCUBA diving. If sampling from a boat or in water deeper than the length of the sampler, extensions may be utilized. If SCUBA diving, all diving activity must be conducted in accordance with EPA's Diving Safety Manual, current version.

2.3 Summary of Procedure

Pore water is collected using a pore water extracting device (Figure 1). The most common type used by LSASD is the PushPoint™ sampler (M.H.E. Products 2003), made out of stainless-steel tubing. The sampling end of the pore water device is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump. The device is suitable for use only in fine-grained material (no gravel or cobble). Other similar devices may be used providing that the integrity of the sample is maintained, and no ambient surface water is allowed in contact with the sample.

2.4 Sampling Equipment

A PushPoint™ or similar sampler typically consists of a pointed tubular stainless-steel tube with a screened zone at one end and a sampling port at the other. The pointed end with the screened zone consists of a series of very fine interlaced machined slots to allow pore water to enter the sampler. A removable guard rod adds rigidity to the sampler during sediment insertion. The length of the screened zone will depend on the site-specific study design. Depending on the data quality objectives (DQO) of the study, filters may be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the device by connecting flexible tubing and using a syringe or peristaltic pump to extract the sample. Teflon® tubing is the preferred tubing to be used for collecting pore water samples. However, other tubing may be used, depending upon the DQOs for the specific application.

There are many modifications that can be incorporated into the procedure to satisfy data quality objectives for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of pore water sampling devices and issues to consider when collecting pore water.

An alternative system is available in LSASD inventory for use in soft sediments in water deeper than wading depth. A well screen and short riser approximately ¾" in diameter are threaded to fasten to the bottom of a custom flange. Internal threads on the screen accept a tubing adapter. The accompanying rimmed flange has a coupling with both top and bottom threads. The well screen is screwed into the bottom of the flange and Teflon® tubing is attached to the tubing adapter threaded into the well screen. For deployment, the tubing is then inserted through a PVC pipe or well casing which is then screwed into the upper threads of the flange. The entire assembly can be deployed in water up to ten feet of depth from a well anchored boat.

2.5 Pore Water Sampler Deployment Considerations

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the sampling end of the pore water device during deployment, surface water may flow down the outside of the device to the screened area and into the intended sample. Therefore, the pore water device should be used with a sampling flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. If pore water is collected from deep in sediments, a flange may not be necessary. When inserted though the flange, the body of the pore water device should form a watertight seal to eliminate surface water intrusion during sample collection. Flanges should include an outer vertical cutting ring to enhance sealing. Flange systems can be augmented by flexible plastic sheeting of appropriate material. The sheeting can be weighted to conform to a stream bottom by objects obtained from other areas of the stream away from the sampling location. Several of the flanges in LSASD inventory have a threaded nut and washer to facilitate sealing the flange to a polyethylene sheet.

The flange can be made of any material that will not cross contaminate the intended sample. If both inorganic and organic analyses are required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. A useful estimate can be made for planning by taking the required water volume, tripling it to assume 33% porosity, and then calculating the dimensions of a cylinder of this volume, based upon the penetration depth of the sampler. The flange should cover at least this estimated volume. If it is not practical to use a large flange, then multiple devices may be deployed, and smaller volumes can be collected from several devices for a composite sample. If multiple devices are deployed, they should be spaced an appropriate distance apart so they will not interfere with one another.

In general, the volume of pore water that can be collected at a given location is limited. Collecting large volumes of pore water will ultimately result in the collection of water from the overlying water body. Often, minimum required volumes must be negotiated with the laboratory to limit the volumes withdrawn.

Where significant differences in parameters such as pH or conductivity exist between the surface water and pore water, a check can be made at the end of sampling to assess whether surface water intrusion has occurred by measuring the pore water parameters at the beginning and conclusion of sampling. Fluorescent dye tracing can also be used for this purpose.

2.6 Pore Water Collection

The flange is first placed at the desired sampling point with the push-point removed to allow any water to escape from under the flange. The flange rim should be carefully worked into the soil or sediment until the flange is flush with the surface. The pore water device should then be inserted through the compression adapter on the flange and into the soil or sediment as carefully as possible (Figure 2). When the sampler is inserted to the desired depth, the compression adapter should be tightened. The push-point's guard rod can then be withdrawn. Do not reinsert the guard rod into the sampler for any reason until the sampler has been cleaned (particles rolled between the two metal surfaces will lock the parts together and permanently damage the sampler.)

When deploying the pore water device, care must be taken not to disturb the sampling area. If the sampler is wading, the sampler should lean out and insert the pore water device as far as possible away from where the sampler is standing to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the pore water device depends on the objectives of the specific investigation.

After the pore water device has been successfully deployed, attach the sample tubing to the sampling port of the pore water device. Short pieces of Silastic® tubing can be used to splice Teflon® sample tubing to a push-point sampler, taking care to butt the tubing to the sampler at the center of the splice. Then attach the other end of the tubing to a sample withdrawing device, such as a syringe or a peristaltic pump (according to LSASD Operating Procedure for Pump Operation, LSASDPROC-203). Before collecting a pore water sample, be sure to purge out all air and surface water from the pore water sampler and sample tubing with the appropriate amount of pore water. This step can be accomplished by calculating the volume of the sampler and attached tubing and pumping this volume plus an additional 10 percent of pore water through the sampler and tubing prior to collecting the sample. If utilizing a syringe for collection, a three-way valve with a side syringe must be utilized for the surface water purge in order not to cross contaminate the sampling syringe.

2.6.1 Peristaltic Pump/Vacuum Jar Collection

The peristaltic pump/vacuum jug can be used for sample collection of organic or inorganic samples because it allows for the sample to be collected without coming in contact with the pump head tubing, maintaining the integrity of the sample. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a pre-cleaned standard 1-liter amber glass container (Figure 3). Teflon® tubing (¼-inch O.D.) connects the container to both the pump and the sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of samples for volatile organic compounds. An alternative method for collecting volatile organics involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the pore water sampler and, then pinched off at the pump in order to maintain the vacuum while it is being disconnected from the pump head tubing. The water is then allowed to carefully drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing, into the sample vials, by reversing the peristaltic pump at very low speed. Great care must still be taken with this method in order not to agitate the sample during the transfer process or to transfer water that has been in contact with the Silastic® tubing into the vials.

Because pore water is typically collected from an anaerobic environment, it is preferable, especially when collecting samples for nutrient analysis, to maintain the integrity of the sample by minimizing exposure to air. This can be accomplished by purging the sample container with an inert gas such as nitrogen or argon prior to sampling. In addition, if analyzing for nutrients or metals, the container can be pre-preserved in order to minimize exposure of the sample to ambient conditions.

An alternative, when collecting samples for metals, nutrients, or other sample analyses not affected by Silastic® tubing and when exposure to air is not a concern, is to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been demonstrated. When collecting samples in this manner, there are several considerations of which to be aware. The pump head tubing (Silastic®, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground or other surface to ensure the integrity of samples collected in this manner.

2.6.2 Syringe

An alternative to using the pump and vacuum container is to use a syringe as the mechanism to draw the pore water through the sampling device. The tubing from the sampling port of the pore water device can be directly attached to a syringe with a three-way valve and a side syringe and the pore water sample can be manually withdrawn. The valve is first switched to the side syringe, which is used for purging air and any ambient surface water in the system prior to sampling. The volume to be purged is determined by the length and diameter of the sampling device and attached tubing. Once the sampler has been purged, the valve is switched to the sampling syringe and the sample is drawn into the syringe. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements. This is the best method to use if the sample is to be collected underwater by SCUBA diving.

2.7 Quality Control

If possible, a control or background sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and reused on-site or, if necessary, to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.8 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect pore water samples shall be cleaned as outlined in the LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205 (most recent version) or LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, LSASDPROC-206 (most recent version) and repaired, if necessary, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

3 Special Sampling Considerations

3.1 Volatile Organic Compounds (VOC)

Pore water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas, unpreserved samples have only a seven day holding time. During most sampling events, preserved vials are used due to their extended holding time. In some situations, however, it may be necessary to use unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used, and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If, after attempting to refill and cap the vial, bubbles are still present, a new vial should be obtained, and the sample should be re-collected.

3.2 Dissolved Metals Sample Collection

If a dissolved metals pore water sample is to be collected, an in-line filtration should be used. The use of disposable, high-capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus is preferred. The high-capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from Section 4.7.3 of the LSASD Groundwater Sampling Procedure (LSASDPROC-301).

3.3 Special Precautions for Pore Water Sampling

- A clean pair of new, non-powdered, disposable latex gloves will be worn each time a different location is sampled, and the gloves should be donned prior to handling sampling equipment. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment. Non-disposable equipment should be pre-cleaned according to procedures contained in LSASD Operating Procedure for Field Equipment Cleaning and Decontamination (LSASDPROC-205), for collection of samples for trace metals or organic compound analyses.

3.4 Sample Handling and Preservation Requirements

- Pore water will typically be collected using a peristaltic pump and placed directly into sampling containers. In some cases, a syringe may be used to collect the pore water and then either left in the syringe as the sample container or transferred into an appropriate container.
- During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
- Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 3.1).
- All samples requiring preservation must be preserved as soon as practically possible, soon after sample collection. If pre-preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. For all other chemical preservatives, LSASD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the LSASD Operating Procedure for Field Sampling Quality Control (LSASDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in the USEPA Laboratory Services Branch *Laboratory Operations and Quality Assurance Manual* (LOQAM).

Figure 1. Pore Water Sampling Device

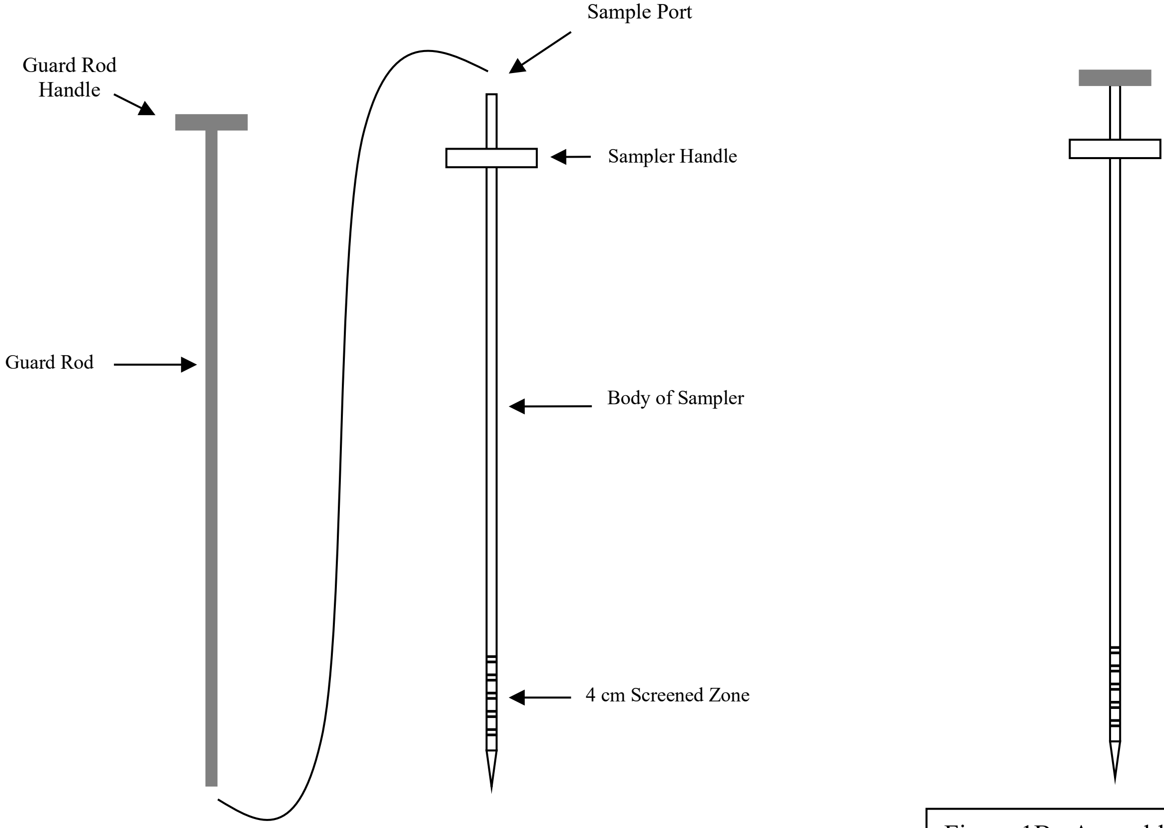
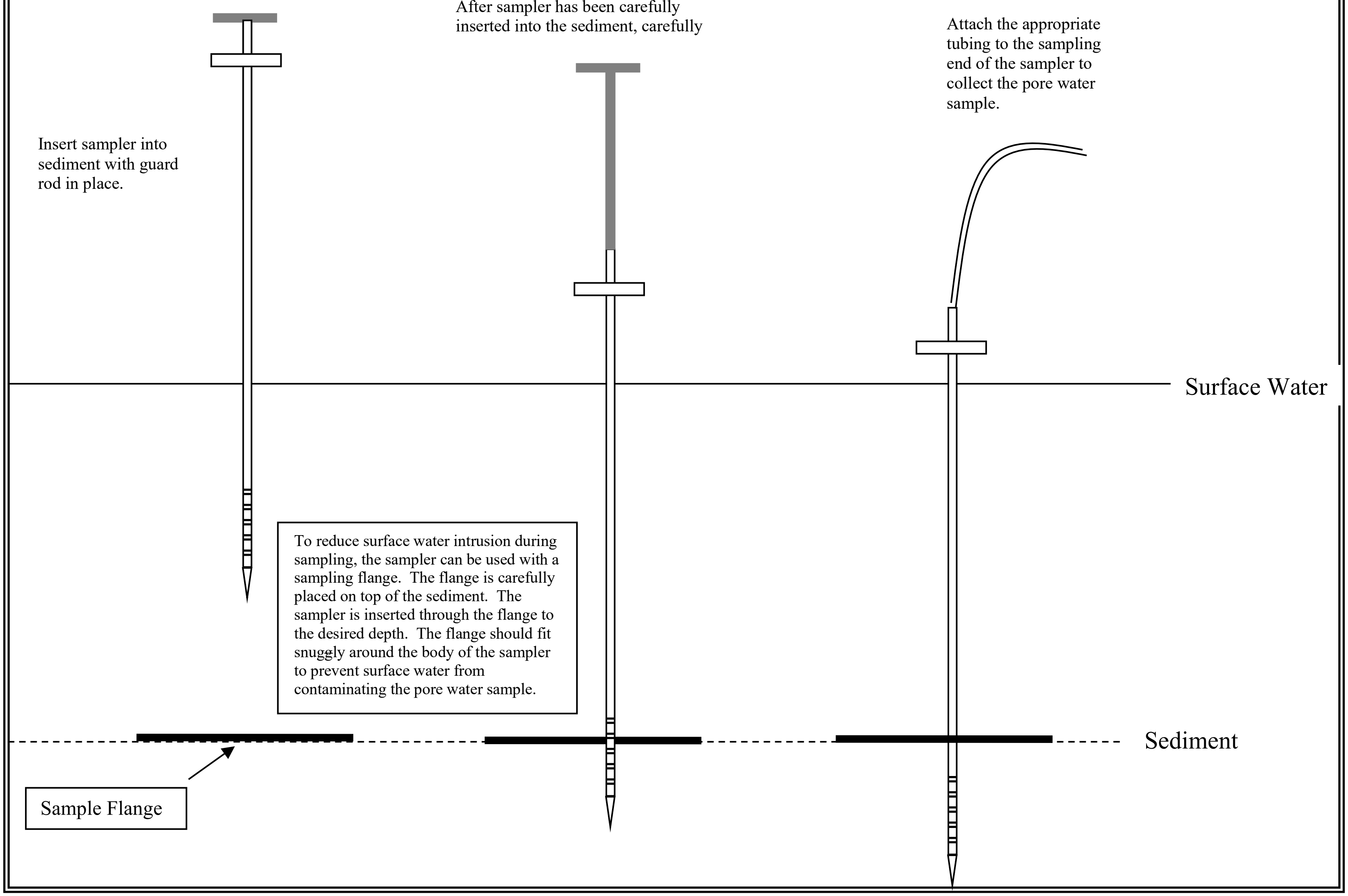


Figure 1A. Disassembled Device

Figure 1B. Assembled Device

Actual length and width of device will vary, depending on sampling needs and site conditions.
Note: cm = centimeter

Figure 2. Pore Water Sampler Deployment Using a Sampling Flange



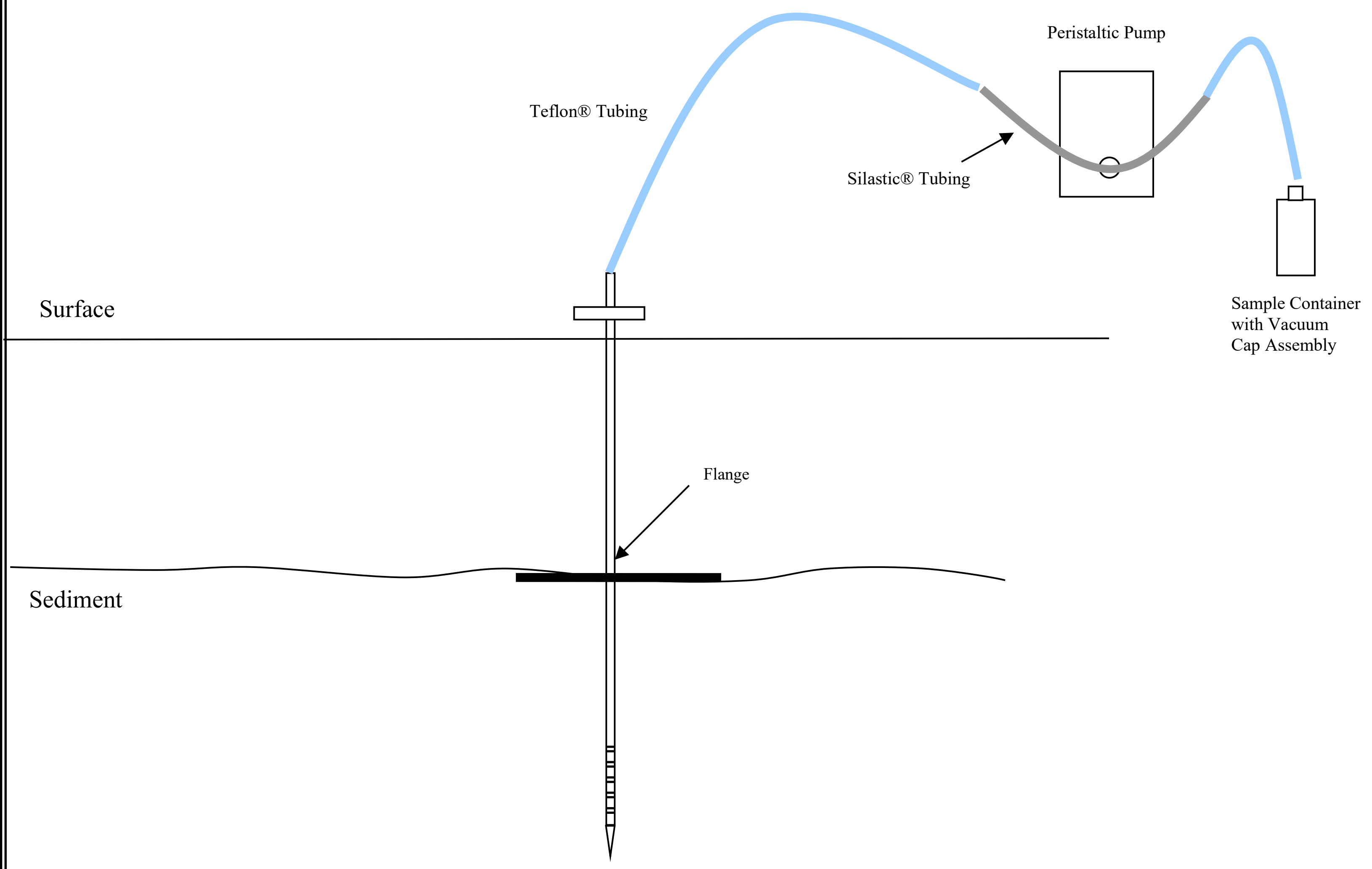
Insert sampler into sediment with guard rod in place.

After sampler has been carefully inserted into the sediment, carefully

Attach the appropriate tubing to the sampling end of the sampler to collect the pore water sample.

To reduce surface water intrusion during sampling, the sampler can be used with a sampling flange. The flange is carefully placed on top of the sediment. The sampler is inserted through the flange to the desired depth. The flange should fit snuggly around the body of the sampler to prevent surface water from contaminating the pore water sample.

Figure 3. Pore Water Sampler Deployment Using a Peristaltic Pump to Sample



References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

M.H.E. Products. 2003. PushPoint Sampler (US Pat. # 6,470,967) Operators Manual and Applications Guide, Version 2.01. East Tawas, MI. http://www.mheproducts.com

LSASD Operating Procedure for Control of Records, LSASDPROC-002, Most Recent Version.

LSASD Operating Procedure for Sample and Evidence Management, LSASDPROC-005, Most Recent Version.

LSASD Operating Procedure for Logbooks, LSASDPROC-010, Most Recent Version.

LSASD Operating Procedure for Surface Water Sampling, LSASDPROC-201, Most Recent Version.

LSASD Operating Procedure for Pump Operation, LSASDPROC-203, Most Recent Version.

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination, LSASDPROC-205, Most Recent Version.

LSASD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, LSASDPROC-206, Most Recent Version.

LSASD Operating Procedure for Groundwater Sampling, LSASDPROC-301, Most Recent Version.

LSASD Operating Procedure for Potable Water Supply Sampling, LSASDPROC-305, Most Recent Version.

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version.

USEPA LSBLOQAM. Laboratory Services Branch Laboratory Operations and Quality Assurance Manual Region 4, Laboratory Services and Applied Science Division, Athens, GA. Most Recent Version.

USEPA SHEMP Safety, Health and Environmental Management Program Procedures and Policy Manual. Laboratory Services and Applied Science Division, Region 4, Athens, GA. Most Recent Version.

LSASD Operating Procedure for Field Sampling Quality Control, LSASDPROC-011, Most Recent Version.

USEPA. 2016. Diving Safety Manual, current version. US Environmental Protection Agency, Washington, DC.

Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the LSASD Document Control Coordinator on the LSASD local area network (LAN).

History	Effective Date
LSASDPROC-513-R4, Pore Water Sampling, *replaces SESDPROC-513-R3.* Laboratory Services and Applied Science Division replaces Science and Ecosystem Support Division **Title Page:** Changed the Field Quality Manager from Hunter Johnson to Stacie Masters. **General:** Corrected typographical, grammatical, and/or editorial errors. Added language to clarify some procedures.	May 13, 2020
SESDPROC-513-R3, Pore Water Sampling, replaces SESDPROC-513-R2. **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Changed the Field Quality Manager from Bobby Lewis to Hunter Johnson. Updated cover page to represent SESD reorganization. John Deatrick was not listed as the Chief of the Field Services Branch	December 16, 2016
SESDPROC-513-R2, Pore Water Sampling, *replaces SESDPROC-513-R1.*	February 28, 2013
SESDPROC-513-R1, Pore Water Sampling, *replaces SESDPROC-513-R0.*	January 29, 2013
SESDPROC-513-R0, Pore Water Sampling, *Original Issue*	February 05, 2007

ARCADIS
Design & Consultancy
for natural and
built assets

ATTACHMENT 2

LABORATORY STANDARD OPERATING PROCEDURES

eurofins

Environment Testing
TestAmerica

Eurofins TestAmerica Knoxville
7 Step Sequential Extraction Procedure Summary

August 13, 2019

Step 1 - Exchangeable Phase: This extraction includes trace elements that are reversibly sorbed to soil minerals, amorphous solids, and/or organic material by electrostatic forces. These forces may be overcome by exposing the soil to a concentrated electrolyte solution, such as 1M $MgSO_4$ that displaces the trace elements from solid surfaces.

Exposure Time/Temperature: 1 hr/ambient.

Step 2 - Carbonate Phase: This extraction targets trace elements that are sorbed or otherwise bound to carbonate minerals. This phase is soluble in a mild acid solution (1M NaOAc solution in 25% HOAc at pH 5).

Exposure Time/Temperature: 3 hr/ambient.

Step 3 – Non-Crystalline Materials Phase: This extraction targets trace elements that are complexed by amorphous minerals (e.g. iron). This phase is extracted with 25 mL of 0.2M ammonium oxalate (pH 3).

Exposure Time/Temperature: 4 hr/ambient.

Step 4 - Metal Hydroxide Phase: Trace elements bound to hydroxides of iron, manganese, and/or aluminum are extracted using a solution of 1M hydroxylamine hydrochloride in 25% v/v acetic acid.

Exposure Time/Temperature: 6 hr/ 95°C ± 5°C.

Step 5 - Organic Phase: This extraction targets trace elements strongly bound via chemisorption to organic material. Oxidation of soil organic matter (using pH 9.5; at 5% NaOCl), will bring into solution metals bound to organic functional groups.

Exposure Time/Temperature: 1 hr/ 95°C ± 5°C.

Step 6 - Acid/Sulfide Fraction: The extraction is used to identify trace elements precipitated as sulfide minerals. Metals associated with sulfide minerals will be extracted by leaching the soils with a 3:1:2 v/v solution of HCl-HNO_3-H_2O to dissolve the metal sulfide minerals.

Exposure Time/Temperature: 1 hr/ 95°C ± 5°C.

Step 7 - Residual Fraction: Trace elements remaining in the soil after the previous extractions will be distributed between silicates, phosphates, and refractory oxides. These residual metals can be removed from the soil through total dissolution with HF, HNO_3, HCl and H_3BO_3.

Exposure Time/Temperature: 3 hr/ 95°C ± 5°C.

eurofins	Always check on-line for validity. **Balance, Syringe, Pipette, Weights and Labware Verification**	Level: **Standard Operating Procedure**
Document number: **QA-SOP11880**		
Old Reference: **1-P-QM-QMA-9015389; DOD - LOM-SOP-ES-235**		
Version: **11**		Organisation level: **5-Sub-BU**
Approved by: **UDM6** Effective Date **31-AUG-2020**	Document users: **4_EUUSLA_ELLE_All_Technical**	Responsible: **5_EUUSLA_Env Quality Assurance_All**

Revision Log
Reference
Cross Reference
Purpose
Scope
Definitions
Personnel Training and Qualifications
Safety Precautions and Waste Handling
Procedure
Documentation
Calculations

Revision Log

Revision:	11	**Effective Date:**	This version
Section	**Justification**	**Changes**	
Revision Log	Formatting requirement	Removed revision logs up to the previous version	
Title	Clarification	Added 'Weights'	
Procedure A	Enhancement	Added the label check following the annual balance calibration. Balances must be re-verified if moved.	
Procedure B	Enhancement	Added directive for potentially damaged weights	
Procedure B	Reflect current process	Evaluation based on the alternate weights that are used during the reclassification period. Weight calibration requirements defined.	

Revision:	10	**Effective Date:**	May 20, 2019
Section	**Justification**	**Changes**	
Reference	Reflect current documents	Added DR-12-SCA-01	
Procedure B	Reflect current requirement	Changed timeframe for reclassification of weights	

Reference

1. *ASTM Standard Specification for Laboratory Weights and Precision Mass Standards,* E 617-97.

2. *ASTM Standard Specification for Laboratory Glass Volumetric Flasks*, E 288-03

3. *ASTM Standard Specification for Laboratory Glass Graduated Cylinders*, E 1272-02

4. *DoD Quality Systems Manual for Environmental Laboratories*, current version

5. *The TNI Standards*, current version

6. *25 Pa 252.306*, current version

7. Center of Expertise in Environmental Analysis of Quebec; *Guidelines for Analytical Work in Chemistry*, DR-12-SCA-01, March 3, 2011.

8. *Chemical Hygiene Plan*, current version

Cross Reference

Document	Document Title
QA-SOP11901	Instrument Maintenance and Calibration

Purpose

To outline the requirements for verifying the accuracy of balances, mechanical pipettes, volumetric syringes, and volumetric labware.

Scope

This procedure addresses the routine maintenance and calibration verification procedures for laboratory balances (analytical and top-loading), volumetric syringes (≤10-µL in size), mechanical pipettes (i.e. eppendorf-style pipettes, repipettors), and the verification of labware to Class A standards. Bottle-top dispensers are verified by this procedure only if used for critical volumes. The requirement for verification applies to equipment where quantitative results are dependent on their accuracy.

Some regulatory programs or special-use equipment have additional requirements for calibration checks and/or maintenance. Because of the wide variety of these devices used by the laboratory, it is recommended that analysts also refer to the manufacturer's instruction manual for additional information.

NOTE: This procedure does not apply to instrument autosampler syringes.

Definitions

1. NIST – National Institute of Standards and Technology

2. ASTM – American Society for Testing and Materials

3. Class 1 Weights (ASTM) – These weights are designated as high-precision standards for calibration. They are used for the monthly (or daily, if desired) calibration check of balances as reference standards for calibrating other weights. These weights are recommended for calibration of balances using optical or electrical methods for accurately weighing quantities below 20 mg.

4. DOD QSM – Department of Defense Quality Systems Manual

5. Sensitivity – The smallest amount that the equipment can measure.

Personnel Training and Qualifications

Personnel performing this procedure must have documentation of reading, understanding and agreeing to follow this version of the SOP.

Analysts must be trained to use the equipment and work with an experienced analyst prior to performing the verification procedure independently.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Procedure

A. Balances

1. Operation

a. Analytical balances must be in an area that is vibration-free in order for the balance to remain level and supply accurate readings. A stabilizing slab can be used on a counter top or in a hood in order to minimize vibrations.

b. Balances must be located away from laboratory traffic and protected from drafts and sudden temperature and humidity changes whenever possible.

c. Special precautions must be taken to avoid spillage of chemicals on the pan or inside the balance case. The interior of the balance housing must be kept scrupulously clean.

d. Before using any balance, check to assure that it is level. In order to obtain accurate readings, a balance must be level. It is the responsibility of each user to ensure that the bubble within the level is fully within the indicator circle each time that they approach the balance for use. If any portion of the bubble is outside of the circle, the balance must be brought back to level and the calibration re-verified prior to use.

e. Do not overload the balance and avoid jarring. Gently place all objects to be weighed on the pan.

f. Each balance is marked with its range and it must not be used for weights outside that range. The total weight placed on the balance is equal to the weight of the tared vessel plus the vessel contents and this total must be within the weight range capacity of the balance. Balance calibration checks (daily and monthly) must span the range of use.

g. A balance could be marked as limited use. The firm that performs the annual check may add a limited use sticker to the balance that will state the limitation such as “corners”. This indicates that the balance does not weigh properly when the load is not placed in the center of the pan. Therefore caution should be used to properly center all loads when weighing.

2. Balance Checks

NOTE: To perform the routine checks, tare the balance with a container representative of the weighing vessel normally used.

a. Daily calibration verification

(1) Check each balance for proper calibration prior to use on each day that it is used. Perform using at least three Class 1 weights that bracket the range of use.

(2) In order to preserve the highly accurate nature of the weights, they must be handled with great care. Ensure the balance pan is free of debris before placing a weight on the pan. Never touch weights with anything other than forceps, and transfer the weights directly from the case to the pan of the balance.

(3) If a balance must be physically moved during the day, perform an additional check of the level and calibration.

b. Acceptance criteria

(1) For **top-loading balances**, the reading must be ±2% or ±0.02g of the true value of the weight, whichever is greater.

(2) For **analytical balances**, the reading must be ±0.1% or ±0.5mg, whichever is greater.

c. Corrective action

(1) For a balance that is not operating within the stated acceptance criteria, first verify the cleanliness of the weights. Clean weights and perform the check again. Some balances are equipped with built-in calibration weights permitting automatic calibration. Consult the manufacturer's manual to see if any adjustments are recommended to correct the problem.

(2) If the problem cannot be resolved, immediately take the balance out of service and place a call to the manufacturer or the professional balance service firm to address the situation. See *QA-SOP11901* in the event of a calibration check failure or the need to take the balance out of service.

(3) Data generated since the last acceptable daily/monthly calibration must be evaluated by management on a case-by-case basis.

d. Annual check

(1) An annual check of the balance must be made by a qualified (ISO *17025 accredited*) professional service firm. This check includes a calibration verification and appropriate preventative maintenance (e.g., cleaning). The Environmental Quality Assurance Department is responsible for scheduling this annual check.

(2) A confirmation of the calibration range defined on the label by the balance is performed following the annual calibration of the balance.

B. Weights

1. If weights are dropped or potentially damaged they need to be checked against alternate weights either from Quality Assurance(QA) or another weight set.

2. Document the comparison in the logbook. Be sure to include the serial number of the alternate weights.

3. All weights must meet the defined acceptance criteria.

4. Reclassification

a. Class 1 weights are available from the Quality Assurance (QA) department for use. The weights must be signed out and returned upon completion of the check. It is the responsibility of QA to have their weights reclassified every 3 years by a qualified (ISO 17025 accredited) calibration firm.

b. If a technical department chooses to purchase their own Class 1 weights, it is their responsibility to ensure they are reclassified every 5 years by a qualified (ISO 17025 accredited) calibration firm.

c. A certificate of reclassification is maintained on file to document traceability to NIST materials used in the recalibration operation. This certification statement, provided with mass standards, certifies that the nominal values of the weights are correct within the prescribed limits. The reclassification certificates for the weights are filed in QA.

d. For weights that are identified on the reclassification certificate as failing the 'As Found tolerance test'

(1) Review the calibration check entries for the department's analytical balance(s) during the time period when the departmental weights were off site for reclassification and the alternate weights were used to perform the balance calibration check. The alternate weights act as a viability check.

(2) Verify that the alternate weights meet the defined acceptance criteria during that period of time.

(3) Document the review including the balance ID, the book # and the status of the alternate weight's acceptance criteria.

(4) Document the reasoning on the reclassification certificate.

C. Syringes

1. Verification

a. Verification is not required for syringes used for handling gas standards.

b. When performing this verification, gloves must be worn to eliminate the possibility of oils from your hands adding weight to the weighing vessel.

c. Verification of Syringes ≥ 50 µL

(1) Pull a volume of reagent water into the syringe that represents ½ the total volume of the syringe (e.g. 25 µL for a 50-µL syringe, 100 µL for a 200-µL syringe).

(2) Ensure that there are no air bubbles and accurately set the syringe to the volume to be measured.

(3) Dispense the reagent water into a tared medicine cup or small vial on an analytical balance.

(4) The weight must fall within the tolerance limits listed in Procedure C.1.d.(4) below.

d. Verification of Syringes <50 µL (20 µL, 10 µL)

(1) Pull a volume of reagent water into the syringe that represents ½ the total volume of the syringe (e.g. 5 µL for a 10-µL syringe, 10 µL for a 20-µL syringe).

(2) Ensure that there are no air bubbles and accurately set the syringe to the volume to be measured.

(3) Dispense the reagent water into a tared, capped GC vial by piercing the cap with the syringe.

(4) The weight must fall within the tolerance limits listed below.

Syringe Tolerance Limits			
Syringe Size (µL)	Volume of Water (µL)	Target Weight (g)	Tolerance Limit (g)
10	5	0.005	(±4%) 0.0048 - 0.0052
20	10	0.010	(±2%) 0.0098 - 0.0102
50	25	0.025	(±2%) 0.0245 - 0.0255
100	50	0.050	(±2%) 0.049 - 0.051
200	100	0.100	(±2%) 0.098 - 0.102
500	250	0.25	(±2%) 0.245 - 0.255
1000	500	0.5	(±2%) 0.49 – 0.51

2. Verification Frequency

a. Initial verification upon receipt:

(1) Perform ten (10) trials as described in Procedure C.1.

(2) If there are several syringes from one lot, test one representative syringe from that lot.

(3) Do not place a syringe (or lot) that is outside the acceptable tolerance into general use.

(4) Notify the department supervisor when new syringes fail and ensure that the vendor is contacted.

(5) If the syringe is within the acceptable tolerance apply the appropriate tape label, using designated color scheme from the table below, and place it in the laboratory for general use.

Syringe Tape Color Code	
Month/Year of calibration	Tape Color
January – even year	Red
July – even year	Blue
January – odd year	Yellow
July – odd year	Green

b. Continuing verification is performed every six months in January and July.

(1) Perform three (3) trials as described in Procedure C.1.

(2) If the syringe is within the acceptable tolerance apply the appropriate tape label and place it in the laboratory for general use.

(3) **Discard any syringe that is outside the tolerance during this verification.**

c. If evidence of wear or deterioration is observed during normal lab operations, recheck the syringe to ensure proper operation and accuracy or discard.

D. Pipettes (eppendorf-style fixed volume or adjustable pipettes, bottle-top dispensers, repipettors)

NOTE: Any pipette found to be contaminated by aspirated liquid or contain a plunger that is excessively “sticky” must be cleaned and verified prior to use.

1. Operation – refer to manufacturer’s instructions for specific steps. The following actions need to be taken into account for any pipette.

a. Pipettes held at an angle will take up more liquid than pipettes held vertically. Pipettes should be held in an upright, vertical position.

b. The density of liquid is dependent on the temperature of the water. Hot and cold liquids can lead to errors in the amount of liquid aspirated. To avoid temperature interferences, pipette liquids that have reached ambient temperature.

c. The measured weight to set volume comparison is made based on the density of reagent water being equivalent to 1 g/mL at ambient temperature.

d. For adjustable pipettes, never use a setting below the lowest volume or above the highest volume of the pipette.

e. Change the pipette tip between each trial.

2. Verification

a. Must be performed each day of use, prior to use. Bottle-top dispensers used for non-critical volumes may have quarterly verification. Daily verification must be used in applications where quantitative results are dependent on their accuracy.

b. Perform and record a minimum of three trials at each nominal volume (volume of use).

(1) The mean must be within ±2% of the nominal volume (bias).

(2) Based on the replicate measurements, RSD must be ≤ 1% of the nominal volume (precision).

(3) If the check fails, the test can be repeated but there must be two passing checks. Otherwise, perform cleaning and maintenance followed by verification.

E. Class B Volumetric labware (e.g. Teflon and HDPE flasks and graduated cylinders that are not used for critical volumes)

1. Verification

a. If the labware is to be used for critical volume preparations (i.e. standard preparation), the labware must be verified to Class A specifications per Procedure F. below.

b. Must be performed by lot, or prior to first use and annually thereafter.

c. Perform and record 10 trials at each nominal volume (volume of use).

(1) The mean must be within ±2% of the nominal volume (bias).

(2) Based on the replicate measurements, RSD must be ≤ 1% of the nominal volume (precision).

(3) If the check fails, the test can be repeated but there must be two passing checks. Otherwise, perform cleaning and maintenance followed by verification.

F. Verification of labware to Class A glassware specifications

1. Tare the clean and dry volumetric on a top-loading balance with at least 0.001 g specificity.

2. Fill volumetric to the mark with reagent water, taking care not to splash water on the walls of the volumetric.

3. Measure and record the weight of the reagent water in the volumetric.

4. Determine the temperature by placing a thermometer in the volumetric after weighing.

5. Calculate the volume (see Calculation section).

6. Verify that the weight meets the tolerances listed in the table below.

7. Engrave an "A" next to the engraved serial number as verification that the volumetric has been checked and meets Class A acceptance criteria.

Required tolerances

Capacity (ml)	**Class A flasks (+/- ml)**	**Class A graduated cylinders (+/- ml)**

eurofins

Document number:
QA-SOP11880
Old Reference:
1-P-QM-QMA-9015389; DOD - LOM-SOP-ES-235
Version:
11
Approved by: UDM6
Effective Date 31-AUG-2020

Always check on-line for validity.
Balance, Syringe, Pipette, Weights and Labware Verification

Document users:
4_EUUSLA_ELLE_All_Technical

Level:
Standard Operating Procedure
Organisation level:
5-Sub-BU
Responsible:
5_EUUSLA_Env Quality Assurance_All

5	0.02	0.05
10	0.02	0.10
25	0.03	0.10
50	0.05	0.25
100	0.08	0.50
200	0.10	0.50
250	0.12	1.00
500	0.20	2.00
1000	0.30	3.00
2500	0.50	6.00

Documentation

A. All the above verifications, any repairs or maintenance must be documented in a maintenance and calibration logbook and/or a verification logbook or acceptable alternative (database, excel spreadsheet, etc.). The acceptance criteria must be noted in the log. Corrective action must be documented for any checks that are out of specification.

B. The unique identification of the equipment (balance, pipette, weight set, etc.) used to perform the various calibrations must also be documented.

Calculations

A. Percent Difference (%D):

$$\%D = \frac{Measured\ Weight - Targeted\ Weight}{Targeted\ Weight} \times 100$$

B. Relative Standard Deviation (RSD):

$$RSD = \frac{Standard\ Deviation\ of\ the\ data\ set}{Mean\ of\ the\ data\ set} \times 100$$

NOTE: The measured weight to set volume (targeted weight) comparison is made based on the density of reagent water being equivalent to 1 g/mL.

C. Volume Calculation for Class A glassware specifications:

$$V_{flask} = \frac{W_{flask+water} - W_{flask}}{\rho_{water}}$$

where:

Vflask = volume of the flask or graduated cylinder

Wflask + water = weight of the water and flask or graduated cylinder

Wflask = weight of the flask or graduated cylinder

pwater = density of water based off the measured temperature

Density of reagent water

Temperature °C	Density (g/ml)
16.0	0.99897
16.5	0.99889
17.0	0.99880
17.5	0.99871
18.0	0.99862
18.5	0.99853
19.0	0.99843
19.5	0.99833
20.0	0.99823
20.5	0.99812
21.0	0.99802
21.5	0.99791
22.0	0.99780
22.5	0.99768
23.0	0.99757

QA-SOP11901 Instrument Maintenance and Calibration

End of document

Version history

Version	Approval	Revision information
9	18.JUN.2018	
10	20.MAY.2019	
11	14.AUG.2020	

Technical Information

Reference Number:	ASTM D5084-16
Test Method Title:	Standard Test Method for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter
Test Property:	Hydraulic conductivity
Test Specimen Size:	Length to diameter ratio of at least 1:1; typically 2.87 or 4.00 inch diameter (from Shelby tube or remolded sample)
Number of Test Specimens:	1
Test Equipment:	Permeameter Pressure panels (hydraulic control system) Vacuum pump Thermometer De-aired water system Trautwein permometer for Method F constant volume test.

Standard Operating Procedure

1. If sample is undisturbed:
   Extrude from tube, cut ends plane and perpendicular, and trim. If voids are present by removing pebbles or removing crumbling areas they may be filled with remolded material from trimmings of the specimen. Prepare such that there is no moisture loss.

2. If sample is remolded:
   Compact specimen to specified density and moisture content using metal mold, scarifying each lift prior to placing the next.

3. Upon completion of sample preparation, measure and record the sample height and diameter in three locations and average. Measure and record the sample mass. Determine the moisture content of the material by using trimmings or unused portion of sample.

4. Place specimen on soaked filter paper disk which sits on a saturated porous stone and placed on base of bottom section of permeameter. Place another soaked filter paper, porous stone and top cap on top of specimen. Using flexible membranes and O-rings, seal specimen, top cap and base plate.

5. Attach cell and fill area surrounding the specimen with water. Begin de-airing specimen with small vacuum. Hook permeameter up to Pressure Panel. Flush out the system by opening flow line valves.

6. Fill pressure panels with de-aired water – tap or bottled, not distilled.

7. Apply cell pressure to 10 psi and backpressure (specimen pressure) to 5 psi (both top and bottom of specimen). Increase pressures by 5 psi measuring and recording changes in volumes. Measure b-value

after each increment. Once a 0.95 b-value is achieved stop increasing pressures. This may take several days. [note: if client requests an effective stress other than 5 psi, the pressure difference between the cell and sample pressure will be changed to accommodate that request]

METHOD A (Constant Head Test)

8. After backpressure saturation, induce flow by increasing bottom sample pressure (inflow). Typically a 2 psi difference is sufficient between bottom and top portions of sample keeping a 5 psi difference (effective stress) between the sample and cell. This difference (which directly affects the gradient) should be checked against recommended gradients in the test standard section 8.5.1.

9. Begin recording inflow and outflow vs time. Calculated permeability for each set of readings.

10. Test is complete when: at least 4 values of permeability have been determined, the ratio of rate of inflow to rate of outflow shall be between 0.75 and 1.25 for the last four consecutive permeability determinations, and the hydraulic conductivity is steady. Steady is defined as when four or more readings fall within 25% of the mean value for permeabilities greater than 1 x 10^{-8} cm/sec or within 50% for permeabilities less than 1 x 10^{-8} cm/sec.

11. Record temperature and correct permeability value to 20 ºC.

12. After completion of permeation, reduce the confining, influent and effluent pressures taking care not to change the volume of the specimen. Disassemble the permeameter cell and remove the test specimen. Measure and record the final height and diameter in three locations and average. Measure the mass of the specimen. Determine the final moisture content by using the entire specimen or using a cross section of the specimen.

13. Calculate permeability as follows:

    $k = QL/Ath * R_t$

    where:

    k = permeability, m/sec
    Q = quantity of flow, taken as the average of inflow and outflow, m^3
    L = length of specimen along path of flow, m
    A = cross-sectional area of specimen, m^2
    t = interval of time, s, over which flow, Q, occurs
    h = difference in hydraulic head across the specimen, m of water
    R_t =temperature correction (IF NEEDED)

METHOD B (Falling Head-Test) – GTX Georgia

14. Create a head using a burette

15. Open valve and record height of water in burette (initial) and start timer

16. Stop flow and stop timer record the height in the burette (final).

17. Continue permeation until a least four values of hydraulic conductivity are obtained over an interval of

time in which the hydraulic conductivity is steady.

METHOD C (Increasing Tailwater Level) – GTX Georgia

18. If the water pressure at the downstream end of the test specimen rises during an interval of time, periodically measure and record either the quantity of inflow and outflow or the change in water level in the influent and effluent burettes.

19. Continue permeation until a least four values of hydraulic conductivity are obtained over an interval of time in which the hydraulic conductivity

METHOD F (Constant Volume) - refer to Figure 2

De-air permometer:

20. Connect line #5 to port #4 and line #6 to port #3. Slowly open port #4 then open port #3.

21. Close port #3. Slowly open valve #8 drawing air bubbles from the line into top reservoir of permometer. Close valve #8 once all air is out of line.

22. Open port #3, close port #4. Slowly open valve #7 to draw air out of top reservoir. The Mercury column will rise during this step. Close valve #7 when the Mercury level reaches 20. Re-open port #4. If necessary, repeat step 4 until all air is visibly removed from top reservoir.

23. Open valve #8 slowly. Leave open for approximately 5 seconds to remove any residual air within line. Close valve #8 and repeat for valve #7 carefully watching Mercury level.

Flow phase:

24. Close port #1. Slowly open valve #7 drawing Mercury to required gradient level (Z_1). See test standard section 8.5.1 for appropriate gradients. Close valve #7 and immediately close port #2.

25. When the top of the Mercury meniscus falls to the next graduation on the column, start timing and record level of Mercury as Z_1. Record for a minimum of 0.2 graduations on the column or at least 30 seconds, whichever takes longer. Record time and Mercury level (Z_2). Record temperature.

26. Calculate gradient as follows:

$i = [(Z_1 \times 12.6) / L]$

where:
i = gradient
L = length of sample, cm

27. Calculate permeability value as follows:

    $k = (-2.395 \text{ x } 10^{-3}) \text{ x } (L/At) \text{ x } (\ln[1-(\Delta Z/Z_1) \text{ x } 1.040953] \text{ x } R_t$

    where:
    k = permeability, cm/sec
    L = length of sample, cm
    A = area of sample, cm^2
    t = time, sec
    R_t =temperature correction (IF NEEDED)

28. Repeat until 4 values of permeability are obtained over a period of time where readings fall within 25% of the mean value for permeabilities greater than $1 \text{ x } 10^{-8}$ cm/sec or within 50% for permeabilities less than $1 \text{ x } 10^{-8}$ cm/sec.

29. After completion of flow phase close all valves, reduce pressures taking care not to change the volume of the specimen. Disassemble the permeameter cell and remove the test specimen. Measure and record the final height and diameter in three locations and average. Measure the mass of the specimen. Determine the final moisture content by using the entire specimen or using a cross section of the specimen.

30. Report: sample identification, descriptive information, specimen type, initial and final dimensions, moisture content, and density of specimen, permeant used, magnitude of cell and sample pressures, effective consolidation stress, gradient used, average hydraulic conductivity over the last four readings.

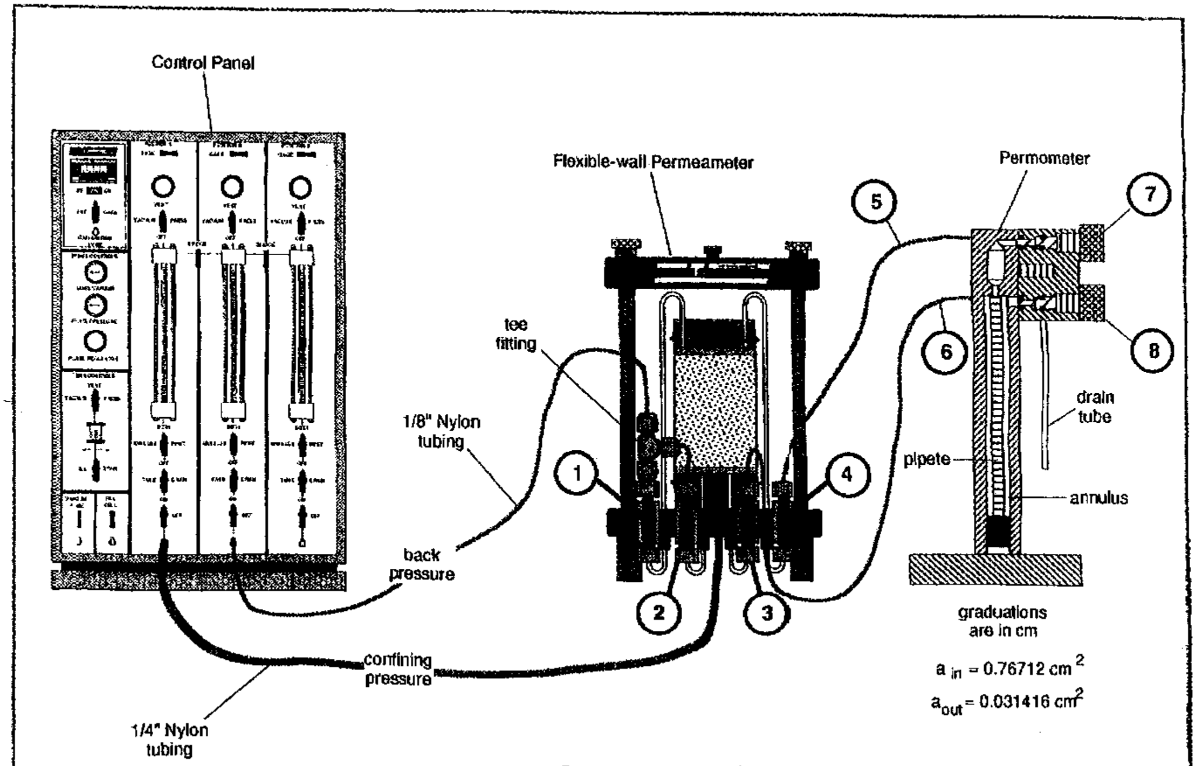


Fig. 2 Permometer Connection to Flexible-Wall Permeameter

Title: Biological Activity Reaction Test (BART™)

Owner: Michael Manning

APPROVALS

11/2/2020

Urooj Sagheer

11/2/2020

Kamash Ramanathan **Date**
Regional Laboratory Director, West

Urooj Sagheer **Date**
Quality Assurance Manager

Copyright Information:

This documentation has been prepared by Eurofins EMLab P&K solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to Eurofins EMLab P&K upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS EMLAB P&K IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS EMLAB P&K IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:

1.0 SCOPE & APPLICATION

1.1 This procedure describes the process for the identification of the presence or absence of various types of bacteria via utilization of an applicable **BART™** test kit. This method is applicable to bulked activated sludge, masses of microbial growth in lakes, rivers, and streams, slime growth in cooling towers, waste water and drinking water. The results are reported as presence or absence.

2.0 SUMMARY

2.1 Water samples are analyzed using the appropriate BART test kit available from Hach (www.hach.com).

3.0 DEFINITIONS

3.1 **Slime Producing Bacteria**: Bacteria capable of producing slime without necessarily having to use any iron. These bacteria generally produce the thickest slime formations under aerobic conditions.

3.2 **Iron Related Bacteria**: Bacteria categorized as having two common features: the
presence of high concentration of ferric (Fe+++) and of high populations of IRB (either as stalked Gallionella, the sheathed IRB or the heterotrophic IRB). These bacteria are able to shunt the iron through oxidative and reductive states through ferric (Fe+++) and ferrous (Fe++) forms respectively.

3.3 **Sulfate Reducing Bacteria:** A group of anaerobic bacteria that, as a part of their
normal activities, generate hydrogen sulfide (H2S). They utilize hydrogen rather than oxygen as the basic driver for many of the metabolic activities. As a result of this, the SRB are anaerobic and are inhibited by the presence of oxygen

3.4 **Denitrifying Bacteria**: Diverse group of bacteria which can reduce nitrate through to nitrite and some continue the nitrification on down to gaseous nitrogen (complete denitrification). In waters, the presence of an aggressive population of denitrifiers can be taken to indicate that there are significant amounts of nitrate in the water.

4.0 INTERFERENCES

4.1 In using the IRB-BART™ to examine waters for the presence of iron related bacteria, it has to be remembered that iron bacteria grow predominantly on surfaces and not directly in the water. When testing water, the BART™ user has to assume that the IRB have detached, are suspended, and possibly are active in the water. As a consequence of this problem, there is a potential for an IRB-BART™ to give a “false” negative since the IRB are absent from the water but are present on the surfaces over which the water is flowing towards the sampling site.

4.2 Sulfate Reducing Bacteria grow deep within biofilms and unless disturbed, may not be present in free flowing water towards the sampling site; thus, false negatives may occur.

5.0 SAFETY

5.1 Wash hands with disinfectant soap before and after handling samples.

5.2 Avoid skin contact, ingestion or inhalation of sample or reagents.

5.3 Observe aseptic precautions when handling specimens.

5.4 Employees must abide by the policies and procedures in the Corporate Safety Manual and this document. There are no materials with a health rating of 3 or 4 used in this method. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS

6.0 EQUIPMENT & INSTRUMENTATION

6.1 Disposable pipettes

6.2 UV Light

7.0 MEDIA & REAGENTS

7.1 Hach BART Test Kit
  7.1.1. SLYM-BART™, for detection of Slime Forming Bacteria
  7.1.2. IRB-BART™, for detection of Iron Related Bacteria

7.1.3. SRB-BART™, for detection of Sulfate Reducing Bacteria
7.1.4. DN-BART™, for detection of Denitrifying Bacteria

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

8.1 Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance and/or specific contract or client requests. This method does not have any referenced holding time.

9.0 QUALITY ASSURANCE

9.1 A trained and qualified technician must analyze all samples. Prior to performing any analyses of client samples, the analyst must have on file with the Quality Assurance Department an Initial Demonstration of Competency (IDOC) demonstrating proficiency with the method and the analyte.

9.2 All samples should be maintained at < 10º C prior to testing.

9.3 Use media and reagents prior to the expiration date

9.4 Collect and store Certificate of Analysis (CoA) from each kit received.

9.4.1. The CoA shall be traceable to client samples by lot/batch number.

9.5 As the manufacturer CoA confirms the following prior to release of the kits, no controls will be processed internally:
9.5.1. Confirmation of sterility for vials and caps.
9.5.2. Approval of the medium as being appropriately formed and acceptable.
9.5.3. Sterility
9.5.4. Confirmation that the kits respond in a typical way to inoculation and incubation using selected defined microbial cultures.

9.6 Store media and reagents at the conditions recommended by the manufacturer, those conditions mandated by regulatory requirements, or those mandated by the specific method. In lieu of any specific requirements, default to the strictest standard.

10.0 PROCEDURE

10.1 SLYM-BART™, IRB-BART™, SRB-BART™, and DN-BART™

10.1.1. Remove inner tube from outer tube

10.1.2. Using the outer tube from the BART, or a different sterile container, collect at least 20 mL of sample. Note: Do not touch or contaminate the inside of the tube or lid. Use aseptic technique.

- **a.** Liquid Samples – Fill the inner tube with sample until the level reaches the "fill" line.
- **b.** Solid Samples:
  - **i.** Weight out 1g of sample and suspend the weighed sample in a sterile conical/capped tube containing 99mL DI water.
  - **ii.** Mix and vortex the sample for 10-15 seconds to obtain a uniform suspension.
  - **iii.** Fill the inner tube with sample until the level reaches the "fill" line.

10.1.3. Tightly screw the cap back on the inner tube. Return the inner tube to the outer tube and screw the outer cap on tightly. Allow the ball to rise at its own speed. DO NOT SHAKE OR SWIRL THE TUBE

10.1.4. Label the outer tube with the date and sample origin.

10.1.5. Place the BART tube away from direct sunlight and allow to incubate at room temperature. Check the BART visually for reaction daily according the timelines below and record observations:

- **a.** SLYM-BART™ - 8 days
  - **i.** Expected reactions: Cloudy; glowing ring around ball under UV and/or slime growing at base of tube
- **b.** IRB-BART™ - 8 days
  - **i.** Expected reactions: Brown slime ring or foam around ball; and/or brown slime growth at base of tube.

- **c.** SRB-BART™ - Standard maximum length for the monitoring of the reaction patterns is commonly ten (10) days. The analyst may find it advantageous to continue observations until the fifteenth (15) day. This is because some SRB do not exhibit reaction patterns until after other bacterial consortia have already grown within the tester (e.g. anaerobic bacteria). This delays the observation of a positive detection for the SRB. In water pipelines and biofouling water wells the time lags can be delayed until days 11 to 15.
  - **i.** Expected reactions: Black slime ring beneath ball; and/or black slime growth at base of tube

- **d.** DN-BART™ - 4 days
  - **i.** Expected reactions: Foam around ball.

11.0 CALCULATIONS

11.1 Qualitative Analysis; only presence/absence to be recorded on the appropriate QC log.

11.2 **Data Qualifiers -** Use data qualifiers to qualify preparatory and/or analytical methods that differ from our standard procedures due to extenuating circumstances (i.e. non-standard sample matrix, non-standard client sampling, damaged samples, etc).

12.0 METHOD PERFORMANCE

12.1 Employees must abide by the training policies and procedures in the Quality Assurance Manual, SOP EM-AD-S-1646 "General Training" and this document.

13.0 POLLUTION CONTROL & WASTE MANAGEMENT

13.1 It is Eurofins EMLab P&K's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantities needed, preparation of reagents based on anticipation usage and reagent stability).Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual and SOP "Procedure for Retention and Disposal of Samples" Document EM-HS-S-1286 for "Waste Management and Pollution Prevention."

14.0 REFERENCES/CROSS-REFERENCES

14.1 BART kit instructions, Hach Company.
14.2 BART user manual (2004), Droycon Bioconcepts, Inc.

15.0 ATTACHMENTS

15.1 Attachment I - BART Instructions

16.0 REVISION HISTORY

16.1 Revision 00, April 2013.

16.2 Revision 01, July 2015
- **16.2.1.** Updated SOP template and reference document numbers.
- **16.2.2.** Updated owner and QA Manager
- **16.2.3.** Changed MSDS to SDS.

16.3 Revision 02, July 2017
- **16.3.1.** Added procedure for solid sample preparation in section 10.3

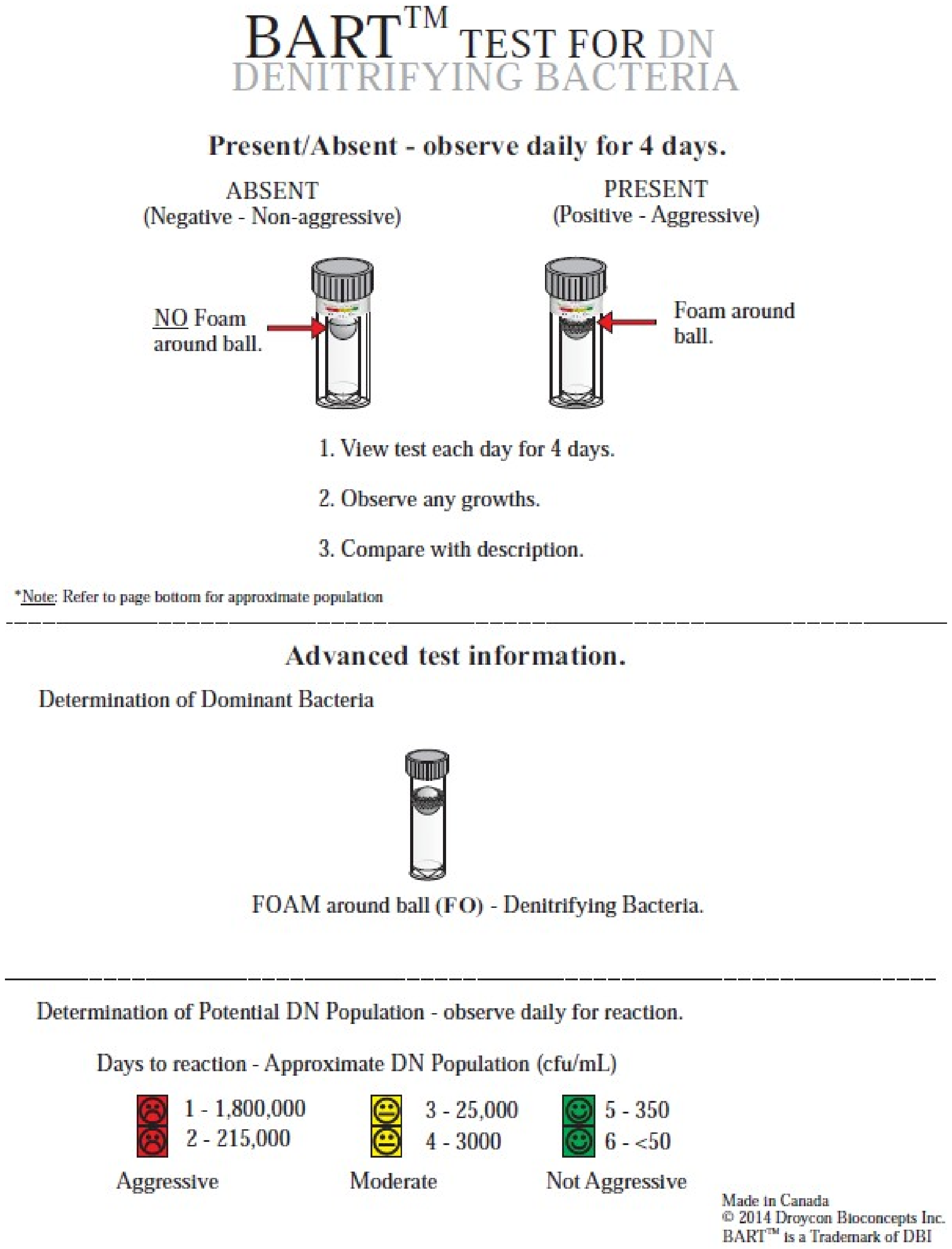
16.4 Revision 03, February 2018
- **16.4.1.** Retitled from Slime Forming Bacteria to BART in order to add additional BART kits (using the same technology) for additional target organisms.

16.5 Revision 04, October 2018
- **16.5.1.** Removal of instructions for performing positive controls
- **16.5.2.** Removal of requirement to perform controls

16.6 Revision 05, November 2020.
- **16.6.1.** Updated for rebranding
- **16.6.2.** Removed reference to 30C incubator
- **16.6.3.** Removed obsolete reagents in 7.1
- **16.6.4.** Review and approval documented in QAzilla 11179

ATTACHMENT 1 – BART Instructions beginning P. 8

ent 1



BART^{TM} TEST FOR DN
DENITRIFYING BACTERIA

Present/Absent - observe daily for 4 days.

ABSENT (Negative - Non-aggressive)	PRESENT (Positive - Aggressive)
NO Foam around ball.	Foam around ball.

1. View test each day for 4 days.
2. Observe any growths.
3. Compare with description.

*Note: Refer to page bottom for approximate population

Advanced test information.

Determination of Dominant Bacteria

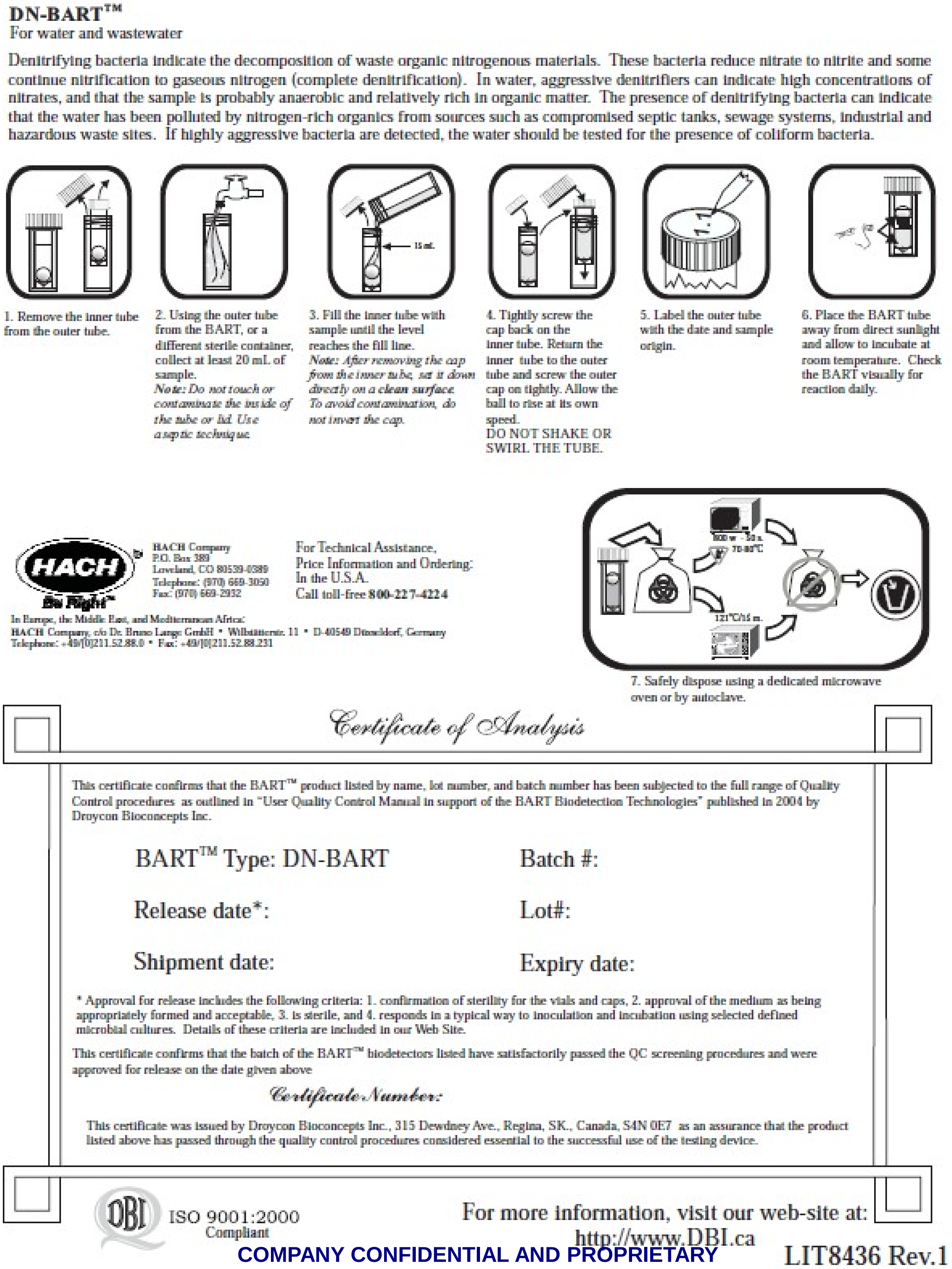
FOAM around ball (FO) - Denitrifying Bacteria.

Determination of Potential DN Population - observe daily for reaction.

Days to reaction - Approximate DN Population (cfu/mL)

☹ 1 - 1,800,000	😐 3 - 25,000	☺ 5 - 350
☹ 2 - 215,000	😐 4 - 3000	☺ 6 - <50
Aggressive	Moderate	Not Aggressive

Made in Canada
© 2014 Droycon Bioconcepts Inc.
BART^{TM} is a Trademark of DBI



DN-BART™

For water and wastewater

Denitrifying bacteria indicate the decomposition of waste organic nitrogenous materials. These bacteria reduce nitrate to nitrite and some continue nitrification to gaseous nitrogen (complete denitrification). In water, aggressive denitrifiers can indicate high concentrations of nitrates, and that the sample is probably anaerobic and relatively rich in organic matter. The presence of denitrifying bacteria can indicate that the water has been polluted by nitrogen-rich organics from sources such as compromised septic tanks, sewage systems, industrial and hazardous waste sites. If highly aggressive bacteria are detected, the water should be tested for the presence of coliform bacteria.

1. Remove the inner tube from the outer tube.

2. Using the outer tube from the BART, or a different sterile container, collect at least 20 mL of sample.
Note: Do not touch or contaminate the inside of the tube or lid. Use aseptic technique.

3. Fill the inner tube with sample until the level reaches the fill line.
Note: After removing the cap from the inner tube, set it down directly on a ***clean surface****.*
To avoid contamination, do not invert the cap.

4. Tightly screw the cap back on the inner tube. Return the inner tube to the outer tube and screw the outer cap on tightly. Allow the ball to rise at its own speed.
DO NOT SHAKE OR SWIRL THE TUBE.

5. Label the outer tube with the date and sample origin.

6. Place the BART tube away from direct sunlight and allow to incubate at room temperature. Check the BART visually for reaction daily.

7. Safely dispose using a dedicated microwave oven or by autoclave.

HACH®
Be Right™

HACH Company
P.O. Box 389
Loveland, CO 80539-0389
Telephone: (970) 669-3050
Fax: (970) 669-2932

For Technical Assistance,
Price Information and Ordering:
In the U.S.A.
Call toll-free **800-227-4224**

In Europe, the Middle East, and Mediterranean Africa:
HACH Company, c/o Dr. Bruno Lange GmbH • Willstätterstr. 11 • D-40549 Düsseldorf, Germany
Telephone: +49/[0]211.52.88.0 • Fax: +49/[0]211.52.88.231

Certificate of Analysis

This certificate confirms that the BART™ product listed by name, lot number, and batch number has been subjected to the full range of Quality Control procedures as outlined in "User Quality Control Manual in support of the BART Biodetection Technologies" published in 2004 by Droycon Bioconcepts Inc.

BART™ Type: DN-BART

Batch #:

Release date*:

Lot#:

Shipment date:

Expiry date:

* Approval for release includes the following criteria: 1. confirmation of sterility for the vials and caps, 2. approval of the medium as being appropriately formed and acceptable, 3. is sterile, and 4. responds in a typical way to inoculation and incubation using selected defined microbial cultures. Details of these criteria are included in our Web Site.

This certificate confirms that the batch of the BART™ biodetectors listed have satisfactorily passed the QC screening procedures and were approved for release on the date given above

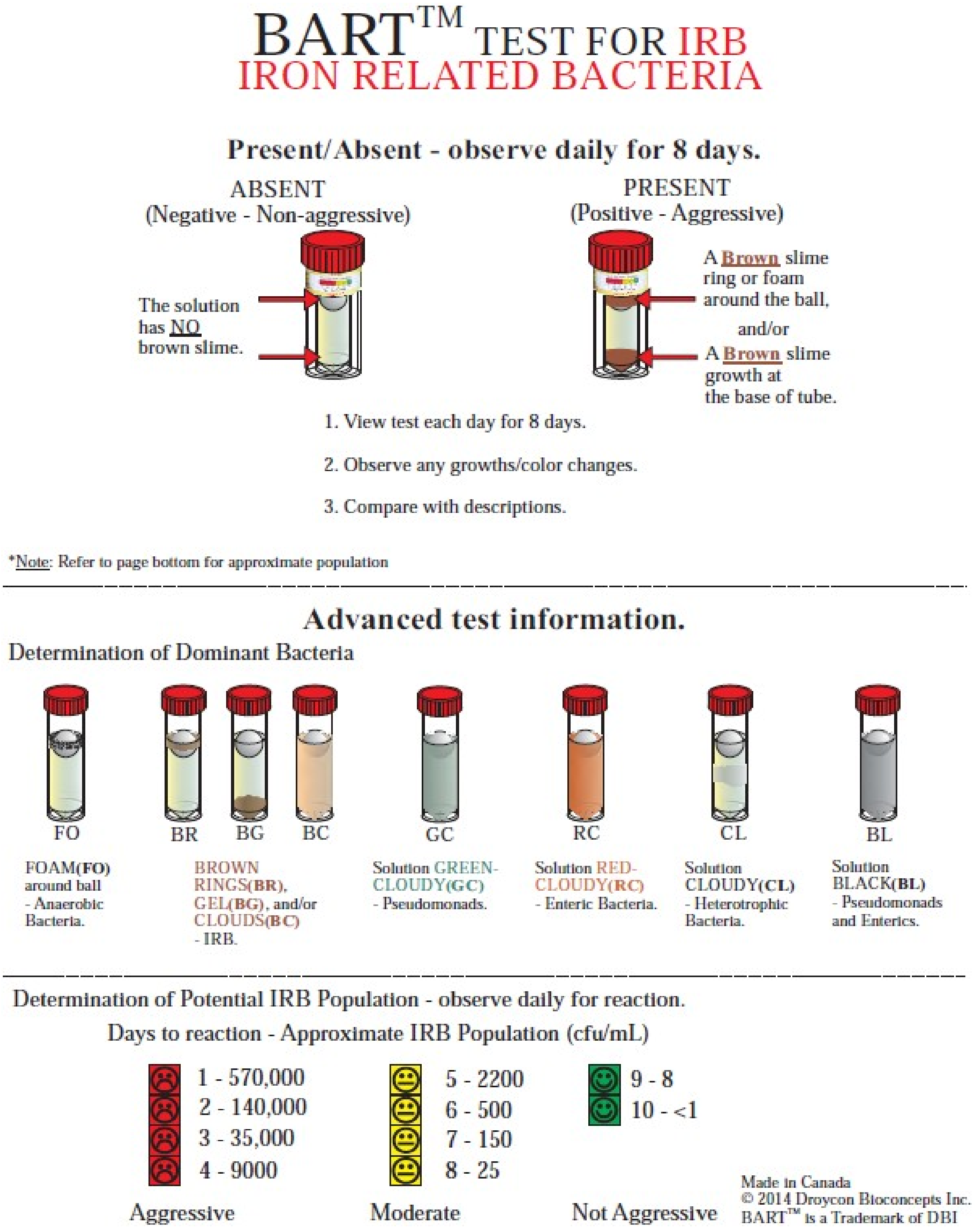
Certificate Number:

This certificate was issued by Droycon Bioconcepts Inc., 315 Dewdney Ave., Regina, SK., Canada, S4N 0E7 as an assurance that the product listed above has passed through the quality control procedures considered essential to the successful use of the testing device.

DBI ISO 9001:2000
Compliant

For more information, visit our web-site at:
http://www.DBI.ca

LIT8436 Rev.1



BART™ TEST FOR IRB
IRON RELATED BACTERIA

Present/Absent - observe daily for 8 days.

ABSENT
(Negative - Non-aggressive)

The solution has NO brown slime.

PRESENT
(Positive - Aggressive)

A Brown slime ring or foam around the ball,

and/or

A Brown slime growth at the base of tube.

1. View test each day for 8 days.
2. Observe any growths/color changes.
3. Compare with descriptions.

*Note: Refer to page bottom for approximate population

Advanced test information.

Determination of Dominant Bacteria

FOAM(**FO**) around ball - Anaerobic Bacteria.

BROWN RINGS(**BR**), GEL(**BG**), and/or CLOUDS(**BC**) - IRB.

Solution GREEN-CLOUDY(**GC**) - Pseudomonads.

Solution RED-CLOUDY(**RC**) - Enteric Bacteria.

Solution CLOUDY(**CL**) - Heterotrophic Bacteria.

Solution BLACK(**BL**) - Pseudomonads and Enterics.

Determination of Potential IRB Population - observe daily for reaction.

Days to reaction - Approximate IRB Population (cfu/mL)

☹ 1 - 570,000
☹ 2 - 140,000
☹ 3 - 35,000
☹ 4 - 9000

Aggressive

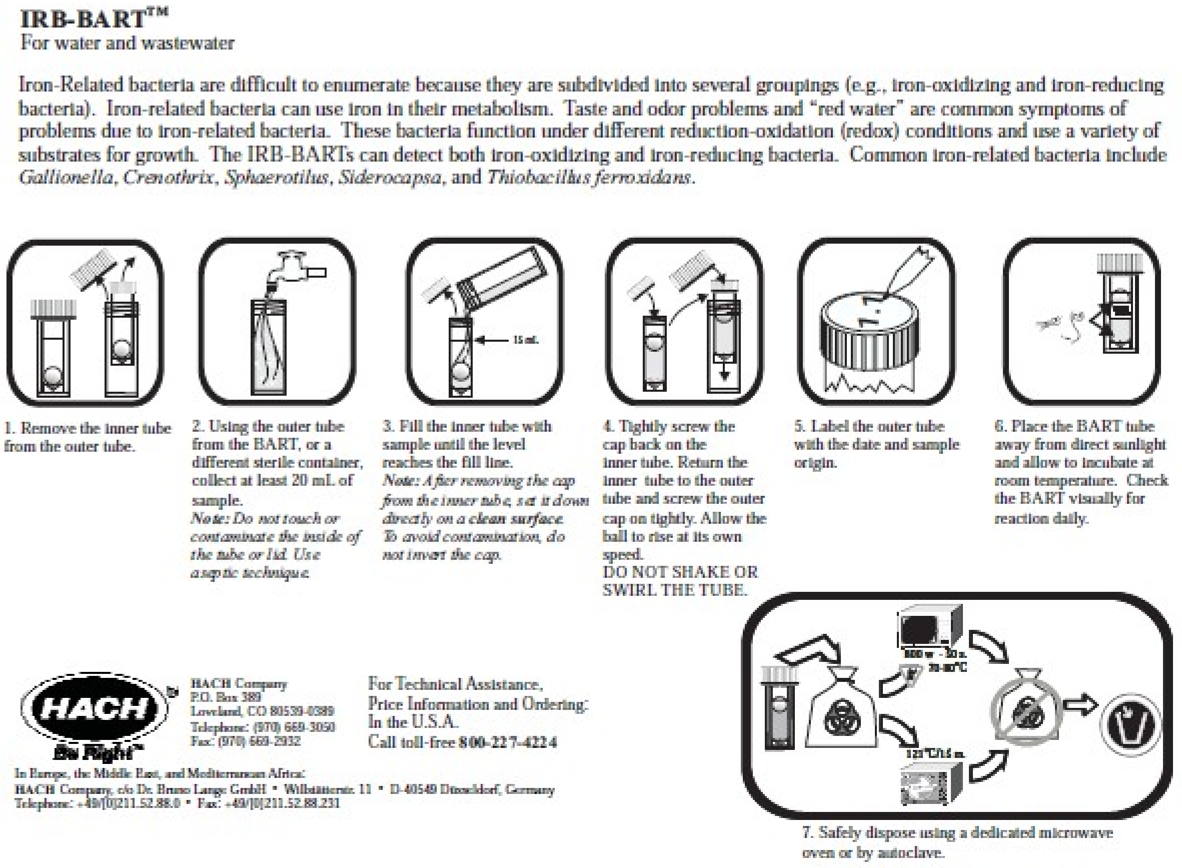
😐 5 - 2200
😐 6 - 500
😐 7 - 150
😐 8 - 25

Moderate

☺ 9 - 8
☺ 10 - <1

Not Aggressive

Made in Canada



IRB-BART™

For water and wastewater

Iron-Related bacteria are difficult to enumerate because they are subdivided into several groupings (e.g., iron-oxidizing and iron-reducing bacteria). Iron-related bacteria can use iron in their metabolism. Taste and odor problems and "red water" are common symptoms of problems due to iron-related bacteria. These bacteria function under different reduction-oxidation (redox) conditions and use a variety of substrates for growth. The IRB-BARTs can detect both iron-oxidizing and iron-reducing bacteria. Common iron-related bacteria include *Gallionella*, *Crenothrix*, *Sphaerotilus*, *Siderocapsa*, and *Thiobacillus ferroxidans*.

1. Remove the inner tube from the outer tube.

2. Using the outer tube from the BART, or a different sterile container, collect at least 20 mL of sample.
Note: Do not touch or contaminate the inside of the tube or lid. Use aseptic technique.

3. Fill the inner tube with sample until the level reaches the fill line.
Note: After removing the cap from the inner tube, set it down directly on a clean surface. To avoid contamination, do not invert the cap.

4. Tightly screw the cap back on the inner tube. Return the inner tube to the outer tube and screw the outer cap on tightly. Allow the ball to rise at its own speed.
DO NOT SHAKE OR SWIRL THE TUBE.

5. Label the outer tube with the date and sample origin.

6. Place the BART tube away from direct sunlight and allow to incubate at room temperature. Check the BART visually for reaction daily.

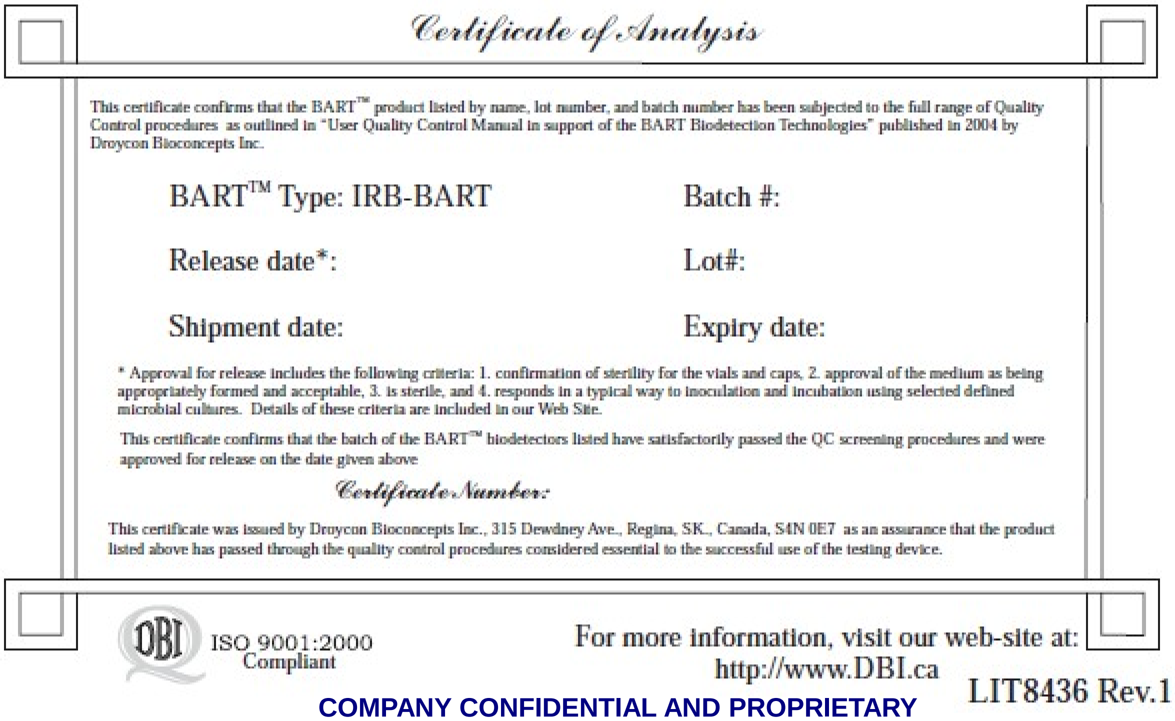
7. Safely dispose using a dedicated microwave oven or by autoclave.

HACH
Be Right™

HACH Company
P.O. Box 389
Loveland, CO 80539-0389
Telephone: (970) 669-3050
Fax: (970) 669-2932

For Technical Assistance,
Price Information and Ordering:
In the U.S.A.
Call toll-free **800-227-4224**

In Europe, the Middle East, and Mediterranean Africa:
HACH Company, c/o Dr. Bruno Lange GmbH • Willstätterstr. 11 • D-40549 Düsseldorf, Germany
Telephone: +49/[0]211.52.88.0 • Fax: +49/[0]211.52.88.231



Certificate of Analysis

This certificate confirms that the BART™ product listed by name, lot number, and batch number has been subjected to the full range of Quality Control procedures as outlined in "User Quality Control Manual in support of the BART Biodetection Technologies" published in 2004 by Droycon Bioconcepts Inc.

BART™ Type: IRB-BART

Batch #:

Release date*:

Lot#:

Shipment date:

Expiry date:

* Approval for release includes the following criteria: 1. confirmation of sterility for the vials and caps, 2. approval of the medium as being appropriately formed and acceptable, 3. is sterile, and 4. responds in a typical way to inoculation and incubation using selected defined microbial cultures. Details of these criteria are included in our Web Site.

This certificate confirms that the batch of the BART™ biodetectors listed have satisfactorily passed the QC screening procedures and were approved for release on the date given above

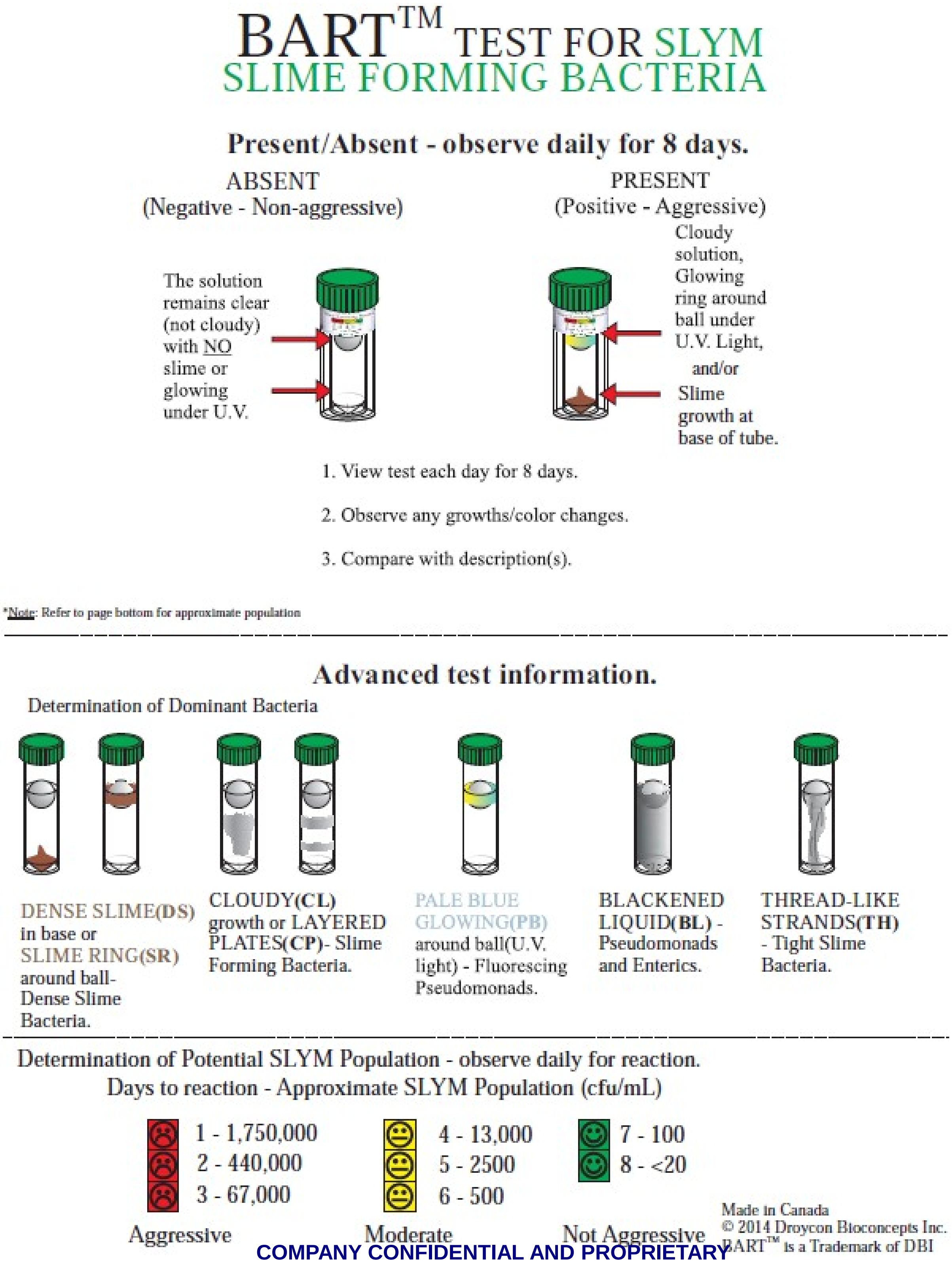
Certificate Number:

This certificate was issued by Droycon Bioconcepts Inc., 315 Dewdney Ave., Regina, SK., Canada, S4N 0E7 as an assurance that the product listed above has passed through the quality control procedures considered essential to the successful use of the testing device.

DBI ISO 9001:2000 Compliant

For more information, visit our web-site at:
http://www.DBI.ca

LIT8436 Rev.1



BART™ TEST FOR SLYM
SLIME FORMING BACTERIA

Present/Absent - observe daily for 8 days.

ABSENT
(Negative - Non-aggressive)

The solution remains clear (not cloudy) with NO slime or glowing under U.V.

PRESENT
(Positive - Aggressive)

Cloudy solution, Glowing ring around ball under U.V. Light,

and/or

Slime growth at base of tube.

1. View test each day for 8 days.
2. Observe any growths/color changes.
3. Compare with description(s).

*Note: Refer to page bottom for approximate population

Advanced test information.

Determination of Dominant Bacteria

DENSE SLIME(**DS**) in base or SLIME RING(**SR**) around ball- Dense Slime Bacteria.

CLOUDY(**CL**) growth or LAYERED PLATES(**CP**)- Slime Forming Bacteria.

PALE BLUE GLOWING(**PB**) around ball(U.V. light) - Fluorescing Pseudomonads.

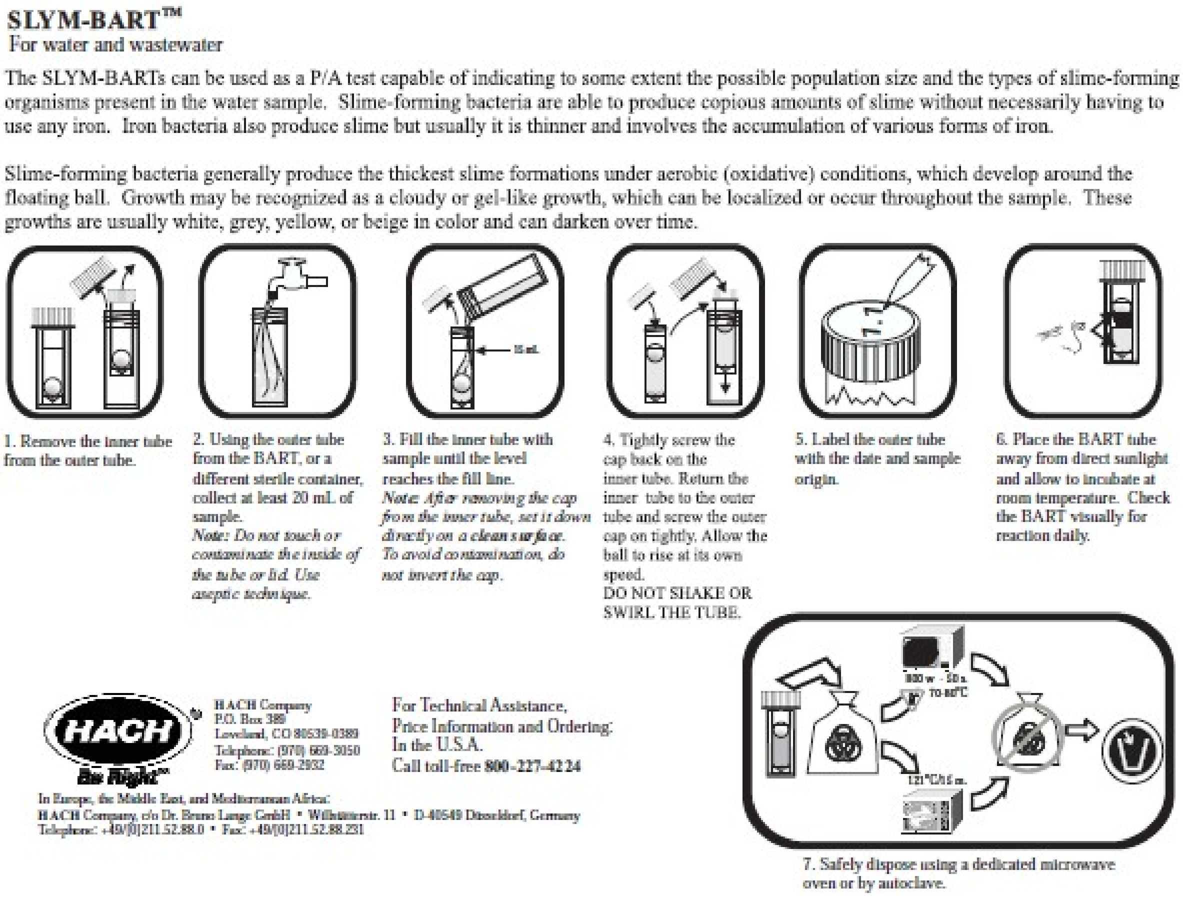
BLACKENED LIQUID(**BL**) - Pseudomonads and Enterics.

THREAD-LIKE STRANDS(**TH**) - Tight Slime Bacteria.

Determination of Potential SLYM Population - observe daily for reaction.
Days to reaction - Approximate SLYM Population (cfu/mL)

Aggressive	Moderate	Not Aggressive
☹ 1 - 1,750,000	😐 4 - 13,000	☺ 7 - 100
☹ 2 - 440,000	😐 5 - 2500	☺ 8 - <20
☹ 3 - 67,000	😐 6 - 500	

Made in Canada



SLYM-BART™
For water and wastewater

The SLYM-BARTs can be used as a P/A test capable of indicating to some extent the possible population size and the types of slime-forming organisms present in the water sample. Slime-forming bacteria are able to produce copious amounts of slime without necessarily having to use any iron. Iron bacteria also produce slime but usually it is thinner and involves the accumulation of various forms of iron.

Slime-forming bacteria generally produce the thickest slime formations under aerobic (oxidative) conditions, which develop around the floating ball. Growth may be recognized as a cloudy or gel-like growth, which can be localized or occur throughout the sample. These growths are usually white, grey, yellow, or beige in color and can darken over time.

1. Remove the inner tube from the outer tube.

2. Using the outer tube from the BART, or a different sterile container, collect at least 20 mL of sample.
Note: Do not touch or contaminate the inside of the tube or lid. Use aseptic technique.

3. Fill the inner tube with sample until the level reaches the fill line.
Note: After removing the cap from the inner tube, set it down directly on a clean surface. To avoid contamination, do not invert the cap.

4. Tightly screw the cap back on the inner tube. Return the inner tube to the outer tube and screw the outer cap on tightly. Allow the ball to rise at its own speed.
DO NOT SHAKE OR SWIRL THE TUBE.

5. Label the outer tube with the date and sample origin.

6. Place the BART tube away from direct sunlight and allow to incubate at room temperature. Check the BART visually for reaction daily.

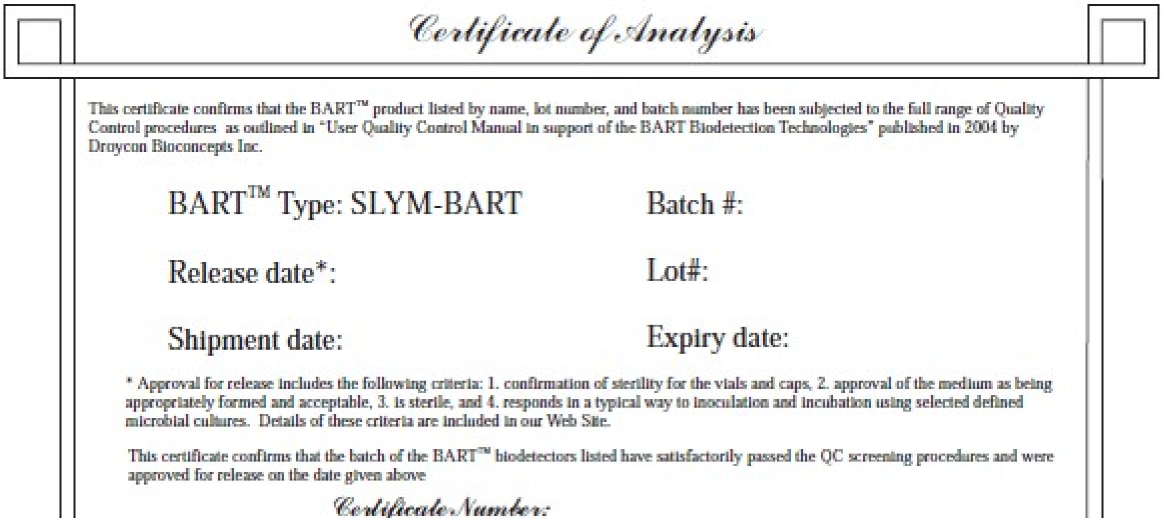
7. Safely dispose using a dedicated microwave oven or by autoclave.

HACH Be Right™

HACH Company
P.O. Box 389
Loveland, CO 80539-0389
Telephone: (970) 669-3050
Fax: (970) 669-2932

For Technical Assistance,
Price Information and Ordering:
In the U.S.A.
Call toll-free **800-227-4224**

In Europe, the Middle East, and Mediterranean Africa:
HACH Company, c/o Dr. Bruno Lange GmbH • Willstätterstr. 11 • D-40549 Düsseldorf, Germany
Telephone: +49/[0]211.52.88.0 • Fax: +49/[0]211.52.88.231



Certificate of Analysis

This certificate confirms that the BART™ product listed by name, lot number, and batch number has been subjected to the full range of Quality Control procedures as outlined in "User Quality Control Manual in support of the BART Biodetection Technologies" published in 2004 by Droycon Bioconcepts Inc.

BART™ Type: SLYM-BART

Batch #:

Release date*:

Lot#:

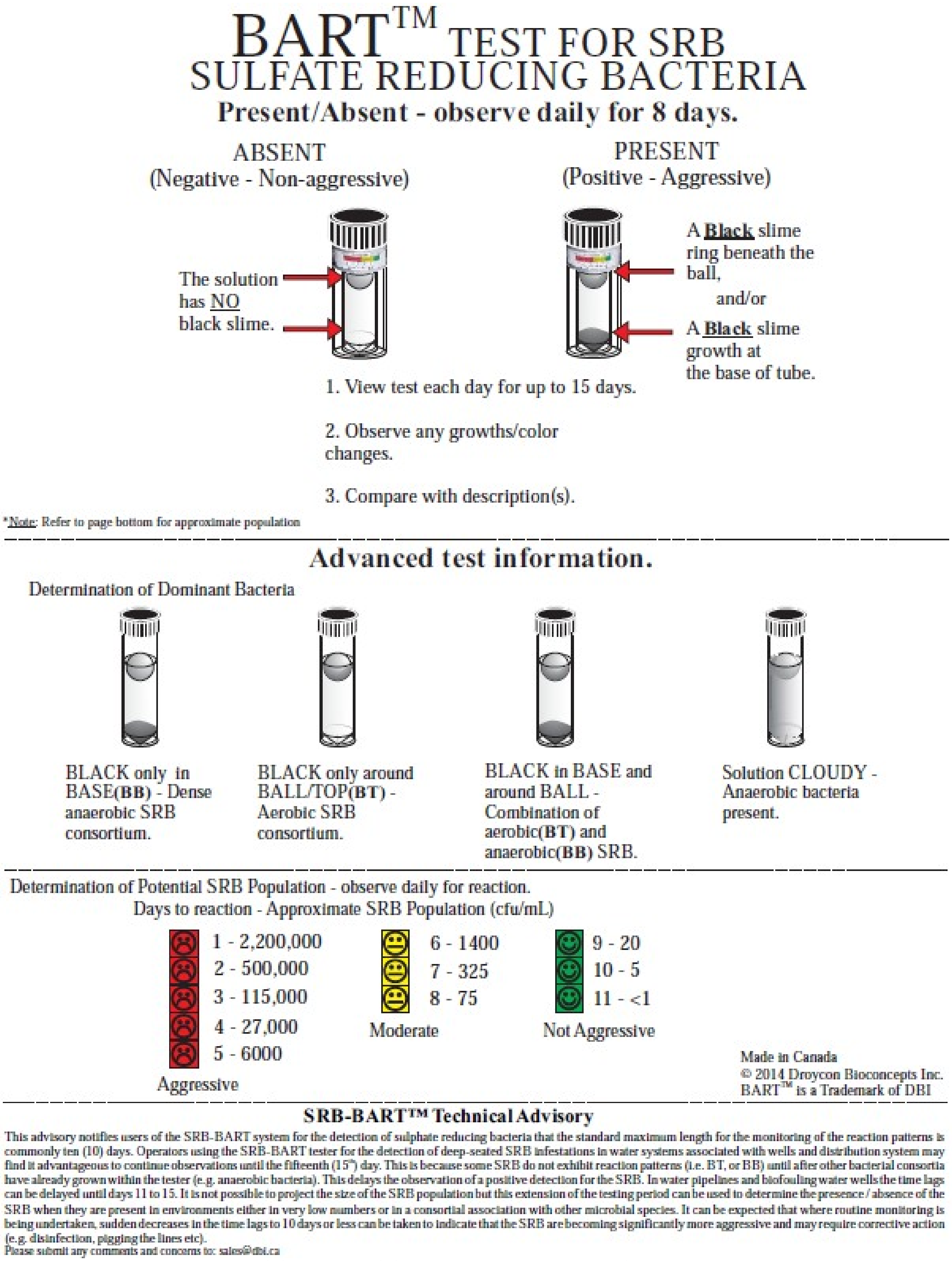
Shipment date:

Expiry date:

* Approval for release includes the following criteria: 1. confirmation of sterility for the vials and caps, 2. approval of the medium as being appropriately formed and acceptable, 3. is sterile, and 4. responds in a typical way to inoculation and incubation using selected defined microbial cultures. Details of these criteria are included in our Web Site.

This certificate confirms that the batch of the BART™ biodetectors listed have satisfactorily passed the QC screening procedures and were approved for release on the date given above

Certificate Number:



BART™ TEST FOR SRB SULFATE REDUCING BACTERIA

Present/Absent - observe daily for 8 days.

ABSENT
(Negative - Non-aggressive)

The solution has NO black slime.

PRESENT
(Positive - Aggressive)

A **Black** slime ring beneath the ball,

and/or

A **Black** slime growth at the base of tube.

1. View test each day for up to 15 days.
2. Observe any growths/color changes.
3. Compare with description(s).

*Note: Refer to page bottom for approximate population

Advanced test information.

Determination of Dominant Bacteria

BLACK only in BASE(**BB**) - Dense anaerobic SRB consortium.

BLACK only around BALL/TOP(**BT**) - Aerobic SRB consortium.

BLACK in BASE and around BALL - Combination of aerobic(**BT**) and anaerobic(**BB**) SRB.

Solution CLOUDY - Anaerobic bacteria present.

Determination of Potential SRB Population - observe daily for reaction.
Days to reaction - Approximate SRB Population (cfu/mL)

☹ 1 - 2,200,000
☹ 2 - 500,000
☹ 3 - 115,000
☹ 4 - 27,000
☹ 5 - 6000
Aggressive

😐 6 - 1400
😐 7 - 325
😐 8 - 75
Moderate

☺ 9 - 20
☺ 10 - 5
☺ 11 - <1
Not Aggressive

Made in Canada

SRB-BART™ Technical Advisory

This advisory notifies users of the SRB-BART system for the detection of sulphate reducing bacteria that the standard maximum length for the monitoring of the reaction patterns is commonly ten (10) days. Operators using the SRB-BART tester for the detection of deep-seated SRB infestations in water systems associated with wells and distribution system may find it advantageous to continue observations until the fifteenth (15th) day. This is because some SRB do not exhibit reaction patterns (i.e. BT, or BB) until after other bacterial consortia have already grown within the tester (e.g. anaerobic bacteria). This delays the observation of a positive detection for the SRB. In water pipelines and biofouling water wells the time lags can be delayed until days 11 to 15. It is not possible to project the size of the SRB population but this extension of the testing period can be used to determine the presence / absence of the SRB when they are present in environments either in very low numbers or in a consortial association with other microbial species. It can be expected that where routine monitoring is being undertaken, sudden decreases in the time lags to 10 days or less can be taken to indicate that the SRB are becoming significantly more aggressive and may require corrective action (e.g. disinfection, pigging the lines etc).

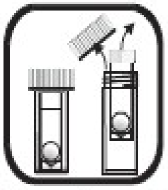
Please submit any comments and concerns to: sales@dbi.ca

SRB-BART™

For water and wastewater

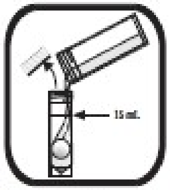
Sulfate-Reducing bacteria are a group of anaerobic bacteria that generate hydrogen sulfide (H_2S). This product can cause a number of significant problems in water. Problems range from "rotten egg" odors to the blackening of equipment, slime formations, and the initiation of corrosive processes. SRB microorganisms are difficult to detect because they are anaerobic and tend to grow deep down within biofilms (slimes) as a part of a microbial community. SRB may not be present in the free-flowing water over the site of the fouling.

If SRB activity is present in the BART, sulfate is reduced to H_2S, which reacts with the diffusing ferrous iron to form black iron sulfide. This sulfide commonly forms either in the base (as black precipitates) and/or around the ball (as an irregular black ring).

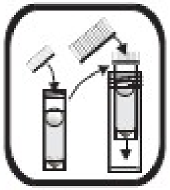


1. Remove the inner tube from the outer tube.

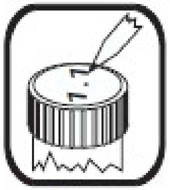
2. Using the outer tube from the BART, or a different sterile container, collect at least 20 mL of sample.
Note: Do not touch or contaminate the inside of the tube or lid. Use aseptic technique.



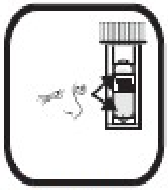
3. Fill the inner tube with sample until the level reaches the fill line.
Note: After removing the cap from the inner tube, set it down directly on a clean surface. To avoid contamination, do not invert the cap.



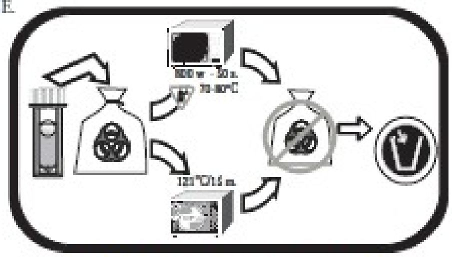
4. Tightly screw the cap back on the inner tube. Return the inner tube to the outer tube and screw the outer cap on tightly. Allow the ball to rise at its own speed.
DO NOT SHAKE OR SWIRL THE TUBE.



5. Label the outer tube with the date and sample origin.



6. Place the BART tube away from direct sunlight and allow to incubate at room temperature. Check the BART visually for reaction daily.



7. Safely dispose using a dedicated microwave oven or by autoclave.

HACH Be Right℠

HACH Company
P.O. Box 389
Loveland, CO 80539-0389
Telephone: (970) 669-3050
Fax: (970) 669-2932

For Technical Assistance,
Price Information and Ordering:
In the U.S.A.
Call toll-free **800-227-42 24**

In Europe, the Middle East, and Mediterranean Africa:
HACH Company, c/o Dr. Bruno Lange GmbH • Willstätterstr. 11 • D-40549 Düsseldorf, Germany
Telephone: +49/[0]211.52.88.0 • Fax: +49/[0]211.52.88.231

Certificate of Analysis

This certificate confirms that the BART™ product listed by name, lot number, and batch number has been subjected to the full range of Quality Control procedures as outlined in "User Quality Control Manual in support of the BART Biodetection Technologies" published in 2004 by Droycon Bioconcepts Inc.

BART™ Type: SRB-BART

Batch #:

Release date*:

Lot#:

Shipment date:

Expiry date:

* Approval for release includes the following criteria: 1. confirmation of sterility for the vials and caps, 2. approval of the medium as being appropriately formed and acceptable, 3. is sterile, and 4. responds in a typical way to inoculation and incubation using selected defined microbial cultures. Details of these criteria are included in our Web Site.

This certificate confirms that the batch of the BART™ biodetectors listed have satisfactorily passed the QC screening procedures and were approved for release on the date given above

Certificate Number:

This certificate was issued by Droycon Bioconcepts Inc., 315 Dewdney Ave., Regina, SK., Canada, S4N 0E7 as an assurance that the product listed above has passed through the quality control procedures considered essential to the successful use of the testing device.

DBI ISO 9001:2000
Compliant

For more information, visit our web-site at:
http://www.DBI.ca

LIT8436 Rev.1

eurofins	Always check on-line for validity. **Particle Size Distribution of Soils and Solids/Grain Size Classification by ASTM D422-63 (reapproved 2007)**	Level: **Work Instruction**
Document number: **T-WC-WI11514**		
Old Reference: **1-P-QM-WI-9014165**		
Version: **11**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **31-JAN-2021**	Document users: **6_EUUSLA_Water Quality_Misc Analysis, 6_EUUSLA_Water Quality_Misc Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control
Table I

Revision Log

Revision 11		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	Reflect current active method	Updated to current LIMS IDs
Cross Reference	Reflect current process	Removed form T_WC_FRM11472 (NLIU)

Revision 11		**Effective Date:** This version
Cross Reference	Higher level documents are not required to be referenced	Removed QA-SOP11880 and QA-SOP11188
Personnel Training and Qualifications	Required wording	Updated to include required wording.

Revision 10		**Effective Date:** 24-Jan 2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Reflect current active method	Added ASTM D422
Cross Reference/throughout document	Reflect current designations in ETQ	Updated to ETQ designation
Reagents and Standards	No longer used	Removed item #2 n-hexane
Procedure B:NOTE;	New requirement	Added that hydrometer will be calibrated yearly by certified vendor.
Procedure F	No longer used	Removed oil extraction section F from SOP
Entire document	Reflect current forms being used	Added form T-WC-FRM11472 (form to use when only analysis 7103)

Reference

1. ASTM D422-63 (reapproved 2007), Standard Test Method for Particle-Size Analysis of Soil

2. ASTM D422, Standard Test Method for Particle-Size Analysis of Soil

3. ASTM D421 - 85, *Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants*.

4. ASTM E868 - 82, “*Conducting Performance Tests on Mechanical Conveying Equipment Used in Resource Recovery Systems*,” Sec 9.9, “*Measuring Bulk Density of Material*.”

5. Method 160.3, *EPA Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020.

6. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
S-SS-WI10697	Moisture (Gravimetric)

Document	Document Title
T-WC-FRM11473	D422 Particle Size Distribution / Grain Size Classification
Q-EQA-WI6815	ETM System Probe Calibration
T-WC-WI9901	Equipment Muffle Furnaces and Ovens

Scope

This procedure is applicable to the determination of the distribution of particle sizes in soils and solids. Particle sizes in the range 75 to 0.075 mm are determined by sieving. Distribution of particle sizes smaller than 0.075 mm is determined by a sedimentation process using a hydrometer.

Basic Principles

The process determines the particle sizes and distribution in a soil/solid sample. The sample is dried, ground, and sieved using sieves of different sizes. Particle sizes in the range 75 to 0.075 mm are determined by sieving. Distribution of particle sizes smaller than 0.075 mm is determined by a sedimentation process using a hydrometer.

Interferences

When high levels of organic material are present in the soil (material with a specific gravity less than one), the hydrometer readings are subject to error. After air-drying the sample, remove as much of the organic material (leaves, roots, sticks, etc.) as possible.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst’s training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Analysts are considered proficient when they have successfully demonstrated competency under supervision of a supervisor or other trained analyst.

Sample Collection, Preservation, and Handling

There is no holding time for this analysis. Samples are stored at 0 to 6^{o}C; not frozen.

1. Place the sample as received in a glass baking dish. Allow to air dry at room temperature for approximately 24 hours or until thoroughly dried. Use enough sample so that the material needed to pass through the No. 8 sieve is approximately 100 g or more.

2. Break and grind the dried sample using the rubber-covered pestle. Continue to do so until the sample is reduced to a fine material. **Do not break rocks that are part of the sample matrix as this is likely to affect the sample sedimentation process.**

All information needs to be recorded on Form *T-WC-FRM11473* as soon as the sample preparation is started.

Apparatus and Equipment

1. Analytical balance, pan balance, or equivalent
2. Mechanically operated sieving device
3. Mechanically operated stirring device
4. Hydrometer - graduated to read in specific gravity of the suspension
5. Sedimentation cylinder – 1000-mL
6. Thermometer - accurate to 0.5°C
7. Sieves - Numbers 4, 6, 8, 16, 30, 50, 100, 200, 0.75", 1.5", and 3"
8. Sieve pan
9. Rubber covered pestle, or equivalent
10. Beakers – 150-mL and 250-mL
11. Specimen collection cups (120-mL), or equivalent
12. Glass baking dishes, or equivalent

13. Glass stir rod, or equivalent
14. Oven maintained at 110° ± 5°C; adjust as needed to stay in this range
15. Whatman Grade 3 filter paper - 6 micron pore size
16. Buchner funnel

Reagents and Standards

Alternate weights may be used as long as the final concentrations remain the same.

1. Sodium hexametaphosphate solution
   a. Using a 1000-mL volumetric flask, dissolve 40 g of sodium hexametaphosphate in reagent water.
   b. Dilute to final volume once dissolved
   c. Store at room temperature in a glass or plastic container
   d. Solution expires one month from date of preparation.

Calibration

Balance calibration must be checked each day before use.

Procedure

A. The sieved analysis of the portion retained on the No. 8 sieve.

Check to be sure that the balance has been calibrated each day before use.

1. Tare the sieving pan, No. 8, No. 6, No. 4, 0.75", 1.5", and the 3" sieved consecutively (to 0.01 g) using a pan balance.
2. Using the prepared sample, place the sample in the tared sieves and begin the sieving operation using the mechanical sieving device. Agitate for approximately one minute. Continue sieving until no more than 1% of the residue on each sieve passes that sieve during 1 minute of sieving.

NOTE: If sample particle size is too great to pass through the sieve, continue to break and grind as needed. Do not break stones or rocks that are part of the sample matrix.

3. Determine the mass retained (Mr) on the 3" sieve. Weigh on the balance and record the combined sieve and sample weight. Continue sieving with the remaining sieves recording all determined sample masses on the sieves.

To calculate the mass retained:

$$M_r = \text{Tare + sample weight value - the tare weight value}$$

4. Retain the sample collected in the sieving pan after weighing.

   a. Weigh approximately 50 g of sample into a 250-mL beaker.

   b. Place approximately 30 g or more of the remaining sample into a specimen cup, for use when performing the moisture and bulk density analyses.

B. Hydrometer analysis

NOTE: The hydrometer used in the process will be calibrated on an annually basis by a certified vendor.
The hydrometer must be visually inspected for any cracks or breaks before each use.

1. Using the sample in the 250-mL beaker add 125 mL of sodium hexametaphosphate solution. Stir the soil-water slurry until it is thoroughly moistened. Cover and label beaker with sample number. Allow this slurry to soak for at least 16 hours, but not to exceed 24 hours.

2. At the end of the soaking period, transfer the soil-water slurry into a dispersion cup using room temperature reagent water. Be sure to rinse all residue from the beaker into the dispersion cup.

NOTE: Extra care needs to be taken when rinsing the sample into another container or when using any apparatus. **All particles need to be rinsed into the new container or a loss in sample weight is possible to occur.**

3. Stir for one minute using the stirring apparatus. **Rinse stirring apparatus well into dispersion cup to prevent loss of sample.**

4. Transfer the soil-water slurry into a glass sedimentation cylinder, rinsing well. Add room temperature reagent water until the total volume is 1000 mL.

5. Cover the open end of the cylinder with parafilm, then placing the palm of the hand over the end, invert the cylinder and agitate for a period of one minute. Place the cylinder in a location where it will not be disturbed or moved during the sedimentation readings (for 24 hours).

6. Remove the parafilm and immediately begin the hydrometer readings, recording the temperature reading after each suspension. Record the readings at the following intervals (measured from the beginning of sedimentation): 2, 5,15, 30, 60, 250, and 1440 minutes. Insert the hydrometer about 20 to 25 seconds before the reading is due to the approximate depth. Clean the hydrometer between readings by placing it into reagent water and twisting with a spinning motion.

NOTE: The basic temperature to be maintained for the hydrometer test is 68°F (20°C). Small variations of temperature do not introduce differences that are of practical significance.

C. Final sieving analysis of the portion of sample passing the No. 8 sieve.

1. After taking the final hydrometer reading, pour the suspension through a No. 200 sieve and rinse with reagent water until the wash water is clear.

2. Transfer the remaining sample from the sieve into a 150-mL beaker, rinsing well with room temperature reagent water and dry in an oven at 110° ± 5°C for at the least 12 hours or longer.

3. After the sample has dried and no liquid is remaining. Remove from the oven and allow to cool. When the sample has reached room temperature use a glass stir rod to scrape the beaker and crush the harden residue into fine particles.

4. Tare the collection pan, the No. 200, No. 100, No. 50, No. 30, and No. 6 sieves consecutively (to 0.01 g) using a pan balance.

5. Place the oven-dried sample into the sieves and cover. The sieves need to be covered at this point of agitation due to the fine and lightweight particle sizes. Agitate for approximately 5 minutes to allow the fine particles to pass within the sieves.

6. Determine the mass retained (Mr) on each sieve by weighing and recording the combined weight of the sieve and the sample.

D. Moisture analysis

Using approximately 5 g of sample from the specimen collection cup (Procedure: Section A.4.b.) Perform a moisture analysis according to *S-SS-WI10697*. Record all information on form *T-WC-FRM11473*.

E. Bulk density analysis

1. Weigh out approximately 15 g of sample from the specimen collection cup (Procedure A.4.b.).
2. Tare a 100 mL volumetric flask and record data on form *T-WC-FRM11473*.
3. Add sample to tared flask.
4. Add approximately 50 mL reagent water and agitate flask until soil is thoroughly mixed.
5. Remove air bubbles by inserting a vacuum tube inside the neck of flask being careful not touch the contents of the flask.
6. Once air bubbles are removed, bring mixture to a final volume of 100 mL using reagent water.
7. Reweigh the flask and its contents and record the reading on the data sheet (form *T-WC-FRM11473*)

F. Grain Size Classification

To report the % Gravel, % Sand, % Silt, and % Clay fractions electronically, a separate grain size classification scan with the % passing at each type as a separate piece will be determined and recorded on form *T-WC-FRM11473*. See below for further information on % classification. For sediments that do not have sufficient sample volume to perform the hydrometer portion of the test, the sum of the % Silt and Clay can be reported as one aggregate value.

1. % Gravel – determined by the amount of particles passing 3-in. and retained on No. 4 sieve (size 4.75-mm).
2. % Sand - determined by the amount of particles passing No. 4 sieve (size 4.75-mm) and retained on No. 200 sieve (size 0.075-mm).
3. % Silt – determined by the amount of particles passing 0.074 to 0.005-mm.
4. % Clay – determined by the amount of particles smaller than 0.005-mm.

Calculations

A spreadsheet using Excel, or an equivalent, has been prepared to facilitate the calculation of data, the plotting of the graph, and the interpolation of the hydrometer results from the graph. It is possible for data to be reported directly from the spreadsheets; however, the plotted hydrometer data may be more accurate than the computer interpolations. Refer to this spreadsheet for the appropriate tables to hand calculate data.

Use the mass retained (Mr) values recorder for each sieve as Mr8, Mr6, Mr4, etc. to determine the percent passing each sieve.

To calculate the mass retained:

Mr = Tare + sample weight value - the tare weight value

A. Sieve analysis values for the portion coarser than the No. 8 sieve

1. Mt = Mc + Mf

   Where:

   Mt = total mass

   Mc = mass of the coarse material

   Mf = mass of the fine material

2. P8 = Mf/Mt x 100

Where:

P8 = percentage passing No. 8 sieve

3.

$$P6 = \frac{Mf + Mr8}{Mt} \times 100$$

Where:

P6 = percentage passing No. 6 sieve

Mr8 = mass retained on No. 8 sieve

4.

$$P4 = \frac{Mf + Mr8 + Mr6}{Mt} \times 100$$

Where:

Mr6 = mass retained on the No. 6 sieve

P4 = percentage passing No. 4 sieve

5. Continue calculations as above for the 0.75", 1.5", and 3" sieves.

B. Percentages of soil in suspension

1. Hf = Os/As

Hygroscopic moisture correction factor

Where:

Hf = hygroscopic moisture correction factor

As = air dry mass of the soil

Os = oven dry mass of the soil = (oven dry weight minus the tare weight)

2. Percentages of soil in suspension

a. Calculate the oven dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass of the soil used in the hydrometer analysis by the moisture correction factor.

b.

$$W = \frac{oven\ dry\ mass\ in\ hydrometer}{P8} \times 100$$

Where:

W = oven dry mass of soil in a total test sample represented by the mass of the soil dispersed, in grams

P8 = percentage passing the No. 8 sieve (from calculation A.2.)

c. P = [(100,000/W) x G/(G-G1)] x (R-G1)

Where:

G = specific gravity of the soil

G1 = specific gravity of the liquid in which the soil was suspended (use 1)

W = oven dry mass of soil in a total test sample represented by the mass of the soil dispersed, in grams

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension

R = Hydrometer reading with composite correction applied

d.

$$D = K\sqrt{L/T}$$

Where:

D = diameter of the particle (in mm)

K = constant using the temperature of the suspension and the specific gravity of the soil particles (see the Excel spreadsheet or equivalent for the Analysis #7103, for the specific “K” values to use)

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured (the effective depth from Table I)

T = interval of time from the beginning of sedimentation to the taking of the reading in minutes

C. Sieve analysis values of the portion finer than the No. 8 sieve

1. Calculate the percentage of particles retained on the No. 8 sieve as follows:

$$Pr8 = 100 - P8$$

Where:

Pr8 = percentage of particles retained on the No. 8 sieve

P8 = percentage of particles passing the No.8 sieve (from calculation A.2.)

2. Calculate the mass retained on the No. 8 sieve represented by the sample volume used in the hydrometer analysis as follows:

$$Mf8 = \frac{Pr8 \times W}{100}$$

Where:

Mf8 = mass retained on the No. 8 sieve represented by the sample volume used in the hydrometer analysis

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

3. Calculate the mass passing the No. 200 sieve as follows:

$$Mp200 = W - (Mr200 + Mr100 + Mr50 + Mr30 + Mr16 + Mf8)$$

Where:

Mp200 = mass passing the No. 200 sieve

Mr200, 100, 50, etc. = mass retained on the sieves used for the portion finer than the No. 8 sieve

Mf8 = mass retained on the No. 8 sieve represented by the mass of the soil used in the hydrometer analysis

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

4.

$$P200 = \frac{Mp200}{W} \times 100$$

Where:

P200 = percentage passing the No. 200 sieve

Mp200 = mass passing the No. 200 sieve (use calculation from C.3.)

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

5.

$$P100 = \frac{Mp200 + Mr200}{W} \times 100$$

Where:

P100 = percentage passing the No. 100 sieve

Mr200 = mass retained on the No. 200 sieve

Mp200 = mass passing the No. 200 sieve (use calculation from C.3.)

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

6.

$$P50 = \frac{Mp200 + Mr200 + Mr100}{W} \times 100$$

Where:

P50 = percentage passing the No. 50 sieve

Mr100 = mass retained on the No. 100 sieve

Mp200 = mass passing the No. 200 sieve

Mr200 = mass retained on the No. 200 sieve

W = oven dry mass of the soil in a total test sample represented by the mass of the soil dispersed (as calculated in B.2.b.)

7. Continue calculations as above for the No. 30 and No. 16 sieves.

D. Prepare a graph of the test results on a logarithmic scale, plotting the diameter of the particles as the abscissa and the percentages smaller than the corresponding diameters as the ordinate. Report (from the graph or directly from the tabulated data) the percentage of particles passing the 3", 1.5", 0.75", No. 4, No. 6, No. 8, No. 16, No. 30, No. 50, No. 100, and No. 200 sieves. Report the hydrometer results (from the graph) as the percentage passing 0.050 mm, 0.020 mm, 0.005 mm, 0.002 mm, and 0.001 mm.

Statistical Information/Method Performance

Not applicable to this procedure.

Quality Assurance/Quality Control

When possible, perform duplicate analyses for particle size on a routine basis.

Batch size is limited to 20 samples or less.

Table I

Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specific Sizes[A]

Hydrometer 151H

Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3
1.001	16.0
1.002	15.8
1.003	15.5
1.004	15.2
1.005	15.0
1.006	14.7
1.007	14.4
1.008	14.2
1.009	13.9
1.010	13.7
1.011	13.4
1.012	13.1
1.013	12.9
1.014	12.6
1.015	12.3
1.016	12.1
1.017	11.8
1.018	11.5
1.019	11.3
1.020	11.0
1.021	10.7
1.022	10.5
1.023	10.2
1.024	10.0
1.025	9.7

Hydrometer 151H

Actual Hydrometer Reading	Effective Depth, L, cm
1.026	9.4
1.027	9.2
1.028	8.9
1.029	8.6
1.030	8.4
1.031	8.1
1.032	7.8
1.033	7.6
1.034	7.3
1.035	7.0
1.036	6.8
1.037	6.5
1.038	6.2

[A] Values of effective depth are calculated from the equation:

$$L = L1 + \tfrac{1}{2} \times \left[L2 - \left(\frac{V_B}{A} \right) \right]$$

Where:

L = effective depth, cm

L1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm

L_2 = overall length of the hydrometer bulb, cm

V_B = volume of hydrometer bulb, cm^3, and

A = cross-sectional area of sedimentation cylinder, cm^2

Values used in calculating the values in Table I are as follows:

For hydrometer 151H:

L_2= 14.0 cm

V_b = 67.0 cm^3

A = 27.8 cm^2

L1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

Q-EQA-WI6815 ETM System Probe Calibration
QA-SOP11188 Reagents and Standards
QA-SOP11880 Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment
S-SS-WI10697 % Moisture Calculation and % Solids Calculation (Gravimetric)
T-WC-FRM11472 #7103 Particle Size Distribution
T-WC-FRM11473 Particle Size Distribution/ Grain Size Classification
T-WC-WI9901 Equipment Muffle Furnaces and Ovens

End of document

Version history

Version	Approval	Revision information	
9	18.AUG.2014		
10	17.JAN.2019		
11	29.JAN.2021		

eurofins

Always check on-line for validity.

pH by EPA 9045C, 9045D and Corrosivity by SW-846 Chap 7 of Solids, Soils, and Solvents using Electrometic Methods

Level:

Work Instruction

Document number:
T-WC-WI11518

Old Reference:
1-P-QM-WI-9011685

Version:
14

Organisation level:
5-Sub-BU

Approved by: **X6TJ**
Effective Date **30-APR-2021**

Document users:
6_EUUSLA_Water Quality_24/48 Hour Analyst, 6_EUUSLA_Water Quality_24/48 Hour Verification

Responsible:
5_EUUSLA_Water Quality_Manager

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

<table>
<tr><td>Revision</td><td>14</td><td>Effective Date:</td><td>This version</td></tr>
<tr><td colspan="2">Section</td><td>Justification</td><td>Changes</td></tr>
<tr><td colspan="2">Revision Log</td><td>Formatting requirement</td><td>Removed revision logs up to the previous version</td></tr>
<tr><td colspan="2">Throughout Document</td><td>Current process</td><td>Update to new LIMS</td></tr>
</table>

Revision 14		**Effective Date:** This version
Cross Reference	Higher level documents are not required to be referenced	Removed *QA-SOP11880*
Procedure 10.	Current process	Added step 10 - to read the 2 and 12 standards when analyzing samples for corrosivity
Procedure	Current Process	Removed previous step 11. Units are reported as S.U.
Quality Assurance/ Quality Control	Clarification Current process	Added acceptance range for CCVs. Removed requirement to analyze the 2 and 12 standards before any samples Removed step to read pH 3 times for corrosivity

Revision 13		**Effective Date:** 08-JUL-2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Procedure 11	Clarification	Added how solid pH is reported (soil pH measured in water at recorded temp at time of analysis)

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 9045C, January 1995.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 9045D, November 2004.

3. Test Methods for Evaluating Solid Wastes, SW-846 Chapter 7.

4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-WC-WI11519	pH Probes and Meters
T-WC-WI10360	Quality Control Data for Wet Chemistry

Scope

This SOP provides the guidelines for analysts performing pH on solid, soil, and solvent samples. This procedure is applicable to solid/soil/solvent samples.

The sensitivity limit for this technique is 0.01 pH units.

Basic Principles

A 1:1 slurry is prepared and the activity of hydrogen ions in the supernatant is measured using a combination pH electrode.

Determination of corrosivity is based on the pH value of the sample.

Reference Modifications

Method 9045C/D has been modified for the analysis of solid, soil, and solvent samples in the following ways:

1. An Automatic Temperature Compensator is used for all samples instead of manually performing calculations to correct measured pH values if the sample and buffer solution temperatures differ by more than 2°C.

2. 25 g of soil to 25 g reagent water is used instead of the 20 g: 20 mL ratio.

3. The samples are tumbled for approximately 30 minutes instead of being stirred with a stir bar for 5 minutes.

These modifications are performed in order to allow for adequate agitation and to provide sufficient supernatant to immerse the pH electrode during analysis.

Interferences

Interferences occur when oily or particulate matter adheres to the electrodes and reduces the response. Gentle wiping or rinsing with reagent water usually corrects this problem. Temperature effects are compensated for by calibrating the pH meter at the temperature of the sample or using a pH meter equipped with temperature compensators. There are no means of controlling temperature effects caused by shifts in ionic equilibria of the sample.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention. Standard safe laboratory procedure must be followed as outlined in the Chemical Hygiene Plan.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst’s training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four pH readings of the 7.00 pH buffer (which is used as the laboratory control standard) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Sample must be collected in an unpreserved container and stored at 0° to 6°C; not frozen, until the time of analysis. There is no published holding time for pH analysis on soil; analysis is performed as soon as possible after sample is received in the laboratory.

Apparatus and Equipment

1. Analytical balance capable of weighing 0.0001 g

2. pH meter equipped with an ATC probe (Automatic Temperature Compensator)

3. Combination electrode or equivalent

4. Stir bar and stir plate

Reagents and Standards

1. 7.00 pH Buffer (ISO 17025 approved vendor) – purchased; see container for shelf life information.

2. Appropriate pH electrode filling solution for electrode, purchased. Store at room temperature. See label for expiration date.

Calibration

Balances must be calibrated each day before use.

Calibrate pH meter as described in *T-WC-WI11519*.

Procedure

1. Make sure the pH meter has been calibrated within the last 24 hours.

2. Weigh 25 ± 0.5 g of sample into a clean specimen cup and add 25 ± 0.5 mL of reagent water (makes a 1:1 slurry).

If the sample absorbs the water, add an additional 25 ± 0.5 mL of reagent water (makes a 1:2 slurry). If a 1:2 slurry does not provide sufficient supernatant to immerse the pH electrode, use less sample and add reagent water in proportion to the weight selected. Enter a comment in LIMS indicating the dilution.

3. Tightly place the screw-cap lid on the sample and mix the slurry in the tumbler for approximately 30 minutes.

4. Remove the sample from the tumbler and allow the sample to settle for about 1 hour.

5. Rinse and shake off any water on the electrodes.

6. Dip the electrodes into the supernatant (aqueous layer) of the sample and allow to equilibrate. If necessary, decant or pipette this layer into another container.

7. Using the calibrated, pH meter note the pH value of the sample after the meter equilibrates, and enter the value in the LIMS.

NOTE: If pH reading is < 4.00 or >10.00, then the pH result will be reported with a qualifying flag.

8. Note the temperature of the sample and record the value in LIMS.

9. Rinse and clean the electrodes before proceeding to the next sample.

10. If corrosivity is to be reported, read the pH 2 and 12 standards and record the pH value.

11. Corrosivity is determined from the sample's pH reading. A "Yes" or "No" response is entered for the corrosivity result, depending on the pH value. A sample is considered corrosive if the pH is <2 or >12, warranting a "Yes" result. A sample with a pH value from 2 to 12 has a corrosivity result of "No".

Calculations

Not applicable.

Statistical Information/Method Performance

Not applicable to this procedure

Quality Assurance/Quality Control

One batch consists of no more than 20 samples.

A Laboratory Control Standard (LCS; 7.00 pH Solution) must be analyzed at the beginning of each batch.

A CCV (7.00 pH Solution) must be analyzed after every ten samples and at the end of the batch. The acceptance range for the CCV is 90 - 110%.

Two matrix duplicates must be analyzed per batch of 20 samples. If 10 or less samples are on a batch then only one matrix duplicate is needed.

When analyzing for Corrosivity, a pH check using buffers 2 and 12 must be analyzed.

If the meter must be re-calibrated during the analysis, a LCS must be analyzed after the calibration is performed.

See LIMS for current quality control acceptance windows.

Refer to *T-WC-WI10360* if any of the QC samples do not meet required specifications.

QA-SOP11880 Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment
T-WC-WI10360 Quality Control Data for Wet Chemistry
T-WC-WI11519 pH Probes and Meters

End of document

Version history

Version	Approval	Revision information	
12	27.JUL.2018		
13	27.JUN.2019		
14	22.APR.2021		

eurofins	Always check on-line for validity. **Total Dissolved Solids (TDS)(Gravimetric) by SM 2540 C-2015, SM 2540 C-2011, SM 2540 C-1997 or EPA 160.1 in Waters and Wastewaters**	Level: **Work Instruction**
Document number: **T-WC-WI11598**		
Old Reference: **1-P-QM-WI-9011630**		
Version: **18**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **31-OCT-2021**	Document users: **6_EUUSLA_Water Quality_Gravimetric Analysis, 6_EUUSLA_Water Quality_Gravimetric Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
Definitions
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 18	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version

Revision: 18	**Effective Date:**	This version
Section	**Justification**	**Changes**
Title	MUR update	Added SM2540 C-2015
Reference	MUR update	Added SM2540 C-2015
Basic Principles	Clarification	Defined analysis start time as the first time samples are placed in the oven.
Procedure 11	Current process	Clarified that the first time in the oven is the start time for the analysis.

Revision: 17	**Effective Date:**	30-APR-2021
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	New LIMS	Update Old LIMS Scan# to new LIMS codes
Throughout Document	No longer performed	Removed analysis 14674 instructions
Apparatus and Equipment 11.	New LIMS Current process	Removed item#11 (LLENS) Added conductivity meter
Procedure 3.	Current process	Added conductivity pre-screen
Procedure 11, 14	Current process	Added step to record date/time/temp. Renumbered accordingly.
Procedure 20.	New LIMS	changed last sentence from "lowest weight" to "final passing weight"
Quality Assurance/ Quality Control	Correction	Changed 'equal' to 'less than' for the method blank

Reference

1. Standard Methods for the Examination of Water and Wastewater, Method 2540 C-2015
2. Standard Methods for the Examination of Water and Wastewater, Method 2540 C-2011
3. Standard Methods for the Examination of Water and Wastewater, Method 2540 C-1997
4. Method 160.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, 1971.
5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title

Document	Document Title
T-WC-WI10359	Instructions for Collecting Data on the LLENS System
T-WC-WI10360	Quality Control Data for Wet Chemistry
T-WC-WI10350	Maintenance of Desiccators
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

This method is applicable for the gravimetric determination of total dissolved solids (TDS) in potable, surface, and saline waters, as well as domestic and industrial wastewaters in a range up to 20,000 mg/L.

The limit of quantitation (LOQ) for this analysis varies with the sample volume.

Refer to *QA-SOP11886* for the current maintained list of state-specific MCLs for TDS.

Basic Principles

A well-mixed sample is filtered through a glass fiber filter. The filtrate is placed in a tared vessel and evaporated to dryness. The vessel is then dried to constant weight in an oven at 180° ± 2°C. The increase in vessel weight represents the TDS.

The start time for the analysis is the time the samples are initially placed in the oven to dry.

Method 160.1_Calcd and 2540C_Calcd require repeat drying cycles of at least one hour followed by weight determinations until the weight change is less than 4% of the previous weight or less than 0.0005g, whichever is less.

Methods 160.1_SingleDry and 2540C_SingleDry record only one weight after a 16 hour drying cycle.

The appropriate analysis scan is typically determined by the sample state of origin.

The pore size is 2 µm or smaller.

Reference Modifications

The differences between SM 2540 C and EPA 160.1 are that SM 2540 C has a ten-minute filtration time limit and samples that do not maintain a homogeneous state after shaking are magnetically stirred while the aliquot is taken.

Definitions

Dissolved solids - the portion of solids that passes through a filter of 2 um or smaller nominal pore size under specified conditions.

Interferences

Highly mineralized samples with a significant concentration of calcium, magnesium, chloride, and/or sulfate may require prolonged drying.

Samples with a high concentration of bicarbonate may also require prolonged drying to convert the bicarbonate to carbonate.

Waters with excessive residue may form a water entrapping crust, therefore residue should be limited to no more than 0.2 g.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples for TDS must be unpreserved, collected in glass or plastic containers and refrigerated at 0° to 6°C, not frozen.

Analyze within 7 days of collection to minimize microbiological decomposition of the solids.

Apparatus and Equipment

1. Glass fiber filters - 2 µm or smaller nominal pore size
2. 0.45 µm filters - client specific
3. Filtration apparatus:
   a. StableWeigh Filling Station, or
   b. Suction flask (at least 500-mL capacity), with reservoir and filter support
4. Weighing vessels
   a. StableWeigh Total Dissolved Solids Disposable Vessels - pre-weighed to 0.1 mg, or
   b. 250-mL glass beakers
5. Hot plates or steam baths
6. Desiccators. Refer to *T-WC-WI10350*

7. Oven maintained at 180° ± 2°C. Adjust as needed to stay in this range.
8. Analytical balance
9. Weighing bracket for StableWeigh vessels
10. Modular rack for StableWeigh vessels
11. Conductivity meter

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same.

1. TDS/TS stock standard (4000 mg/L)
    a. Dry Potassium Chloride (KCl) for at least 1 hour in oven at 104° ± 1°C.
    b. Desiccate approximately 1 hour.
    c. Dissolve 4.0 ± 0.05 g KCl in reagent water and dilute to 1000 mL in volumetric flask.
    d. Store at 0° to 6°C, not frozen. Stable 6 months.
    e. Use as prepared for spiking solution.
2. TDS/TS working standard (200 mg/L)
    a. Pipette 50 mL of TDS/TS stock standard into a volumetric flask
    b. Dilute to 1000 mL with reagent water.
    c. Store at 0° to 6°C, not frozen.
    d. Stable 6 months from preparation date of TDS/TS stock standard.
    e. Use as prepared for Laboratory Control Sample (LCS).

Calibration

A balance calibration check and leveling check must be performed each day before use.

Procedure

1. Batch samples for analysis in the LIMS.
2. Preparation of beakers to obtain tare weights. If using the StableWeigh system, proceed to Step 3.
    a. Place clean beakers in oven for at least 1 hour at 180° ± 2°C.

b. Cool the beakers in a desiccator to balance temperature.

(1). Check desiccant to be sure that the indicator crystals are blue and not pink.

c. Check to make sure the balance has been calibrated and is level each day before use.

d. Use tongs to place beaker on a zeroed balance.

e. Record beaker designation and tare weight.

f. Store beakers in a clean, dry environment until needed.

3. Conductivity Pre-screen

a. Shake sample well.

b. Pour a small amount of sample into a small beaker and read the conductivity.

c. Record the conductivity reading in the batch worksheet. Use this reading to determine the volume of sample to use for the analysis.

4. Assemble filtration apparatus.

For beakers:

a. Place a glass fiber filter on the filter holder and assemble the filtration apparatus.

b. Apply vacuum and wash with at least three 20-mL portions of reagent water.

c. Continue suction to remove all traces of water.

d. Discard washings and rinse suction flask with reagent water.

e. Reassemble filtration apparatus and apply vacuum.

f. For the StableWeigh system: the filling station holds the vessel and replaces the filter flask.

5. Preparation of QC samples

a. Analyze a blank using 200 mL of reagent water.

b. Analyze a laboratory control sample (LCS) using 100 mL of the 200 mg/L TDS/TS working solution.

c. Prepare a duplicate sample by analyzing two equal aliquots of the sample.

d. Prepare a matrix spike (MS) by adding 10 mL of spiking solution (4000 mg/L TDS/TS stock solution) to another equal aliquot of a sample. If the sample aliquot is 10 mL then 20 mL of spiking solution must be used.

6. Analysis of samples

a. Shake sample vigorously.

b. **If the sample does not maintain a homogeneous state until the aliquot is taken, then magnetically stir the sample while pipetting the appropriate aliquot onto the filter.**

c. Using a Class A graduated cylinder or wide-mouth pipette, measure 200 mL of sample or a smaller portion if the sample matrix dictates. An appropriate aliquot should be sufficient to contain between 0.0025 and 0.2 g total dissolved solids.

d. Rapidly transfer the aliquot to the filtration apparatus.

e. Still under vacuum, filter the sample.

7. Rinsing

a. Wash the filter with three 10-mL portions of reagent water (rinsing inside of the graduated cylinder and funnel).

b. If complete filtration takes more than 10 minutes, decrease the sample volume or increase the filter diameter (Practical filter diameters are 2.2 to 12.5 cm).

c. Allow complete drainage between washings.

d. Continue suction at least 3 minutes after filtration is complete.

NOTE: This step is important to ensure that dissolved solids are rinsed through, and not left behind in the filter.

8. Filtration Completion

a. If using the StableWeigh system, the sample filters directly into the weighing vessel.

(1). Remove vessel from the filling station and transfer to the modular rack.

(2). The modular rack will hold vessels through evaporation and drying.

b. If using beakers for the analysis, transfer the total filtrate with washings to a tared beaker.

(1). Rinse suction flask with two small portions of reagent water

(2). Transfer each rinse to the beaker.

9. Record sample aliquot volume and vessel designation, along with other requested information, on the batch worksheet.

10. Evaporate the sample to dryness on a hot plate or steam bath.

11. Record the date, time and temperature in the LIMS each time the samples are placed in or removed from the oven. The first time the samples are placed in the oven is the documented start time for the analysis.

12. When evaporation is complete, place the vessel or rack of vessels containing the sample residue in an oven.

13. Sample drying times

   a. Samples analyzed under 160.1_Calcd and 2540C_Calcd must dry at least 1 hour at 180° ± 2°C.

   b. Samples analyzed under 160.1_SingleDry and 2540C_SingleDry must dry at least 16 hours at 180° ± 2°C.

14. Remove the vessels containing dried residue from the oven. Use tongs to remove individual beakers. Record date, time and temperature.

15. Cool the vessels containing dried residue in a desiccator to balance temperature. Check desiccant to ensure that the indicator crystals are blue and not pink.

16. Again, check to make sure that the balance has been calibrated and leveled before use.

17. Using tongs, place the vessel containing dried residue on a zeroed balance. Use the weighing bracket to keep the StableWeigh vessel upright.

18. Record the weight. Samples for 160.1_SingleDry and 2540C_SingleDry require only one weight after a 16-hour drying cycle.

19. Return vessels for 160.1_Calcd and 2540C_Calcd to the oven and dry at least 1 hour at 180° ± 2°C. Remove vessels containing dried residue from the oven. Use tongs to remove beakers. Cool vessels in a desiccator to balance temperature.

20. Using tongs, place vessel containing dried residue on a zeroed balance. Record the weight. Repeat this drying, desiccating, and reweighing cycle until weight change is less than 4% of the previous weight, or less than or equal to 0.0005 g, whichever is less. The final passing weight is used for the calculation.

21. If an inappropriate aliquot was selected, repeat process using more or less sample volume. Minimum volume should be at least 10 mL to ensure analysis of a representative sample. Maximum volume is 200 mL.

Calculations

$$mg\ total\ dissolved\ solids/L = \frac{(A - B) \times 1000 \times 1000}{Sample\ Volume\ (mL)}$$

Where:

A = Weight of residue and beaker or dish (in grams) after drying

B = Tare weight of empty beaker or dish (in grams)

NOTE: Occasionally, weight gain is exhibited during the analysis due to oxidation of the sample.

Statistical Information/Method Performance

Generate the method detection limit (MDL) and limit of quantitation (LOQ) annually by following the procedure in *QA-SOP11892*. The department maintains annual study data and updates to LIMS are made as needed by the QA department.

Generate the quality control acceptance windows by following the procedure outlined in *QA-SOP11896*. The limits are evaluated annually and updated as needed. These limits are maintained in LIMS for the relevant analysis number(s).

Quality Assurance/Quality Control

Batch size is limited to 20 samples or less.

A method blank must be analyzed on each batch. The method blank must be less than the LOQ. If the method blank is greater than the LOQ, then all samples on the batch must be reanalyzed.

A 200 mg/L laboratory control standard (LCS) must be analyzed on each batch. See LIMS for the current acceptance range.

A matrix spike (MS) must be analyzed with each batch.

A sample matrix duplicate must be analyzed with each group of not more than 10 samples. Duplicate determinations should agree within 5% of their average weight.

When sample volume does not permit the analysis of either the MS or the required duplicates, then an LCSD must be analyzed to demonstrate method precision.

Samples from West Virginia require a usable precision measurement for each batch. Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: an MS/MSD, a non-program deleted duplicate or an LCSD.

If the residue weight of a sample is less than 0.0025 gram and sample volume permits, repeat the sample using up to 1000 mL of sample. Samples from the state of South Carolina have a minimum limit of 0.01 gram.

The maximum residue limit is 0.2 gram. Samples with results above this limit are reanalyzed with smaller volumes.

See LIMS for current quality control (QC) acceptance windows. Follow guidelines in *T-WC-WI10360* for outlier QC data.

QA-SOP11886 Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-SOP11896 Establishing Control Limits
T-WC-WI10350 Maintenance of Desiccators
T-WC-WI10359 Instructions for Collecting Data on the LLENS System
T-WC-WI10360 Quality Control Data for Wet Chemistry

End of document

Version history

Version	Approval	Revision information	
16	25.MAR.2019		
17	30.APR.2021		
18	29.OCT.2021		

eurofins	Always check on-line for validity. **Total Suspended Solids (TSS)-Gravimetric by SM 2540 D-2011 or SM 2540 D-1997 and Total Filtered: Total Volume Test by NJDEP in Waters**	Level: **Work Instruction**
Document number: **T-WC-WI11604**		
Old Reference: **1-P-QM-WI-9012778**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **31-OCT-2021**	Document users: **6_EUUSLA_Water Quality_Gravimetric Analysis, 6_EUUSLA_Water Quality_Gravimetric Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision:	12	**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Throughout document	new LIMS		update to new LIMS IDs
Throughout Document	deactivated method		Removed all references to analysis 14115 - Total Filtered: Total Volume analysis

Revision: 12	**Effective Date:**	This version
Reference	Reflects current method references	Added 2540 D-2015, removed NJDEP
Cross Reference	Update to D4 format Reflects current process	re-identified SOP Analysis 207, 208 Removed *T-WC-WI10359*
Basic Principles	Clarification	Defined analysis start time as the first time samples are placed in the oven.
Apparatus and Equipment 7.	new LIMS	Removed LLENS refence
Procedure	new LIMS	Renamed columns in worksheet to match new LIMS
Procedure 8.g	Method criteria	removed when sample matrix allows
Procedure 11, 13	Current process	Added instructions to record time and temperature, noting the first time in the oven is the start time for the analysis.
Attachment I	deactivated process	Removed Attachment I

Revision: 11	**Effective Date:**	24-DEC-2018
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Document Title	Addition	Added SM 2540 D-2011
Throughout document	Update to D4	Changed Cross References 2-5 to D4 document numbers
Reference 1.	Correction	Added SM 2540 D-1997
Basic Principles	Clarification	Rewritten to explain drying/weighing cycle. Added "weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less."
Interferences	Clarification	Included how samples over the residue limit are treated
Reagents and Standards A.	Requirement	Added reagent grade and storage container.
Procedure 8	Clarification	Changed name of step from analysis to filtration
Procedure A.8.b,e,f	New process	In addition to the volume used, the initial and all subsequent volumes of the filtering process will be recorded.
Procedure A.8.e-g	New process	Reorganized steps to match current process
Procedure A.8.h	Reflects current process	Removed original step A.8.h. Renumbered step 9 as step A.8.h
Procedure A.8.h, i (previously A.9,10)	This is part of the filtration process	Renumbered A.9,10 as step A.8.h and i. Renumbered following steps accordingly.
Procedure A.9.	New process	Added instructions for calculating the sample volume filtered.
Procedure A.12, 14 (previously (A.14,16)	Clarification	Distinguished between the two analyses drying requirements.

Revision: 11	**Effective Date:**	24-DEC-2018
Procedure A.13 (previously A.15)	Reflects method reference	Changed cooling time to "cool to balance temperature".
Attachment I	Addition	Added comment #9388

Reference

1. *Standard Methods for the Examination of Water and Wastewater*, Method 2540 D-2015.

2. *Standard Methods for the Examination of Water and Wastewater*, Method 2540 D-2011.

3. *Standard Methods for the Examination of Water and Wastewater*, Method 2540 D-1997.

4. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-WC-WI10610	Fixed Suspended Solids (Gravimetric) (#207) Volatile Suspended Solids (Gravimetric) (#208) by SM 2540 E - 1997
T-WC-WI10360	Quality Control Data for Wet Chemistry
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

Methods 2540D and 2540D_SingleDry are used for the determination of total suspended solids (TSS) in potable, surface and saline water, domestic and industrial wastewater in a range up to 20,000 mg/L. The reporting limit for this analysis varies with the sample volume. LIMS may round the reporting limit during the calculation routine.

Basic Principles

For Methods 2540D and 2540D_SingleDry, a well-mixed sample is filtered through a glass fiber filter and the residue on the filter is dried in an oven at 103° to 105°C. The increase in weight of the filter represents the TSS.

The start time for the analysis is the time the samples are initially placed in the oven to dry.

Method 2540D requires repeat drying, cooling, desiccating and weighing cycles until the weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less.

Method 2540D_SingleDry records only one weight after a 16 hour drying cycle.

The appropriate analysis scan is typically determined by the sample state of origin.

Interferences

Samples high in dissolved solids may yield positive interferences. If the sample is over the residue limit of 0.2 g, the sample is reanalyzed at a reduced volume.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst’s training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples must be unpreserved, collected in glass or plastic containers and refrigerated at 0° to 6°C, not frozen.

Analyze samples within 7 days of collection to minimize microbiological decomposition of the solids.

Exclude non-representative, nonhomogeneous materials, such as leaves and sticks, from the analysis if their inclusion is not desired in the final result.

Apparatus and Equipment

1. ProWeigh Filters from Environmental Express – pre-weighed 47 mm glass fiber filters
2. ProWeigh Filters from Environmental Express – pre-weighed **volatile** 47 mm glass fiber filters or equivalent
3. Filtration apparatus
4. Desiccators.
5. Oven maintained at 104° ± 1°C. Adjust as needed to stay in this range.

6. Analytical balance.

Reagents and Standards

Alternate weights and volumes are acceptable as long as final concentrations remain the same.

A. TSS Working Standard (150 mg/L)

1. Dry 1,8 Naphthalimide, 98%, for approximately 1 hour at 104° ± 1°C.
2. Desiccate approximately 1 hour.
3. Dissolve 0.1500 ± 0.005 g Naphthalimide in approximately 700-mL of reagent water in a 1000-mL volumetric flask.
4. Mix very well.
5. Dilute to 1000 mL with reagent water.
6. Store at room temperature.
7. Stable 30 days.
8. Prior to use, Naphthalimide can be weighed out and stored in small glass vials at room temperature.

Calibration

A balance calibration check and leveling check must be performed each day before use.

Procedure

1. Batch samples for analysis in the LIMS.
2. Assemble filtration apparatus.
3. Assign a pre-washed, pre-weighed filter to each client sample and each QC sample on the batch. If fixed or volatile suspended solids are to be performed use a pre-weighed **volatile** filter and follow instructions in T-WC-WI10610.
4. Remove the assigned filter from its aluminum carrying dish and place on the filtration unit.
5. Apply vacuum to the filtration apparatus.
6. Wet filter with small amount of reagent water to seat it.
7. Quality Control samples preparation
    a. Analyze the method blank by using 1000 mL of reagent water.

b. Analyze the LCS/LCSD using 1000 mL of the 150 mg/L TSS working standard for each QC standard.

c. Prepare duplicates by analyzing equal portions of sample.

8. Sample filtration

a. Shake sample vigorously.

NOTE: If the sample does not maintain a homogeneous state until the aliquot is taken, then magnetically stir the sample while pipetting the appropriate aliquot onto the filter.

b. Using a graduated cylinder or wide mouth pipette, measure 1000 mL of sample or a smaller portion if the sample matrix dictates. Record this volume in the 'Notes' column.

c. Rapidly transfer the aliquot to the filtration unit.

d. Still under vacuum, filter the sample.

e. Repeat steps 8b-d until an appropriate total volume for the sample matrix has been filtered. Record each volume filtered in the 'Notes' column.

f. After all of the sample's volumes have been filtered, rinse the graduated cylinder. Rinse sufficiently to ensure all of the solids inside the graduated cylinder are transferred to the filtration apparatus.

(1) If the entire volume was used for the analysis, then rinse the inside of the sample container with at least three 10-mL portions of reagent water and transfer the rinses to the filtration apparatus. Rinse sufficiently to ensure all of the solids inside the bottle are transferred to the filtration apparatus.

g. Wash the filter with at least three 10 mL portions of reagent water. Allow complete drainage between washings. If complete filtration takes more than 10 minutes, reanalyze the sample decreasing the sample volume.

h. If necessary, perform a final rinse to remove any residue from the sides of the filtration unit.

i. Continue suction at least 3 minutes after filtration is complete.

NOTE: This step is important to ensure that dissolved solids are rinsed through, and not dried in the filter.

9. The total sample volume filtered ('initial Amount' column on the raw data worksheet) is calculated by adding all the volumes from the 'Notes' column. Record the total sample volume used in the 'Initial Amount' column on the worksheet.

10. Transfer the filter back into its aluminum carrying dish and place in an oven maintained at 104° ± 1°C.

11. Record the oven temperature and the time the samples were placed in the oven in the batch information window. The first time the samples are placed in the oven is the start time for the analysis.

12. Dry the filter for the appropriate amount of time according to the analysis selected.

a. Method 2540D - dry the filter for at least one hour.

   b. Method 2540D_SingleDry - dry the filter for at least 16 hours.

13. After the drying time, transfer the aluminum dish into a desiccator and cool to balance temperature. Record the time and the temperature.

14. After cooling, weigh the filter and record the oven dried weight of the filter.

   a. Method 2540D - repeat this drying desiccating and weighing cycle until weight change is less than 4% of the previous weight or ≤ 0.0005g, whichever is less.

   b. Method 2540D_SingleDry - require only one weight after a 16-hour drying cycle.

Calculations

$$mg\ Total\ Suspended\ Solids / L = \frac{(A - B) \times 1000 \times 1000}{Sample\ Volume\ (mL)}$$

Where:

A = Weight of filter, (in grams) after drying

B = Tare weight of filter (in grams)

Statistical Information/Method Performance

Generate the method detection limit (MDL) and limit of quantitation (LOQ) annually by following the procedure in *QA-SOP11892* . The department maintains annual study data and updates to LIMS are made as needed by the QA department.

Generate the quality control acceptance windows by following the procedure outlined in *QA-SOP11896*. The limits are evaluated annually and updated as needed. These limits are maintained in LIMS for the relevant analysis number(s).

Quality Assurance/Quality Control

Batch size is limited to 20 samples or less.

One method blank and one 150-mg/L LCS must be analyzed with each TSS batch.
A sample matrix duplicate must be analyzed with each group of not more than 10 samples.

Duplicate determinations should agree within 5% of their average weight.

When sample volume does not permit the analysis of the required duplicate(s), then a 150 mg/L LCSD must be analyzed to demonstrate method precision.

The maximum residue limit is 0.2 gram.

Samples from West Virginia require a usable precision measurement for each batch.

Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: a non-program deleted duplicate or an LCSD.

See LIMS for current quality control (QC) acceptance windows. Follow guidelines in *T-WC-WI10360* for outlier QC data.

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
T-WC-WI10359 Instructions for Collecting Data on the LLENS System
T-WC-WI10610 Fixed Suspended Solids (Gravimetric) (#207) Volatile Suspended Solids (Gravimetric) (#208) by SM 2540 E - 2011 or EPA 160.4 in Water

---

End of document

Version history

Version	Approval	Revision information	
10	28.SEP.2016		
11	10.DEC.2018		
12	29.OCT.2021		

eurofins	Always check on-line for validity. **Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS**	Level: **Work Instruction**
Document number: **T-MET-WI8636**		
Old Reference: **1-P-QM-WI-9015160**		
Version: **26**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **11-MAY-2021**	Document users: **6_EUUSLA_Metals_ICP Prep, 6_EUUSLA_Metals_ICP Prep Verifiers, 6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Purpose
Scope
Basic Principles
Definitions
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Procedure
Block Digestor Instructions
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

	Revision: 26	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
LIMS ID	Enhancement	Added TALS method codes
Cross Reference	Enhancement	Added Data Verification SOP
Definitions	Clarification	Removed redundant definitions; added SQAR
Apparatus and Equipment	Reflects current procedure	Removed reference to LLENS
Procedure	Reflects current procedure	Removed reference to the LLENS
Quality Assurance/Quality Control	Reflects current procedure	Removed reference to individual analysis SOPs; added in reference to Data Verification SOP

Revision 25	**Effective Date:**	06.MAR.2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
LIMS ID	Follow current procedure	Added analysis number for sieving
Apparatus and Equipment	Follow current procedure	Added sieves and changed temperature to 90C-100C
Procedure	Follow Current Procedure	Clarified if sample is not split
Procedure	Follow current procedure	Added section on how to dry and sieve samples
Procedure	Update to Method	Updated 90C-95C to be 90C-100C
LIMS ID	Follow Current Procedure	Removed old analysis numbers
Quality Assurance/Quality Control	Clarification	Added sentence about sample and batch QC must be the same

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3050B, December 1996.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-MET-WI11933	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)
T-MET-WI11931	Metals by Inductively Coupled Plasma Atomic Emissions Spectroscopy for SW-846 Methods 6010B/C (aqueous, solid, tissue) and EPA 200.7(aqueous)
T-MET-WI9082	Working Instructions for Prep Solutions and Standards

Document	Document Title
T-MET-WI35797	Metals Data Verification Process

Purpose

This digestion procedure is for the preparation of solid samples for analysis by ICP and ICP/MS following SW-846 protocol.

Scope

This method is used for preparation of metals in solid samples for analysis by ICP and ICP/MS.

Basic Principles

A representative sample is digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). Hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. The resultant digestate is diluted and analyzed.

This method is not a total digestion technique for most samples; it is a very strong acid digestion that dissolves almost all elements that could become "environmentally available." By design, elements bound in silicate structures are not normally dissolved by this procedure.

Definitions

1. SQAR = Sludge Quality Assurance Regulations

Interferences

When analyzing sample by ICP-MS using this digestion procedure we follow the instrument manufacturer's guidelines to eliminate polyatomic interferences typically caused by Chlorine. The process we follow involves the use of a collision/reaction cell on the ICP-MS. Below is a description of how the collision/reaction cell works.

Reaction Process - The primary method of interference removal is through a reaction event. When using a reaction gas, either the target interference is more reactive than the target analyte, leading to preferential removal of the interferent or (less commonly) the target analyte is more reactive and is converted to a new species at a different mass which is free from any existing or newly-formed overlap.

Collision Process - The primary method of interference removal is through a non-reactive event. This process of interference removal is kinetic energy discrimination (KED). Energy Discrimination is most commonly used with an inert gas, which means the interference removal process is not affected by reactions in the cell.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO3)** – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.
>
> **Hydrochloric acid (HCl)** – This acid can cause skin burns. Never mix HCI with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood or use the designated dispensing equipment.
>
> **Hydrogen peroxide (H2O2)** - This is a strong oxidizing agent and causes severe burns. Avoid contact with skin.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds. Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four LCSs that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four LCSs or one blind sample.

Sample Collection, Preservation, and Handling

Solid samples require no chemical preservation.

Samples must be submitted in glass or plastic containersand stored at 0° to 6°C, not frozen, prior to digestion. Samples must be analyzed within 6 months of sample collection.

Digested samples are stored in polypropylene bottles at room temperature.

Apparatus and Equipment

1. Polypropylene containers and covers (digestion vessels) - certified clean and Class A equivalent
2. Whatman No. 41 filter paper or equivalent
3. Funnels
4. Environmental Express HotBlock (block digestor) - adjustable and capable of maintaining a temperature of 90 to 100°C
5. Balance capable of reading 0.01 g
6. Chemware Ultra-Pure PTFE boiling stones, or equivalent.
7. Computer and software TALS
8. Sieves size depending upon client request

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form *T-MET-WI9082*.

1. Nitric acid (HNO3) – Fisher, Trace Metal Grade, or equivalent. Store at room temperature. Re-evaluate annually.
2. Nitric acid (1:1) – Add 500 mL of HNO3 to 500 mL of reagent water. Store in polypropylene at room temperature. Expires 6 months from date of preparation. (Different volumes are acceptable but ratios must stay the same.)
3. Hydrogen peroxide, 30% (H2O2) – Fisher, Certified ACS or equivalent. Store at room temperature. Re-evaluate annually.
4. Hydrochloric acid (HCl) – Baker Instra-Analyzed, or equivalent. Store at room temperature. Re-evaluate annually.

NOTE: It is acceptable to prepare using multiples of indicated weights and volumes if ratios are maintained.

Procedure

A. Soil Sample Digestion

1. Turn block digestor on and allow block to reach the Control Point setting that provides 90° to 95°C sample temperature. (The block temperature setting is not necessarily the sample temperature.) See below for **Block Digestor Instructions** section.
2. If sample is not located in split bin, weigh 1.00 to 1.50 g (to the nearest 0.01 g) of a well-mixed sample into a polypropylene digestion vessel. (If the sample is watery use 5.00 to 5.50 grams for analysis. Additional information on non-standard matrices is found at the end of the procedure section.) Add 1.00 to 1.49 g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Enter the blank and LCS weight as 1.0000 to 100.0000 final volume in theworksheet tab of the TALS prep batch. In the Notes field, record the true weight measured.For sample batch spiking procedures see *T-MET-WI9082*. All spiking must be performed prior to starting the digestion procedure.
3. Add 10mL of (1:1) HNO_3, swirl to mix, and cover with a polypropylene cover.
4. Place sample vessel in block digestor. Heat (reflux) the sample at 90°to 100°C for 10 to15 minutes without boiling.
5. Add 5 mL of concentrated HNO_3. Add 20 mL of reagent water. Replace cover, return vessel to digestion block and heat for 30 minutes.
6. **NOTE**: If brown fumes are generated (indicating oxidation of the sample by HNO_3) continue the process of adding 5mL HNO_3 and heating until no brown fumes are given off by the sample. This indicates that the reaction with HNO_3 is complete. Add the same amount of HNO_3 to the entire digestion batch.
7. With cover on, heat at 90° to 100°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).
8. Remove vessel from digestion block and allow sample to cool.
9. Add 2 mL of reagent water and 3 mL of 30% H_2O_2. With cover on, return vessel to digestion block and heat until effervescence subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.
10. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.
11. **NOTE:** Do not add more than a total of 10mL 30% H_2O_2.
12. With cover on, continue heating the acid-peroxide digestate at 90°to 100°C without boiling for 2hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).
13. Remove sample vessel from digestion block and allow to cool.
14. Add 10 mL of HCl. With the cover on, return vessel to digestion block and heat at 90°to 100°C for 15 minutes.
15. Remove sample vessel from digestion block.
16. If floating particulate is evident after digestion, the sample must be filtered.
    a. Filter through Whatman No. 41 filter paper into a polypropylene container.
    b. Wash sample vessel, residue, and paper thoroughly with reagent water.

c. If any samples are filtered, the MB and LCS must also be filtered.

17. Adjust volume to the 100 mL mark on the digestion vessel with reagent water and mix. Seal vessel with a screw cap. The sample is now ready for analysis.

NOTE: When special limits of quantitation are required by the client, use more sample weight.

B. For wipe samples:

When wipes are digested by this method, one blank media each must be used for the batch preparation blank, the LCS, and the LCSD. Refer to Form *T-MET-WI9082* for the spiking of the LCS and LCSD. Digest wipes in their own batch. Use reagent water to rinse any particulate matter from the wipe container into the vessel containing the wipe before digesting. If brown fumes are evolved during wipe sample digestion, perform only two 5 mL HNO_3 additions with 30-minute refluxing each; add the same amount of HNO_3 to the entire batch. Proceed with digestion.

C. For tissue samples:

When fish tissues or other tissue samples are digested by this method, refer to Form *T-MET-WI9082* for the spiking of the LCS, LCSD (if needed), matrix spike (MS), and matrix spike duplicate (MSD). Add 1.00 to 1.49g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Digest tissue samples in their own batch.

D. For soil samples that need to be dried and sieve prior to digestion.

1. Lay out sample on a clean surface of either glass or aluminum foil.
2. Allow sample to dry. When sample is done drying, but is dried in a large chunk take appropriate clean tools to break down the large chunk in order to sieve the sediment.
3. Pour entire sample that has been laid out to dry into the appropriate sieve size defined by client and shake sieve to get sample to fall through.
4. Take the sieved sample and place into the appropriate bottle needed for analysis.
5. Rinse the sieve and the sieve filter with reagent water until no particulate is left on sieve.
6. Dry sieve in open air or in an oven until no reagent water is present.

Block Digestor Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.
2. Wait about 8 seconds until controller display indicates current block temperature.
3. PRESS and hold STAR (*) key.
4. The display shows Control Point temperature.
5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.
6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used. If necessary, adjust Control Point temperature to the proper setting as instructed below.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations
Not applicable.

Statistical Information/Method Performance
Not applicable to this procedure. See analysis method.

Quality Assurance/Quality Control

For sample batch spiking instructions see form *T-MET-WI9082*. Refer to ICP section when prepping ICP analysis. Refer to ICP/MS section when prepping ICP/MS analysis. Prepare a method blank, sample duplicate, MS, MSD, and LCS with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP. If any samples are filtered the prep blank and LCS must also be filtered.

Any extra digestion step done on one sample in a batch must be done to batch QC as well. Example if 1 mL of H_2O_2 is added to a sample it must also be added to the batch QC.

Refer to *T-MET-WI35797* for sample batch quality control requirements, acceptance criteria and corrective action.

T-MET-WI11931 Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)
T-MET-WI11933 Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B(aqueous, solid, tissue) and EPA 200.8 (aqueous)
T-MET-WI35797 Metals Data Verification Process
T-MET-WI9082 Working Instructions for Prep Solutions and Standards

---

End of document

Version history

Version	Approval	Revision information	
24	13.FEB.2018		
25	06.MAR.2019		
26	11.MAY.2021		

eurofins	Always check on-line for validity. **Carbon Dioxide in Water Using Headspace Sampling Techniques and GC-TCD, Method RSK-175 or 8015**	Level: **Work Instruction**
Document number: **T-GC-WI9644**		
Old Reference: **1-P-QM-WI-9015187**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **02-DEC-2019**	Document users: **6_EUUSLA_EPH/Misc. GC_All Management/Verifier/Prin, 6_EUUSLA_EPH/Misc. GC_Volatiles Headspace Chemist**	Responsible: **5_EUUSLA_EPH/Misc. GC_Manager**

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

	Revision: 12	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Reflects current reference	Removed 8000B and 8000C as references – only need 8000D
Cross Reference	Enhancement	Added WI9683
GC Analysis Conditions	Formatting requirement	Combined with Apparatus and Equipment
Reagents and Standards	Clarification	CO_2 concentration is approximate
Calibration	Clarification	Clarified the linear acceptance criteria
Procedure	Clarification	Clarified that MS is spiked with same solution used to prepare ICAL standards
Statistical Information/Method Performance	Reflects current practice	Removed ‘When 8000D is referenced’
Quality Assurance/Quality Control	Reflects current practice	Added ‘or less than the MDL for MDL reporting’

	Revision: 11	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Reflects current references	Added 8000B/C/D
Calibration	Reflects current practice	Added 8000D criteria. Added action when 2 consecutive CCVs fail. Added criteria and corrective actions for the ICAL MDL standard
Procedure	Clarification Reflects current practice	Added calibrated pipette throughout section. Added additional information on preparing the MS/MSD Changed from 20 samples associated with a batch to 15 samples in a batch.
Statistical Information/ Method Performance	Reflects current practice	Added similar verbiage as other department SOPs.
Quality Assurance/ Quality Control	Reflects current practice	Changed from 20 samples associated with a batch to 15 samples in a batch.
Apparatus and Equipment	Reflects current practice	Corrected system reference to GC-TCD

Reference

1. *Test Methods for Evaluating Solid Wastes, SW-846 Method 8015C, Rev. 3, February 2007.*
2. *Test Methods for Evaluating Solid Wastes SW-846, Method 8015D, Rev. 4, June 2003.*
3. Sample Preparation and Calculations for Dissolved Gas Analysis in Water Samples Using a GC Headspace Equilibration Technique, RSKSOP-175, Rev. 5, Oct. 2010, modified.
4. Determinative Chromatographic Separations, SW-846, Method 8000D, July 2014
5. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-GC-WI9689	Maintenance and Troubleshooting Procedures for GC-FID Instrumentation
T-GC-WI9657	Common Equations Used During Chromatographic Analyses
T-GC-WI9749	QC Data Acceptability and Corrective Action
T-GC-WI9698	Monitoring QC Data Acceptance Limits
T-GC-WI9683	Interpretation and Integration of Chromatographic Data
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation

Scope

This method is applicable to the quantitation of gaseous phase carbon dioxide (CO_2) in water samples at a limit of quantitation (LOQ) of 12,000 µg/L. The current MDL and LOQ is updated as needed in the LIMS.

Basic Principles

A 5 mL aliquot of sample is transferred into a 10 mL headspace sampling vial which is then placed in an incubator at 35°C. After loading a portion of the headspace into a fixed volume loop it is analyzed by capillary gas chromatography using thermal conductivity detection (GC-TCD).

Interferences

A TCD is used for this analysis and can detect various molecules. (Changes in thermal conductivity, such as when organic molecules displace some of the carrier gas, cause a temperature rise in the element which is sensed as a change in resistance and recorded as the signal/peak during the chromatographic analysis.) However, there is little chromatographic interference using this method for CO_2 analysis. The CO_2 is measured in the headspace generated from a water sample, so competing or coeluting molecules are not routinely generated in that headspace.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Safety glasses must be worn when handling any contents under high pressure. Safety glasses, a lab coat, and latex rubber gloves (or equivalent) must be worn when working with samples. Upon completion of analysis, sampling vials and GC vials must be placed in a container, such as a box, and discarded in the designated area for GC vials. Read and follow all safety and handling procedures accompanying the custom blended gas standard.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the chromatography data system to set up sequences, perform the calculations, interpret chromatograms, perform instrument maintenance, and enter data into the LIMS. Proficiency is measured through documented audits of the tasks listed and over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four laboratory control samples or one blind sample.

Sample Collection, Preservation, and Handling

Water samples are collected in 40-mL unpreserved vials with zero headspace. Samples are stored refrigerated at 0° - 6°C, not frozen, and must be analyzed within 14 days from collection.

Other preservation is not recommended. Variation in sample pH including preservation may affect CO_2 values.

Apparatus and Equipment

1. GC-TCD system such as an HP5890, or equivalent

The conditions listed serve as a guideline and are typically the optimum operating conditions. However, the laboratory GC conditions may differ, to optimize the sensitivity, linearity, run time, resolution, and overall chromatography on each instrument and column used for analysis as long as initial and continuing calibration criteria and quality assurance criteria for this analysis are met.

Oven Temperature:	40°C for 0.1 min; 40°C/min to 80°C hold 1 min; 50°C/min to 150°C hold 1 min

Carrier Gas:	Helium at approximately 30 mL/min ±2 mL/min
Injector:	100°C
Detector:	Thermal conductivity detector (TCD)
TCD Detector:	200°C
TCD adjustments:	LG settings, Hc setting
Flows:	Balanced at 28 mL/min ± 2 mL/min
Range:	0
Attenuation:	1

NOTE: It is highly recommended that the TCD be turned on at least 30-60 minutes prior to starting a run/ICAL.

2 CTC Analytics CombiPAL headspace sampler or equivalent. The sampler must be operated by conditions similar to the following:

Incubate temperature	35°C
Incubate time	5 min
Agitator	Run 5 Stop 3
Agitator RPM	1600
Sample extraction	01
Run time	7 min. 20 sec.
Syringe	45°C
Volume	1000 µL
Syringe fill delay	2 sec.
Fill speed	300 µL/sec.
Inject speed	3000 µL/sec.
Inject delay post	10 sec.
Bake out	5°C for 20 sec.
Flush	1 min, 35 sec.

3. A PC-based data acquisition system such as Chrom Perfect by Justice Innovations or equivalent

4. 10-mL capacity headspace sampling vials with silicone Teflon-lined crimp caps

5. Assorted gastight syringes, pipettes, and syringe adapters

6. GC column – Alltech CTR1 column 6’ x 1/4” x 0.210” SS, Alltech Reference #252928R, or equivalent

Reagents and Standards

1. Stock standard – A certified gas standard at 100%$CO_2$is purchased from an appropriate supplier such as Air Products Catalog# 9151, or equivalent.

   a. The standard may be stored at room temperature indefinitely.

   b. A second gas standard of 100% CO_2 is purchased as a second source for the initial calibration verification (ICV).

2. Working Calibration standards

a. At least 5 levels of calibration standards, a method detection limit verification (MDL) standard, and an initial calibration verification (ICV) standard are prepared just prior to calibrating the instrument according to the dilution scheme listed below (see 2.d.).

b. Autosampler vials containing 5 mL of reagent water are prepared, one for each standard, and capped with the crimp top. The appropriate aliquot of CO_2 gas standard for each working standard is removed from the tank using a gas tight syringe. The needle of the syringe is then pushed through the crimp top of the vial and placed into the water in the vial. The gaseous aliquot is then dispensed into the water.

c. See the Spiking Technique outlined in Procedure Section C for a detailed explanation of how to measure out the aliquots of standard.

d. These standards are used once on the analytical run and are then discarded.

Standard ID	**Standard Used**	**Amount of Standard (µL)**	**Final Volume (DI water)**	**CO_2 Approx. Concentration (ppb)**	**Description**
CO2_1	100% CO2	30	5	10800	Level 1 calibration
CO2_2	100% CO2	60	5	21600	Level 2 calibration
CO2_3	100% CO2	100	5	36,000	Level 3 calibration
CO2_4	100% CO2	250	5	90,000	Level 4 calibration
CO2_5	100% CO2	500	5	180,000	Level 5 calibration
CO2_6	100% CO2	1000	5	360,000	Level 6 calibration
CO2MX	100% CO2	20	5	7200	MDL
CO2CX	100% CO2	320	5	35008	ICV

3. Continuing calibration verification (CCV) standards

a. The initial calibration standard level 3 is used as the CCV.

b. These are prepared fresh each time samples are analyzed.

c. They are used once on the analytical run and are then discarded.

d. See above for the dilution schemes (see 2.d.).

4. Reagent water – water in which an interferent is not observed at or above the reporting limit for parameters of interest. In general, the deionized water supplied at the taps in the laboratory meets this criterion. If the reagent water does not meet the requirements, see your supervisor for further instructions.

Calibration

1. To prepare the initial/continuing calibration standards, use a syringe to add 5-mL of reagent water into the appropriate number of 10-mL headspace vials. Crimp caps onto the vials tight enough so that the caps cannot be rotated easily.

2. Using the calibration scheme described in the Reagents and Standards section, add the appropriate amount of spiking standards to each vial. Use the spiking standard technique described above.

3. Prepare a sequence using the following suggested order of injections:

    1. IBLK

2. CO2_1
3. CO2_2
4. CO2_3
5. CO2_4
6. CO2_5
7. CO2_6
8. Water blank
9. CO2MX (MDL Standard)
10. ICV standard
11. Blank
12. LCS
13. 1234567
14. 1234567MS
15. 1234567MSD

16.-20. Continue with samples
21. CO2_3
22.-31. 10 samples
32. CO2_3

Continue running groups of 10 samples with a CCV between sample groups.

4. Inject conditioner to prime the system.

a. Conditioner is best utilized when the GC has not been running and there is a gap in time prior to starting a set of injections.

b. The conditioner is typically a standard or sample that has already been injected.

5. Water blanks can be run to allow the GC to go through some temperature programs and/or to check the cleanliness of the system.

6. Initial Calibration (ICAL)

a. The system is calibrated using the 6 concentration levels of CO_2.

b. An external standard calibration is used with average response factor (AVGRF) if the %RSD is ≤20%.

(1) Alternatively, when this criteria is not met, a calibration curve is used. A linear fit is tried first. The coefficient of determination (r^2) must be >0.99.

(2) However, if the correlation coefficient is <0.99 a quadratic fit will be tried.

(a) A 6-point calibration must be run to use quadratic fit.

(b) Prepare a sixth point somewhere within the established calibration range listed in the standards preparation section.

(3) For either curve type, extrapolate or force zero is not allowed. Set the zero to ignore.

(4) If the 0.99 curve coefficient cannot be met:

(a) Inspect the data points to see if one or more calibration levels appear to be off. A specific calibration level may have concentrated due to solvent evaporation or degraded over time.

(b) Remake the standard if this is the cause. Otherwise, the instrument may need maintenance.

(c) See *T-GC-WI9689* for troubleshooting linearity problems.

When curve fits are used in the initial calibration, percent error (%E) must be calculated for each calibration level. Standard levels at or below the LOQ must meet +/- 50%, standards above the LOQ must meet +/-30%. (%error = (true concentration - calculated concentration)/true concentration)*100).

7. An initial calibration verification (ICV) standard is analyzed and the % difference of the concentration for CO_2 must be within 15% difference of the nominal concentration for 8015B/RSK-175 and 20% for 8015C/8015D.

8. Continuing Calibration Verification (CCV)

a. Analyze a continuing calibration verification (CCV) standard every ten injections.

b. Use the level 3 standard from the initial calibration.

c. The concentration quantitated for the CCV must be within ±15% of the nominal concentration for 8015B/RSK-175 and 20% of the nominal concentration for 8015C/8015D.

d. Samples must be bracketed with compliant standards.

(1) Exception: If, however, the standard following a sample is outside the ±15% for 8015B/RSK-175 or ±20% for 8015C/8015D but exhibits increasing response, the samples before it do not have to be re-injected if the target analytes are not detected. A comment must be added to the analytical report.

(2) If two consecutive/sequential CCVs fail, corrective action must be taken, which can include performing injection port maintenance, baking the instrument or other types of maintenance. Two consecutive/sequential CCVs must meet the criteria or an initial calibration must be performed before sample analysis can continue.

(3) If these criteria are not met; then all samples since the last compliant CCV and before the next compliant CCV must be reanalyzed.

9. Retention time (RT) windows

a. Established as 3x the standard deviation determined over a 72-hour period, or at no less than ±0.03 min, applied to the mid-point initial calibration standard.

b. If the RTs for a CCV fall outside the windows, update the midpoint RT using that standard.

(1) Save this under an appropriate name to indicate an update has occurred.

(2) All subsequent continuing standards run within a 24-hour period must fall within this window.

(3) RTs cannot be updated more than once per day.

c. If RTs are not consistent, the cause must be investigated and corrective action taken.

10. Ensure the CO_2peak in each standard is labeled properly and the scaling of the plot is such that the concentration at the MDL exhibits a peak about 2 to 3mm in height.

11. MDL Standard Criteria

Verify the peak on the MDL standard is integrated by the data system correctly. The CO_2peak in the MDL standard must be detected to be acceptable.

Take corrective actions if the CO_2 peak is not detected in the MDL standard. Troubleshoot the system to determine the source of the problem and perform system maintenance as required. The initial calibration sequence must be reanalyzed once the problem has been resolved.

Procedure

A. Sample preparation procedure

NOTE: Prior to sample preparation, it is recommended to have the system calibrated and the blank, laboratory control spike (LCS), and laboratory control spike duplicate (LCSD) if needed, analyzed and within specifications.

1. Remove samples from cold storage and allow them to warm to room temperature.

2. Crimp a labeled cap onto an empty vial tight enough so that the cap cannot be rotated easily.

3. Using helium displacement (supplied through bulk tanks piped into lab area), withdraw a 5-mL aliquot of the sample through the septum of the 40-mL vial using a 10-mL gastight syringe.

a. Refer to *Diagram 1* for a pictorial description of the helium displacement set-up.

b. Insert a 10-mL gastight syringe into the 40-mL sample vial through the septum.

c. Insert the helium needle into the sample vial septum and allow the 10-mL syringe to fill with 5 mL of sample.

d. Add the sample to the 10-mL headspace vial through the septum.

(1) Vent the pressure of the sample vial that accumulated from the addition of the sample while the syringe is still through the septum of the vial.

(2) If a dilution is necessary, the vial must contain sufficient reagent water so the final volume after sample addition is 5 mL.

4. Equilibrate the sample at 35°C for at least 5 minutes.

B. QC preparation

1. Method Blank

a. One method blank is analyzed per batch of up to 15 samples.

b. To prepare a method blank: Use a calibrated pipette or syringe to add 5-mL of reagent water into a 10-mL headspace vial.

c. Crimp a labeled cap onto the vial tight enough that the cap cannot be rotated easily.

2. Laboratory Control Sample (LCS)

a. One laboratory control spike is analyzed per batch of up to 15 samples. An LCSD is also prepared if no MS/MSD is available.

b. To prepare an LCS:

(1) Use a calibrated pipette or syringe to add 5-mL of reagent water into a 10-mL headspace vial.

(2) Crimp a cap onto the vial tight enough so that the cap cannot be rotated easily.

(3) Use a gastight syringe to add 100 µL of the 100% CO_2 (Calibration Stock) through the septum using the “spiking standard addition technique” described below.

3. Matrix Spike (MS)

a. Prepare an MS/MSD for every batch of up to 15 samples and/or every day.

b. Choose a sample with 4 or more vials at random and prepare two additional aliquots. If only 3 vials are available, prepare a MS and LCSD. If there are specific paid QC samples, there should be sufficient sample for those.

c. Crimp cap onto vial tight enough that cap cannot be easily rotated.

d. Use calibrated syringe to add 5ml of sample into capped 10ml headspace vial, from sample vial through the septa, using the displacement apparatus (*Diagram 1*)

C. Spiking standard technique (for calibration standards, LCS/LCSD, and MS/MSD)

1. Attach a syringe adapter to the standard gas cylinder.

2. Flush the adapter with gas standard by inserting a gastight syringe without the plunger for approximately 2 seconds.

3. Rinse a gastight syringe with three syringes full of standard. Expel the rinse gas each time as opposed to pushing the contents of the syringe back into the gas cylinder.

This will prevent contamination of the gas standard as well as preventing plating out of the compounds on the walls of the syringe.

4. Remove an aliquot of gas that is at least 10% greater than the volume that will be used for the standard. Expel the excess gas.

By this time, the precisely measured amount of gas standard in the syringe is at atmospheric pressure.

5. Press the syringe needle through the septum of the headspace vial until the syringe needle penetrates the water in the vial. Expel the contents of the syringe into the water at a constant rate of speed then quickly remove the syringe.

D. Sample Analysis:

1. Samples are analyzed according to the sequence in the calibration section.

2. The retention time of the peak in the sample is compared to the standard RT window. Peaks within the RT window are quantitated according to the calculations section below.

3. Dilutions – Any sample with a response greater than that of the high-level standard must be diluted and reanalyzed. If possible, a fresh 40-mL vial must be used to prepare the dilution or make reinjections.

Calculations

A.Gas standard concentration calculation

Converting CO2 stock of 100% to mg/L 1000000 ppm(v) to mg/L:

$$\frac{1000000\,\mu L\,CO_2}{lL}\,x\,\frac{1mL}{1000\,\mu L}\,x\,\frac{1mmol}{*24.45mL}\,x\,\frac{**44mg\,CO_2}{mmolCO_2}\,x\,\frac{1L}{1000000\,\mu L}=\frac{1.7996E=3mg\,CO_2}{1\mu L}$$

Hence, the concentration (in ppb) of carbon dioxide for level 1 in a 10-mL vial containing 5 mL of water:

$$\frac{1.7996E\text{ - }3\text{ mg }CO_2}{1\ \mu L\ CO_2}\times\frac{***25\ \mu L\ CO_2}{5\ mL\ H_2O}\times\frac{1000\ mL\ H_2O}{1L\ H_2O}=\frac{9.00\ mg\ CO_2\ (ppm)}{L\ H_2O}\times\frac{1000\ \mu g}{1mg}=\frac{9500\ \mu g\ CO_2}{L}$$

***NOTE:** PV=nRT; V=nRT/P

Using the ideal gas law, we can calculate the volume occupied by 1 mole of an ideal gas at room temperature. Assuming room temperature to be 25°C.

1 mole x 0.082056 L atm $mol^{-1}K^{-1}$ x 298K/a atm = 24.45 L

****NOTE:** The molecular weight of carbon dioxide is 44 mg CO2/mmole CO2

*****NOTE:** A 25-µL aliquot of standard was used for this particular dilution for Standard Level 1.
B.A 6-point calibration is established for each component using peak height versus concentration in µg/L. A linear curve with equal weighting is used with zero set to ignore, or alternatively the average RF. Results are calculated as follows:

Using curve:

$$\frac{\text{Sample height - Y intercept}}{\text{Slope}}\times(FV/IV)\times DF=\mu g/L$$

Using Average RF:

$$\frac{\text{Sample height}}{\text{Average RF}}\ x\ \left(\frac{FV}{IV}\right)\ x\ DF=\mu g/L$$

Where:

FV = Final volume of the extract

IV = Initial volume

DF = Dilution factor

$$AVG\ RF = \frac{(RF\ calib\ 1 + RF\ calib\ 2 + RF\ calib\ 3 + RF\ calib\ 4 + RF\ calib\ 5)}{5}$$

$$RF = \frac{STD\ Height}{Std\ conc.\left(\mu g / L\right)}$$

Also see *T-GC-WI9657* for details on calculations.

Statistical Information/Method Performance

LCS, MS, and RPD are compared to the limits stored in the LIMS. The limits for LCS and MS are statistically derived. Historical data for MS/Ds, LCS/Ds, measurement of uncertainty, is reviewed at least annually. Refer to *T-GC-WI9698*. Reporting limits including method detection limits (MDLs) and limits of quantitation (LOQs) are set according to EPA method requirements and are evaluated annually. Refer to QA-SOP11892 for specific guidelines and procedures. Updates to the LIMS are made as needed by the QA Department and only as directed by the manager. The department database is updated via a download from the LIMS.

An annual lower limit of quantification (LLOQ) verification is required annually on at least one instrument. All instruments must have one at least every three years.

Quality Assurance/Quality Control

A blank and an LCS are analyzed with each batch (samples analyzed within a 24-hour period) of 15 or less samples. A Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are analyzed with every batch of 15 or less samples. An LCSD is prepared if sufficient volume is not available for performing an MS/MSD.

CO_2 in the blank must be <LOQ for LOQ reporting or <MDL for MDL reporting.

If the LCS and/or LCSD are out-of-specification, then the LCS/LCSD and/or calibration must be repeated until all analytes are in specifications.

If the MS and/or MSD fall outside the acceptance windows (RPD>20%), the analyst may repeat the outlier (if improper spiking technique or other random error is suspected), or the sample data can be accepted on the basis that the LCS and LCSD are within specifications. If criteria is not met, the problem must be corrected and samples affected by the outlier must be reanalyzed under a different batch.

See *T-GC-WI9749* for details on QC acceptance criteria and corrective action.

Attachment:
Diagram 1 (.doc)

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
T-GC-WI9657 Common Equations Used During Chromatographic Analyses
T-GC-WI9683 Interpretation and Integration of Chromatographic Data
T-GC-WI9689 Maintenance and Troubleshooting Procedures for GC-FID Instrumentation
T-GC-WI9698 Monitoring QC Data Acceptance Limits
T-GC-WI9749 QC Data Acceptability and Corrective Action
Attachment: Diagram 1 (doc)

End of document

Version history

Version	Approval	Revision information	
10.1	17.APR.2017		
11	03.DEC.2018		
12	02.DEC.2019		

eurofins	Always check on-line for validity. **Multi-Parameters in Solids and Waters by ManTech Multi-Parameter System**	Level: **Work Instruction**
Document number: **T-WC-WI11475**		
Old Reference: **1-P-QM-WI-9013112**		
Version: **15**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **30-APR-2021**	Document users: **6_EUUSLA_Water Quality_24/48 Hour Analyst, 6_EUUSLA_Water Quality_24/48 Hour Verification**	Responsible: **5_EUUSLA_Water Quality_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control
Table I

Revision Log

Revision	15	**Effective Date:**	This version
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Throughout document	Current process		Update to new LIMS
Cross-Reference	Higher level documents are not required to be referenced		Removed QA-SOP-11880, *QA-SOP11188*, *QA-SOP11913*
Scope / Reference	Deactivated analysis		Removed SM2340A, 13414(total acidity prep)
Apparatus and Equipment 3.	Deactivated analysis		Deleted step 3.
Calibration C, D, E	Current process		Added instructions to attach calibration data to the analytical batches
Procedure I.B	Deactivated analysis		Removed total acidity prep instructions
Calculation G.	Deactivated analysis		Deleted non-carbonate hardness calculation. Moved Un-ionized ammonia to G, correcting for the 2 H. calculations

Revision	14	**Effective Date:**	20-Mar-2020
Section	**Justification**		**Changes**
Revision Log	Formatting requirement		Removed revision logs up to the previous version
Calibration C.9.	Calibration confirmation		A pH buffer must be analyzed after the calibration
Table I	Addition		Added columns for frequency and acceptance criteria

Reference

1. Standard Methods for the Examination of Water and Wastewater, Method 2310 B-2011.
2. Standard Methods for the Examination of Water and Wastewater, Method 2310 B-1997.
3. Standard Methods for the Examination of Water and Wastewater, Method 2320 B-2011.
4. Standard Methods for the Examination of Water and Wastewater, Method 2320 B-1997.
5.
6.
7. Standard Methods for the Examination of Water and Wastewater, Method 2340 C-2011.
8. Standard Methods for the Examination of Water and Wastewater, Method 2340 C-1997.
9. Standard Methods for the Examination of Water and Wastewater, Method 2510 B-2011.
10. Standard Methods for the Examination of Water and Wastewater, Method 2510 B-1997.
11. Standard Methods for the Examination of Water and Wastewater, Method 2550 B-2010.
12. Standard Methods for the Examination of Water and Wastewater, Method 4500-CO2 D-2011.
13 Standard Methods for the Examination of Water and Wastewater, Method 4500-F B-2011.
14 Standard Methods for the Examination of Water and Wastewater, Method 4500-F C-2011.
15. Standard Methods for the Examination of Water and Wastewater, Method 4500-F C-1997.
16. Standard Methods for the Examination of Water and Wastewater, Method 4500 H+ B-2011.
17. Standard Methods for the Examination of Water and Wastewater, Method 4500-H+ B-2000.
18. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 B-2011.
19. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 D-2011.
20. Standard Methods for the Examination of Water and Wastewater, Method 4500 NH3 D-1997.
21. Standard Methods for the Examination of Water and Wastewater, 8010 F-2015 (Table 8010:VI.)
22. Method 120.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, rev 1982
23. Method 130.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
24. Method 150.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.
25. Method 170.1 Methods for Chemical Analysis of Water and Wastes USEPA 600 rev 1974
26. Method 305.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.
27. Method 305.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
28. Method 310.1, Methods for Chemical Analysis of Water and Wastes USEPA 600, rev 1974
29. Method 340.2, Methods for Chemical Analysis of Water and Wastes USEPA 600.
30. Method 350.3, Methods for Chemical Analysis of Water and Wastes USEPA 600.
31. Test Methods for Evaluating Solid Wastes, SW-846 Method 9040B.
32. Test Methods for Evaluating Solid Wastes, SW-846 Method 9040C.
33. Test Methods for Evaluating Solid Wastes, SW-846 Method 9050A, December 1996.
34. Test Methods for Evaluating Solid Wastes, SW-846 Method 9214.
35. ManTech Associates, Inc. PC-Titration Plus Instruction and Operation manual, January 25 2000.
36. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title

Document	Document Title
T-WC-WI10425	Bellack Distillation for Fluoride in Waters and Solids by SM 4500 F B–1997 or EPA 340.1 Procedure 6.1
T-WC-WI11519	pH Probes and Meters
T-WC-WI10348	Fixed Volume Hand-Held Pipettes
T-WC-WI9897	Adjustable Volume Handheld Pipettes
T-WC-WI11585	Standardization of 0.02 Normal Sodium Hydroxide
T-WC-WI10362	Standardization of 0.02 and 0.1 Normal Sulfuric Acid
T-WC-WI10360	Quality Control Data for Wet Chemistry
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope

This SOP uses the ManTech Multi-Parameter System for sample analyses. The ManTech Multi-Parameter System is an instrument used for the determination of the following analytes (see table below) in drinking waters, surface waters, and wastewater samples.
The table below lists the name and LIMS ID number of the analyses that are included in this SOP and the method references associated with each.

Table of Analyses and Reference Methods

Analysis Name	LIMS ID	Analysis Method
Acidity to pH 3.7 and pH 8.3 (titrimetric)	305.2	EPA 305.2
Fluoride	340.2 4500_F_C	SM 4500-F C-2011 SM 4500-F C-1997 EPA 340.2
Fluoride (distilled)	340.2_Dist 4500_F_B	SM 4500-F C-2011 SM 4500-F C-1997 EPA 340.2 SW-846 9214
Total Acidity	SM2310B 305.1	SM 2310 B-2011 SM 2310 B-199 EPA 305.1
Specific Conductance	2510B 120.1 9050A	SM 2510 B-2011 SM 2510 B-1997 EPA 120.1 SW-846 9050A
Total Hardness	2340C 130.2	SM 2340C-2011 SM 2340C-1997 EPA 130.2
Carbonate Alkalinity	2320B	SM 2320B-2011 SM 2320B-1997
Bicarbonate Alkalinity	2320B	SM 2320B-2011 SM 2320B-1997
Total Alkalinity	2320B 310.1	SM 2320B-2011 SM 2320B-1997 EPA 310.1
Temperature of pH	170.1 SM2550B	EPA 170.1, SM 2550B-2010
pH	SM4500_H+ 9040C 9040B	SM 4500-H+ B-2011 SM 4500-H+ B-2000 SW-846 9040B SW-846 9040C
pH	150.1	EPA 150.1
Ammonia-Nitrogen	SM4500NH3_D 350.3	SM 4500-NH3D-2011 SM 4500-NH3 D-1997 EPA 350.3
Ammonia-Nitrogen Distilled	SM4500NH3_D 350.3	SM 4500-NH3 D-2011 SM 4500-NH3 D-1997 EPA 350.3
Phenolphthalein Alkalinity	2320B 310.1	SM 2320B-2011 SM2320B-1997 EPA 310.1
Preparation /Calculations		**Method**
Free Carbon Dioxide	SM4500_CO2_D	SM 4500-CO2 D-2011
Hydroxide Alkalinity	2320B	SM 2320 B-2011, SM 2320 B-1997
Fluoride distillation	4500_F_B 340.2_Dist	SM 4500-F B-2011, SM 4500-F B-1997 EPA 340.1
Unionized Ammonia	SM8010F_NH4	SM 8010 F-2011 (Table 8010:VI)

Ammonia Distillation	SM4500NH3_B	SM 4500-NH3 B-2011 EPA 350.3

Basic Principles

Samples are analyzed using the ManTech Multi-Parameter System, which has been designed to test for specific analytes. The system is able to be configured in different ways to optimize sample volumes required, and analysis times. The ManTech system is able to analyze for one parameter or multi-parameters at the same time.

Reference Modifications

The following analyses have reference modifications:

1. Method 2340C - Total Hardness:
    a. The procedures listed in SM 2340C and Method 130.2 are modified by using potassium cyanide (KCN) as the inhibitor instead of sodium cyanide (NaCN).
    b. SM2340C and EPA 130.2 state to dilute 25 mL of sample to 50 mL with reagent water prior to titration. This procedure uses 50 mL sample.
2. Method 4500_F_C, 340.2 – Fluoride and Distilled Fluoride
SM 4500-F-C states that the electrodes are inserted before stirring the sample. For EPA 340.2 begin stirring the sample before the electrodes are inserted.
3. Method SM4500NH#_D, 350.3 – Ammonia-Nitrogen and Distilled Ammonia-Nitrogen
Instead of using NaOH in order to pH adjust the samples during the ammonia analysis, this procedure uses alkaline reagent. Alkaline reagent is a purchased reagent containing NaOH that is currently recommended by the manufacturer of the ammonia electrode.

Interferences

Sample matrix is likely to be the largest cause of interference for each analysis.

Samples containing oily layers, soaps, suspended solids, or precipitates may impede the electrode response. The electrode must be cleaned according to the manufacturer's instructions.

Total Hardness – excessive amounts of heavy metals can interfere, causing a faded or indistinct endpoint by the stoichiometric consumption of EDTA. If this interference is not resolved after multiple dilutions on the ManTech automated procedure, then the sample must be analyzed using the manual procedure. See SM 2340C for more discussion on interferences and how to treat them.

Ammonia Nitrogen – volatile amines are a positive interference. Mercury and Silver also interfere with this procedure. Electrode responds slowly at levels <1 mg NH3-N/L; use longer electrode immersion times.

Fluoride – several polyvalent cations, notably aluminum, iron and silicon interfere by forming complexes with fluoride. Adding TISABIII provides a uniform ionic strength background, adjusts pH and breaks up complexes so that the fluoride ion activity can be measured. Fluoborates are widely used in industrial processes. At concentrated levels, as in electroplating wastes, fluoborates do not hydrolyze to release fluoride without using the Bellack distillation (Analysis 01448) prior to analyzing the sample distillate by ISE. SM 4500 F C-2011 1.b requires to distill fluoride if the Total Dissolved Solids concentration is greater than 10,000 mg/L.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples for pH and Temperature of pH are to be collected in unpreserved glass or plastic containers and analyzed as soon as possible upon receipt at the laboratory. Avoid sample agitation and prolonged exposure to air.

Samples for Total Alkalinity, Total Acidity, and Acidity to pH 3.7 and pH 8.3 are to be collected with minimum aeration and analyzed within 14 days of collection.

Samples for Specific Conductance and Fluoride are to be collected in unpreserved glass or plastic containers and analyzed within 28 days of collection.

Samples for Ammonia must be collected in a glass container, preserved with concentrated sulfuric acid to a pH < 2. Analyze within 28 days of collection.

Samples for Total Hardness must be collected in a glass or plastic container, preserved with nitric acid to a pH < 2. Analyze within 6 months of collection.

All samples must be stored at 0° to 6°C, not frozen. Prior to analysis, samples must be brought to room temperature.

Apparatus and Equipment

1. ManTech Multi-Parameter System instrument:

   The instrument is equipped with systems to analyze for pH, temperature of pH, alkalinity, acidity, conductivity, fluoride, ammonia and total hardness. The methods for each procedure is pre programmed and the analyst will not need to make any adjustments.

   Each test can be performed individually or in tandem with others. The analyst will determine the combination of analyses for each run.

   a. pH Electrode
   b. Fluoride ion selective electrode
   c. Interface
   d. Burivar I/2 (25 mL burette)
   e. TitraRinse pump
   f. TitraRinse slow speed reagent addition pump
   g. Reversible Peri-Max pump
   h. ManTech photometric detector with a 620nm filter
   i. Conductivity/TDS Meter, Model 4510
   j Ammonia electrode
   k. Autosampler - MEGA, 197 sample locations

2. Ammonia Distillation LIMS ID SM4500NH3_B

   a. Kjeldahl distillation rack
   b. Refrigerated recirculator (Coolflow) with a recirculating pressure set at 20 psi and the temperature adjustment set at 10°C, or equivalent.
   c. Glass distillation apparatus with connector and 800 mL Kjeldahl flask
   d. pH indicator strips, purchased
   e. Porous boiling chips, purchased
   f. 500 mL class A graduate
   g. 400 mL (approx.) distillation collection cups
   h. 800 mL (approx.) sample collection cups
   i. Kjeldahl flasks
   j. 5 mL class A pipette

Reagents and Standards

Alternative weights and volumes are acceptable as long as final concentrations remain the same.

A. Reagents and Standards needed for ManTech Analyses

1. Standardized Sulfuric acid, 0.02
    - a. Dilute 0.6 mL of concentrated sulfuric acid to 1000 mL with reagent water.
    - b. Store at room temperature in glass or plastic container and re-evaluate yearly.
    - c. Standardize according to *T-WC-WI10362*.
    - d. Re-standardize titrant before use if more than 30 days have passed since the last standardization date.
2. Sodium hydroxide, 10 N - Purchased; see shelf life for information. Or prepare:
    - a. Prepared - Dissolve 400 g NaOH pellets in 1000 mL volumetric flask containing approximately 800 mL reagent water. Solution becomes very warm.
    - b. Cool to room temperature and dilute to 1000 mL with reagent water.
    - c. Store at room temperature in plastic or glass container and re-evaluate yearly.
3. Sodium hydroxide, 1.0 N
    - a. In a 1-L glass volumetric flask containing approximately 500 mL reagent water, dissolve 40.00 ± 0.05 g NaOH.
    - b. Cool and dilute to volume.
    - c. Store at room temperature in plastic or glass container and re-evaluate yearly.
4. Standardized Sodium Hydroxide, 0.02 N
    - a. In a 1-L glass volumetric flask, add approximately 500 mL reagent water.
    - b. Add 20 mL of 1 N NaOH to the flask and dilute to volume with reagent water.
    - c. Store at room temperature in glass or plastic container and re-evaluate yearly.
    - d. Standardize using instructions as per T-WC-WI11585.
    - e. Re-standardize titrant before use if more than 30 days have passed since the last standardization date.
5. Total alkalinity standard (9400 mg/L as CaCO3)
    - a. Dry sodium carbonate (Na2CO3) for at least 4 hours at approximately 250°C.
    - b. Desiccate approximately 2 hours.
    - c. Dissolve 9.954g ± 0.002g Na2CO3 in reagent water and dilute to 1000 mL in a volumetric flask.
    - d. Solution expires 3 months from the date of preparation.
    - e. Store in a glass container at 0º to 6ºC, not frozen.
    - f. Dilute for other concentrations.
6. pH 4, 7, and 10 buffer solutions- purchased; see container for shelf life information.

NOTE: A second source 7.00 pH buffer solution that is from ISO 17025 approved vendor must be purchased; see container for shelf life information. This is to be used for Methods SM4500_H+, 9040C, 9040B Laboratory Control Standard (LCS). It is also to be used for Methods SM4500_H+, 9040C, 9040B, 2320B and 310.1 Continuing Calibration Verification Standards (CCVs)

7. 1413 µhoms/cm cell constant; purchased. See container for shelf life information
8. 0.1 M KCl (12,900 µhoms/cm) [Solution I]
    - a. In a 1 L glass volumetric flask, dissolve 7.4555 ± .0001 g predried KCl (2 hours at 105° ± 2°C) in approximately 500 mL reagent water.
    - b. Dilute to volume with reagent water.
    - c. Solution expires 3 months from the date of preparation.
    - d. Store at room temperature in a glass or plastic
9. 0.01 M KCL (1413 µhoms/cm) [Solution II

a. In a 1-L volumetric flask, dissolve 0.7456 ± 0.0001 g predried KCl (2 hours at 105° ± 2°C) in approximately 500 mL reagent water.

b. Dilute to volume with reagent water.

c. Solution expires 3 months from the date of preparation.

c. Store at room temperature in a glass or plastic container.

10. 5 umhos/cm low-level check standard

    a. In a 1 L glass volumetric flask, containing approximately 500 mL reagent water, dissolve 0.0025 g of NaCl.

    b. Prepare fresh before use.

11. Fluoride standard stock solution (100 mg/L)

    a. Dissolve 0.2210 ± .0005 g of anhydrous sodium fluoride (NaF) in reagent water and dilute to 1000 mL in a volumetric flask.

    b. Stable for 6 months.

    c. Store in a plastic container at room temperature.

    d. Dilute for other concentrations.

NOTE: A separate standard fluoride stock solution must be prepared using a **different source of NaF** when preparing a calibration curve.

12. TISAB III, purchased. See container for storage conditions.

13. Ammonia nitrogen standard (100 m/L NH3-N)

    a. Dry ammonium chloride (NH4Cl) at 103°C to 105°C for approximately 2 hours.

    b. Desiccate approximately 1 hour.

    c. Dissolve 0.3819 ± 0.0002 g NH4Cl in approximately 800 mL reagent water.

    d. Once dissolved, preserve the solution with concentrated sulfuric acid to a pH < 2. Dilute to 1000 mL in a volumetric flask.

    e. Stable for 6 months.

    f. Store at 0º - 6º C, not frozen in a glass or plastic container.

    g. Dilute as needed for other concentrations.

    NOTE: The Ammonia nitrogen standard used to prepare the calibration curve must be prepared using a **different source** of NH4Cl.

14. Ammonia electrode storage solution- purchased. See container for shelf life information.

15. Standard EDTA titrant (0.02N) - Purchased; see container for shelf life information. Or prepare:

    a. Dissolve 3.723 g ± 0.005 g of disodium EDTA in reagent water and dilute to 1000 mL with reagent water in a volumetric flask.

    b. Stable for 3 months.

    c. Store at room temperature in plastic bottle.

    d. Standardize daily before use. Refer to Calibration section B.1. for instructions for standardizing.

16. pH 10 hardness buffer solution – purchased; see container for shelf life information. Or prepare:

    a. Dissolve 16.9 g ammonia chloride (NH4Cl) in 143 mL of concentrated ammonium hydroxide (NH4OH).

    b. Separately, dissolve 1.179 ± 0.0005 g of disodium EDTA and 0.78 ± 0.005 g of (MgSO4 · 7 H2O) in 50 mL of reagent water (this solution becomes turbid).

    c. Combine the above two solutions and dilute to 250 mL with reagent water in a volumetric flask.

    d. Solution is stable 30 days.

    e. Store at room temperature in plastic container.

17. Calmagite indicator solution (0.1 % w/v) – Purchased; Store at room temperature. See manufacturer's label for expiration date.

18. Calcium reference standard (1000 mg/L Ca = 2497 mg/L CaCO3) – Purchased; store at room temperature. See manufacturer's label for expiration date.

19. 1000 mg/L CaCO3 intermediate calcium standard

    a. Pipette 20.0 mL of purchased reference standard into 50 mL volumetric flask and dilute to volume with reagent water.

    b. Stable for 6 months.

    c. Store at room temperature in glass or plastic container.

20. 15% Sodium Acetate Solution – dissolve 15.0 g of sodium acetate anhydrous in 85 mL of reagent water in a 150 mL beaker. Prepare fresh before use.

21. Total acidity standard (5000 mg/L as CaCO3)

    a. In a 1-L glass volumetric flask containing approximately 500 mL of reagent water; add 20.4000 ± 0.0005 g potassium hydrogen phthalate, dried at 120 degrees C for 2 hours. Cool in desiccator for approximately 1 hour.

    b. Allow to dissolve and dilute solution to volume.

    c. Store at 0°to 6°C, not frozen, in a glass container.

    d. Solution expires 6 months from the date of preparation.

22. Hydrogen peroxide (H2O2, 30% solution) – purchased. Store at room temperature. See label for expiration date.

23. pH 8.00 buffer Solution; purchased. Store at room temperature. See container for shelf life information.

B. **Reagents needed for LIMS ID SM4500NH3_B – Ammonia distillation:**

1. Borate buffer solution – Dissolve 19.0 ± 0.1 g sodium borate, 10-hydrate (Na2B4O7·10 H2O) in approximately 1000 mL of reagent water. Add 176 mL of 0.1N NaOH to the solution and dilute to 2000 mL with reagent water in a volumetric flask. Store at room temperature in a glass container. Solution stable for 6 months.

2. 0.1N H2SO4 – Dilute 3.0 mL of concentrated sulfuric acid to 1000 mL with reagent water in a volumetric flask. Store at room temperature in a glass or plastic container and re-evaluate yearly.

3. 4% H2SO4 Solution – Dilute 380 mL of 0.1N H2SO4 to 1000 mL with reagent water in a volumetric flask. Store at room temperature in a plastic container and re-evaluate yearly.

4. Antifoam B; purchased. Store at room temperature. See manufacturer's label for expiration date.

5. Sodium hydroxide, 5N – Dissolve 200 ± 0.5 g sodium hydroxide (NaOH) pellets in approximately 800 mL of reagent water. Solution becomes very warm. Cool to room temperature and dilute to 1000 mL with reagent water in a volumetric flask. Store at room temperature in plastic container and re-evaluate yearly. (This is used to pH adjust samples.)

6. Sodium hydroxide, 0.1N – Dilute 40 mL of 5N NaOH solution to 2000 mL with reagent water in a volumetric flask. Store at room temperature in a plastic container and re-evaluate yearly. (This is used in borate buffer solution.)

7. Dilute Hydrochloric (HCl) acid – To prepare, add 50 mL of concentrated HCl to approximately 3.5 liters of reagent water. Slowly invert. Store at room temperature in a plastic container. (This is used to rinse tubing prior to steam cleaning the distillation apparatus.)

8. Ammonia-nitrogen standard, 500 mg/L NH3-N – In a 1 L volumetric flask containing approximately 500 mL reagent water, dissolve 1.9095 ± .0002 g NH4Cl (which has been dried at 105° ± 2°C for at least 2 hours). Dilute to 1 L using reagent water. Preserve the solution with concentrated sulfuric acid to a pH <2. Store the standard at 0º to 6ºC, not frozen in a glass or plastic container. Standard expires 6 months from the date of preparation.

C. Standards Preparation Needed for Linearity Check for LIMS ID 2510B, 120.1 and 9050A

1. Prepare Solution I (12,900 µhoms/cm) and II (1413 µhoms/cm) as indicated above under the reagents and standards section.

2. Prepare the following 8 standards in 100-mL volumetric flasks using Solutions I or II as indicated in the table below. Bring to volume with reagent water. Prepare fresh before use.

Solution to use	**Standard**	**mL of Solution**	**umhos/cm**
Solution II	0.0001 M	1	14.94
Solution II	0.0005 M	5	73.9
Solution II	0.001 M	10	147
Solution II	0.005 M	50	717.8
Solution II	0.01 M	100	1,413

Solution I	0.02 M	20	2,767
Solution I	0.05 M	50	6,668
Solution I	0.1 M	100	12,900

Calibration

Calibration is performed daily. It is only necessary to calibrate for analyses that will be performed on that day.

Balances must be calibrated each day before use.

NOTE: For all calibrations, 50 mL of standard is poured into a sample tube. For each analysis, a predetermined and programmed sample volume is used (to be taken from the 50 mL in the sample tube).

NOTE: Recalibration is necessary for each analysis if acceptance criteria is not met.

A. LIMS ID 2510B,120.1, 9050A Conductivity Calibration

1. Determine cell constant daily:

    a. Place 1413 μhoms/cm calibration standard into autosampler rack.

    b. The conductivity cell is rinsed with reagent water in the rinse station.

    c. The cell moves to the 1413 μhoms/cm standard and is dipped 3 times in the solution, remaining fully immersed on the third dip.

    d. Solution stabilizes for 25 seconds. The meter prompts to press calibration key twice.

    e. The cell constant is displayed. Record cell constant in maintenance logbook.

2. Conductivity Meter Linearity Check:

    a. Linearity check of the conductivity meter must be performed on the instrument at least once per year using the following concentrations below. Refer to Reagents and Standards section A for preparation instructions.
    0 .0001M, 0.0005M, 0.001M, 0.005M, 0.01M, 0.02M, 0.05M, 0.1M

    b. Fill tubes with standards in accending order by concentration and schedule the Mantech instrument to perform conductivity on all the standards.

    c. Print out data from standards and keep in designated calibration folder for Conductivity.

    d. Correlation Coefficient must be = 0.995. If this acceptance criteria is not met, the linearity check must be repeated.

B. LIMS ID 2340C, 130.2 - Total Hardness Calibration:

1. Manual Calibration:

    *Standardization of EDTA Titrant:

    a. Weigh 1.000 ± 0.0005 g $CaCO3$ and transfer to a 500-mL Erlenmeyer flask.

    b. Add 1 + 1 HCl gradually until the $CaCO3$ dissolves.

    c. Add 200 mL reagent water and boil for a few minutes to expel $CO2$.

    d. Allow the flask to cool.

    e. Add a few drops of methyl red indicator and adjust to an intermediate orange color with 3 N $NH4OH$ or 1 + 1 HCl, as needed.

    f. Dilute to 1000 mL with reagent water in a volumetric flask. (This solution is 1000 mg/L $CaCO3$.)

    g. Dilute 5.0 mL (pipette) of this solution to 50 mL and titrate to a blue endpoint following the steps in the Procedure Section of this analysis.

    h. Perform duplicate trials. Use the average of two trials to calculate the constant.

$$\textbf{EDTA constant} = \frac{5mL\ CaCO_3}{mL\ of\ EDTA\ titrant}$$

2. Automated Calibration: Not Applicable to this analysis

C. pH Calibration - Analyses that require pH calibration: LIMS ID# 305.2, SM2310B, 305.1, SM4500_H+, 9040C, 9040B, 2320B, 310.1

1. Place pH 4, 7, and 10 buffers into autosampler rack and run a schedule to calibrate pH electrode.
2. Sampler moves to rinse station where the sample transfer line and Titrasip vessel are rinsed with reagent water.
3. Sampler moves to pH 4 buffer position and sample transfer line is primed with pH 4 buffer solution.
4. 10 mL of buffer solution is pumped into Titrasip cell and stirrer is turned on.
5. pH of buffer solution is measured and recorded.
6. Steps b. to e. are repeated for pH buffers 7 and 10.
7. Acceptance criteria: Slope of pH calibration must be within the range of -54.0 to -60.0.mV for pH performed on the ManTech.

8. Record pH electrode ID, instrument ID, Employee ID, and date on calibration print out. Export and attach the calibration to the analytical batch in LIMS. Store in designated calibration folder for pH calibration sheets.

9. Immediately after calibration, analyze one of the pH buffers again. The result must be within ±0.1 pH units of the true value of the buffer.

D. LIMS ID 340.2, 4500_F_C Fluoride Calibration:

1. Curve:

a. Using the fluoride concentrations listed below, analyze a new calibration curve daily.

NOTE: When analyzing samples from the state of North Carolina, each analyst performing the analytical procedure must produce a standard curve (Ref 15A NCAC 2H .0805 (a) (7) (l)).

Concentration (mg/L)	mL Stock Standard (100 mg/L)	Final Volume (mL)
0.10	1	1000
0.50	5	1000
1.0	10	1000
1.5	15	1000
2.0	20	1000

NOTE: Before analyzing distillate batches from LIMS ID 4500_F_B or 340.2_dist, add 1 mL of 15% of Sodium Acetate to all curve standards.

b. Place calibration standards 0.10, 0.50, 1.0, 1.5, 2.0 mg/L, into autosampler rack in ascending order and run a schedule to calibrate fluoride probe.

1.) Fluoride curves for distillate batches (from LIMS ID 4500_F_B or 340.2_dist), must have 1 mL of 15% of Sodium Acetate added to all curve standards.

c. Sampler moves to rinse station where sample transfer line and Titrasip vessel are rinsed with reagent water.

d. Sampler moves to first standard position.

e. Sample transfer line is primed with standard solution.

f. 30 mL of standard and 3 mL of TISAB is pumped into the Titrasip vessel, and stirrer is turned on.

g. Fluoride concentration is measured and recorded.

h. Steps c. – g. are repeated for remaining standards.

i. Acceptance criteria: The slope of the curve must be between -54.0 to -60.0 mV.

j. Record pH electrode ID, instrument ID, TISAB III lot number, calibration standards documentation, Employee ID and date on calibration print out. Store in designated calibration folder.

k. Export and attach the curve to the analytical batch in LIMS.

E. LIMS ID SM4500NH3_D, 350.3 Ammonia Calibration

1. Curve: Prepare a calibration curve daily using the concentrations listed below.

The curve must be analyst specific.
Conc. (mg/L NH3-N) mL 100 mg/L ® 100 mL

0.1	0.1
0.5	0.5
2.5	2.5
5.0	5.0
10.0	10.0

a. Ensure electrode is filled to line with appropriate fill solution (NH4Cl).

b. Dilute stock standard to obtain the following concentrations: 0.1, 0.5, 2.5, 5.0, 10.0 mg/L and place calibration standards into the sampler rack in ascending order, and pH 4 buffer into dip rinse (second rinse) position.

c. Sampler moves to flowing rinse station where electrode is rinsed with reagent water.

d. Sampler moves to dip station and electrode remains immersed in pH 4 buffer for 15 seconds.

e. Sampler moves to flowing rinse station where electrode is rinsed again with reagent water.

f. Sampler moves to first calibration standard position and stirrer is turned on.

g. 0.45 mL of ISA (10 N NaOH) is added to sample to adjust pH to 11 or greater, ensuring all NH4 is converted to NH3.

h. Ammonia electrode is allowed to stabilize and reading is recorded.

i. Steps c. - h. are repeated for remaining calibration standards.

j. Acceptance criteria: The slope of the curve must be between -54.0 to -60.0 mV

k. Record pH electrode ID, instrument ID, Employee ID, and date on calibration print out. Store in designated calibration folder.

l. Export and attach the curve to the analytical batch in LIMS.

Procedure

I. Preparation Procedures: Ammonia Distillation

A. LIMS ID SM4500NH3_B - Ammonia Distillation (for use with LIMS ID SM4500NH3_D)

1. Before each distillation, rinse tubes with dilute HCl. Add approximately 500 mL of reagent water to an 800-mL Kjeldahl flask to steam clean the distillation apparatus.

2. Connect Kjeldahl flask to glass adapter and hose. Turn on the Coolflow. Turn on the temperature control and distill until approximately 150 mL of water is collected in the beaker at the bottom of the setup. Remove distillate from delivery tube immediately after turning off heat source to avoid back wash during the cool-down phase.

3. Measure 500 mL of sample (or an aliquot diluted to 500 mL with reagent water) using a Class A graduated cylinder. Pour into a beaker and adjust the pH of the sample to 9.5 with 5N NaOH. Check pH of adjusted sample with purchased pH strips to ensure the pH is 9.5, and then add 25 mL of borate buffer solution.

4. Transfer the solution to an 800-mL Kjeldahl flask. Add a pinch of boiling chips and approximately 5 mL of antifoam B to the solution. Connect flask to glass adapter and hose. Turn on the Coolflow.

5. Measure 50 mL of 4% H2SO4 solution into a 400-mL beaker. Make sure the delivery tube is below the level of the 4% H2SO4 solution (receiving solution).

6. Begin heating. Collect approximately 220 mL of distillate. Distill at a rate of 6 - 10mL/min. Turn off heat and immediately remove the distillate beakers from the delivery tube. Turn off the Coolflow.

7. Pour the distillate into a 500-mL Class A graduated cylinder and dilute to final volume of 500 mL with reagent water. Store distillate at 0°C - 6°C; not frozen, prior to analyzing

8. Analyze distillate for NH3-N according to Method SM4500NH3_D procedure.

II. ManTech Procedure:

NOTE: Before operating the analyst must ensure that all required reagents and reagent water containers needed for each test are in adequate supply and are not expired.

A. To operate the instrument, open the PC-titrate software menu from the computer desktop.

Each day before any analysis is performed; the daily calibration associated with each analysis must be completed and evaluated to ensure that the acceptance criteria are met. (See the calibration section for the required concentrations and acceptance criteria for each calibration). Daily calibration schedules are created and saved. If a specific analysis is not being performed that day, the calibration can be removed from the schedule for that day.

B. Performing daily calibration:

Open the daily start up tab and load the standards according to the schedule for each calibration. Click on the start button and the instrument will proceed with the calibration. Once the calibration is complete the calibration data is automatically sent to the assign printer and is printed. Ensure the calibration data meets requirements before proceeding. See the calibration section for the required concentrations and acceptance criteria for each calibration. Store calibration sheets in designated ManTech folders.

C. Analyzing samples:

NOTE: Before analyzing distilled fluoride samples (requiring Method 4500_F_C with 4500_F_B), add 1 mL of 15% of Sodium Acetate to all standards (including the curve), samples, and Blank.

NOTE: For Fluoride, the LCS must be at or below the Maximum Contaminant Level (MCL) when analyzing regulatory drinking waters.

NOTE: When referencing EPA 150.1, perform additional measurements on successive volumes of sample until the values differ by **less than** 0.1 pH units (two or three measurements are usually sufficient).

To analyze samples, select "Run titration" from the PC-titrate menu. A blank schedule will appear.

1. Click on the add row buttons to add the required number of rows for that run.

2. Double click on the blank space under the schedule header. The menu of analyses will appear. Select the analyses to be performed and click ok.

3. When completing the schedule, each analysis must be bracketed by its own Continuing Calibration Verification standard (CCV) of alternating concentrations and a Continuing Calibration Blank (CCB). The CCV's and CCB's ID's are pre–programmed and are to be scanned in from the charts provided at each instrument. Barcodes are provided for each CCV and CCB. See Table I.

4. The Laboratory Control Standard (LCS) for each analysis is pre-programmed and is to be scanned in from the charts provided at each instrument. See Table I.

5. Scan the sample numbers directly from the label of the container. Sample numbers and QC identifiers can be typed. Use a "^" to designate the dilution factor. 410-12345-6^10 indicates a 10 times dilution.

6. Fill the sample tubes with Method Blank, LCS, each CCV, CCB and samples. Ensure the sample tubes are loaded according to the order on the schedule.

7. Click on the auto-generate order number to add the order number to the schedule.

8. Once the schedule is completed, click start and the instrument will proceed with the analysis until the entire schedule of samples is complete.

9. Save the data and send to the PC –Titrate export file before importing to the LIMS system.
10. Create PDFs of all calibrations and raw data associated with the batch. Attach the PDFs to the "Docs" tab in the batch in the LIMS.

D. Routine Maintenance of ManTech multi-parameter system

1. Calibration of Titrasip Pumps: Flow Rate Check:

   a. Manual Control of Autosampler

      (1) From the PC-Titrate main menu, click on Titrator, then on Manual Control (autosampler tab)

      (2) Next, click on Load Tray From Folder button and then double click on Automax 197 Tube Sampler file

      (3) Then, under Zone select tubes. Under Tubes select tube position and place a tube in that position on the autosampler tray.

      (4) Click on Home Sampler

      (5) Click on Go To This Location XYZ button. The autosampler will then move to the tube position.

      (6) Click OK button in order to get back to the PC-Titrate main menu

   b. Flow Rate Check:

      (1) From the PC-Titrate menu select the pump to be calibrated (Click on Pump Cal button) and follow the prompts.

      (2) Calibration must be performed monthly and documented in the maintenance logbook.

   NOTE: When completed, Home sampler (follow instructions under II. ManTech Procedure D1a. steps 1-4)

2. Calibration of Zero and Span for the Hardness detector and monthly buffer volume check for Hardness Detector

NOTE: Turn on Hardness Detector in order for the detector to warm up before calibration.

NOTE: Ensure reagent water is in titration vessel cup containing the photo cell

a. Accessing Zero and Span Check screen and set-up:

(1) From the PC-Titrate main menu, click on Titrator, then on Manual Control

(2) Click on Analog

(3) Click on Scan Analog, making sure the continuous button is checked

(4) The Hardness Detector is on Monitor Electrode # 4

b. Zero Check/Calibration:

(1) Unscrew the Zero (careful not to look directly into the bright light). The Zero must be as close as possible to 0 mV.

(2) If not, adjust the screw for Zero on the hardness detector until reading is as close as possible to 0 mV.

(3) Check/Calibration must be performed monthly and documented in the maintenance logbook.

c. Span Check/Calibration:

(1) The Span reading must be -400 mV ± 25 mV.

(2) If not, adjust the screw for Span on the hardness detector until reading is within range.

(3) Check/Calibration must be performed monthly and documented in the maintenance logbook.

d. Monthly buffer volume check on Hardness Detector:

**Perform two trials:

(1) Place buffer dispenser titrant line into a 10 mL graduated cylinder.

(2) From the PC-titrate main menu, click on run titration.

(3) Following steps outlined in procedure section C above, schedule a vial of reagent water to be analyzed for Hardness and click on START.

(4) Once the buffer has been dispensed into the graduated cylinder, click on STOP on the computer screen.

(5) Record the amount of buffer solution collected in the graduated cylinder in the maintenance logbook for the instrument. Repeat steps 1-5 once more.

(6) Average two trials and record average result in maintenance logbook

(7) Acceptance criteria is 2.0 mL +/- 0.1 mL.

(8) If averaged amount falls outside the acceptance range, then repeat the buffer volume check. If the check fails again then seek guidance from supervisor.

3. Changing and Entering Acid Normalities on ManTech:

a. Changing Titrant –Purging titrant: Titrant must be changed each time a new stock of titrant is prepared.

NOTE: Disconnect acid titrant bottle from dispenser line

(1) Click on PC-Titrate, Titrator, Manual Control, Serial Devices

(2) Click on Burivar II TIS1 tab; Titrasip pump#1/Burette #1

(3) Purge Buret one time (empty cup-use manual button on side of pump #1 at the top of cup)

(4) Fill titrant bottle with new standardized 0.02N H_2SO_4 titrant and connect dispenser line to bottle

(5) Purge system two more times

(6) Click OK

b. Entering Acid Normalities: Acid Normalities must be updated in ManTech software each time the acid is standardized and the standardization yields a new normality.

(1) Click on PC-Titrate, Set-up, Titration Method, Load

(2) Click Alkalinity

(3) Under Titrant Information-Concentration, change Normality to current value.

(4) Click Save

(5) Click Alkalinity-Low and follow steps (3) and (4) above

E. Maintenance of all other systems:

1. Maintenance of the Ammonia Ion-Selective Electrode – See manufactures instruction manual for maintenance and operation of electrode

2. Maintenance of pH electrodes- refer to *T-WC-WI11519* for maintenance of pH electrodes

3. Perform Maintenance on all other systems as needed.

F. Retrieving Historical Raw Data on ManTech:

1. Find run number of sample in question.

2. Open PC-titrate software menu from computer desktop

3. Click on Titrator, Replay Titration, Load, Sort by order and sample name

4. Click on sample (go by time), Select, Print, Equation Result Report (rplyeqn.srw), Print.

Calculations

A. Fluoride, pH, Temperature of pH, Conductivity, and Ammonia are measured directly and therefore no calculations are needed.

B. Alkalinity: (LIMS ID 2320B, 310.1)

To pH of 8.3:
Alkalinity, mg/L $CaCO_3$ = (mL of titrant pH 8.3) x Normality x 50,000) / mL of sample

To pH of 4.5:

Alkalinity, mg/L $CaCO_3$ = (Total mL of titrant x Normality x 50,000) / mL of sample

C. Low Alkalinity (< 20 mg/L $CaCO_3$):

[(2 X B - C) X N X 50,000] / mL of sample

where:
B = mL titrant to first pH
C = Total mL titrant to pH 0.3 units lower
N = Normality of 0.02 N H_2SO_4

D. Carbonate, Bicarbonate, and Hydroxide Alkalinity are calculated from the total alkalinity result and the phenolphthalein alkalinity value (LIMS ID 2320B)

Phenolphthalein Alkalinity Value	Bicarbonate Alkalinity Calculation	Carbonate Alkalinity Calculation	Hydroxide Alkalinity Calculation
< MDL	Total Alkalinity	< MDL	< MDL
< 1/2 x Total Alkalinity	Total Alkalinity - (2 x Phenolphthalein Alkalinity)	2 x Phenolphthalein Alkalinity	< MDL
= 1/2 x Total Alkalinity	< MDL	2 x Phenolphthalein Alkalinity	< MDL
> 1/2 x Total Alkalinity	< MDL	2 x (Total Alkalinity - Phenolphthalein Alkalinity)	(2 x Phenolphthalein Alkalinity) - Total Alkalinity
= Total Alkalinity	< MDL	< MDL	Total Alkalinity

E. Free Carbon Dioxide Calculation (LIMS ID SM4500_CO2_D)

The calculation assumes that the total alkalinity of the sample is due almost entirely to hydroxides, carbonates or bicarbonates.

The total dissolved solids of the sample may not be greater than 500 mg/L for this calculation.

mg/L CO_2 = 2.0 x B x 10^(6-pH)

Where:
B = bicarbonate alkalinity (from LIMS ID 2320B)

NOTE: Total carbon dioxide can be calculated, if requested, according to the calculations found in SM 4500 CO2 D-2011

F. Total Hardness (LIMS ID 2340C, 130.2):
Hardness (mg/L as $CaCO_3$) = [(mL EDTA used x 1000)/ mL of sample] x EDTA Constant

G. Un-ionized Ammonia (LIMS ID SM8010F_NH4):

mg un-ionized ammonia/L = (ammonia nitrogen raw result mg/L) x (% un-ionized ammonia at a given pH and temperature)

Refer to table below for % un-ionized ammonia:

Table 8010: VI.
Percentage of Ammonia Un-ionized in Distilled Water*

Temp °C	**Percentage Un-ionized at a given pH***								
	6.0	6.5	7.0	7.5	8.0	8.5	9.0	9.5	10.0
5	0.01	0.04	0.11	0.40	1.1	3.6	10	27	54
10	0.02	0.06	0.18	0.57	1.8	5.4	15	36	64
15	0.03	0.08	0.26	0.83	2.6	7.7	21	45	72
20	0.04	0.12	0.37	1.2	3.7	11.7	28	55	80
25	0.05	0.17	0.51	1.7	5.1	14	35	63	84
30	0.07	0.23	0.70	2.3	7.0	19	43	70	88

*Prepared from data given in Sillen and Martell.

H. Total Acidity (LIMS ID SM2310B, 305.1)

$$\text{Acidity as mg / mL } CaCO_3 \quad \frac{[(A \times B) - (C \times D)] \times 50{,}000}{\text{mL of sample}}$$

Where:
A = Vol. of std. NaOH used in titration
B = Normality of std. NaOH
C = Vol. of std. H_2SO_4 used to reduce pH to less than 4
D = Normality of std. H_2SO_4

I. Acidity to pH 3.7 and pH 8.3 (LIMS ID 305.2)

mg/L as $CaCO_3$ = (Normality)(50,000)(mL of titrant) / (mL of sample volume)

Statistical Information/Method Performance

The method detection limit (MDL) is determined annually by following the procedure outlined in *QA-SOP11892*.

The quality control acceptance windows are generated annually by following the procedure outlined in *QA-SOP11896*.

Quality Assurance/Quality Control

The batch size is limited to 20 samples or less. For analyses with more than 10 samples on a batch, two duplicates are needed (excluding ammonia and fluoride – only one duplicate is needed per batch 20 samples –unless a batch contains an NPDES sample(s) from South Carolina and the batch contains more than 10 samples, then two duplicates are needed).
A Laboratory Control Standard (LCS) must be analyzed for each analysis per batch. See Table I for each analysis LCS concentration.

For alkalinity, Total Acidity and total hardness one matrix spike (MS) must be analyzed per batch of 20 samples or less.

For ammonia, fluoride and Acidity to pH 3.7 and 8.3 (total acidity spikes only), a matrix spike (MS) and a matrix spike duplicate (MSD) must be analyzed per batch of 20 samples or less.

When sample volume does not permit the analysis of either a sample matrix duplicate or an MS and MSD, then a LCSD must be analyzed to demonstrate method precision.

For Fluoride, pH, Ammonia by ISE, and specific conductance a Certified Reference Material (CRM) must be analyzed annually. The acceptance range for the CRM is 90% to 110% of the true value.

Total Acidity (LIMS ID#4530) samples with results > 600 mg/L as $CaCO_3$ must be diluted and reanalyzed.
Samples from West Virginia require a usable precision measurement for each batch.

Analyzing one of the following on a batch containing a West Virginia sample fulfills this requirement: an MS/MSD, a non-program deleted duplicate, or an LCSD.

For Specific Conductance, if the batch contains samples from the state of North Carolina a low-level check standard of 5 umhos/cm NaCl must be analyzed.

For Total Hardness, samples that are designated NPDES require digestion. Confirm whether this analysis is appropriate for samples designated NPDES.

For Fluoride, the LCS must be at or below the MCL when analyzing regulatory drinking waters.

For each analysis, a continuing calibration verification (CCV) standard must be analyzed at the beginning of each batch, after every 10 samples and at the end of each batch. The concentration of the CCV must alternate in a repeating wave pattern. All CCVs must have an acceptance criteria of 90% to 110%. If any CCV's are outside this range, then the samples bracketed by the out of specification CCV must be repeated. Refer to Table I for CCV concentrations for each analysis.

A continuing calibration verification blank (CCB – a reagent water blank) must be analyzed after each CCV standard on the batch. The CCB must be less than the LOQ for the analysis.

See LIMS for current quality control (QC) acceptance windows. Follow guidelines in *T-WC-WI10360* for outlier QC data.

Table I

Analysis Name and Number	CCV Barcodes/Concentrations	Frequency	Acceptance Criteria
Total Alkalinity (12150) Carbonate (12148) Bicarbonate (12149)	CCVPH2 = 7.00 pH buffer (ISO 17025 approved) LCSAK = 188 mg/L LCSPH = 7.00 pH buffer (ISO 17025 approved) PBW = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Total Acidity (4530)	CCVPH8 = 8.00 pH buffer LCSHACD = 250 mg/L PBW = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Acidity to pH 3.7 and pH 8.3 (0475, 0476)	CCVPH8 = 8.00 pH buffer LCSACD = 250 mg/L PBW = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
pH (12152) pH (15052) Temperature of pH (12151)	CCVPH2 = 7.00 pH buffer (ISO 17025 approved) LCSPH = 7.00 pH buffer (ISO 17025 approved) CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Specific Conductance (12146)	CCVSC1 = 147 uhoms/cm CCVSC2 = 1413 umhos/cm CCVSC3 = 12,900 umhos/cm LCSSC = 147 umhos/cm PBW = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Fluoride (12678, 2200)	CCVFL1 = 0.5 mg/L CCVFL2 = 1.0 mg/L CCVFL3 = 1.5 mg/L LCSFL = 1.0 mg/L PBW = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Ammonia (12677) Distilled Ammonia (12679)	CCVNH1 = 1.0 mg/L CCVNH2 = 2.5 mg/L CCVNH3 = 7.5 mg/L LCSNH = 5 mg/L PBWNH = Method Blank (12679-distilled) PBW = Method Blank (12677) CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows
Total Hardness (12147)	CCVTH1 = 20 mg/L CCVTH2 = 40 mg/L CCVTH3 = 60 mg/L LCSTH = 40 mg/L PBWTH = Method Blank CCB = reagent water blank	CCV/CCB - beginning/end of batch, after every 10 samples	CCVs ±10% of true value PBW/CCBs <LOQ LCS - see LIMS for current QC acceptance windows

---

QA-SOP11188 Reagents and Standards
QA-SOP11880 Balance, Syringe, Pipette, Weights and Labware Verification
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
QA-SOP11913 Laboratory Notebooks, Logbooks, and Documentation
T-WC-WI10348 Fixed Volume Hand-Held Pipettes
T-WC-WI10360 Quality Control Data for Wet Chemistry
T-WC-WI10362 Standardization of 0.02 and 0.1 Normal Sulfuric Acid
T-WC-WI10425 Bellack Distillation for Fluoride in Waters and Solids by SM 4500 F B-2011, EPA 340.1 Procedure 6.1 or SM 4500 F B-1997
T-WC-WI11519 pH Probes and Meters
T-WC-WI9862 Accumet Model AB30 pH/Ion/Conductivity Meter
T-WC-WI9897 Adjustable Volume Handheld Pipettes
T-WC-WI9898 Calibration of Hach 2100AN Turbidimeter

End of document

Version history

Version	Approval	Revision information	
13	16.JUL.2019		
14	20.MAR.2020		
15	22.APR.2021		

eurofins	Always check on-line for validity. **Anions by Ion Chromatography in Waters and Soil by EPA 300.0, SW 846 9056, and SW 846 9056A**	Level: **Work Instruction**
Document number: **T-WC-WI11626**		
Old Reference: **1-P-QM-WI-9011637**		
Version: **23**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **30-APR-2021**	Document users: **6_EUUSLA_Instrumental Water Quality _IC Analyst, 6_EUUSLA_Instrumental Water Quality _IC Verifier**	Responsible: **5_EUUSLA_Instrumental Water Quality_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
References
Cross Reference
Scope
Basic Principles
Interferences
Safety Precautions and Waste Handing
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Quality Control Standards
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control
Table I
Table II

Revision Log

Revision: 23		**Effective Date:** This version
Section	**Justification**	**Changes**
Throughout document	Current process	Updated to current LIMS IDs Added TALS reagent IDs Removed low level reagents
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	Current Instrumentation Process	Removed unnecessary sample information that was used for old instrumentation.
Title	Enhancement	Removed 'Determination of'
Procedure 7	Current process	Samples and Standards are diluted with eluent, not DI
Quality Assurance, Quality Control A5	Enhancement	changed "must" to "should"

Revision:	22	**Effective Date:**	This version

Revision:	22	**Effective Date:**	This version

Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Throughout Document	Analysis Clarification	Removed unused analysis and added the DOD analysis to document
Throughout Document	Update Reference SOPs	Updated to current D4 numbers
Calibration and QC standards	Enhancement and traceability	Rewrote sections and updated standard names
Quality Assurance/ Quality Control	Method Change	Updated the frequency of calibration
Quality Assurance/ Quality Control	Analysis update	Added the source and procedure for preforming the matrix Spike

References

1. Method 300.0, Methods for Chemical Analysis of Water and Wastes USEPA 600, Revision 2.1, 1993

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 9056, September 1994.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 9056A, February 2007

4. ICS-1000, *Chromatography System Operator's Manuel*, Dionex Corporation, Document No.031879, Revision 02, September 2005.

5. Method 4110, *Standard Methods for the Examination of Water and Wastewater*, 21st Edition, 2005, pp. 4-3 to 4-7.

6. Chromeleon®, *Chromatography Management System*, Tutorial and User Manual 6.70, April 2005, or 6.80, March 2007, Dionex Corporation.

7. *Installation Instructions and Troubleshooting Guide for the Ionpac AG14 Guard Column and AS14 Analytical Column*, Dionex Corporation, Document No. 031199, Revision 04, December 10, 1998.

8. *Installation Instructions and Troubleshooting Guide for the Anion Self-Regenerating Suppressor (ASRS-ULTRA)*, Dionex Corporation, Revision 04, Document No. 031367, April 8, 1999.

9. *Chemical Hygiene Plan*, current version.

10. ICS-1100, *Chromatography System Operator's Manual*, Dionex Corporation, Document No. 065289, Revision 01, March 2009.

11. Department of Defense Quality Systems Manual (DOD QSM) revision 5.3, 2019

Cross Reference

Document	Document Title
T-WC-WI10083	Reagent Water Extraction (as Preparation for Analysis on the Ion Chromatograph or Autoanalyzer)
T-WC-WI11652	Quality Control for Analyses Preformed in Insturmental Water Quality
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11886	Processing Regulatory Compliance (i.e. SDWA, NPDES) Samples
QA-SOP11188	Reagents and Standards

Scope

This method covers the determination of the following inorganic anions in reagent water, drinking water, surface water, wastewater, and groundwater using an ion chromatography system: bromide, chloride, fluoride, nitrate-N, nitrite-N, and sulfate. The limits of quantitation and method detection limits are found in the analysis information file.

This method is used on solids after a reagent water extraction. See analysis 1352. The limits of quantitation for anion analysis of a solid are also found in the analysis information file.

For EW samples: MCL violations must be reported within regulatory guidelines. Refer to QA-SOP11886 for MCLs and reporting requirements.

Basic Principles

A small volume of sample is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, separator column, suppressor, and conductivity detector. Separations are due to differences in the equilibrium distribution of sample components between the mobile phase (eluent) and stationary phase (column exchange resin). Retention times for various anions depend on several factors such as ionic charge, ionic size, pH, resin type, eluent concentration, and flow rate.

Interferences

Interferences occur when substances with retention times that are similar to and overlap those of the anion of interest are present. Often, large amounts of an anion interfere with the peak resolution of an adjacent anion. Use sample dilution to solve most interference problems.

Potential sources of interference which lead to false readings or elevated baselines are contaminated reagents, glassware, or other sample-processing apparatus. Samples containing particulate matter >0.45 microns require filtration to prevent damage to instrument columns and flow systems. If any samples on a batch are filtered, the corresponding PBW and LCSW must also be filtered.

Known coelution is caused by carbonate and other small organic anions such as acetate and formate. At certain fluoride concentrations or in certain sample matrices, this interference is not significant. It is the analyst's responsibility to perform a spike in any sample matrix that has interferences potentially affecting the fluoride determination. If repeatability and accuracy are not obtained in the sample matrix, it is recommended that this method not be used for fluoride.

Safety Precautions and Waste Handing

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

There are no special safety precautions for this method. Follow routine laboratory safety steps. Discard all acid waste in the acid waste containers.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst’s training records.

Initially, each technician performing these techniques must work with an experienced technician for a period of time until they can independently perform the procedure. Proficiency is measured through an Initial Demonstration of Capability (IDOC).

The IDOC and the DOC consists of four laboratory control spikes (or alternatively, one blind sample for the DOC) that are carried through all steps of the procedure and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation.

Sample Collection, Preservation, and Handling

Samples must be collected in unpreserved, clean glass or polyethylene containers. Samples for fluoride must be collected in plastic. Samples must be stored at 0° to 6°C, not frozen. The holding time for samples is 28 days except for samples requiring nitrate nitrogen and/or nitrite nitrogen analysis. Samples requiring one or more of these analyses must be run within 48 hours of collection.

Waters – Samples with particulate matter >0.45 microns must be filtered before analysis. The corresponding LCSW and PBW of filtered samples on a batch must also be filtered.

Blanks (rinse blanks – RB, equipment blanks – EB, field blanks – FB) and GLP samples must initially be run at a dilution factor of one. Most other samples are initially analyzed at a dilution factor of five, which increases the reporting limits by a factor of five. The analyst must increase this dilution factor further (if necessary) to obtain raw results that are less than the most concentrated calibration curve standard for the anion(s) of interest.

NOTE: Refer to Project Notes in Parallax for client/project specific requirements for each sample.

Highly alkaline or highly acidic samples must be neutralized before injection. Often, sample dilution is used in lieu of neutralization.

If appropriate, samples are prescreened for chloride and sulfate. For sulfate, add approximately 0.2 to 0.3 g of $BaCl_2$ using a measuring spoon (1 scoop) to one culture tube of sample. The amount of sulfate in the sample is estimated based upon the resulting turbidity. For chloride, add a few drops of 0.1 N $AgNO_3$ to one culture tube of sample. The chloride is estimated based upon the resulting turbidity. For nitrate nitrogen and nitrite nitrogen, estimate the concentration of the sample using nitrite/nitrate test strips.

Apparatus and Equipment

1. Dionex system ICS-1000, or ICS-1100 Ion Chromatograph
2. PC, with Windows XP (or equivalent)
3. Hard disk, monitor, keyboard, mouse, and printer
4. Chromeleon® software, Version 6.70 (or newer), Dionex Corporation, 2005
5. Lancaster Laboratories' LIMS system with IDAT
6. Automatic sampler
7. Anion guard column, 4 × 50 mm, Dionex AG14, or equivalent
8. Anion analytical column, 4 × 250 mm, Dionex AS14, or equivalent
9. Anion suppressor, Dionex anion self-regenerating suppressor, ASRS-Ultra, or equivalent
10. Conductivity cell detector
11. Analytical balance, capable of accurately weighing to 0.0001 g

Reagents and Standards

NOTE: All chemicals used must be ACS reagent grade unless otherwise noted. See QA-SOP11188 for the appropriate labeling and documentation of reagents and standards. It is acceptable to use different volumes or weights in the preparation of reagents or standards as long as the ratios remain equivalent.

1. Concentrated eluent solutions, 0.5 M $NaHCO_3$ (WC_IC_BCrbSTK) and 0.5 M Na_2CO_3 (WC_IC_CrbSTK)

Sodium bicarbonate ($NaHCO_3$)	42.00 ± 0.05 g

Sodium carbonate (Na2CO3)	53.00 ± 0.05 g

a. Prepare separate stocks for each component by dissolving each reagent listed above in reagent water and diluting to 1 L in a volumetric flask.

b. Store stock solutions in amber glass at 0° to 6°C not frozen.

c. Stable for 30 days.

2. Working eluent for AS14 column, 1.0 mM NaHCO3/3.5 mM Na2CO3

   a. Combine 32 mL of sodium bicarbonate concentrated eluent and 112 mL of sodium carbonate concentrated eluent.

   b. Dilute the solution to 16 L with reagent water.

   c. Store in plastic carboy. Stable for 7 days at room temperature.

3. BaCl2 (anhydrous) – Purchased. Store at room temperature. See label for expiration date.

4. 0.1 N AgNO3 – Purchased. Store at room temperature. See label for expiration date.

5. Nitrate/Nitrite test strips – Purchased. Store at rom temperature. See label for expiration date.

Calibration

Calibration

A. Stock standards for calibration - These anion standards are purchased with a concentration of 1000 mg/L each. Store purchased standards at room temperature. See label for expiration date. As an option, below is the procedure for preparing stock standards:

Cal Standard 1

Sodium fluoride (NaF)	2.2100 g
Potassium nitrate(KNO_3)	7.2179 g
Sodium nitrite($NaNO_2$)	4.9257 g

Cal Standard 2

Sodium chloride (NaCl)	1.6485 g

Cal Standard 3

Potassium bromide (KBr)	1.4893 g

Cal Standard 4

Sodium sulfate(Na_2SO_4)	1.4787 g

a. Dry each of the above compounds at 105°C ± 5°C for 30 minutes (± 5 minutes). Weigh each compound to ± 0.0003 g.

b. Weigh out and quantitatively transfer into a 1000-mL volumetric flask.

c. Dilute to volume with reagent water. The concentration of each of the standard solutions is 1000 mg/L. Store at 0° to 6°C not frozen. The solutions are stable for 30 days.

NOTE: Salt standards that are stored in a dessicator must be covered with parafilm, not ground glass stoppers.

B. Waters and Soils Calibration Standards

1. Detection limit standard (WC_IC_C_Det)

Anion (1000 mg/L stock)	Initial Volume (mL)	Final concentration (mg/L)
Fluoride (WC_IC_C_F)	0.5	10
Nitrite (WC_IC_C_NO2)	0.5	10
Nitrate (WC_IC_C_NO3)	0.5	10
Chloride(WC_IC_C_Cl)	2.0	40
Bromide (WC_IC_C_Br)	2.5	50
Sulfate (WC_IC_C_SO4)	5.0	100

a. Using a volumetric pipette, add the above volumes to a 50-mL volumetric.

b. Dilute to volume with reagent water.

c. Prepared fresh each time the instrument is recalibrated and store at room temperature.

2. Intermediate calibration standard (WC_IC_C_Int)

Anion (1000 mg/L stock)	Initial Volume (mL)	Final concentration (mg/L)
Fluoride (WC_IC_C_F)	0.5	10
Nitrite (WC_IC_C_NO2)	0.5	10
Nitrate (WC_IC_C_NO3)	0.5	10
Chloride (WC_IC_C_Cl)	1.0	20
Bromide (WC_IC_C_Br)	2.5	50
Sulfate (WC_IC_C_SO4)	2.5	50

a. Using a volumetric pipette, add the above volumes to a 50-mL volumetric.

b. Dilute to volume with reagent water.

c. These standards are stable for 24 hours at room temperature.

3. Working calibration standards and continuing calibration verification (CCV)

Pipette the specified volume of the appropriate calibration standard into a volumetric flask. Dilute to volume with reagent water. Prepare fresh daily according to the table below:

Regular Anion				Mg/L - Anions			
	Std	Std Volume (mL)	Final Volume (mL)	F/ NO_2, NO_3	Cl	Br,	SO_4
Cal 1	Det. Limit Std	0.5	50	0.1	0.4	0.5	1.0
Cal 2	Int. Cal Standard	2.0	50	0.4	0.8	2.0	2.0
Cal 3	Int. Cal Standard	5.0	50	1.0	2.0	5.0	5.0
Cal 4	Int. Cal Standard	10.0	50	2.0	4.0	10.0	10.0
Cal 5	Int. Cal Standard	15.0	50	3.0	6.0	15.0	15.0
CCV/LCS	Int. QC Standard	15.0	50	0.75	3.0	7.5	7.5

NOTE: A reagent water blank is analyzed prior to the calibration and must contain no anions of interest. Therefore all anions must be< the MDL or re-calibration must occur.

Quality Control Standards

A. QC Standards – pipet the below standards in 50-ml volumetrics and bring to volume with reagent water. Stable for 24 hours at room temperature.

1. Soil QC Standard (WC_IC_DISPIKE)

Anion (1000 mg/L stock)	**Initial Volume (mL)**	**Final concentration (mg/L)**
Fluoride (WC_IC_Q_F)	**0.5**	**10**
Nitrite (WC_IC_Q_NO2)	**0.5**	**10**
Nitrate (WC_IC_Q_NO3)	**0.5**	**10**
Chloride (WC_IC_Q_Cl)	**1.0**	**20**
Bromide (WC_IC_Q_Br)	**2.5**	**50**
Sulfate (WC_IC_Q_SO4)	**2.5**	**50**

2. Intermediate QC standard (WC_IC_QC2)

Anion (1000 mg/L stock)	**Initial Volume (mL)**	**Final concentration (mg/L)**
Fluoride (WC_IC_Q_F)	**0.25**	**5**
Nitrite (WC_IC_Q_NO2)	**0.25**	**5**
Nitrate (WC_IC_Q_NO3)	**0.25**	**5**
Chloride (WC_IC_Q_Cl)	**1.0**	**10**
Bromide (WC_IC_Q_Br)	**2.5**	**50**
Sulfate (WC_IC_Q_SO4)	**2.5**	**50**

B. Working control standards

1. ICV/CCV2 and LCSW:

   a. Pipette 7.5 mL of the intermediate QC standard into a 50-mL volumetric flask, which contains about 80-mL reagent water.

   b. Dilute to volume with reagent water.

   c. This standard must be prepared daily. True values of this standard are:

NO2, NO3, F	0.75 mg/L
Cl	3.0mg/L
Br, SO4	7.5 mg/L

2. The LCSS is spiked before digestion of samples using the Soil QC Standard. See the preparation SOP T-WC-WI10083.

Procedure

1. Check chromatography setup for proper installation according to the following flow diagram:

Anion Self-Regenerating Suppression

injection valve ® guard column ® anion column ® ASRS suppressor
® conductivity cell ® ASRS suppressor ®waste

2. Check working eluent reservoirs. If levels are getting low, replenish as needed.

3. Start up the ion chromatograph using Table I as a guide.

NOTE: Prime the analytical pump before turning on if the system has not been used for a few days or if the working eluent has been changed.

4. Do not begin any injections until the pressure of the system has equilibrated and the conductivity reading has stabilized.

5. Using Chromeleon®, set up the ion chromatograph with the method, program file, and sequence files to be used. Refer to the *Chromeleon® User's Guides* for more detailed instructions on the use of the software.

   a. A new sequence table is started with each daily batch of samples. The daily sequence table is identified with a six-digit number containing the following information:

```
      03    360     X
RUN#
      |     |       |
      yr    |       |
      Julian day    |
             Where x is the system #
```

   b. A program file is identified as the template as this information does not change from the sequence under which the system was calibrated. Calibrations last for up to one month.

   c. All data files are saved in the sequence with which they are run.

6. Inject aliquots of reagent water until a stable baseline results.

7. Prepare standards and sample dilutions. Use eluent for the diluent.

8. The dilution factor must be entered for each sequence file. For solids, the sample weight is entered into the volume column. The dilution factor for solids is calculated by multiplying the final volume of the extract (typically 50-mL) by the dilution performed.

9. When calibrating the system, use a blank and the working calibration standards. After the calibration standards, analyze the following: batch QC, ICV, ICB, independent QC standard.

10. Samples are injected. A CCV and CCB are required after every 10 injections.

11. All injections must be documented with the following:

   a. Sample name

   b. Dilution factor

c. Auto-sampler Position

Calculations

This analytical method has been validated using peak areas for quantitation. The calibration fit is linear, equally weights all calibration points, and ignores zero. The equation of the line is y = K0 + K1x, where y = the area, K1 = the slope, x = the concentration, and K0 = the y intercept. Chromeleon® software performs the above determinations each time the calibration is updated. After the calibration is completed, Chromeleon® determines x (concentration) using the above equation. For the waters report format, Chromeleon® automatically multiplies this result by the dilution factor that has been entered by the analyst. For the solids report format, Chromeleon® performs the calculation using the analyst's inputted values for sample weight in grams and dilution factor. (The value entered in the dilution factor column is the volume extracted [normally 50 milliliters] × any dilution made by the analyst).

Statistical Information/Method Performance

1. The method detection limit (MDL) must be determined every 6 months by following the procedure outlined in QA-SOP11892

2. The quality control acceptance windows must be generated by following the procedure outlined in QA-SOP11896.

Quality Assurance/Quality Control

Check standards are used to establish retention times at the beginning of each calibration. The retention window assigned to each analyte is set with each calibration. Refer to the Initial Calibration for each analyte’s specific window. The windows are set from 0.200AG to 1.00 AG. The window is +/- a set time from the greatest peak within the window around each established retention time. The time input is in minutes. For example, 1.00 AG corresponds to 60s, therefore 0.200 AG corresponds to 12 seconds.

A. For waters and solids:

1. Calibration

a. The ion chromatography system should be recalibrated every 30 days. Instruments must be re-calibrated,whenever the calibration verification requirements of ±10% are not met or if the retention time windows are exceeded.

b. For each analyte of interest, a blank and calibration standards at 5-concentration levels must be used to calibrate the instrument.

(1) The concentrations for cal 1 must recover within 50% of the true value. The concentrations for this cal are equal to the reporting limit for each anion.

(2) The calibration curves for the anion(s) of interest must have a correlation coefficient (r) of greater than or equal to 0.995 for the curve to be valid. The r^2 value must be greater than or equal to 0.99.

NOTE: Chromeleon® returns an r^2 value for the correlation coefficient. The square root of this value is the “r” value, which is used to evaluate the curve.

c. The linearity of the calibration range must be verified every 6 months or whenever a significant change in instrument response is observed or expected. A minimum of 3 calibration standards must be analyzed. The results of these standards must not deviate from the true values by >10%. If this criterion is not met, linearity must be re-established. The values of the three standards are taken from the calculated values of the standards used for the calibration curve.

2. An ICV standard must be analyzed after every calibration. An acceptable result is a result which does not deviate from the true value by >±10%. If this criterion is not met for any anion(s) of interest, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the ICV

reanalyzed. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, the instrument must be recalibrated. If the acceptance criterion is met, the analysis is in control. Any samples affected by the original out-of-spec ICV must be repeated. All corrective action taken must be documented. Refer to T-WC-WI11652.

NOTE: If referencing Method 9056, results for the anions must not deviate from the true value by >±5%.

3. An ICB must be analyzed after every calibration. An acceptable result is a result < the MDL for the anion(s) of interest. If this criterion is not met for the anion(s) of interest, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the ICB reanalyzed. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, the instrument must be recalibrated. If the acceptance criterion is met, the analysis is in control. Any samples affected by the original out-of-spec ICB must be repeated. All corrective action taken must be documented. T-WC-WI11652.

4. Samples must be analyzed in batches not exceeding 20 samples per batch.

5. Sample results must not exceed the concentration of the highest standard. If a sample result falls above this value, the sample is diluted and run again. The sample result for any diluted sample should be >10% of the concentration of the highest standard for it to be considered acceptable data.

6. A batch blank (PBS, or PBW) must be analyzed for the anion(s) of interest in every batch. An acceptable result is a result < the MDL for the anion(s) of interest. If this criterion is not met, the cause for the out-of-spec condition must be determined and corrective action taken, if possible. The batch blank is reanalyzed once. Upon reanalysis, if the acceptance criterion still is not met for the anion(s) of interest, all samples associated with the batch blank must be re-prepared and reanalyzed. If the acceptance criterion has been met for a reanalyzed blank, all sample results associated with the batch blank are valid. All corrective action taken must be documented. Refer to T-WC-WI11652.

7. A LCSS or LCSW must be prepared and analyzed for the anions of interest in every batch. The current acceptance window of the LCSW is 90% to 110%. See Lims for the acceptance windows for DOD analyses - 10697, 10698, 10699, 10700, 10701, and 10702. If this criterion is not met, the cause for the out-of-spec condition must be determined if possible. Corrective action must be taken and the LCS reanalyzed. Upon reanalysis, if the acceptance criterion is still not met, all samples associated with that LCS must be re-prepared and reanalyzed.

If the acceptance criterion is met, the sample results are valid. All corrective action that was taken must be documented. Refer to T-WC-WI11652.

8. Based upon client requirements (when appropriate) a LCSDS or LCSDW is also prepared and analyzed under the same conditions as the LCS. The acceptable range is the same as that of the LCS. If this criterion is not met, the cause for the out-of-spec condition must be determined, if possible. Corrective action must be taken and the LCS and LCSD reanalyzed. Upon reanalysis, if the acceptance criterion is still not met, all samples associated with the LCS and LCSD must be re-prepared and reanalyzed. If the acceptance criterion is met, the sample results are valid. All corrective action that was taken must be documented. For the acceptable RPD between the LCS and LCSD, refer to the current statistical windows in the LIMS. If the RPD is outside these criteria, consult your group leader to determine if reanalysis is necessary.

9. A CCV and CCB must be analyzed after every 10 injections. An acceptable result for the CCV is a result which does not deviate from the true value by> ±10%. An acceptable result for the CCB is a result < the MDL. If the CCV and/or CCB do not meet this acceptance criterion, all samples since the last compliant CCV/CCB and before the next compliant CCV/CCB must be reanalyzed for the out-of-spec analyses. The cause of the out-of-spec condition must be determined and corrective action, if taken, must be documented. Refer to T-WC-WI11652.

NOTE: If referencing Method 9056, results for the anions must not deviate from the true value by >±5%.

10. A duplicate must be prepared and analyzed for every 10 samples in the batch (not to exceed 20 samples). For the acceptable RPD, refer to the LIMS. The cause of the out-of-spec condition must be determined if possible and corrective action, if taken, must be documented.

11. A MS must be analyzed for every 10 samples in the batch (not to exceed 20 samples). The MS is prepared by adding 0.5 mL of the intermediate QC STD to a 5 mL volumetric and bringing to volume with the appropriate sample. (The MS can be prepared into any volume volumetric as long as the amount of intermediate QC added is adjusted to keep the ratio of 1:10 the same.) See LIMS for current windows.

Table I

Ion Chromatographic Operating Conditions

		ICS-1100	ICS-1000
System Power		On	On
Service Air Valve (In-house air)		N/A	N/A
Helium Pressure Valve		N/A	N/A
High Limit Switch		3000 psi	3000 psi
Analytical Pump Flow Rate		1.2 mL/min	1.2 mL/min
Low Limit Switch		0 psi	0 psi
ASRS current		24 mA	39 mA
Detector Range (Full Scale)		10 µS	10 uS
Operating Pressure		1200-2200 psi	1200-2200 psi
Temperature Compensation		1.7 %/ºC	1.7 %/°C
Sample Loop		Approx. 50 µL	Approx. 50 µL
Analytical Pump		On	on

Table II

Ion Chromatography Shutdown Conditions

	ICS-1000/ICS-1100
Analytical Pump	Off
Service Air Valve (In-house air)	Closed
Helium Pressure Valve	N/A
ASRS Suppressor Current	N/A
System Power	Off
Analytical Pump	Off

End of document

Version history

Version	Approval	Revision information	
21	26.MAY.2015		
22	10.DEC.2018		
23	22.APR.2021		

eurofins	Always check on-line for validity. **TOC and TC in Solids and Sludges by Combustion by SM 5310B, EPA 415.1, SW-846 9060/9060A, Lloyd Kahn**	Level: **Work Instruction**
Document number: **T-WC-WI11627**		
Old Reference: **1-P-QM-WI-9013418**		
Version: **17.1**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **06-JUL-2021**	Document users: **6_EUUSLA_ Instrumental Water Quality _TOC Analyst, 6_EUUSLA_ Instrumental Water Quality _TOC Verifier**	Responsible: **5_EUUSLA_Instrumental Water Quality_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Scope
Basic Principles
Reference Modifications
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Sample Cup Preparation
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 18	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	MUR	Added method reference to comply with MUR
Throughout Document	LIMs update	Corrected IDs to match current LIMs

Revision: 17	**Effective Date:**	July 09, 2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version.
Scope	Current Process	Removed LIMS analyses 6623 and 11763

Revision: 17		**Effective Date:** July 09, 2019
Reference Modifications	Method Requirement	Removed 10% dup instead of a quadruplicate per batch
Procedure 8.	Current Process	Removed the process of analyzing three blanks prior to calibration.
Procedure 10.	Current Process	Removed the concentration of the CCV, this changes dependent on the CRM provided by the manufacturer
QA/QC	Method Requirement	One quadruplicate per batch for Lloyd Kahn

Reference

1. Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005, Method 5310B-2011,

2. Standard Methods for the Examination of Water and Wastewater, Method 5310B-2014.

3. *Method 415.1, Methods for Chemical Analysis of Water and Wastes USEPA 600.*

4. *Test Methods for Evaluating Solid Wastes, SW-846 Method 9060, September 1986.*

5. Test Methods for Evaluating Solid Wastes, SW-846 Method 9060A, November 2004

6. *The Primacs SNC-100 Analyzer Manual*, Skalar..

7. *Determination of Total Organic Carbon in Sediment, U. S. EPA, Region II, July 27, 1988. ("Lloyd Kahn Method.").*

8. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11188	Reagents and Standards

Scope

This method is applicable for the determination of total organic carbon (TOC) in soils and other samples not easily analyzed by the TOC waters method. The limit of quantitation (LOQ) for this method can be found in the analysis information file. Quantitative TOC results up to 1,000,000 mg/kg may be obtained by this method.

Basic Principles

TOC is determined by acidifying a sample and heating it to remove the TIC. An aliquot of sample (1 mg to 1 g) is weighed into a sample cup. The sample is then heated to 900°C for combustion of the remaining TOC. The resulting carbon dioxide from the TOC is detected by a nondispersive infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. The mass is proportional to the mass of TOC in the sample. Samples analyzed by this method include solids such as soils or sediments, slurries, sludges, brines, and corrosives.

The TC measurement is identical to the TOC measurement, with the exception that there is not an acidification and heating step to remove the TIC.

TIC is the calculation subtracting the TOC soil result from the result obtained when analyzing the TC.

Reference Modifications

There is no referenced method for the determination of Total Carbon in solids the analysis references a modified TOC method. The calibration standards, QC standards, and LCS employed for this analysis are composed of only organic sources of carbon (Sucrose for the calibration and QC standards, and a purchased standard for the LCS solid). Therefore, the Total Organic Carbon content is equal to the Total Carbon content in these standards.

Interferences

Carbon is ubiquitous in nature. Therefore, extra care must be taken to avoid contamination of reagents, glassware, and any other materials that come in contact with the sample. Samples which are light in weight may need to be analyzed at smaller aliquots to fit within a sample cup. These samples are reported with an elevated LOQ.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

1. Normal laboratory practices for safety must be followed.

2. Extreme caution must be used when handling sample cups after they have been analyzed.

3. Discard acid waste in acid waste containers.

4. See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.
Analysts are considered proficient when they have successfully completed a Demonstration of Capability for the analysis. A Demonstration of Capability consists of four laboratory control standards that are carried through all steps of the analysis and that meet the acceptance criteria for the LCS and LCSD. Documentation for these studies are in each individual’s training records.

Demonstration of Capability is performed annually and is maintained in the analyst's training records.

Sample Collection, Preservation, and Handling

Samples must be collected and stored in glass containers unpreserved. Samples must be stored under refrigeration at 0° to 6°C, not frozen.

Because very small amounts (1 mg to 1 g) of sample are used for the analysis, the sample must be as homogeneous as possible.

The holding time for analysis by SM 5310 B, EPA 415.1 and SW-846 9060/9060A is 28 days. The holding time for analysis by the Lloyd Kahn method is 14 days.

Apparatus and Equipment

1. Skalar-Primacs SC100

2. PC and SNAccess software

3. 2-stage gas regulator (two required)

4. Sample cups

5. Fiber quartz wool

6. Analytical balance, capable of accurately weighing to 1. mg

7. Glassware – General laboratory glassware as needed for preparing reagents and standards

8. Microliter syringe, various volumes

Reagents and Standards

NOTE: All chemical used must be ACS reagent grade unless otherwise noted.
Different volumes or weights may be used provided the ratios remain equivalent.
See *QA-SOP11188*, for the appropriate labeling and documentation of reagent and standard preparation.

1. Ultra pure nitrogen gas (60 psi)

2. Ultra pure oxygen gas (60 psi)

3. Hydrochloric acid, concentrated (HCI) – Purchased. Store at room temperature.

4. 1+1 Phosphoric acid (WC_TOC_1:1PA)

Phosphoric acid (H3PO4)	50 mL
Reagent water	50 mL

Take 50 mL of H3PO4 and add slowly while swirling to a 100-mL volumetric flask containing 50 mL reagent water. Store at room temperature. Prepare fresh every 6 months.

5. TOC stock calibration standard (sucrose containing 30% carbon) (WC_TOC_30%SUC), purchased. See label for expiration date. Store at room temperature.

6. TOC LCS standard, purchased (WC_TOC_SLCSCC). See label for expiration date, and the certified concentration. Store at room temperature.

Sample Cup Preparation

Sample cups must be conditioned prior to analysis. This is achieved by heating the cups to 750°C for a period of 2 to 5 minutes.

Calibration

1. Working standards for calibration
Pipette a volume of the TOC stock calibration standard into the sample cups as follows:

Working Std. (mg C)	Vol. Stock TOC (µL)
0.30	1.0
0.90	3.0
3.0	10.0
6.0	20.0

Prepare fresh daily. Store at room temperature.

2. Continuing calibration verification standard (CCV)

This is a purchased standard, and the acceptable range is specified by the manufacturer.

Procedure

1. Turn on nitrogen to 60 psig.

2. Turn on oxygen to 60 psig.

3. Add water and a drop of HCI to the scrubber.

4. Measure the carrier gas (oxygen) flow rate and adjust to 200 mL/min.

5. Measure the dryer gas (nitrogen) flow rate and adjust to 160 to 300 mL/min.

6. Power up instrument.

7. Program in a sequence file.

8. Run the calibration sequence as follows: 0.30, 0.90, 3.0, and 6.0 mg C (Performed monthly).

9. The instrument automatically calculates the R and list the calibration data.

10. Samples are analyzed along with the appropriate laboratory control standard and preparation blank. A check standard (CCV) and a continuing calibration blank (CCB) must be run at the beginning of each run and after every ten samples.

11. Add sufficient glass wool to each cup to cover the bottom surface.

12. Weigh samples into sample cups (up to 1000 mg). Add 1+1 H_3PO_4 drop wise until effervescence stops. Heat at 75°C for 15 minutes. Record the oven ID, time and temperature of this step in the log book.

NOTE: This procedure will convert inorganic carbonates and bicarbonates to carbon dioxide and eliminate it from the sample. The addition of H_3PO_4 and the heating to 75°C is not performed for analysis 10065.

13. Analyze the residue according to the instrument manufacturer's instructions for the remaining TOC result.

Calculations

1. To determine mg/kg TOC or TC

   mg/kg = (Raw result in mg C) x (1000/weight in mg) x (1000)

2. To determine LOQ/MDL factors

$$\text{Factor} = \frac{\text{Maximum sample aliquot weight }(1000.\text{mg})}{\text{weight of sample }(\text{mg})}$$

3. To determine Total Inorganic Carbon (TIC)

   TIC = Total Carbon (TC) - Total Organic Carbon (TOC)

Statistical Information/Method Performance

1. The method detection limit (MDL) is determined annually by following the procedure outlined in *QA-SOP11892*.

2. The quality control acceptance windows are generated annually by following the procedure outlined in *QA-SOP11896*.

Quality Assurance/Quality Control

1. A calibration must be performed every 30 days. The acceptable range for the calibration is R = 0.995 or greater. If this criteria is not met, the instrument must be recalibrated.

2. A batch must contain no more than 20 field samples.

3. A batch blank (MB) must be analyzed every batch or each day samples are prepared (not to exceed 20 samples). An acceptable result is < the limit of quantitation. If the PBS does not meet this criterion, it must be rerun twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be repeated.

4. A laboratory control standard (LCSS) must be analyzed every batch or each day samples are analyzed (not to exceed 20 field samples). For TOC this is a purchased standard and the acceptable range is specified by the manufacturer. The LCSS should undergo the same steps as the samples. If the LCSS does not meet the acceptable criterion, it must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples in the batch must be reanalyzed.

5. Based upon client requirements, a laboratory control standard duplicate (LCSD) may need prepared and analyzed under the same conditions as the LCS. The acceptance criterion for the LCSD is the same as that of the LCS. The relative percent difference between the LCS and the LCSD is calculated statistically.

6. 9060 - a duplicate is analyzed every 10 samples. Lloyd Kahn - one sample is analyzed in quadruplicate every batch. The acceptable relative percent difference is statistically determined. The duplicate relative percent differences must be tracked to continually monitor method performance.

7. 9060, 5310B - A spike must be analyzed for every 10 samples. The sample is spiked with 3 ul of the purchased TOC stock calibration standard. The acceptance range is determined statistically. The spike recoveries must be tracked to continually monitor method performance.

8. Based upon client requirements, a matrix spike duplicate (MSD) may need prepared and analyzed under the same conditions as the MS. The acceptance criterion for the MSD is the same as that of the MS. The relative percent difference between the MS and the MSD is calculated statistically.

9. A check standard (CCV) and blank (CCB) must be run after every ten injections (including blanks and standards). The acceptable range for the CCV determined by the manufacturer. An acceptable CCB result is < the limit of quantitation. If either, or both, of these injections do not meet the acceptance criterion, the unacceptable original must be repeated twice. If either of the two additional trials do not meet the acceptance criterion, all samples since the last compliant CCV/CCB must be reanalyzed.

10. A CCV and CCB shall be analyzed at the beginning and the end of each run. At any time when the instrument has been idle for a period of 4 hours or more, a CCV and CCB must be analyzed. If either of these parameters cannot meet specifications, the instrument must be recalibrated.

QA-SOP11188 Reagents and Standards
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits

End of document

Version history

Version	Approval	Revision information	
16	17.OCT.2018		
17	03.JUL.2019		
17.1	22.JUN.2021		

eurofins	Always check on-line for validity. **Total Organic Carbon (TOC), Dissolved Organic Carbon (DOC), and Total Inorganic Carbon (TIC) by SM 5310C or EPA 415.1 in Waters**	Level: **Work Instruction**
Document number: **T-WC-WI11637**		
Old Reference: **1-P-QM-WI-9011651**		
Version: **17**		Organisation level: **5-Sub-BU**
Approved by: **X6TJ** Effective Date **06-JUL-2021**	Document users: **6_EUUSLA_ Instrumental Water Quality _TOC Analyst, 6_EUUSLA_ Instrumental Water Quality _TOC Verifier**	Responsible: **5_EUUSLA_Instrumental Water Quality_Manager**

Revision Log
Reference
Cross Reference:
Scope:
Basic Principles:
Reference Modifications:
Interferences:
Safety Precautions and Waste Handling:
Personnel Training and Qualifications:
Sample Collection, Preservation, and Handling:
Apparatus and Equipment
Reagents and Standards
Calibration:
Procedure:
Calculations:
Statistical Information/ Method Performance:
Quality Assurance/Quality Control:

Revision Log

Revision: 17		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	MUR	Updated SM to confirm to MUR
Throughout Document	Updated LIMS	Added nomenclature to match current LIMS IDS
Throughout Documnet	ISO Standard Update	updated Guide 34 to ISO 17034

Revision: 16		**Effective Date:** Sept 25, 2018
Section	**Justification**	**Changes**
Calibration	Updated procedure	Corrected the process to make CAL and QC Stds.
Quality Assurance/Quality Control	Updated procedure	Updated process for 25 TIC std. Updated reference for QC outliers Added the use of pH adjusted reagent blanks

Revision: 16		**Effective Date:** Sept 25, 2018
Reference	Update Standard Methods Edition	Updated the Standard Methods Edition to the 22nd Edition, 2011.

Reference

1. Standard Methods for the Examination of Water and Wastewater, 22nd Edition, Method 5310 C - 2000

2. . Standard Methods for the Examination of Water and Wastewater, Method 5310 C- 2011

3. Standard Methods for the Examination of Water and Wastewater, Method 5310 C -2014.

4. Method 415.1, Revision 1 (1974), Methods for Chemical Analysis of Water and Wastes USEPA 600.

5. *Model 1030 Total Organic Carbon Analyzer Operating Procedures and Service Manual,* O.I. Corporation, 2005.

6. *Chemical Hygiene Plan*, current version.

7. QC Laboratories Inc.SOP QC0590.

Cross Reference:

Document	Document Title
T-WC-WI11644	Maintenance of the OI Analytical Model 1030 Total Organic Carbon Analyzer
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits

Scope:

This procedure is applicable for the determination of total organic carbon, Dissolved Organic Carbon, or Total Inorganic Carbon in water and wastewaters by SM 5310 C-2000 or EPA 415.1.

Swabs can be accepted and analyzed for TOC as a special request by the client.

The current limit of quantitation (LOQ) and method detection limit (MDL) for this analysis can be found in the laboratory information management system (LIMS).

Basic Principles:

The procedure outlined in this method pertains to the O.I. Model 1030 analyzer. This model uses the persulfate oxidation technique.

A sample aliquot of water or wastewater is injected into a reaction vessel at 100°C where metered amounts of phosphoric acid are added. The sample is purged with ultrapure nitrogen gas to remove inorganic carbon as carbon dioxide and any purgeable organic carbon (POC). Following the purging of the inorganic fraction, a metered amount of sodium persulfate is added to the reaction vessel, and the sample is heated. The persulfate oxidizes any organic carbon to carbon dioxide. Following the completion of the persulfate digestion, the carbon dioxide is carried to the IR detector by the nitrogen stream.

Samples for DOC are passed through a 0.45-µ membrane filter prior to analysis. TIC can be determined by calculating the difference between total carbon and TOC.

Clients can send swabs for the determination of TOC. These samples are reported as mg/swab. Multiply the concentration of TOC found in the extract by the swab extract volume.

Reference Modifications:

There are no procedural differences between EPA 415.1 and SM 5310 C.

Interferences:

This method is generally free of interferences when normal wastewater and water samples are analyzed. However, high concentrations of chloride can interfere and several other problems can arise from certain sample matrices. These are listed below.

1. Chloride can react with the persulfate, which can cause erroneously low TOC results. Samples high in chloride may require that extra persulfate be added and the reaction time extended. The chlorine gas that is produced is also damaging to the molecular sieve. For these reasons, samples must not be preserved with hydrochloric acid.

2. Samples with high levels of inorganic carbon may require extra acid and longer purge time to ensure all the carbonates and bicarbonates are removed from the sample. A smaller sample volume may also be used.

3. Samples with high levels of organic carbon may require that extra sodium persulfate be added and that the reaction time is extended to ensure that all the organic carbon is oxidized by the persulfate. A smaller sample volume may also be used. Spiking the sample with a known amount of carbon and calculating recovery can be used to verify the oxidation efficiency.

4. When sequentially analyzing samples, carryover may occur when a sample high in carbon is followed by a sample low in carbon. This will produce erroneously high results for low-level TOC samples. This is caused by adsorption of organic carbon on the Teflon™ tubes and overloading of the molecular sieve. If an excessively high-level sample is accidentally introduced into the system, it may be necessary to clean the digestion vessel with reagent water before analyzing any more samples. This is accomplished by cycling reagent water and the reagents through the system until TOC values drop to less than the normal detection limit.

Safety Precautions and Waste Handling:

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Normal laboratory safety practices must be followed when performing this method. Extra care must be taken when handling the persulfate reagent (which is a strong oxidant) and unknown samples that could contain toxic or carcinogenic chemicals. Inspect all glassware before use. Discard or send for repair any glassware that is chipped, flawed, or broken.

Personnel Training and Qualifications:

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Proficiency is measured through an Initial Demonstration of Capability (IDOC) that consists of four laboratory control samples (LCS) that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four LCS's or one blind sample.

Sample Collection, Preservation, and Handling:

All samples must be collected in glass containers and stored at 0°to 6°C not frozen. Samples for TOC must be acidified with concentrated phosphoric acid or sulfuric acid to a pH <2. The holding time for these samples is 28 days.

Apparatus and Equipment

1. O.I. Corporation Model 1030 TOC analyzer

2. O.I. Corporation Model 1088 Rotary Autosampler

3. 2-stage gas regulator

4. Analytical balance capable of accurately weighing to 0.0001 g

5. Automatic pipettes

Reagents and Standards

A. All chemicals used must be ACS reagent grade unless otherwise noted.
B. Different volumes or weights may be used provided the ratios remain equivalent.

C. Reagents and Standards
1. Ultra-pure nitrogen gas (30 psi)

2. (WC_TOC_SodPer) Oxidizing reagent - Sodium persulfate ($Na_2S_2O_8$)
 a. Dissolve 100 ± 1 g of sodium persulfate in approximately 500 mL of reagent water in a 1000-mL volumetric flask.

 b. Dilute to volume with reagent water.

 c. Purge with nitrogen gas for 1 hour to release inorganic carbon present in reagent.

 d. This solution is stable for 6 months and is stored at room temperature in a plastic container.

3. (WC_TOC_5%Phos) Phosphoric acid (5% w/w)

 a. Add 59 mL of 85% H_3PO_4 to about 500 mL of reagent water in a 1000-mL volumetric flask.

 b. Mix thoroughly and dilute to volume with reagent water.

 c. This solution is stable for 6 months and is stored at room temperature in a plastic container.

4. (WC_TIC_QC) Total Inorganic Standard (500 mg/L)

Sodium bicarbonate ($NaHCO_3$) 0.1750 g ±0.0005 g

Sodium carbonate (Na_2CO_3) 0.2209 g ±0.0005 g

 a. Dissolve above reagents in a 100-mL volumetric flask containing about 50 mL of reagent water.

b. Dilute to volume with reagent water.

c. Prepare monthly. Do not acidify. Store in the flask at 0°to 6°C not frozen.

5. ISO 17034 Certified Reference Material (CRM): Purchased. See certificate of analysis for true value supplied by the vendor.

Calibration:

1. (WC_TOC_CALSTD) TOC stock calibration standard (500 mg C/L)

Potassium hydrogen phthalate (KHP) 1.063 ± 0.005 g
Phosphoric acid, concentrated (H3PO4) 20 drops

a. Dissolve 1.063 ± 0.005 g of KHP a 1000-mL volumetric flask containing about 500 mL of reagent water.

b. Add concentrated phosphoric acid (H3PO4) to the 1000-mL volumetric flask until the pH is <2.

c. Bring to volume with reagent water

d. Prepare every 30 days. Store in an amber glass bottle at 0º to 6º C, not frozen.

2. (WC_TOC_QC_STD) TOC stock QC standard (500 mg C/L)

Prepare identically to TOC stock calibration standard using KHP from a different source.

3. Working standards for calibration and QC

Pipette a volume of TOC stock standard into the appropriate volumetric flask that contains reagent water and three drops of concentrated phosphoric acid. Dilute to volume with reagent water. Prepare daily, as needed, according to the following table:

Volume Stock CAL Standard (mL)	Standard Concentration (mg/L)	Final Volume (mL)
20	100	100
10	50	100
10	25	200
2.0	10	100
3.0	7.5	200
0.2	1.0	100
Volume Stock QC Standard (mL)	Standard Concentration (mg/L)	Final Volume (mL)
10.0	25 –CCV2/LCSW	200

Procedure:

1. General (more detailed instructions for each step can be found in the instrument manual)

2. Start up the instrument in the following manner:
   a. Turn on the power switch located at the back of the instrument. The system loads the startup screen and User Login dialog box. Turn on the nitrogen gas flow.

   b. Prime the syringe and all reagents by following the manufacturer's instructions located in the instrument manual.

3. Load the active sequence.

4. Each sample is injected in duplicate. The RPD of the duplicate injections must be< 10% when the concentrations are greater than five times the LOQ.

5. For DOC, the samples and all quality control samples are filtered through a 0.45 micron filter that has been rinsed two times with reagent water. There is no subtraction of the filter blank in the calculation for DOC.

6. For swab samples, add 20 to 50 mL of reagent water to the container holding the swab. Record the amount of reagent water added. Shake the sample for 5 minutes. Filter the extract through a 0.45 micron filter as described abouve in #5.

7. Load samples onto the autosampler trays.

8. Press Start.

Calculations:

The TOC result (in mg/L) is performed as below using the average of the two injections:

raw result (mg/L from instrument) x dilution factor = final TOC (mg/L)

For swab samples report the TOC as mg per swab by the following:

TOC, mg per swab = T x L

Where:

T = the concentration of TOC found in the extract in mg/L.

L = swab extract volume (L) = (mL used for the extract/1000).

Statistical Information/ Method Performance:

1. The method detection limit (MDL) is determined annually by following the procedure outlined in *QA-SOP11892*. The 7 replicates must be analyzed over a period of at least 3 days.

2. The quality control acceptance windows are generated annually by following the procedure outlined in *QA-SOP11896*.

Quality Assurance/Quality Control:

1. A calibration of 6 standards and a reagent water blank must be run monthly as the instrument calibration. The correlation coefficient of the curve must be ≥0.995.

2. An initial calibration blank (ICB) and an initial calibration verification standard (ICV) must be run after every calibration. The acceptable result is < the LOQ (limit of quantitiation) for the ICB and +/- 10% for the ICV.
The ICB is reagent water brought to a pH of <2 with phosphoric acid.

3. A CCV and CCB must be analyzed after every 10 injections. The acceptable range for the CCV is ±10% of the true value. An acceptable CCB result is < the LOQ. The CCB is reagent water brought to a pH of <2 with phosphoric acid.

4. A batch contains no more than 20 samples.

5. A batch blank (MB) is analyzed every batch. An acceptable result is less than the LOQ. The Batch blank is reagent water brought to a pH of <2 with phosphoric acid.

6. A laboratory control standard (LCS) must be analyzed every batch. The LCS is the 25-mg/L QC standard (CCV2). The acceptable range for this standard is statistical and is found in the analysis information file.

7. Based upon client requirements, when appropriate, a laboratory control standard duplicate (LCSD) must also be prepared and analyzed under the same conditions as the LCS. The relative percent deviation between the LCS and LCSD can be found in the LIMS. The acceptable range of the LCSD is the same as the LCS. If the LCSD does not meet this acceptance criterion, see T-WC-WI11652 for handling outliers and the appropriate corrective action must be taken. If the RPD between the LCS and LCSD is not within specifications, see your manager or group leader to determine if reanalysis is needed.

8. If any of the QC does not meet acceptance criterion, see T-WC-WI11652 for handling outliers and the appropriate corrective action that must be taken.

9. Sample results may not exceed the concentration of the highest standard. If a sample result falls above this value, the sample must be diluted with reagent water and run again.

10. A duplicate must be analyzed every batch at a frequency of 1 duplicate per 10 samples. The acceptance criterion is calculated by the LIMS statistical program. If the relative percent difference falls outside of the acceptance criteria, see your group leader to determine if reanalysis is needed.

11. A matrix spike (MS) must be analyzed at a frequency of 1 spike per 10 samples. This spike must be prepared at a concentration level that is at least four times greater than the concentration of the unspiked sample. See LIMS for current quality control acceptance criteria. If the recovery is outside of the acceptance criteria, consult your group leader to determine if reanalysis is necessary.

12. Based upon client requirements, a MSD is prepared and analyzed under the same conditions as the MS. The acceptance criteria and corrective action are the same as the MS. The relative percent difference between the MS and MSD is statistical and can be found in the LIMS. If the RPD is outside of these criteria, consult your group leader to determine if reanalysis is necessary.

13. The instrument must be calibrated at least every 30 days, or whenever acceptable check standards can no longer be obtained. At any point where the instrument has been idle for a period of 4 hours or more, a CCB and CCV must be analyzed. If either of these parameters is not within specifications, the instrument must be recalibrated and a new run started.

14. A Total Inorganic Carbon (TIC) check standard must be prepared daily to determine the efficiency of inorganic carbon removal when performing the total organic carbon analysis. The LCSW sample must be spiked with the same level of TIC and analyzed with each run. This standard is prepared by adding 1.25 ml of the TOC QC stock and 1.25 ml of the TIC QC stock to a 25 ml volumetric and bringing to volume with reagent water.The acceptable recovery range for this standard is 80% to 120%.

15. Annually, an ISO 17034 certified reference material (CRM) must be analyzed as a check standard. The CRM must recover within the supplier’s acceptance criteria, or within 10% of the known value, whichever is tighter.

QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
T-WC-WI11644 Maintenance of the OI Analytical Total Organic Carbon Solids Analyzer

End of document

Version history

Version	Approval	Revision information	
15	22.MAY.2015		
16	11.SEP.2018		
17	22.JUN.2021		

eurofins	Always check on-line for validity.	Level:
Document number: **T-MET-WI8636**	**Sample Prep of Sediments, Sludges, Soils, and Tissues by SW846 3050B for ICP and ICP-MS**	**Work Instruction**
Old Reference: **1-P-QM-WI-9015160**		
Version: **26**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **11-MAY-2021**	Document users: **6_EUUSLA_Metals_ICP Prep, 6_EUUSLA_Metals_ICP Prep Verifiers, 6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Purpose
Scope
Basic Principles
Definitions
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Procedure
Block Digestor Instructions
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

	Revision: 26	**Effective date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
LIMS ID	Enhancement	Added TALS method codes
Cross Reference	Enhancement	Added Data Verification SOP
Definitions	Clarification	Removed redundant definitions; added SQAR
Apparatus and Equipment	Reflects current procedure	Removed reference to LLENS
Procedure	Reflects current procedure	Removed reference to the LLENS
Quality Assurance/Quality Control	Reflects current procedure	Removed reference to individual analysis SOPs; added in reference to Data Verification SOP

Revision 25	**Effective Date:**	06.MAR.2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
LIMS ID	Follow current procedure	Added analysis number for sieving
Apparatus and Equipment	Follow current procedure	Added sieves and changed temperature to 90C-100C
Procedure	Follow Current Procedure	Clarified if sample is not split
Procedure	Follow current procedure	Added section on how to dry and sieve samples
Procedure	Update to Method	Updated 90C-95C to be 90C-100C
LIMS ID	Follow Current Procedure	Removed old analysis numbers
Quality Assurance/Quality Control	Clarification	Added sentence about sample and batch QC must be the same

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3050B, December 1996.

2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
T-MET-WI11933	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)
T-MET-WI11931	Metals by Inductively Coupled Plasma Atomic Emissions Spectroscopy for SW-846 Methods 6010B/C (aqueous, solid, tissue) and EPA 200.7(aqueous)
T-MET-WI9082	Working Instructions for Prep Solutions and Standards

Document	Document Title
T-MET-WI35797	Metals Data Verification Process

Purpose

This digestion procedure is for the preparation of solid samples for analysis by ICP and ICP/MS following SW-846 protocol.

Scope

This method is used for preparation of metals in solid samples for analysis by ICP and ICP/MS.

Basic Principles

A representative sample is digested with repeated additions of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2). Hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed. The resultant digestate is diluted and analyzed.

This method is not a total digestion technique for most samples; it is a very strong acid digestion that dissolves almost all elements that could become "environmentally available." By design, elements bound in silicate structures are not normally dissolved by this procedure.

Definitions

1. SQAR = Sludge Quality Assurance Regulations

Interferences

When analyzing sample by ICP-MS using this digestion procedure we follow the instrument manufacturer's guidelines to eliminate polyatomic interferences typically caused by Chlorine. The process we follow involves the use of a collision/reaction cell on the ICP-MS. Below is a description of how the collision/reaction cell works.

Reaction Process - The primary method of interference removal is through a reaction event. When using a reaction gas, either the target interference is more reactive than the target analyte, leading to preferential removal of the interferent or (less commonly) the target analyte is more reactive and is converted to a new species at a different mass which is free from any existing or newly-formed overlap.

Collision Process - The primary method of interference removal is through a non-reactive event. This process of interference removal is kinetic energy discrimination (KED). Energy Discrimination is most commonly used with an inert gas, which means the interference removal process is not affected by reactions in the cell.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO3)** – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.
>
> **Hydrochloric acid (HCl)** – This acid can cause skin burns. Never mix HCI with concentrated H2SO4 to avoid a violent reaction. Always use in a fume hood or use the designated dispensing equipment.
>
> **Hydrogen peroxide (H2O2)** - This is a strong oxidizing agent and causes severe burns. Avoid contact with skin.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds. Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A powder or equivalent to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an Initial Demonstration of Capability (IDOC).

The IDOC consists of four LCSs that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Various options are available for a DOC and can include four LCSs or one blind sample.

Sample Collection, Preservation, and Handling

Solid samples require no chemical preservation.

Samples must be submitted in glass or plastic containersand stored at 0° to 6°C, not frozen, prior to digestion. Samples must be analyzed within 6 months of sample collection.

Digested samples are stored in polypropylene bottles at room temperature.

Apparatus and Equipment

1. Polypropylene containers and covers (digestion vessels) - certified clean and Class A equivalent
2. Whatman No. 41 filter paper or equivalent
3. Funnels
4. Environmental Express HotBlock (block digestor) - adjustable and capable of maintaining a temperature of 90 to 100°C
5. Balance capable of reading 0.01 g
6. Chemware Ultra-Pure PTFE boiling stones, or equivalent.
7. Computer and software TALS
8. Sieves size depending upon client request

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form *T-MET-WI9082*.

1. Nitric acid (HNO3) – Fisher, Trace Metal Grade, or equivalent. Store at room temperature. Re-evaluate annually.
2. Nitric acid (1:1) – Add 500 mL of HNO3 to 500 mL of reagent water. Store in polypropylene at room temperature. Expires 6 months from date of preparation. (Different volumes are acceptable but ratios must stay the same.)
3. Hydrogen peroxide, 30% (H2O2) – Fisher, Certified ACS or equivalent. Store at room temperature. Re-evaluate annually.
4. Hydrochloric acid (HCl) – Baker Instra-Analyzed, or equivalent. Store at room temperature. Re-evaluate annually.

NOTE: It is acceptable to prepare using multiples of indicated weights and volumes if ratios are maintained.

Procedure

A. Soil Sample Digestion

1. Turn block digestor on and allow block to reach the Control Point setting that provides 90° to 95°C sample temperature. (The block temperature setting is not necessarily the sample temperature.) See below for **Block Digestor Instructions** section.
2. If sample is not located in split bin, weigh 1.00 to 1.50 g (to the nearest 0.01 g) of a well-mixed sample into a polypropylene digestion vessel. (If the sample is watery use 5.00 to 5.50 grams for analysis. Additional information on non-standard matrices is found at the end of the procedure section.) Add 1.00 to 1.49 g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Enter the blank and LCS weight as 1.0000 to 100.0000 final volume in theworksheet tab of the TALS prep batch. In the Notes field, record the true weight measured.For sample batch spiking procedures see *T-MET-WI9082*. All spiking must be performed prior to starting the digestion procedure.
3. Add 10mL of (1:1) HNO_3, swirl to mix, and cover with a polypropylene cover.
4. Place sample vessel in block digestor. Heat (reflux) the sample at 90°to 100°C for 10 to15 minutes without boiling.
5. Add 5 mL of concentrated HNO_3. Add 20 mL of reagent water. Replace cover, return vessel to digestion block and heat for 30 minutes.
6. **NOTE**: If brown fumes are generated (indicating oxidation of the sample by HNO_3) continue the process of adding 5mL HNO_3 and heating until no brown fumes are given off by the sample. This indicates that the reaction with HNO_3 is complete. Add the same amount of HNO_3 to the entire digestion batch.
7. With cover on, heat at 90° to 100°C without boiling for 2 hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).
8. Remove vessel from digestion block and allow sample to cool.
9. Add 2 mL of reagent water and 3 mL of 30% H_2O_2. With cover on, return vessel to digestion block and heat until effervescence subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.
10. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.
11. **NOTE:** Do not add more than a total of 10mL 30% H_2O_2.
12. With cover on, continue heating the acid-peroxide digestate at 90°to 100°C without boiling for 2hours. Maintain a covering of solution over the bottom of the vessel at all times (add reagent water if necessary).
13. Remove sample vessel from digestion block and allow to cool.
14. Add 10 mL of HCl. With the cover on, return vessel to digestion block and heat at 90°to 100°C for 15 minutes.
15. Remove sample vessel from digestion block.
16. If floating particulate is evident after digestion, the sample must be filtered.
    a. Filter through Whatman No. 41 filter paper into a polypropylene container.
    b. Wash sample vessel, residue, and paper thoroughly with reagent water.

   c. If any samples are filtered, the MB and LCS must also be filtered.

17. Adjust volume to the 100 mL mark on the digestion vessel with reagent water and mix. Seal vessel with a screw cap. The sample is now ready for analysis.

NOTE: When special limits of quantitation are required by the client, use more sample weight.

B. For wipe samples:

When wipes are digested by this method, one blank media each must be used for the batch preparation blank, the LCS, and the LCSD. Refer to Form *T-MET-WI9082* for the spiking of the LCS and LCSD. Digest wipes in their own batch. Use reagent water to rinse any particulate matter from the wipe container into the vessel containing the wipe before digesting. If brown fumes are evolved during wipe sample digestion, perform only two 5 mL HNO_3 additions with 30-minute refluxing each; add the same amount of HNO_3 to the entire batch. Proceed with digestion.

C. For tissue samples:

When fish tissues or other tissue samples are digested by this method, refer to Form *T-MET-WI9082* for the spiking of the LCS, LCSD (if needed), matrix spike (MS), and matrix spike duplicate (MSD). Add 1.00 to 1.49g of Chemware Ultra-Pure PTFE boiling stones to the digestion vessel for the blank and LCS. Digest tissue samples in their own batch.

D. For soil samples that need to be dried and sieve prior to digestion.

1. Lay out sample on a clean surface of either glass or aluminum foil.
2. Allow sample to dry. When sample is done drying, but is dried in a large chunk take appropriate clean tools to break down the large chunk in order to sieve the sediment.
3. Pour entire sample that has been laid out to dry into the appropriate sieve size defined by client and shake sieve to get sample to fall through.
4. Take the sieved sample and place into the appropriate bottle needed for analysis.
5. Rinse the sieve and the sieve filter with reagent water until no particulate is left on sieve.
6. Dry sieve in open air or in an oven until no reagent water is present.

Block Digestor Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.
2. Wait about 8 seconds until controller display indicates current block temperature.
3. PRESS and hold STAR (*) key.
4. The display shows Control Point temperature.
5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.
6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used. If necessary, adjust Control Point temperature to the proper setting as instructed below.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations

Not applicable.

Statistical Information/Method Performance

Not applicable to this procedure. See analysis method.

Quality Assurance/Quality Control

For sample batch spiking instructions see form *T-MET-WI9082*. Refer to ICP section when prepping ICP analysis. Refer to ICP/MS section when prepping ICP/MS analysis. Prepare a method blank, sample duplicate, MS, MSD, and LCS with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP. If any samples are filtered the prep blank and LCS must also be filtered.

Any extra digestion step done on one sample in a batch must be done to batch QC as well. Example if 1 mL of H_2O_2 is added to a sample it must also be added to the batch QC.

Refer to *T-MET-WI35797* for sample batch quality control requirements, acceptance criteria and corrective action.

T-MET-WI11931 Metals by ICP for Methods SW-846 6010B/C/D (aqueous, solid, tissue) and EPA 200.7 (aqueous)
T-MET-WI11933 Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B(aqueous, solid, tissue) and EPA 200.8 (aqueous)
T-MET-WI35797 Metals Data Verification Process
T-MET-WI9082 Working Instructions for Prep Solutions and Standards

---

End of document

Version history

Version	Approval	Revision information	
24	13.FEB.2018		
25	06.MAR.2019		
26	11.MAY.2021		

eurofins	Always check on-line for validity. **Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A/6020B(aqueous, solid, tissue) and EPA 200.8 (aqueous)**	Level: **Work Instruction**
Document number: **T-MET-WI11933**		
Old Reference: **1-P-QM-WI-9018443**		
Version: **11**		Organisation level: **5-Sub-BU**
Approved by: **XL3S** Effective Date **12-NOV-2020**	Document users: **6_EUUSLA_Metals_ICP-MS Analysis, 6_EUUSLA_Metals_ICP-MS Analysis Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Scope
Routine Methods
Basic Principles
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control
Figure I - Daily Performance Acceptance Criteria and Tuning Solution Concentrations

Revision Log

Revision: 11	**Effective Date:**	This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Reference	Reflects current protocol	Removed 7500 information
Cross Reference	Enhancement	Added *T-MET-WI35797* Metals Data Verification Process
Routine Methods	Added/Deleted	Removed old LIMs analysis numbers
Basic Principles	Reflects current protocol	Corrected instrument and equipment information. Removed 7500 information, added 7900 information. Corrected gas mode order.

Revision: 11	**Effective Date:**	This version
Section	**Justification**	**Changes**
Interferences	Reflects current protocol	Removed 7500 and 7700 references.
Safety Precautions and Waste Handling	Reflects current protocol	Removed responsibilities for chemical spills that are now taken care of by the ERT.
Apparatus and Equipment	Reflects current protocol	Removed 7500 information, added 7900 information. Corrected instrument/equipment information. Removed references to Parallax and updated to reflect procedures for TALS. Removed adjustable pipette information.
Reagents and Standards	Reflects current protocol	Corrected standard/reagent name(s) from Parallax to TALS formatting.
Calibration	Reflects current protocol	Corrected standard/reagent name(s) from Parallax to TALS formatting. Added LRC
Procedure	Reflects current protocol	Removed procedure for old LIMS. Added the procedure for new LIMS. Corrected standard/reagent and QC name(s) from Parallax to TALS formatting.
Quality Assurance/Quality Control	Reflects current protocol	Corrected QC name(s) from Parallax to TALS formatting. Removed 7500 information and added 7900 information. Updated 7700 usage. Removed verification process and added cross reference to *T-MET-WI35797*
Figure 1	Enhancement	Removed erroneous information and replaced with correct tuning parameters/concentration(s).
Table I	Enhancement	Removed table and cross referenced to *T-MET-WI35797*.
Table II	Enhancement	Removed table and cross referenced to *T-MET-WI35797*.

Revision:10	**Effective Date:**	05.JUL.2019
Section	**Justification**	**Changes**
Revision Log	Formatting requirement	Removed revision logs up to the previous version
Table II - ICPMS Metals by EPA 6020/6020A/6020B	Update to method requirements	Internal standard requirements

Reference

1. Method 200.8 (rev 5.4), Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Mass Spectrometry, USEPA 600/R-94/111 May 1994.

2. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020, September 1994.

3. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020A, February 2007.

4. Test Methods for Evaluating Solid Wastes, SW-846 Method 6020B, Rev. 2, July 2014.

5. ESI SC Manual.

6. Agilent 7700 and 7900 Series ICP-MS MassHunter Workstation Guide and Hardware Maintenance Manual.

7. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Q-EQA-FRM6869	Nonconformance Form
T-MET-WI12065	Working Instructions for Preparation of ICP-MS Solutions and Standards
S-SS-WI10695	Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
QA-SOP11892	Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896	Establishing Control Limits
QA-SOP11901	Instrument Maintenance and Calibration
T-MET-WI18027	Instrument Operations for Agilent 7700
T-MET-WI18029	Instrument Maintenance for Agilent 7700
T-MET-WI21589	Instrument Operations for Agilent 7900
T-MET-WI21590	Instrument Maintenance for Agilent 7900
T-MET-WI35797	Metals Data Verification Process

Scope

This procedure applies to analyses performed at Eurofins Lancaster Laboratories Environmental (ELLE) using ICP-MS for identification and quantitation of metallic constituents.

Aqueous undigested samples and digestates (aqueous and solid matrices) are analyzed via this SOP.

Limits of Quantitation (LOQs), Method Detection Limits (MDLs) and linear ranges are maintained in the LIMS. These are subject to change without notification.

Routine Methods

Analyte	Mass
Be	9
B	11
Na	23
Mg	24
Al	27
K	39
Ca	44
Ti	47
V	51
Cr	52
Mn	55
Fe	57
Co	59
Ni	60
Cu	63
Zn	66
As	75
Se*	78
Se**	82

Sr	88
Mo	98
Ag	107
Cd	111
Sn	120
Sb	121
Ba	137
Tl	203
Pb 206	206
Pb 207	207
Pb 208***	208
U	238
*Se 78 is the mass reported from collision cell ICPMS only **Se 82 is the mass reported from non-collision cell ICPMS only ***For Pb masses 206, 207 and 208 are summed for calibration and analysis	

Basic Principles

ICP-MS is an analytical instrument that uses the energy of inductively coupled plasma to generate ions to be analyzed in the mass spectrometer.

The instruments use a discrete sampling system that first loads the sample into a Teflon sample loop. The sample is then pushed by a carrier solution using a computer controlled peristaltic pump that delivers the sample from the loop into the nebulizer attached to a cooled Scott double pass spray chamber.

The Agilent 7700 (E05) and 7900 (E07/E08/E09) can analyze samples in 3 different modes of operation, although typically analysis is done in 3 mode or No Gas mode only:

1. Mode 1 – no gas. This mode does not use a collision or reaction gas. Elements with a low atomic mass or that have no interferences can be analyzed using the "no gas" mode.

2. Mode 2 – Hydrogen (H_2) mode. This mode uses H_2 as a reaction gas, since it reacts quickly and efficiently with the Argon-based interfering species, but reacts slowly or not at all with the analyte(s) of interest. In this mode, interferences can be reduced to the level of baseline noise, allowing lower detection limits to be achieved for some difficult elements.

3. Mode 3 – Helium (He) mode. This mode uses He as a collision gas for reliable, predictable removal of unknown matrix interferences. No new interferences are formed in the cell, and no analytes are lost by a reaction.

Interferences

ICP-MS interferences include isobaric elemental interferences and polyatomic ion interferences derived from the plasma gas, reagents, and/or sample matrix. The Agilent 7900 utilize helium as an inert collision gas to reduce or eliminate many types of polyatomic ion interferences.

Physical interferences caused by the change in sample matrix affecting sample transport and/or nebulization must be compensated for using internal standardization.

Memory interference is the contribution of analyte signal from a previous sample onto the next sample analysis. Adequate rinse time with an appropriate rinse solution of the sample introduction system overcomes any memory interference.

Safety Precautions and Waste Handling

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state and local laws and regulations.
See the *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

Hydrochloric acid (HCl) – This acid can cause skin burns. Only mix with approved solutions. Always use this chemical in a fume hood.

Hydrofluoric acid (HF) – This acid is very toxic and absorbs through the skin without pain. This acid is found at trace levels in standard solutions.
Hydrogen Peroxide 30% (H_2O_2) **-** This oxidizer can cause skin burns. Always use in a fume hood.

Nitric acid (HNO_3) – This acid can cause skin burns and burning of the respiratory tract. Add nitric acid to samples in a fume hood to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.
Store concentrated acids in the prep room acid cabinets. Only acids are to be stored in these cabinets. Flammable solvents are stored in flammable liquid storage cabinets. Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Large spills are cleaned by HSE. For large spills of concentrated acid, immediately evacuate the area and dial 1-1-1. HSE will use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.)

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and an annual documented Demonstration of Capability (DOC) which is maintained in the analyst's training records.

Initially, each analyst performing the instrumental analysis must work with an experienced analyst for a period of time until they can independently calibrate the instrument, use the sequence editor to set up the run, perform calculations, interpret raw data, and enter/transfer data into the LIMS. Proficiency is measured through documented audits of the tasks listed, and checking of data as well as an IDOC (Initial Demonstration of Capability) and continued DOCs (Demonstration of Capability) as defined in *QA-SOP11178*.

Sample Collection, Preservation, and Handling

Aqueous undigested samples, aqueous digestates, and solid digestates are analyzed via this SOP. Samples must be analyzed within 6 months of collection for all methods in this SOP.

Aqueous samples are collected in plastic or glass containers. Drinking Water samples are collected in 1-L plastic or glass containers. Aqueous samples are preserved with nitric acid with a pH of < 2 and stored at 0° to 6°C, not frozen.

Aqueous digestates are digested per the method in SOP *T-MET-WI11939* (aqueous samples). Aqueous samples are stored in 50 ml plastic vials at room temperature.

Solid samples are collected in glass containers and stored at 0° to 6°C, not frozen. Refer to the protocols in the digestion SOP T-MET-WI8636. Solid samples are stored in 100 ml plastic vials at room temperature.

Store standards and digestates separately.

The general practice in the metals group is to discard the digestions after all the required metals from a batch of samples have been analyzed and verified in the LIMS. Samples which require the digestate to be held for long term storage are periodically evaluated for discard.

Apparatus and Equipment

A. The following is a list of the hardware used in the Agilent 7700 and 7900 ICP-MS systems.

1. Inductively Coupled Plasma Mass Spectrometers (ICP-MS)

a. The Agilent 7700 and 7900 - The sample introduction system consists of an ESI FAST discrete sampling system, Teflon nebulizer and cooled spray chamber attached to a concentric quartz tube plasma torch.

2. Autosampler

a. The Agilent 7700 and 7900 (E08) systems use an ESI SC-8 FAST autosampler. The Agilent 7900 (E07 & E09) systems use an ESI SC-14 FAST autosampler. The autosampler parameters for each automated run are entered into the Sample List in MassHunter software.

3. Coolflow

a. The Agilent 7700 and 7900 G3292A Recirculating Chillers are set up to deliver cooling liquid to the ICP-MS at a regulated pressure of ~40 psi.

4. Computer

a.The Agilent 7700 and 7900 are controlled by a Windows-based IBM compatible PC with MassHunter software installed.

5. Vacuum Pumps

a. The Agilent 7700 and 7900 both have a 2-stage vacuum system. A rotary pump evacuates the interface chamber; a turbo molecular pump evacuates the ion lens chamber and the analyzer chamber.

B. The following is a list of the apparatus necessary for the setup of an ICP-MS run for analysis:

1. ICP-MS prep batch sheets.
2. Test tube racks
3. Graduated 15-mL polypropylene tubes and caps
4. 50-mL polypropylene screw cap tubes
5. FilterMate 2 µm filters and plungers
6. 10-mL sterile disposable syringes
7. 25-mm syringe filters, PTFE, 0.45µm
8. 30-mL polypropylene medicine cups
9. Fixed volume hand-held pipettes (25 – 2000 µL) - Eppendorf or equivalent.

NOTE: For routine operation, calibration, and maintenance of fixed-volume, hand-held pipettes, see *QA-SOP11880*.

Reagents and Standards

Refer to Form *T-MET-WI12065* for reagent and standard information and the preparation of the following standards and solutions

1. ICP-MS Calibration Standards and ICP-MS LOQ Check Standard Solution (CRI)
2. ICP-MS Initial and Continuing Calibration Verification Standards
3. Interference Check Solutions
4. Rinse/Carrier, Tuning, and Calibration Solutions
5. Internal Standard Solution
6. PDS Solutions
7. General Acids and Chemicals

Calibration

Before calibration is performed, allow the instrument to warm up for the recommended time, usually at least 15 minutes, if the plasma has been off for 15 minutes or less, or at least 30 minutes if the plasma has been off for more than 15 minutes. Daily tuning requirements must be performed prior to calibration of the instrument and after warm up occurs. Refer to forms *T-MET-WI18027*, and *T-MET-WI21589* for detailed tuning and calibration information. Full Tuning and Optimization is done as needed, or when required by the Group Leader.

Calibration is performed daily. Calibrations consist of a blank and one to three standards, followed by analysis of an ICV, ICB, CCV, CRI, ICSA/ICSAB, LRC, CCV, and CCB.

A single point calibration, consisting of a blank and one standard, is used for all analysis except samples requiring a multi-point calibration (either MA MCP or CT RCP protocol or a lab note indicating a multi-point calibration is needed). For samples requiring a multi-point calibration the calibration consists of a blank, “S1”, “S2”, and “S3” standards. The concentrations of the calibration standards are defined in *T-MET-WI12065*.

For acceptance criteria and corrective action, see *T-MET-WI35797*.

Procedure

A. Setting up an ICP-MS run

The procedure is the same for the Agilent 7700 (E05) and 7900 (E07/E08/E09) instruments.

1. In MassHunter open the method batch file used for analysis.

2. Enter the calibration standards and lot numbers in the order of: ICV/ICB/CRI/ICSA/ICSAB/LRC(s)/Rinse(s)/CCV/CCB

3. Scan all samples into the sample list and add PDS/SD and/or dilutions as needed.

4. Add a CCV/CCB block after every 10 samples (or ending the run) as well as an ending CRI/ICSA/ICSAB as needed.

5. Enter the locations of the samples to match the rack in which they will loaded onto the auto-sampler. Ensure that the order of the samples on the batch paperwork/re-read sheet match the order in which they are loaded.

6. Right click on the sample list and print a copy.

7. Use the print out to confirm the sample ID(s) and locations of the samples on the auto-sampler.

8. Load the auto-sampler based off of the printed out sample list. Sign off on the sample list with your initials, employee number and the date.

9. Keep the sample list with the batch(es) paperwork until the batch is imported into the LIMS. Note the analytical and prep batch(es) numbers on the sample list and give to the Group Leader.

B. Pouring an ICP-MS run (Waters only in 50 mL digestion tubes):

The procedure is the same for the Agilent 7700 (E05) and 7900 (E07/E08/E09) instruments.

It is important to minimize any chance of contamination, to both yourself and the samples. Keep your hands and the work area clean at all times. Wear appropriate PPE at all times to prevent exposure to chemicals and samples.

NOTE: See Form *T-MET-WI12065* for standards and solutions used during the analytical run.

1. Choose the appropriate prep batch sheet.

NOTE: When retrieving the batch from the shelf for the first time, record the batch location on the cap of the prep blank in black sharpie. This saves time when returning the batch to the proper location after analysis and shows that the batch has been poured before and verified to be in the correct order. To prevent more than one batch from being assigned to the same location, the digestion trays all have the location written on the foam holder.

2. Verify that each digestion tube is in the correct order according to the prep sheet. If anything is missing, correct the prep sheet and notify PREP or the Group Leader to correct the LIMS and put the sample back in for digestion. If the order is incorrect, reorder the sample digestion tubes to match the paperwork.

3. For batches prepared in hot blocks in 50-mL digestion tubes:

    a. The only tubes that typically need to be poured for the initial run are the post digestion spike (PDS) and serial dilution (SD)samples. These are prepared using the background sample (BKG) or the duplicate background sample (DUP) when limited sample volume is available.

    b. All other samples remain in the 50-mL digestion tubes for analysis.

4. Samples that need to be poured into test tubes:

    a. Obtain the appropriate number of tubes.

    b. Write the last four digits of the sample number and the dilution factor if greater than 1 on each tube. If the MB or LCS must be in tubes, write MB/LCS and the prep batch number/location on the tubes.

    c. Place tubes in test tube racks in the same order as the samples on the prep batch cover sheet and/or re-read.

    d. The analyst must ensure that tubes are labeled correctly, that dilutions are performed accurately, and that the dilutions are recorded on the prep cover sheet and/or re-read.

5. Post-digest spike (PDS):

    a. A PDS is required for each batch (sample volume permitting).

    b. A PDS is prepared by pipetting 0.2 mL of the appropriate PDS solution into a 14-mL graduated test tube, and bringing it to a volume of 10 mL with the background sample, "BKG". If the PDS must be diluted, pipette 0.2 mL of the PDS solution into a 14 mL graduated test tube, add the appropriate amount of background sample, "BKG", and dilute to 10 mL using matrix rinse. If there is limited volume for the background sample, "BKG", then the "DUP" may be used for dilutions of the PDS or SD.

    c. Record the lot number of PDS solution used on the prep batch cover sheet and/or re-read.

6. Serial dilution (SD)

    a. Prepare the SD by diluting the background sample at a dilution that is equal to 5× the dilution factor of the PDS sample (i.e.if PDS= DF1, SD must = DF5; if PDS=DF5, SD must=DF25).

7. Filtering samples

    a. Samples that are cloudy or have particulate suspended in solution must be filtered prior to analysis to prevent clogging of the sample introduction system, which causes run failures and instrument down time to correct the problem.

    b. Samples that are digested using the 50-mL hotblock tubes need to be filtered using the FilterMate filtration devices and plunger.

    c. Attach a filtration device to the plunger and very carefully insert the filter into the hotblock tube until the filter reaches the bottom of the digestion tube.

    d. Remove and discard the plunger.

    e. An alternative for samples in any type of vessel is to filter using a 10-mL sterile disposable syringe fitted with a 0.45μm PTFE syringe filter.

    f. If any samples are filtered, the MB and LCS must also be filtered. If any QC sample is filtered (BKG, DUP, MS, or MSD), then all QC including the MB and LCS must be filtered.

    g. Document all filtrations on the prep batch cover sheet.

NOTE: It is not necessary to filter all samples (in hotblock tubes only) that contain particulate, as long as all of the particulate is settled to the bottom of the tube below the 5-mL mark in 50-mL digestion vessels. The sampling depth of the autosampler probe is set such that the tip of the probe is approximately 100 mm above the bottom of the digestion tube.

8. Verify that all samples in the hotblock tray are in the correct position and/or pour each sample (or sample filtrate) into the appropriate tube. Usually, the order of the batch QC is MB, LCS, (LCSD), Bkg, PDS, DUP, MS, (MSD), and SD. Most importantly, the actual positions of the samples in the tray must match the autosampler table used for analysis.

NOTE: Immediately prior to beginning analysis, the analyst must visually verify that each sample is in the position indicated in the autosampler table used for the run.

9. If not being analyzed immediately,cap any poured tubes to prevent contamination of the samples (hotblock tubes and dilutions prepared in graduated test tubes must be capped tightly except during pouring and/or analysis).

10. Return samples to sample storage, being sure to place the batch back into the location recorded on the prep batch sheet.

11. Record the following on the prep batch cover sheet: initials, employee number, and the date.

12. NOTES:

    a. A PDS and a SD are performed on one sample in each digestion batch. Typically, the background sample is chosen. If the batch QC is split between two samples, the PDS is performed on the background sample accompanied by a matrix spike; the SD is performed on the background sample accompanied by a matrix duplicate. If sample volume is limited, it is acceptable to use the duplicate for the PDS and SD.

    b. Batches with only field blanks or equipment blanks do not need a PDS or a SD.

    c. Air filter batches need only a SD on one sample in the batch (a PDS is not required).

    d. “As Received” samples are analyzed with a blank and LCS, LCSD (prepared by the analyst).

    e. Documentation is of utmost importance. Verify all entries.

Calculations

1. Final Result

    a. Water sample

$$\begin{matrix}\text{Instrument}\\ \text{Reading}\end{matrix} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Volume}}$$

    b. Solid sample (mg/kg)

$$\begin{matrix}\text{Instrument}\\ \text{Reading}\end{matrix} \times \frac{\text{Dilution Volume}}{\text{Aliquot Volume}} \times \frac{\text{Final Volume}}{\text{Sample Weight (grams)}}$$

2. Relative percent different (RPD)

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

Where:

S = first sample value

D = duplicate sample value

3. Spike recovery

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

Where:

SSR = spiked sample result

SR = sample result

SA = spike added

4. Correlation Coefficient

$$r = \frac{\sum XY - \frac{\sum X \sum Y}{N}}{\sqrt{\left(\sum X^2 - \frac{(\sum X)^2}{N}\right)\left(\sum Y^2 - \frac{(\sum Y)^2}{N}\right)}}$$

Where :

X = the known concentration

Y = the instrument response

N = the total number of data points

5. Serial Dilution

$$\% \text{ Difference} = \frac{(5 \times SDR) - SR}{SR} \times 100$$

Where:

SDR = serial dilution result

SR = sample result

6. Methods of standard additions (MSA)
Take either 4 identical aliquots (for 3 point MSA) or 2 identical aliquots (for one point MSA) of the same sample. Leave one unspiked. Spike the other 3 aliquots with different levels of a standard solution (for 3 point MSA) and spike the other aliquot at approximately the indigenous concentration of the sample (for one point MSA). Add blank solution to sample aliquots so that the final volume is the same for all. Use small volumes of spiking solution to avoid diluting the sample more than 10%. Analyze the 4 aliquots or 2 aliquots and record the instrument readings in absorbance. Use the readings and spike values to find the slope and x- and y- intercepts. The x- intercept is the result.

$$\text{Slope} = m = \frac{\sum x_i y_i - (\sum x_i \sum y_i)/n}{\sum x_i^{\,2} - (\sum x_i)^2/n}$$

$$\text{Y-Intercept} = b = \overline{y} - m\overline{x}$$

$$\text{Result} = -\frac{b}{m}$$

$$\text{Correlation Coefficient} = r = \frac{\sum\{(x_i - \overline{x})(y_i - \overline{y})\}}{\sqrt{\left[\sum (x_i - \overline{x})^2\right]\left[\sum (y_i - \overline{y})^2\right]}}$$

The correlation coefficient (r) for the least squares fit must be ³0.995. If the r value is <0.995, the MSA must be repeated at the same dilution. If the r value is again low, the result with the higher r value is verified and both are flagged with a "+" in the data package. If the r value is <0.990, the sample is run at an interference dilution to overcome matrix effects. This usually requires a raised limit of quantitation. If a client requests a particular limit of quantitation that prohibits further dilution, then the sample is repeated at the same dilution and the best of the two results is verified.

7. Average, Standard Deviation and Relative Standard Deviation

The average result, $\overline{x}$, is calculated by summing the individual results and dividing this sum by the number (n) of individual values:

$$\overline{x} = \frac{x_1 + x_2 + x_3 + x_4 + \ldots x_n}{n}$$

Standard deviation is a measure of how precise the average is or how well the individual numbers agree with each other.

$$\text{Standard deviation, S} = \sqrt{\frac{(x_1 - \overline{x})^2 + (x_2 - \overline{x})^2 + (x_3 - \overline{x})^2 + \ldots + (x_n - \overline{x})^2}{n - 1}}$$

Relative standard deviation (RSD) is expressed in percent and is obtained by multiplying the standard deviation by 100 and dividing this product by the average.

$$\text{Relative standard deviation, RSD} = \frac{100 \times S}{\overline{x}}$$

Statistical Information/Method Performance

Generate MDLs and LOQs according to *QA-SOP11892*.

The department supervisor maintains annual study data. The department supervisor requests that a Quality Assurance Specialist update to the LIMS as needed. Update the department database via a download from the LIMS.

Refer to *QA-SOP11896* for statistical information.

Quality Assurance/Quality Control

A. For 6020, 6020A and 6020B, each digestion batch (up to 20 samples) must contain a method blank, LCS, and either an BKG, DUP, MS, MSD or an LCS/LCSD.

B. For 200.8, each digestion batch (up to 10 samples) must contain a method blank, LCS, and either an BKG, DUP, MS or an LCS/LCSD.

C. QC limits for MS/MSD, and LCS/LCSD are established through statistical analysis of historical data.

1. The limits are maintained in the LIMS for the relevant analysis numbers.

2. The limits are evaluated every 6 months and updated as needed.

3. The limits are subject to change without notification.

D. Batch Quality Control

1. For the preparation and concentrations of Batch Quality Control see *T-MET-WI12065*.

2. For the frequency, acceptance criteria and corrective action see Tables I and II.

E. Raw data quality checks

1. Make sure that the run is correctly labeled, and dated and that the corresponding prep sheet is attached to the run.

2. Verify that the appropriate Tuning Report is with the run.

3. For run and batch QC/Calibration frequency, acceptance criteria and corrective action, see Method Specific Tables I (EPA 200.8), and II (EPA 6020). For information on statistical windows refer to *QA-SOP11896*.

4. Spike levels of batch QC are available in the LIMS and on T-MET-WI9082.

5. LOQs are available to analysts in the LIMS.

6. Check to make sure that all results are not> 90% of the Linear Range. If a sample reading is above 90% of the linear range, then reread the sample at a dilution sufficient to bring the sample concentration to approximately the middle of the calibration range.

7. Check that the **absolute** value of all nondetected analytes is less than the LOQ. A technical decision must be made as to whether a reread is warranted for readings <|-LOQ|.

8. Check for carryover between samples. Sample RSD >20%, with a concentration > the LOQ decreasing progressively over time (i.e., Reading 3<2<1). Flag any suspect samples for reread.

NOTE: Whenever a sample is encountered that exceeds the calibration range, the following sample must be checked for carryover for the over range analyte (s) and any suspect results must be deselected and reanalyzed. During reanalysis the sample order must be modified as needed to avoid carryover from any over range sample(s).

9. For all EW (samples from public drinking water sources), check the results against the MCL (maximum contaminant level). If an analyte **exceeds** the MCL notify a verifier at once. An automated email is sent to the Client Service Representative and the state for the analytes listed below with the exception of Pb and Cu which follow the 90th percentile rule (the CSR tracks the lead and copper and notifies the supplier when necessary). Suppliers must be notified within 24 hours.

10. To avoid the risks associated with polyatomic interferences in wastewater and soil matrices all analytes must be analyzed on the collision cell ICP-MS instruments which include Agilent 7900, Equipment #27813, Equipment #30647, Equipment #30921).

   a. Because EPA 200.8 rev. 5.4 does not currently allow the analysis of drinking water samples using collision cell ICP-MS all drinking water samples must be analyzed using a method with no gas mode for all analytes on the Agilent 7700, Equipment #19204.

11. Check the internal standard level for the entire run. If the internal standard reading for any sample is out of the acceptance range of the S0 reading, evaluate whether this is a result of the sample matrix or drift from other samples in the run. Only reread the sample at a dilution if the internal standard appears to be out of range due to the sample matrix. For samples where an internal standard is above the acceptance range it is acceptable to reprocess the data using an alternate internal standard. This is the preferred approach in order to avoid raised reporting limits. The acceptance criteria differ based on the method being used. Refer to Table I, and II for the method acceptance criteria.

NOTE: The internal standard is added in equal concentration to all of the samples and standards via a dedicated line on the peristaltic pump. The analytical lines referenced to an internal standard report a corrected concentration value based on the ratio of analyte to internal standard intensities. All the calculations for determining concentration are based on Intensity Ratio (IR). The IR is defined as the background corrected intensity signal of the analyte line (Ia) divided by the internal standard value (Iis). IR= Ia/Iis.

12. For TCLP and SPLP samples, an MSA (method of standard additions) is required if:

   a. The sample concentration falls between 80% and 100% of the regulatory limit.

   b. If the TCLP or SPLP matrix spike (QA) recovers <20%, all samples in the leachate batch must be reanalyzed using the method of standard additions for that analyte.

13. When items 1 – 12 are complete, check the following:

   a. All samples requiring reread/redigestion are listed on the reread/redigestion schedule forms.

   b. The data are uploaded to LIMS via IDAT by the reviewer.

Figure I - Daily Performance Acceptance Criteria and Tuning Solution Concentrations

Be (9)	10 or 100 PPB
Mg (24)	10 or 100 PPB
Co (59)	10 or 100 PPB
In (115)	10 or 100 PPB
Pb (208)	10 or 100 PPB
CeO/Ce	<3%
Ce++/Ce	<3%

Background	<30 cps @ Mass 220
RSD	<5%
Peak Width at 10% Peak Height	<0.9 AMU
Res/Axis	< +/- 0.1 AMU from unit mass

Q-EQA-FRM6869 Nonconformance Form
QA-SOP11178 Demonstrations of Capability
QA-SOP11880 Laboratory Equipment Verifications - Balance, Syringe, Pipette, Weights, and Other Equipment
QA-SOP11892 Determining Method Detection Limits and Limits of Quantitation
QA-SOP11896 Establishing Control Limits
QA-SOP11901 Instrument Maintenance and Calibration
S-SS-WI10695 Liquid Sample Preservation, Sample Splitting, and Turbidity for metals by EPA Methods 200.7 and 200.8
T-MET-FRM8822 ICPMS Analysis
T-MET-WI11939 Digestion by EPA 200.8 for the Analysis of Total Recoverable Metals in Water by ICPMS
T-MET-WI12065 Working Instructions for Preparation of ICP-MS Solutions and Standards
T-MET-WI18026 Instrument Operations for Agilent 7500
T-MET-WI18027 Instrument Operations for Agilent 7700
T-MET-WI18028 Instrument Maintenance for Agilent 7500
T-MET-WI18029 Instrument Maintenance for Agilent 7700
T-MET-WI21589 Instrument Operations for Agilent 7900
T-MET-WI21590 Instrument Maintenance for Agilent 7900
T-MET-WI35797 Metals Data Verification Process

End of document

Version history

Version	Approval	Revision information	
9	21.MAR.2019		
10	05.JUL.2019		
11	12.NOV.2020		

eurofins	Always check on-line for validity. **Sample Preparation of Leachates and Other Wastewater for Analysis of Total Metals by Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)**	Level: **Work Instruction**
Document number: **T-MET-WI11937**		
Old Reference: **1-P-QM-WI-9015165**		
Version: **12**		Organisation level: **5-Sub-BU**
Approved by: **UBFR** Effective Date **19-JAN-2015**	Document users: **6_EUUSLA_Metals_ICP-MS Prep, 6_EUUSLA_Metals_ICP-MS Prep Verifiers, 6_EUUSLA_Metals_Management**	Responsible: **5_EUUSLA_Metals_Manager**

This documentation has been prepared by Eurofins Lancaster Laboratories Environmental LLC and its affiliates ("Eurofins"), solely for their own use. The user of this document agrees by its acceptance to return it to Eurofins upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use if for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF EUROFINS IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY EUROFINS IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES.

Revision Log
Reference
Cross Reference
Purpose
Scope
Basic Principles
Reference Modifications
Definitions
Interferences
Safety Precautions and Waste Handling
Personnel Training and Qualifications
Sample Collection, Preservation, and Handling
Apparatus and Equipment
Reagents and Standards
Calibration
Procedure
Block Digestion Instructions
Calculations
Statistical Information/Method Performance
Quality Assurance/Quality Control

Revision Log

Revision: 12		**Effective Date:** This version
Section	**Justification**	**Changes**
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Reference	No longer applicable to the procedure	Removed 3010A Modified
Reference Modifications	No longer applicable to the procedure	Removed 3010A modifications
Procedure A and B	Clarification	Reworded numbers 2, 5 and 6 to clarify steps found in the EPA procedure for 3020A

Revision: 11		**Effective Date:** Nov 25, 2013
Section	**Justification**	**Changes**

Revision: 11		**Effective Date:** Nov 25, 2013
Revision Log	Formatting requirement per 1-P-QM-QMA-9017356	Removed revision logs up to the previous version
Throughout Document	Reflect re-identification of documents in EtQ	Replaced all prior Level 1, 2, 3, and 4 document numbers (analyses excluded) with EDR numbers
Cross Reference	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,…
Sample Collection, Preservation, and Handling	Process change	Changed sample storage temperature from 4° ± 2° C to 0° to 6° C but not frozen, prior to digestion.
Safety Precautions and Waste Handling	No longer used	Deleted text pertaining to Hydrofluoric Acid.
Reagent and Standards	Reflect current procedure	Added text pertaining to preparing solutions using different volumes, is acceptable, if exact ratios are maintained.
Procedure A	Reflect current procedure	Deleted text, in NOTE, pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text, to NOTE, pertaining to samples concentration
Procedure A.1	Reflect current procedure	Added text pertaining to adding spike solution, after the sample has been poured. Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.
Procedure B	Reflect current procedure	Deleted text in NOTE pertaining to using a smaller sample aliquot if insufficient sample is submitted. Deleted text to NOTE pertaining to samples concentration
Procedure B.1	Reflect current procedure	Added text pertaining to reference to Analysis #6142, 6123, 6125,… for batch quality control
Block Digestor Instructions	Reflect current procedure	Clarified instruction steps. Deleted text pertaining to the difference between sample temperature and display temperature.
Quality Assurance/Quality Control	Reflect current procedure	Added reference to Analysis #6142, 6123, 6125,… for batch quality control requirements.

Reference

1. Test Methods for Evaluating Solid Wastes, SW-846 Method 3020A, July 1992.
2. *Chemical Hygiene Plan*, current version.

Cross Reference

Document	Document Title
Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …	Metals by Inductively Coupled Plasma Mass Spectrometry for SW-846 Methods 6020/6020A (aqueous, solid, tissue) and EPA 200.8 (aqueous)
1-P-QM-FOR-9009182	Working Instructions for Prep Solutions and Standards
1-P-QM-QMA-9015390	Demonstrations of Capability

Purpose

This digestion procedure is used to prepare leachate and other wastewater samples for measurement of total metals by inductively coupled plasma-mass spectrometer (ICP-MS) following SW 846 protocol.

Scope

This acid digestion procedure is used by the Metals Department of the Environmental Sciences Division to prepare leachate, wastewater, surface water, and groundwater samples for measurement of total recoverable metals by inductively coupled plasma-Mass Spectrometer (ICP-MS) following SW-846.

Basic Principles

A mixture of nitric acid and the sample is refluxed in a covered beaker/digestion vessel at low volume to dissolve metals. It is cooled and brought up to volume with reagent water.

Reference Modifications

1. A 50–mL sample aliquot and final volume is used instead of 100-mL to improve digestion throughput, conserve sample usage, and limit waste generation. Because all reagents are also adjusted so that concentrations are equivalent to a 100–mL aliquot, there is no impact on the data.

2. Ribbed watch glasses are not used; samples are evaporated without watch glasses in nonmetallic hoods to speed evaporation. No contamination trends have been observed in prep blanks evaporated without watch glasses.

Definitions

1. ACS – American Chemical Society
2. ASTM – American Society of Testing and Materials
3. D – Sample Duplicate
4. DOC – Demonstration of Capability
5. IDOC – Initial Demonstration of Capability
6. LCS/LCSD – Laboratory Control Sample/ Laboratory Control Sample Duplicate
7. LCSW – Laboratory Control Sample Water
8. LLENS – the computer program that integrates a PC with an analytical balance to collect data directly from the balance. The program organizes the data and transmits the readings to the LIMS.
9. LIMS – Laboratory Information Management Systems
10. LLI Sample ID – unique 7-digit number assigned to a client sample.
11. LOQ – Limit of Quantitation
12. MDL – Method Detection Limit
13. MS (R) – Matrix Spike
14. MSD (M) – Matrix spike duplicate
15. PB/PBW – Preparation Blank/ Preparation Blank Water
16. QC – Quality Control

17. Method Blank – equivalent to a Preparation Blank. A designated sample designed to monitor for sample contamination during the analysis process. A volume of reagent laboratory water is typically used to monitor water sample analysis, while solids blanks consist of a purified solid matrix or just the reagents used in the test. The blank demonstrates that no artifacts were introduced during the analysis process.

18. SOP – Standard Operating Procedure

19. SPLP – Synthetic Precipitation Leaching Procedure

20. STLC – Soluble Threshold Limit Concentration

21. TCLP – Toxicity Characteristic Leaching Procedure

22. U or US – unspiked background sample

Interferences
Not applicable to this procedure.

Safety Precautions and Waste Handling
All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations.

See *Chemical Hygiene Plan* for general information regarding employee safety, waste management, and pollution prevention.

Preparing samples for inorganic analysis involves working with concentrated acids and other chemicals which are dangerous if not handled carefully:

> **Nitric acid (HNO_3)** – This acid can cause skin burns. Add nitric acid to samples in a hood or use the designated dispensing equipment to avoid exposure to toxic fumes.

When diluting strong acids, never add water to acid; always add acid to water.

Store concentrated acids in the prep room acid lockers. Only acids are to be stored in these lockers. (Store solvents in the flammable liquid storage cabinet.) Some concentrated acids are kept in the acid reagent bottles on prep room counters. Fill reagent bottles in an operating fume hood using caution to avoid spills.

Perform acid digestions in hoods that are turned on and have active alarms. Notify a supervisor immediately if the hood is malfunctioning or the alarm sounds.

Samples that contain dust may be hazardous. Open in a fume hood.

When a hazardous flag is added indicating possible cyanide, special precautions are required to avoid exposure to hydrogen cyanide gas. Contact your supervisor prior to adding acid. Always open these samples and add the acid in a hood.

Use spill pillows to absorb large acid spills (small spills are cleaned with wet paper towels.) Use SPILL-X-A , soda ash or equivalent, to neutralize any remaining acid and then rinse the area thoroughly with water. Spill pillows and SPILL-X-A are stored on the prep room shelf. Soda ash is located in the stairwell adjacent to the prep room.

Dispose of acid waste properly. Collect all acid digestions, waste solutions, and expired reagent solutions in waste containers. When the acid waste containers are full, a designated acid waste handler transfers the waste to the acid neutralization tank.

Personnel Training and Qualifications

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability for this or an equivalent procedure.

Initially, each employee performing this digestion procedure must work with an experienced employee for a period of time until they can independently set up batches and perform the necessary steps outlined in this procedure. Proficiency is measured through documentation of the critical steps in this procedure, over checking of data as well as an IDOC.

The IDOC and the DOC consists of four laboratory control samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The criteria include the calculation of mean accuracy and standard deviation. Refer to 1-P-QM-QMA-9015390, for specific requirements. A DOC is performed annually and is maintained in the analyst’s training records.

Sample Collection, Preservation, and Handling

Samples are collected in plastic containers and preserved to a pH of <2 with HNO_3. (Samples to be analyzed for soluble metals requiring filtration at the lab must be submitted unpreserved. The sample is run through a 0.45-micron filter within 5 days of receipt and then preserved.) The pH is checked upon receipt and adjusted as necessary by Sample Support; samples that are pH adjusted at the lab must not be digested for a minimum of 24 hours. If samples fail to maintain a pH of <2 the Client Service Representative is notified for further direction. Samples are stored at 0° - 6°C, but not frozen, prior to digestion. Samples must be digested within 6 months of collection. Digested samples are stored in plastic at room temperature and have a 6 month holding time.

Apparatus and Equipment

1. Polypropylene containers (digestion vessels) – certified clean and Class A equivalent

2. Watch glasses or reflux caps

3. 50–mL graduated cylinders or other appropriate graduated cylinders if necessary

4. 50–mL volumetric flasks or other appropriate Class A volumetric flasks if necessary

5. 250-mL beakers or other appropriate beakers

6. Hotblocks or hot plates, adjustable and capable of maintaining a temperature of 90° to 95°C

Reagents and Standards

For reagent preparation, shelf life, and storage conditions, see Form 1-P-QM-FOR-9009182. .

Nitric acid, HNO_3 – Fisher, Trace Metal Grade, or equivalent. Store at room temperature and re-evaluate annually.

NOTE: It is acceptable to prepare solutions using multiples of indicated volumes if exact ratios are maintained.

Calibration

Not applicable to this method.

Procedure

This SOP has been set up to outline the procedures for both hotblock and hot plate digestions (see below). Choose the procedure that corresponds to the sample heating technique being used for sample digestion.

A. Hotblock

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Transfer 50 mL of well mixed sample to a 68-mL digestion vessel. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1-P-QM-FOR-9009182. For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

   NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluids are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the vessel in the hotblock at 90° to 95°C, and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the digestion vessel is allowed to go dry.

3. Cool the digestion vessel and add another 1.5–mL portion of HNO_3.

4. Cover the digestion vessel with a reflux cap and return to the hotblock. Increase the temperature of the hotblock so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and re-prepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the digestion vessel and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the digestion vessel to go dry.

7. Remove the digestion vessel and add approximately 5 mL of reagent water, mix, and continue warming for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Adjust volume to the 50-mL mark on the digestion vessel with reagent water and mix. Seal vessel with screw cap.

9. The sample is now ready for analysis.

B. Hot Plates

NOTE: When insoluble matter is present in the digested sample, allow it to settle by gravity or filter prior to introduction to the instrument. If any samples are filtered, the prep blank and LCS must also be filtered.

NOTE: For soluble metals analysis, filter unpreserved sample through 0.45-micron filter paper. Adjust the filtered sample to pH <2 with nitric acid preserving solution. Measure the volume of sample, as stated in this procedure, and digest as normal. The prep blank and spiked LCS must also be prepared with filtered reagent water.

NOTE: If the sample contains high solids, use a smaller aliquot of the sample and bring sample to final volume as stated in this procedure. Make appropriate acid, reagent, and spike volume adjustments based on sample final volume.

1. Shake sample well. Use a 50 mL graduated cylinder to transfer 50 mL of well mixed sample into a 250 mL beaker. After the sample has been poured, add the spiking solution. For sample batch spiking procedures see form 1-P-QM-FOR-9009182. For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

NOTE: For leachate samples, use the appropriate extraction fluid for the PBW and LCS. The extraction fluid are as follows: TCLP, SPLP, STLC, ASTM, Filtration, and Elutriate.

2. Add 1.5 mL of HNO_3. Place the beaker on a hot plate and cautiously evaporate to low volume (about 5 mL), making certain that the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry.

3. Cool the beaker and add another 1.5–mL portion of HNO_3.

4. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that gentle reflux action occurs.

NOTE: If a sample is allowed to go to dryness, low recoveries result. If this occurs, discard the sample and reprepare in a new batch.

5. Continue heating (refluxing), adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).

6. When digestion is complete, uncover the beaker and evaporate to low volume (about 3 mL). Do not allow any portion of the bottom of the beaker to go dry.

7. Remove the beaker and add approximately 5 mL of reagent water, mix, and continue warming the beaker for 10 to 15 minutes to allow additional solubilization of any residue to occur.

8. Allow to cool. Transfer the solution to a 50–mL volumetric flask. Adjust volume to the 50 mL mark with reagent water and mix.

9. Transfer to a polypropylene bottle.

10. The sample is now ready for analysis.

Block Digestion Instructions

1. Turn block digestor on by pressing rocker switch located on the cord.

2. Wait about 8 seconds until controller display indicates current block temperature.

3. PRESS and hold STAR (*) key.

4. The display shows the Set Point Temperature.

5. The digits can be changed to the desired value by pressing the up and down arrow keys while holding the (*) key.

6. Confirm Control Point temperature is set to the block temperature that provides 90° to 95°C.

NOTE: See HotBlock Control Point Temperature Logbook to obtain control point temperature setting for the HotBlock being used for digestion. If necessary, adjust Control Point temperature to the proper setting.

NOTE: Polypropylene containers must not be heated above 130°C.

Calculations

Not applicable to this procedure.

Statistical Information/Method Performance

Not applicable to this method. See analysis procedure.

Quality Assurance/Quality Control

A method blank, sample duplicate, sample matrix spike, sample matrix spike duplicate, and laboratory control sample must be performed with every digestion batch (20 samples or less). Each piece of batch QC is digested following the procedure in this SOP.

For sample batch quality control requirements see Analysis #6142, 6123, 6125, 10801, 6126, 6127, 6129, 6128, 6132, 6131, 6133, 6134, 6140, 6136, 6137, 6138, 6143, 6139, 6135, 6124, 6141, 6146, 6144, 6147, 6145, …

End of document

Version history

Version	Approval	Revision information	
12	19.JAN.2015		

ATTACHMENT 3

LABORATORY CERTIFICATIONS

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL LLC
2425 New Holland Pike
Lancaster, PA 17601
Dorothy M. Love   Phone: 717-556-7327

ENVIRONMENTAL

Valid To: November 30, 2022   Certificate Number: 0001.01

In recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Demands				
COD	---------------	EPA 410.4	---------------	---------------
Total Organic Carbon	---------------	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310C-2011	EPA 9060A SM 5310 B-2011
Anions				
Ammonia	---------------	EPA 350.1	EPA 350.1	SM 4500-NH3 B/C-2011
Fluoride	---------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrate (as N)	---------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Nitrite (as N)	---------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Bromide	------------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Chloride	------------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Sulfate	------------------	EPA 300.0 EPA 9056A	EPA 9056A	EPA 9056A EPA 300.0
Wet Chemistry				
Alkalinity	------------------	SM 2320B-2011	---------------	---------------
Corrosivity	------------------	---------------	SW-846 Chapter 7	SW-846 Chapter 7
Cyanide	------------------	EPA 9012B	EPA 9012B	EPA 9012B
Filterable Residue (TDS)	------------------	SM 2540C-2011	---------------	---------------
Flashpoint	------------------	---------------	EPA1010A	EPA 1010A
Grain Size	------------------	---------------	---------------	ASTM D422
Hexavalent Chromium Digestion	------------------	---------------	---------------	EPA 3060A
Hexavalent Chromium	------------------	EPA 7196A EPA 7199	EPA 7196A EPA 7199	EPA 7196A EPA 7199
Ignitability	------------------	---------------	40 CFR 261.21	40 CFR 261.21
Nitrate/Nitrite	------------------	---------------	EPA 353.2	---------------
Non-filterable Residue (TSS)	------------------	SM 2540D-2011	---------------	---------------
pH	------------------	SM 4500 H+B-2011 EPA 9040B EPA 9040C	EPA 9040B EPA 9040C	EPA 9045C EPA 9045D
Phenol	------------------	EPA 9066	EPA 9066	---------------
Reactivity	------------------	---------------	SW-846 Chapter 7.3	SW-846 Chapter 7.3
Sulfide	------------------	EPA 376.1 EPA 376.2 SM 4500 S2D-2011 SM 4500 S2F-2011	---------------	---------------
Total Residue	------------------	SM 2540B-2011	---------------	SM 2540G-2011
Metals				
Metals Digestion	------------------	EPA 3005A EPA 3010A EPA 3020A	EPA 3010A EPA 3020A	EPA 3050B
Aluminum	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Antimony	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Arsenic	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Barium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Beryllium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Boron	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Cadmium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Calcium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Chromium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Cobalt	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Copper	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Iron	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lead	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Lithium	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Molybdenum	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Magnesium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Manganese	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Mercury	------------------	EPA 7470A	EPA 7470A	EPA 7471A EPA 7471B
Nickel	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Potassium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Selenium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Silicon	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Silver	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sodium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Strontium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Sulfur	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Thallium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Thorium	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tin	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Titanium	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Tungsten	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Uranium	------------------	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B	EPA 6020A EPA 6020B
Vanadium	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zinc	------------------	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B	EPA 6010C EPA 6010D EPA 6020A EPA 6020B
Zirconium	------------------	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D	EPA 6010C EPA 6010D
Purgeable Organics (Volatiles)				
Volatile Preparation	------------------	EPA 5030C	EPA 5030C	EPA 5035A
Acetone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acetonitrile	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrolein	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Acrylonitrile	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Allyl chloride	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Alcohol	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Amyl Methyl Ether	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Alcohol	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butyl Formate	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Benzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromodichloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromoform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Bromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Butanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
sec-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
tert-Butylbenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon disulfide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Carbon tetrachloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloro-1,3-butadiene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroacetonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1-Chlorobutane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chlorodifluoromethane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chloroethyl Vinyl Ether	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloroform	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	**Drinking Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
1-Chlorohexane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Chloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Chlorotoluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Cyclohexanone	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Di-Isopropyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dibromochloromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromo-3-chloropropane	EPA 524.2	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
Dibromomethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dibromoethane (EDB)	------------------	EPA 8260C/D EPA 8011	EPA 8260C/D EPA 8011	EPA 8260C/D
1,2-Dichlorobenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,4-dichloro-2-butene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorodi-fluoromethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Dichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,2-Dichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Dichlorofluoromethane	EPA 524.2	---------------	---------------	---------------
1,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2,2-Dichloropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1-Dichloropropene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
cis-1,3-Dichloropropene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
trans-1,3-Dichloropropene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Dioxane	------------------	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM	EPA 8260C/D EPA 8260C/D SIM
Ethanol	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethylbenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Ethyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Freon-113	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Gasoline Range Organics (GRO) [Volatile Petroleum Hydrocarbons (VPH)]	------------------	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101	EPA 8015C EPA 8015D EPA 8260C/D NW TPH-Gx MA VPH AK101
Heptane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Hexanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Hexachlorobutadiene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Hexachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isobutyl Alcohol	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropyl Alcohol	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Isopropylbenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,4-Isopropyltoluene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylacrylonitrile	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acetate		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Acrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Iodide	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Ethyl ketone	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylene Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Isobutyl Ketone	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Methacrylate	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methyl Tert-Butyl Ether	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
4-Methyl-2-pentanone	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Methylcyclohexane		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
2-Nitropropane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Naphthalene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Pentachloroethane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Propionitrile	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
n-Propylbenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Styrene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tert-Amyl Ethyl Ether		EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2,2-Tetrachloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrachloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Tetrahydrofuran	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Toluene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trichlorobenzene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,1-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,1,2-Trichloroethane	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Trichloroethene	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Trichlorofluoromethane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,3-Trichloropropane	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2,4-Trimethylbenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3,5-Trimethylbenzene	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Vinyl Acetate	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Vinyl Chloride	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
Xylenes, Total	------------------	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,2-Xylene (o-Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D
1,3+1,4-Xylene (m+p Xylene)	EPA 524.2	EPA 8260C/D	EPA 8260C/D	EPA 8260C/D

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Extractable Organics (Semivolatiles)				
Acenaphthene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acenaphthylene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Acetophenone	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Acetylaminofluorene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Alkylated PAHs	------------------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
4-Aminobiphenyl	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Amino-4,6-dinitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
4-Amino-2,6-dinitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Aniline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Anthracene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Atrazine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzaldehyde	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzoic acid	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Benzo (a) anthracene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (b) fluoranthene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (k) fluoranthene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (ghi) perylene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (a) pyrene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Benzo (e) pyrene	------------------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Benzyl Alcohol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Biphenyl	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Chloroethoxy) Methane	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Chloroethyl) Ether	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
bis (2-Chloroisopropyl) Ether	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
bis (2-Ethylhexyl) Phthalate	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
4-Bromophenylphenyl Ether	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Butyl benzyl Phthalate	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
Caprolactam	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbazole	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Carbon Range Organics C8-C44 (including subsets of this range i.e. HRO, MRO, ORO, RRO)	------------------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
4-Chloroaniline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chloro-3-methylphenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chlorobenzilate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Chloronaphthalene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chloronaphthalene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Chlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Chlorophenyl phenyl ether	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Chrysene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Cresols (Methyl phenols)	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
cis-/trans-Diallate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Diamino-6-nitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
2,6-Diamino-4-nitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Dibenzo (a,h) acridine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dibenzo (a,h) anthracene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Dibenzofuran	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,2-Dichlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3-Dichlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,4-Dichlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,3-Dichlorobenzidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diesel Range Organics (DRO) [Extractable Petroleum Hydrocarbons (EPH)]	------------------	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103 AK102/103-SV	EPA 8015C EPA 8015D NWTPH DX MA EPH TX1005/1006 AK102/103
2,4-Dichlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,6-Dichlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diethyl Phthalate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dimethoate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
p-Dimethylaminoazobenze	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
7,12-Dimethylbenz (a) anthracene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dimethylphenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Dimethyl Phthalate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,3'-Dimethylbenzidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Di-n-butyl Phthalate	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Di-n-octyl phthalate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3,5-Dinitroaniline	------------------	EPA 8330B	EPA 8330B	EPA 8330B
1,3-Dinitrobenzene	------------------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
1,4-Dinitrobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4-Dinitrotoluene	------------------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
2,6-Dinitrotoluene	------------------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
1,4-Dioxane	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Diphenylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Diphenyl ether	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2-Diphenylhydrazine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Ethyl Methane Sulfonate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Fluoroanthene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Fluorene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobenzene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Hexachlorobutadiene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachlorocyclo-pentadiene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloroethane	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexachloropropene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Indeno (1,2,3-cd) Pyrene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Isodrin	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Isophorone	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Isosafrole	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Methycholanthrene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Methyl-4,6-dinitrophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Methyl methane sulfonate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Methylnaphthalene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylnaphthalene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
2-Methylphenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Methylphenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Naphthalene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
1,4-Naphthoquinone	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1-Naphthylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Naphthylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroquinoline-1-oxide	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitroaniline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
3-Nitroaniline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitroaniline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Nitrobenzene	------------------	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B	EPA 8270D/E EPA 8330B
Nitroglycerin	------------------	EPA 8330B	EPA 8330B	EPA 8330B
2-Nitrophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
4-Nitrophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Nitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
3-Nitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
4-Nitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
5-Nitro-o-toluidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitroso-di-n-butylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiethylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodimethylamine	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
n-Nitrosomethylethylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosomorpholine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodi-n-propylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosodiphenylamine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopiperidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
n-Nitrosopyrrolidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	------------------	EPA 8330B	EPA 8330B	EPA 8330B

Parameter/Analyte	Potable Water	Non-Potable Water	Solid Hazardous Waste	
			Aqueous	Solid
2,2-Oxybis (1-chloropropane)	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachloronitrobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentachlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pentaerythritol Tetranitrate (PETN)	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Perylene	------------------	EPA 8270D/E SIM	EPA 8270D/E SIM	EPA 8270D/E SIM
Phenacetin	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Phenanthrene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Phenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2-Picoline	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pronamide	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Pyrene	------------------	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM	EPA 8270D/E EPA 8270D/E SIM
Pyridine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Safrole	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4,5- Tetrachlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,3,4,6-Tetrachlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethyl dithiopyrophosphate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetraethy lead	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Tetryl	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Thionazin	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
o-Toluidine	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,2,4-Trichlorobenzene	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
1,3,5-Trinitrobenzene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
2,4,5-Trichlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trichlorophenol	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
O,O,O-Tri-ethylphosphorothioate	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
2,4,6-Trinitrotoluene	------------------	EPA 8330B	EPA 8330B	EPA 8330B
Organochlorine Pesticides				
Aldrin	------------------	EPA 8081B	EPA 8081B	EPA 8081B
alpha-BHC	------------------	EPA 8081B	EPA 8081B	EPA 8081B
beta-BHC	------------------	EPA 8081B	EPA 8081B	EPA 8081B
delta-BHC	------------------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-BHC (Lindane)	------------------	EPA 8081B	EPA 8081B	EPA 8081B

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
alpha-Chlordane	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Chlordane (Technical)	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Chlorobenzilate	------------------	EPA 8081B	EPA 8081B	EPA 8081B
2,4’-DDD	------------------	EPA 8081B	EPA 8081B	EPA 8081B
2,4’-DDE	------------------	EPA 8081B	EPA 8081B	EPA 8081B
2,4’-DDT	------------------	EPA 8081B	EPA 8081B	EPA 8081B
4,4’-DDD	------------------	EPA 8081B	EPA 8081B	EPA 8081B
4,4’-DDE	------------------	EPA 8081B	EPA 8081B	EPA 8081B
4,4’-DDT	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Diallate	------------------	EPA 8081B	EPA 8081B	EPA 8081B
1.2-Dibromo-3-chloropropane (DBCP)	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Dieldrin	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Dinoseb	------------------	EPA 8270D/E	EPA 8270D/E	EPA 8270D/E
Endosulfan I (alpha)	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan II (beta)	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Endosulfan Sulfate	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Aldehyde	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Endrin Ketone	------------------	EPA 8081B	EPA 8081B	EPA 8081B
gamma-Chlordane	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Heptachlor Epoxide	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorobenzene	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Hexachlorocyclopentadiene	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Isodrin	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Methoxychlor	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Mirex	------------------	EPA 8081B	EPA 8081B	EPA 8081B
Toxaphene	------------------	EPA 8081B	EPA 8081B	EPA 8081B
PCBs (Aroclors)				
PCB-1016 (Arochlor)	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1221	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1232	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1242	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1248	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1254	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1260	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1262	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB-1268	------------------	EPA 8082A	EPA 8082A	EPA 8082A
PCB congeners (209)	------------------	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C	EPA 1668A EPA 1668C

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Herbicides				
2,4,5-T	------------------	EPA 8151A	EPA 8151A	EPA 8151A
2,4,5-TP (Silvex)	------------------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-D	------------------	EPA 8151A	EPA 8151A	EPA 8151A
2,4-DB	------------------	EPA 8151A	EPA 8151A	EPA 8151A
Dalapon	------------------	EPA 8151A	EPA 8151A	EPA 8151A
Dicamba	------------------	EPA 8151A	EPA 8151A	EPA 8151A
Dichlorprop	------------------	EPA 8151A	EPA 8151A	EPA 8151A
Dinoseb	------------------	EPA 8151A	EPA 8151A	EPA 8151A
MCPA	------------------	EPA 8151A	EPA 8151A	EPA 8151A
MCPP	------------------	EPA 8151A	EPA 8151A	EPA 8151A
Pentachlorophenol	------------------	EPA 8151A	EPA 8151A	EPA 8151A
PCB Homologues				
Monochlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Dichlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Trichlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Tetrachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Pentachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Hexachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Heptachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Octachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Nonachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Decachlorobiphenyls	------------------	EPA 680	EPA 680	EPA 680
Dioxins/Furans				
2,3,7,8-TCDD	EPA 1613B	EPA 8290A	EPA 8290A	EPA 8290A
2,3,7,8-TCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,7,8-PeCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8-PeCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8-HxCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
2,3,4,6,7,8-HxCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,-HxCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,6,7,8-HxCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,7,8,9-HxCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,7,8,9-HpCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
1,2,3,4,6,7,8-HpCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
OCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
OCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total HpCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total HxCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total PeCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A

Parameter/Analyte	**Potable Water**	**Non-Potable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Total PeCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDD	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Total TCDF	------------------	EPA 8290A	EPA 8290A	EPA 8290A
Misc. Headspace Analysis				
Carbon dioxide	------------------	RSK-175	RSK-175	------------------
Ethane	------------------	RSK-175	RSK-175	------------------
Ethene	------------------	RSK-175	RSK-175	------------------
Methane	------------------	RSK-175	RSK-175	------------------
Hazardous Waste Characteristics				
Toxicity Characteristic Leaching Procedure	------------------	------------------	EPA 1311	EPA 1311
Synthetic Precipitation Leaching Procedure	------------------	------------------	EPA 1312	EPA 1312
ASTM Leaching Procedure	------------------	------------------	ASTM D3987-85	ASTM D3987-85
Other				
Perchlorate	------------------	EPA 6850	EPA 6850	EPA 6850
Hydrazine	------------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Formaldehyde	------------------	------------------	EPA 8315A	EPA 8315A
Methylhydrazine	------------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	------------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	------------------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction/Cleanup	------------------	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3510C EPA 3511 EPA 3660B, 3620C, 3665A	EPA 3546 EPA 3550C EPA 3660B, 3620C, 3665A, 3640A

Parameter/Analyte	**Drinking Water**	**Nonpotable Water**	**Solid Haz.Waste**
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl Perfluorooctane-Sulfonamidoacetic Acid (NetFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
N-methyl Perfluoroctane-Sulfonamidoacetic Acid (NMeFOSAA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorobutanesulfonic Acid (PFBS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorodecanoic Acid (PFDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	**Drinking Water**	**Nonpotable Water**	**Solid Haz.Waste**
Perfluorododecanoic Acid (PFDoDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroheptanoic Acid (PFHpA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexanesulfonic Acid (PFHxS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexanoic Acid (PFHxA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorononanoic Acid (PFNA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanesulfonic Acid (PFOS)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanoic Acid (PFOA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorotetradecanoic Acid (PFTeDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorotridecanoic Acid (PFTrDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroundecanoic Acid (PFUnDA)	EPA 537 EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoro-n-butanoic Acid (PFBA)	---------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoro-n-pentanoic Acid (PFPeA)	---------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	**Drinking Water**	**Nonpotable Water**	**Solid Haz.Waste**
8:2 Fluorotelomersulfonic Acid (8:2FTS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
4:2 Fluorotelomersulfonic Acid (4:2-FTS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoropentanesulfonic Acid (PFPeS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
6:2 Fluorotelomersulfonic Acid (6:2-FTS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluoroheptanesulfonic Acid (PFHpS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorononanesulfonic Acid (PFNS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorodecanesulfonic Acid (PFDS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
10:2 Fluorotelomersulfonic Acid (10:2-FTS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorododecanesulfonic Acid (PFDoDS)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorohexadecanoic Acid (PFHxDA)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctadecanoic Acid (PFODA)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
Perfluorooctanesulfonamide (PFOSA)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
N-methylperfluoro-1-octanesulfonamide (NMePFOSA)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15
(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

Parameter/Analyte	Drinking Water	Nonpotable Water	Solid Haz.Waste
N-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	----------------------	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15	PFAS by LCMSMS Compliant with QSM 5.3 Table B-15

End of DoD ELAP section of scope

Start of KY, WY, and ISO 17025 section of scope

In addition, in recognition of the successful completion of the A2LA evaluation process (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and for the test methods applicable to Kentucky Statute KRS 224.60-130(2)(a), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP-MS Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.-Electronic Probes (pH, F^-, O_2), Oxygen Demand, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, TCLP, Total Organic Carbon, Turbidity, Liquid Chromatography/Mass Spectrometry/Mass Spectrometry, High Resolution Gas Chromatography/Mass Spectrometry

Parameter/Analyte	Tissue	Nonpotable Water	Solid Hazardous Waste	
			Aqueous	Solid
Other				
Perchlorate	Food & Food Products EPA 6850	EPA 6850	EPA 6850	EPA 6850
Hydrazine	--------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Methylhydrazine	--------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
1,1-Dimethylhydrazine	--------------	EPA 8315A MOD	EPA 8315A MOD	EPA 8315A MOD
Volatile Preparation	--------------	EPA 5030A EPA 5030C	EPA 5030A EPA 5030C	EPA 5035 EPA 5035A
Organic Extraction/ Cleanup	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3510C EPA 3511 EPA 3660B EPA 3620C EPA 3665A	EPA 3546 EPA 3550C EPA 3660B EPA 3620C EPA 3665A EPA 3640A
Kentucky UST Program				
Metals				
Arsenic	--------------	------------------	EPA 6010B	EPA 6010B
Barium	--------------	------------------	EPA 6010B	EPA 6010B

Parameter/Analyte	**Tissue**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Cadmium	--------------	------------------	EPA 6010B	EPA 6010B
Chromium	--------------	------------------	EPA 6010B	EPA 6010B
Lead	--------------	------------------	EPA 6010B	EPA 6010B
Mercury	--------------	------------------	EPA 7470A	EPA 7471A
Selenium	--------------	------------------	EPA 6010B	EPA 6010B
Silver	--------------	------------------	EPA 6010B	EPA 6010B

Parameter/Analyte	**Tissue**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Purgeable Organics (Volatiles)				
Diesel Range Organics (DRO)	--------------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Gasoline Range Organics (GRO)	--------------	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D	EPA 8015C EPA 8015D
Wyoming Storage Tank Program				
Metals				
Cadmium	--------------	------------------	EPA 6010C	EPA 6010C
Chromium	--------------	------------------	EPA 6010C	EPA 6010C
Chromium (Total, hexavalent)	--------------	------------------	EPA 7196A	EPA 7196A
Lead	--------------	------------------	EPA 6010C	EPA 6010C
Purgeable Organics (Volatiles)				
Volatile Preparation	--------------	------------------	EPA 5030C EPA 5030C	EPA 5035 EPA 5035A
Benzene	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
1,2-Dichloroethane	--------------	------------------	EPA 8260D	EPA 8260D
1,2-Dibromoethane	--------------	------------------	EPA 8011	EPA 8011
Diisopropyl Ether	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl Benzene	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Ethyl tert-butyl Ether	--------------	------------------	EPA 8260D	EPA 8260D
Methyl tert-butyl Ether	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Naphthalene	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Toluene	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Tert-amyl Methyl Ether	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D
Tert-butyl Alcohol	--------------	------------------	EPA 5030C EPA 8260D	EPA 8260D

Parameter/Analyte	**Tissue**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**
Xylenes, total	-------------	-----------------	EPA 5030C EPA 8260D	EPA 8260D
Gasoline Range Organics (GRO C6-C10)	-------------	-----------------	EPA 5030C EPA 8260D	EPA 8260D
Extractable Organics (Semivolatiles)				
Diesel Range Organics (DRO C10-C32)	-------------	-----------------	EPA 8015C w/ EPA 3630 cleanup	EPA 8015C w/ EPA 3630 cleanup

Food and Feed (WHO 29)	**Food/Feed**			
2,3,7,8-TCDD	EPA 1613B	-----------------	--------------	--------------
2,3,7,8-TCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,7,8-PeCDF	EPA 1613B	-----------------	--------------	--------------
2,3,4,7,8-PeCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,7,8-PeCDD	EPA 1613B	-----------------	--------------	--------------
1,2,3,4,7,8-HxCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,6,7,8-HxCDF	EPA 1613B	-----------------	--------------	--------------
2,3,4,6,7,8-HxCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,7,8,9-HxCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,4,7,8-HxCDD	EPA 1613B	-----------------	--------------	--------------
1,2,3,6,7,8-HxCDD	EPA 1613B	-----------------	--------------	--------------
1,2,3,7,8,9-HxCDD	EPA 1613B	-----------------	--------------	--------------
1,2,3,4,6,7,8-HpCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,4,7,8,9-HpCDF	EPA 1613B	-----------------	--------------	--------------
1,2,3,4,6,7,8-HpCDD	EPA 1613B	-----------------	--------------	--------------
OCDF	EPA 1613B	-----------------	--------------	--------------
OCDD	EPA 1613B	-----------------	--------------	--------------
Total HpCDD	EPA 1613B	-----------------	--------------	--------------
Total HpCDF	EPA 1613B	-----------------	--------------	--------------
Total HxCDD	EPA 1613B	-----------------	--------------	--------------
Total HxCDF	EPA 1613B	-----------------	--------------	--------------
Total PeCDD	EPA 1613B	-----------------	--------------	--------------
Total PeCDF	EPA 1613B	-----------------	--------------	--------------
Total TCDD	EPA 1613B	-----------------	--------------	--------------
Total TCDF	EPA 1613B	-----------------	--------------	--------------
6 marker PCBs (PCB28, PCB52, PCB101, PCB138, PCB153, and PCB180)	EPA 1668A EPA 1668C	-----------------	--------------	--------------

Parameter/Analyte	**Tissue**	**Nonpotable Water**	**Solid Hazardous Waste**	
			Aqueous	**Solid**

12 Dioxin-like PCBs (dl-PCBs)/coplanar PCBs (PCB77, PCB81, PCB105, PCB114, PCB118, PCB123, PCB126, PCB156, PCB157, PCB167, PCB169, and PCB189)	EPA 1668A EPA 1668C	------------------	---------------	---------------

Parameter/Analyte	**Drinking Water**	**Nonpotable Water**	**Solid Haz.Waste**
Per and Polyfluoroalkyl Substances (PFAS)			
N-ethyl perfluorooctane-sulfonamidoacetic acid (NetFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-methyl perfluorooctane-sulfonamidoacetic acid (NMeFOSAA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorobutanesulfonic acid (PFBS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorodecanoic acid (PFDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorododecanoic acid (PFDoDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroheptanoic acid (PFHpA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexanesulfonic acid (PFHxS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexanoic acid (PFHxA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorononanoic acid (PFNA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanesulfonic acid (PFOS)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanoic acid (PFOA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorotetradecanoic acid (PFTeDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorotridecanoic acid (PFTrDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroundecanoic acid (PFUnDA)	EPA 537 Ver. 1.1 EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid (HFPODA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	EPA 537.1	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoro-n-butanoic acid (PFBA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoro-n-pentanoic acid (PFPeA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
8:2 Fluorotelomersulfonic acid (8:2FTS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

Parameter/Analyte	**Drinking Water**	**Nonpotable Water**	**Solid Haz.Waste**
4:2 Fluorotelomersulfonic acid (4:2-FTS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoropentanesulfonic acid (PFPeS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
6:2 Fluorotelomersulfonic acid (6:2-FTS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluoroheptanesulfonic acid (PFHpS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorononanesulfonic acid (PFNS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorodecanesulfonic acid (PFDS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
10:2 Fluorotelomersulfonic acid (10:2-FTS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorododecanesulfonic acid (PFDoDS)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorohexadecanoic acid (PFHxDA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctadecanoic acid (PFODA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
Perfluorooctanesulfonamide (PFOSA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMePFOSAE)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-methylperfluoro-1-octanesulfonamide (NMePFOSA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtPFOSAE)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod
N-ethylperfluoro-1-octanesulfonamide (NEtPFOSA)	---------------------	EPA 537 Ver.1.1 Mod	EPA 537 Ver.1.1 Mod

End of KY, WY, and ISO 17025 section of scope

Accredited Laboratory

A2LA has accredited

EUROFINS LANCASTER LABORATORIES ENVIRONMENTAL, LLC

Lancaster, PA

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated *April 2017*).

Presented this 28th day of January 2021.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 1.01
Valid to November 30, 2022

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

GEOTESTING EXPRESS, INC.
125 Nagog Park
Acton, MA 01720
Joe Tomei   Phone: 978 635 0424

Valid To: March 31, 2022   Certificate Number: 2965.01

GEOTECHNICAL

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests under the ASTM recommended practice D3740:

Test Method:	**Test Description:**
Soils:	
ASTM D421	Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constant
ASTM D422 (Withdrawn 2016)[1]	Particle Size Analysis of Soils
ASTM D7928	Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis
ASTM D698	Moisture-Density Relations (Standard Proctor)
ASTM D854	Specific Gravity of Soils
ASTM D1140	Amount of Material in Soils Finer than No. 200 Sieve
ASTM D1557	Moisture-Density Relations (Modified Proctor)
ASTM D1883	CBR (California Bearing Ratio) of Laboratory-Compacted Soils
ASTM D2166/D2166M	Unconfined Compressive Strength of Cohesive Soil
ASTM D2216	Water Content of Soil, Rock & Soil-Aggregate Mixtures
ASTM D2434-68 (2006)[1]	Permeability of Granular Soils (Constant Head)
ASTM D2435/D2435M	One-Dimensional Consolidation Properties of Soils
ASTM D2487	Classification of Soils for Engineering Purposes
ASTM D2488	Description and Identification of Soils (Visual-Manual Procedure)
ASTM D2850	Undrained, Unconsolidated Strength in Triaxial Compression
ASTM D2974	Moisture, Ash, and Organic Matter of Peat and Other Organic Soils
ASTM D3080/D3080M	Direct Shear Test of Soils Under Consolidated Drained Conditions
ASTM D4015	Modulus and Damping of Soils by Resonant-Column Method
ASTM D3999	Determination of the Modulus and Damping Properties of Soils Using the Cyclic Triaxial Apparatus
ASTM D4186	One-Dimensional Consolidation Properties of Saturated Cohesive Soils Using Controlled-Strain Loading
ASTM D4253	Maximum Index Density and Unit Weight of Soils Using a Vibratory Table
ASTM D4254	Minimum Index Density and Unit Weight of Soils and Calculation of Relative Density
ASTM D4318	Liquid Limit, Plastic Limits & Plasticity Index of Soils

Test Method:	**Test Description:**
ASTM D4373	Rapid Determination of Carbonate Content of Soils
ASTM D4546	One-Dimensional-Swell or Settlement Properties of Cohesive Soils
ASTM D4718	Correction of Unit Weight and Water Content for Soils Containing Oversize Particles
ASTM D4767	Consolidated Undrained Triaxial Compression Test for Cohesive Soils
ASTM D4829	Expansion Index of Soils
ASTM D4972	pH of Soils
ASTM D5334	Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure
ASTM G51	Standard Test Method for Measuring pH of Soil for Use in Corrosion Testing
ASTM D5084	Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter
ASTM D5311	Load Controlled Cyclic Triaxial Strength of Soil
ASTM D6467	Torsional Ring Shear Test to Determine Drained Residual Shear Strength of Cohesive Soils
ASTM D6528	Consolidated Undrained Direct Simple Shear Testing of Cohesive Soils
ASTM D6913	Particle-Size Distribution (Gradation) of Soil using Sieve Analysis
ASTM D6938[2]	In-Place Density and Water Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth)
ASTM D7181	Consolidated Drained Triaxial Compression Test for Soils
ASTM D7263	Laboratory Determination of Density (Unit Weight) of Soil Specimens
ASTM D7608	Standard Test Method for Torsional Ring Shear test to determine Drained Fully Softened Shear Strength and Nonlinear Strength Envelope of Cohesive Soils for Slope with No Preexisting Shear Surfaces
ASTM D8296	Consolidated Undrained Cyclic Direct Simple Shear Test under Constant Volume with Load Control or Displacement Control
ASTM G57	Soil Resistivity Using the Wenner Four-Electrode Method
AASHTO T307	Determining the Resilient Modulus of Soils and Aggregate Materials
ISO/TS 17892-1	Determination of Water Content
ISO/TS 17892-3	Determination of Particle Density - Pycnometer Method
ISO/TS 17892-5	Incremental Loading Oedometer Test
ISO/TS 17892-7	Unconfined Compression Test on Fine-Grained Soil
ISO/TS 17892-9	Consolidated Triaxial Compression Tests on Water-Saturated Soil
ISO/TS 17892-11	Determination of Permeability by Constant and Falling Head
ISO/TS 17892-12	Determination of Atterberg Limits
Rock:	
ASTM D2845	Laboratory Determination of Pulse Velocities and Ultrasonic Elastic Constants of Rock
ASTM D2936	Direct Tensile Strength of Intact Rock Core Specimens
ASTM D3967	Splitting Tensile Strength of Intact Rock Core Specimens
ASTM D4543	Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances
ASTM D4644	Slake Durability of Shales and Similar Weak Rocks
ASTM D5607	Performing Laboratory Direct Shear Strength Tests of Rock Specimens Under Constant Normal Force

Test Method:	**Test Description:**
ASTM D5731	Determination of the Point Load Strength Index of Rock and Application to Rock Strength Classifications
ASTM D5873	Determination of Rock Hardness by Rebound Hammer Method
ASTM D6032	Determining Rock Quality Designation (RQD) of Rock Core
ASTM D7012	Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperature
ASTM D7625	Laboratory Determination of Abrasiveness of Rock Using the CERCHAR Method
Handewith (2000)	Punch Penetration
ISRM Part 1	Water Content of Rock
ISRM Part 2	Porosity/Density
ISRM Part 3	Saturation/Buoyancy

[1] This laboratory's scope contains withdrawn or superseded methods. As a clarifier, this indicates that the applicable method itself has been withdrawn or is now considered "historical" and not that the laboratory's accreditation for the method has been withdrawn.

[2] This laboratory meets A2LA *R104 – General Requirements: Accreditation of Field Testing and Field Calibration Laboratories* for these tests.

Accredited Laboratory

A2LA has accredited

GEOTESTING EXPRESS, INC.

Acton, MA

for technical competence in the field of

Geotechnical Testing

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

SEAL 1978

Presented this 2nd day of June 2020.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2965.01
Valid to March 31, 2022

For the tests to which this accreditation applies, please refer to the laboratory's Geotechnical Scope of Accreditation.

AASHTO
ACCREDITED

CERTIFICATE OF ACCREDITATION

AMERICAN ASSOCIATION
OF STATE HIGHWAY AND
TRANSPORTATION OFFICIALS
AASHTO

Atlantic Testing Laboratories, Limited

in

Canton, New York, USA

has demonstrated proficiency for the testing of construction materials and has conformed to the requirements established in AASHTO R 18 and the AASHTO Accreditation policies established by the AASHTO Committee on Materials and Pavements.

The scope of accreditation can be viewed on the Directory of AASHTO Accredited Laboratories (aashtoresource.org).

Jim Tymon,
AASHTO Executive Director

Moe Jamshidi.

Moe Jamshidi,
AASHTO COMP Chair

This certificate was generated on 05/04/2021 at 10:19 AM Eastern Time. Please confirm the current accreditation status of this laboratory at aashtoresource.org/aap/accreditation-directory

SCOPE OF AASHTO ACCREDITATION FOR:

Atlantic Testing Laboratories, Limited
in Canton, New York, USA

Quality Management System

Standard:		Accredited Since:
R18	Establishing and Implementing a Quality System for Construction Materials Testing Laboratories	10/26/2011
C1077 (Aggregate)	Laboratories Testing Concrete and Concrete Aggregates	03/12/2014
C1077 (Concrete)	Laboratories Testing Concrete and Concrete Aggregates	03/01/2012
D3740 (Soil)	Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction	03/01/2012
E329 (Aggregate)	Standard Specification for Agencies Engaged in the Testing and/or Inspection of Materials Used in Construction	03/12/2014
E329 (Concrete)	Standard Specification for Agencies Engaged in the Testing and/or Inspection of Materials Used in Construction	03/01/2012
E329 (Soil)	Standard Specification for Agencies Engaged in the Testing and/or Inspection of Materials Used in Construction	03/01/2012
E329 (Sprayed Fire-Resistive Material)	Standard Specification for Agencies Engaged in the Testing and/or Inspection of Materials Used in Construction	05/06/2015

Scope of AASHTO Accreditation for:

Atlantic Testing Laboratories, Limited

in Canton, New York, USA

Soil

Standard:		Accredited Since:
R58	Dry Preparation of Disturbed Soil and Soil Aggregate Samples for Test	10/10/2008
T88	Particle Size Analysis of Soils by Hydrometer	10/10/2008
T89	Determining the Liquid Limit of Soils (Atterberg Limits)	10/10/2008
T90	Plastic Limit of Soils (Atterberg Limits)	10/10/2008
T99	The Moisture-Density Relations of Soils Using a 5.5 lb [2.5 kg] Rammer and a 12 in. [305 mm] Drop	10/10/2008
T100	Specific Gravity of Soils	06/23/2011
T180	Moisture-Density Relations of Soils Using a 10 lb [4.54 kg] Rammer and an 18 in. [457 mm] Drop	10/10/2008
T216	One-Dimensional Consolidation Properties of Soils Using Incremental Loading	10/26/2011
T265	Laboratory Determination of Moisture Content of Soils	10/10/2008
D421	Dry Preparation of Disturbed Soil and Soil Aggregate Samples for Test	10/10/2008
D422	Particle Size Analysis of Soils by Hydrometer	10/10/2008
D698	The Moisture-Density Relations of Soils Using a 5.5 lb [2.5 kg] Rammer and a 12 in. [305 mm] Drop	10/10/2008
D854	Specific Gravity of Soils	06/23/2011
D1140	Amount of Material in Soils Finer than the No. 200 (75-µm) Sieve	10/10/2008
D1557	Moisture-Density Relations of Soils Using a 10 lb [4.54 kg] Rammer and an 18 in. [457 mm] Drop	10/10/2008
D1883	The California Bearing Ratio	06/03/2015
D2166	Unconfined Compressive Strength of Cohesive Soil	06/03/2015
D2216	Laboratory Determination of Moisture Content of Soils	10/10/2008
D2435	One-Dimensional Consolidation Properties of Soils Using Incremental Loading	10/26/2011
D2487	Classification of Soils for Engineering Purposes (Unified Soil Classification System)	10/10/2008
D2488	Description and Identification of Soils (Visual-Manual Procedure)	10/10/2008
D2850	Unconsolidated, Undrained Compressive Strength of Cohesive Soils in Triaxial Compression	06/03/2015
D4318	Determining the Liquid Limit of Soils (Atterberg Limits)	10/10/2008

SCOPE OF AASHTO ACCREDITATION FOR:

Atlantic Testing Laboratories, Limited
in Canton, New York, USA

Soil (Continued)

Standard:	Accredited Since:
D4318 Plastic Limit of Soils (Atterberg Limits)	10/10/2008
D4767 Consolidated-Undrained Triaxial Compression Test on Cohesive Soils	06/03/2015
D5084 Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter	01/30/2018
D6938 In-Place Density and Moisture Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth)	10/10/2008

SCOPE OF AASHTO ACCREDITATION FOR:

Atlantic Testing Laboratories, Limited
in Canton, New York, USA

Aggregate

Standard:		Accredited Since:
R90	Sampling Aggregate	05/07/2013
T19	Bulk Density (“Unit Weight”) and Voids in Aggregate	01/15/2002
C29	Bulk Density (“Unit Weight”) and Voids in Aggregate	01/15/2002
C40	Organic Impurities in Fine Aggregates for Concrete	01/15/2002
C117	Materials Finer Than 75-µm (No. 200) Sieve in Mineral Aggregates by Washing	01/15/2002
C127	Specific Gravity and Absorption of Coarse Aggregate	01/15/2002
C128	Specific Gravity (Relative Density) and Absorption of Fine Aggregate	01/15/2002
C136	Sieve Analysis of Fine and Coarse Aggregates	01/15/2002
C566	Total Moisture Content of Aggregate by Drying	01/15/2002
C702	Reducing Samples of Aggregate to Testing Size	01/15/2002
D75	Sampling Aggregate	05/07/2013
D4791	Flat Particles, Elongated Particles, or Flat and Elongated Particles in Coarse Aggregate	01/15/2002

SCOPE OF AASHTO ACCREDITATION FOR:

Atlantic Testing Laboratories, Limited
in Canton, New York, USA

Sprayed Fire-Resistive Material

Standard:	Accredited Since:
E605 Thickness and Density of Sprayed Fire-Resistive Material(SFRM) Applied to Structural Members	06/23/2011
E736 Cohesion/Adhesion of Sprayed Fire-Resistive MaterialsApplied to Structural Members	06/23/2011

SCOPE OF AASHTO ACCREDITATION FOR:

Atlantic Testing Laboratories, Limited
in Canton, New York, USA

Concrete

Standard:		Accredited Since:
C31 (Cylinders)	Making and Curing Concrete Test Specimens in the Field	01/15/2002
C39	Compressive Strength of Cylindrical Concrete Specimens	01/15/2002
C138	Density (Unit Weight), Yield, and Air Content of Concrete	01/15/2002
C143	Slump of Hydraulic Cement Concrete	01/15/2002
C172	Sampling Freshly Mixed Concrete	01/15/2002
C173	Air Content of Freshly Mixed Concrete by the Volumetric Method	01/15/2002
C231	Air Content of Freshly Mixed Concrete by the Pressure Method	01/15/2002
C511	Moist Cabinets, Moist Rooms, and Water Storage Tanks Used in the testing of Hydraulic Cements and Concretes	08/26/2011
C617 (8000 psi and below)	Capping Cylindrical Concrete Specimens	02/12/2019
C1064	Temperature of Freshly Mixed Portland Cement Concrete	01/15/2002
C1231 (7000 psi and below)	Use of Unbonded Caps in Determination of Compressive Strength of Hardened Concrete Cylinders	08/26/2011

Appendix B

Comments and Responses

US Army Corps
of Engineers ®

New England District
696 Virginia Road
Concord, Massachusetts
01742-2751

Project Name:	Former Fort Devens Army Installation	Date:	15 February 2022
Location:	Devens, Massachusetts	Reviewer:	Multiple
Document Name: Internal Draft Barrier Wall Performance Monitoring Work Plan, Shepley's Hill Landfill (SHL)			
Prepared By: Seres Arcadis 8(a) JV			

No.	**Ref. Page / Para.**	**COMMENT**	**RESPONSE**
		EPA Comments	
G1		Please identify groundwater/aquatic sample locations where/when mentioned throughout the document.	Table 3 identifies which samples are groundwater, surface water, porewater, and sediment. References to this table will be added as appropriate to the text.
G2		To avoid confusion with the other 28 proposed sample locations near the barrier wall / in Red Cove, please identify the eight sample locations in the PSP area with the words "PSP" and/or as the "reference locations" and use consistently throughout the document.	The document was revised so the Red Cove samples are prefixed "RC" and the reference locations have "PS" as a prefix. A note was added to the text/tables/figures to clarify the nomenclature.
G3		EPA recommends that a full synoptic groundwater level measurement round be collected to help to provide context to the analytical porewater and surface water results. This should include all available monitoring wells located within the upgradient, downgradient, and aquatic environment areas shown in Figure 1, as well as any surface water staff gauges.	Synoptic groundwater level measurements coinciding with the two sampling events will be collected from existing LTM wells upgradient of Red Cove, in addition to piezometers and surface water gauges established as part of this study. Wells that will be used for water level gauging are shown on Figure 3 and Table 3.
G4		With the installation of the barrier wall, is groundwater from SHL forced into the underlying fractured bedrock, which then migrates into the surficial sand aquifer and discharges into PSP? If so, could this increase the flux of dissolved arsenic from bedrock fractures into the sand aquifer and finally into PSP?	While assessing change in flux from the surficial sand aquifer to bedrock and back to the surficial aquifer after construction of the barrier wall is outside the scope of work for this study, information collected from the clustered wells as part of this work plan will provide information that can be used for this assessment if needed.
1	Page II, Table of Contents, Tables	Please indicate that Table 10 is located within the text.	A note was added to the table of contents to clarify that Table 10 is embedded in the text.

No.	Ref. Page / Para.	COMMENT	RESPONSE
2	Page 1, § 1.0, ¶ 3	Indicate that sediment toxicity testing will not be conducted (as recommended by the EPA) and the reasoning for this adjustment up front rather than learning about this in Section 2.2 (Project Goals). Provide a date and reference for the Scope of Work issued by the U. S. Army to SERES-Arcadis JV.	The following text was added to Section 1.0 to clarify: "Although sediment toxicity testing was recommended, collection of these data is not needed to assess the effectiveness of the barrier wall installation for the reduction of arsenic flux. Therefore, collection and testing of sediment for benthic toxicity are not included in this Work Plan."
3	Page 1, § 1.1, ¶ 1, bullet list	Please include the excavation of contaminated Red Cove sediments as described in Section 1.0 to have occurred after the barrier wall installation.	Text was added as requested.
4	Page 2, § 1.1, first full ¶	The second sentence states that the soil-bentonite barrier wall was constructed "through the landfill cap and a thin mantling of waste". Based on historical documents, the soil-bentonite barrier wall was not installed in an area underlying the landfill cap. Please amend.	Text was revised as requested.
5	Page 3, § 1.2, ¶ 2	Please update the first sentence to "Mass flux of arsenic across the barrier wall and to Red Cove has, pre- and post-barrier installation, been estimated over segments of the surficial aquifer ….".	Text was revised as requested.
6	Page 4, § 1.3, ¶ 4 and Page 7, § 2.1, 2nd bullet	A statement is made that a portion of dissolved iron and arsenic precipitated within the aquifer east of the barrier wall and west of Red Cove. However, to date there has been no sampling and characterization of aquifer solids to demonstrate the accumulation of these precipitates in the aquifer prior to groundwater discharge into Red Cove. Since the potential accumulation of these precipitates has direct influence on the rate of decline in arsenic flux to Red Cove, it is highly recommended that one or more core samples of aquifer solids be collected to support determination of potential accumulated amorphous ferric oxyhydroxide mass (along with co-precipitated arsenic). Two possible mechanisms that may control declining groundwater iron and arsenic concentrations	The Work Plan has been revised to include a soil sampling section under Section 3.0 that includes additional soil cores as outlined below. These changes are also reflected in revised Figure 3 and Table 3. Soil cores from the surficial sand aquifer will be collected adjacent to three existing well locations between the barrier wall and Red Cove: 1. Area with elevated (greater than 2x groundwater standard of 10 µg/L) dissolved As in groundwater: • SHL-4 (3-13 ft bgs) • SHP-01-38A (1.5-6.5 ft bgs)

No.	Ref. Page / Para.	COMMENT	RESPONSE
		include flushing with less-contaminated groundwater and/or precipitation of iron and arsenic onto aquifer solids. Assessment of the relative control(s) on groundwater concentrations in this portion of the aquifer is important to more reliably assess cleanup timeframe within the aquifer.	2. Area with lower dissolved As in groundwater: • SHL-19 (20-30 ft bgs) Samples will be collected as 5-foot composites from the ground surface to top of bedrock using a Direct Push Rig (DPT refusal will be used as the indication that the depth of the top of bedrock has been reached.) Soil core samples will be analyzed for metals (As, Fe, Mg, Mn, Al, Ca), TOC, pH, total sulfur, and grain size. Two samples from each location will be collected for sequential extraction procedure (SEP): one within the well screen interval and the other from the core above the contact with bedrock. Groundwater samples will be collected from the three wells prior to soil core sampling.
7	Page 5, § 1.3, last ¶, 4th sentence	Please revise the sentence to "The methods used and results from these investigations have served as a guide in development of this Work Plan to allow for direct comparison of <u>performance data to be collected under this work plan with historical data</u>".	Text was revised as suggested.
8	Page 7, § 2.1, ¶ 1, last sentence before bullets	Please put "historical" or similar description prior to "geochemical characterization".	Text was revised as recommended.
9	Page 7, § 2.1, ¶ 1, bullets	EPA DQO Process guidance specifies that Step 1 should *summarize the contamination problem that will require new environmental data and identify the resources available to resolve the problem/develop conceptual site model*. The bullets identify the problem, but do not clearly identify the resources available (i.e., evaluation to be conducted) to resolve the problem. For example, for the 2nd bullet, does the 2nd	The DQO process is being used to, "conserve resources by determining which data collection and analysis methods are most appropriate for the data quality needs of the study" (EPA QA/G-4HW Section 0.4.3). The resources to develop the approach, collect the data, and evaluate the data will be provided by the Army.

No.	Ref. Page / Para.	COMMENT	RESPONSE
		sentence describe the additional data to be collected to resolve the problem? Also, the first bullet does not clearly state the resources available to resolve the problem. Please update the bullets to clearly identify the resources to resolve the problem.	The “evaluation to be conducted” is described in Steps 2 through 7 of DQO process.
10	Page 8, Table 1, Goal / DUO #1	The work plan does not propose conducting evaluation of benthic toxicity of sediment samples collected from Red Cove. However, EPA recommends that this data collection approach be considered if sediment arsenic concentrations are observed that are comparable with pre-remedy concentrations that were linked to benthic toxicity.	Please see response to Comment #2.
11	Page 9, Table 2	In the Laboratory Analysis/Field parameter column for Surface water samples and Groundwater samples, after major cation please include “(includes arsenic/iron)”.	Table 2 was revised as requested.
12	Page 10, § 2.4, ¶ 1	Please change “study objective” to “study goals” in the first sentence since there are 4 project goals identified in Section 2.2.	Text was revised as requested.
13	Page 10, § 2.4, ¶ 1	Provide a brief summary of the locations to be included in the monitoring program. For example, “The 28 proposed locations near the barrier wall/Red Cove include groundwater sampling locations (4 existing wells upgradient of the barrier wall and 4 existing downgradient wells from the barrier wall) and aquatic environment sampling locations (20 in red cove). Also, eight reference locations are proposed in the PSP area for aquatic environment monitoring. These are shown on Figure 3 and presented in Table 3”.	Text was revised as suggested.
14	Page 10, § 2.4.1, ¶ 1	Please include additional reasons why the selected existing wells were chosen (e.g., elevated arsenic elevations, full range of groundwater characteristics), if applicable, and provide their screen depth intervals in the text and/or Table 3.	Table 3 was adjusted to add the screen-depth intervals for the selected wells and additional reasons why they were chosen.
15	Page 10, § 2.4.3, ¶ 1,	Typo. Change “statical” to “statistical” and add “.” at the end of the sentence.	Text was revised as requested.

No.	Ref. Page / Para.	COMMENT	RESPONSE
	2nd sentence		
16	Page 10, § 2.4.3, ¶ 1	Please describe the number of piezometers in each cluster.	Text was revised as requested to indicate it will be clusters of two piezometers.
17	Page 11, § 2.5, Table 4, row 1	For the Decision Rule and Metric for Goal/DUO #1, if the answer to the Decision Question is yes, the Decision Rule and Metric states that additional study/monitoring may be required and that 95% UCLs will be used to describe current sediment conditions. What action will be taken if the 95% UCLs are exceeded? Another removal?	The text in Table 4 was revised as follows: "If no, no further action is required for arsenic. If yes, additional study/monitoring may be required and would be conducted under the revised LTMMP. Remedial recommendations/actions are outside the scope of this study/evaluation.
18	Page 11, § 2.5, Table 4, row 2	For the Decision Rule and Metric for Goal/DUO #2, if the answer to the Decision Question is yes, the Decision Rule and Metric states that additional study/monitoring may be required. If a flux is determined to exist, why would additional monitoring/study be required? The question to be answered is more likely, if a larger than expected flux was encountered or if results were inconclusive, additional study/monitoring may be required. Please consider and adjust the appropriate text accordingly.	Text in Table 4 was revised for DUO #2 as suggested.
19	Page 11, § 2.5, Table 4, row 3	For the Decision Rule and Metric for Goal/DUO #3, if the answer to the Decision Question is yes, in addition to what is stated, it is recommend adding that if the sufficient evidence for stable or a decreasing trends are not present, then additional study/monitoring may be required and would be conducted under the revised LTMMP.	Text was added as suggested.
20	Page 12, § 2.6, last ¶, 1st sentence	Please identify/elaborate on the "*process* [to ensure the program is designed to accomplish the data goals and objectives]."	Text was added after "process" to include "(i.e., design of the sampling approach, field collection practices, laboratory analysis, data validation, and data evaluation)".
21	Page 13, § 2.7, ¶ 1, 6th sentence and § 3.1.4	Are there provisions for installing a shallower "deep" piezometer if refusal is encountered?	Text was revised as follows: "Alternate locations will be attempted if refusal is encountered. Based on current well logs, there is no reason to assume refusal will happen. Up

No.	Ref. Page / Para.	COMMENT	RESPONSE
			to two additional attempts within a 10-feet radius will take place if refusal is encountered."
22	Page 15, § 3.1.1, Header	Typo. Spelling of "Measurements" should be corrected.	The misspelling was corrected.
23	Page 15, § 3.1.1, last ¶	Please specify that the staff gauge will be tied into a benchmark elevation on land to determine water surface elevation.	Text was added as suggested.
24	Page 15, § 3.1.2, ¶ 1, 2nd sentence	"DUO2" should be removed from the list of measurements.	Text was deleted as recommended.
25	Page 16, § 3.1.3, 1st sentence	Typo. Please include closing parentheses.	The text was revised as suggested.
26	Page 16, § 3.1.3, 2nd sentence	The text states that instantaneous flow into and out of the Plow Shop Pond will not be measured within 48 hours of a preceding precipitation event. It is recommended that the same cutoff be used for all surface water and porewater measurements and samples and consider using a similar cutoff for all groundwater measurements and samples. The cutoff for groundwater may be tied to a "significant" precipitation event, such as greater than 1 inch in 24 hours, as groundwater is not expected to be as sensitive to precipitation.	Text was revised to add a similar cutoff timeframe for all surface water, porewater, and groundwater samples.
27	Page 16, § 3.1.4	The use of clustered piezometers to assess vertical gradients and estimate the direction and magnitude of vertical groundwater flux will provide important information to aid in determining the magnitude and differentiating source(s) of arsenic flux. It is recommended that assessment of horizontal gradients also be calculated using three-point estimates based on groundwater levels in the deepest of the clustered piezometers. This presents an opportunity to more fully map out the groundwater flow field under Red Cove with respect to flow gradients in the aquifer between the barrier wall and the	The Work Plan has been revised to include the addition of the assessment of horizontal gradients as outlined below. These changes are also reflected in revised Figure 3 and Table 3. Assessment of horizontal gradients will be included in the study. The methodology that uses three-point estimates as used in the LTM reporting will be followed. Pressure transducers will be installed in:

No.	Ref. Page / Para.	COMMENT	RESPONSE
		cove. Since vertical and horizontal gradients are highly dependent on surface water level in the cove (and PSP), it is recommended that at least a portion of these piezometers be instrumented with logging pressure transducers to assess variations over the period of data collection (12 weeks). Continuous measurements of this nature will help facilitate clearly defining periods when groundwater is likely discharging into the cove. Likewise, it is recommended some degree of continuous water level monitoring in Red Cove be carried forward into the updated LTMMP. It is also recommended that piezometer locations RSK37, RSK15, RSK12, RSK19 and RSK7 be included as part of the network of locations for water level measurements to be conducted under this work plan.	1. Four of the well clusters (eight transducers) in Red Cove, 2. One well cluster in the reference area in PSP, 3. PSP stilling well 4. Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (N2-P1/P2 and N3-P1/P2 well clusters) adjacent to Red Cove 5. Two well clusters (4 transducers) in the surficial sand aquifer and bedrock (PZ-12-06 and SHM-11-02 cluster; RSK-28 and SMH-11-07 cluster) upgradient of the barrier wall. Water levels in all wells monitored as part of the LTM in the vicinity of Red Cove and the Barrier Wall will be included in the synoptic water level measurements (see response to Comment #G3).
28	Page 17, § 3.2.1, ¶ 1	Please identify which “collected data” in the second sentence.	The text was revised to clarify it is porewater and groundwater collected data.
29	Page 17, § 3.2.2, ¶ 2, last sentence	Please indicate that the wells observed with iron floc were from historical well development events or clarify that the proposed well locations will be adjusted based on floc observed during well development to be conducted.	Text was revised to indicate that the proposed well locations will be adjusted based on floc observations made during well development.
30	Page 18, § 3.3.1, ¶ 1, 3rd sentence	Please note that sample intervals may also be adjusted based on sample recovery.	Text was revised as suggested.
31	Page 18, § 3.3.2, ¶ 1, 1st sentence	Please change Figure 2 to Figure 3.	Text was revised as requested.
32	Page 18, § 3.3.2	Use of a core collection method will facilitate the process of preserving the redox status of iron-bearing minerals and associated arsenic in sediment. EPA recommends that care be taken to minimize oxygen exposure during core sectioning, especially for core sub-sections to be submitted for sequential	The Army is willing to discuss whether/how this research-effort can be incorporated into the field program.

No.	Ref. Page / Para.	COMMENT	RESPONSE
		extraction procedure (SEP) analyses. If feasible, EPA/ORD is interested in acquiring sub-samples of cores collected from proposed locations RC-01, RC-02, RC-03 and RC-05 for analysis using x-ray absorption spectroscopy. This information could supplement results from SEP analyses and allow comparison to sediment characterization results conducted prior to barrier wall installation. EPA would appreciate the opportunity to discuss whether coordination for acquiring sub-samples is feasible prior to commencement of field activities.	
33	Page 19, § 3.3.2, 1st bullet	Please clarify which specific metals/analytes will be determined by SEP.	Text has been revised to indicate the following will be analyzed by SEP: Cations: As, Fe, Mn, Al, Ca, Mg Anions: Sulfur Organic matter: DOC
34	Page 21, § 3.3.2, Table 8	Please amend the table to include ammonia as anion.	Ammonia was added to the porewater analysis and table as requested.
35	Page 22, § 3.5.1, ¶ 1	EPA agrees that existing monitoring wells should be redeveloped if needed; however, the text does not specify the timeframe between redevelopment and sampling. Please provide a minimum time between redevelopment and sampling.	Text specifying a timeframe of 24 hours between redevelopment and sampling was added to Section 3.5.1.
36	Page 28, Table 10	Please update the anticipated project schedule to reflect the December 31, 2021 federal holiday and EPA's submission of comments on January 3, 2022.	Schedule was revised as requested.
37	Page 25, Figure 2: Generalized Conceptual Site Model Cross Section AA'	The figure depicts possible arsenic contributions to Plow Shop Pond (PSP) including the attenuated flux from the Shepley's Hill Landfill (SHL) through the barrier wall and contributions of dissolved arsenic from the fractured bedrock into the surficial sand aquifer. Is the bedrock currently a significant dissolved arsenic source now that the contribution from SHL has been diminished? Can the bedrock contribution be estimated with available information, or can the current investigation evaluate the contribution? This question could be part of Goal/DUO #3.	The CSM figure will be revised to reflect the following based on groundwater elevation measurements from well clusters post-barrier wall construction: 1. Upgradient of the Barrier Wall: a net downward potential gradient from the surficial sand aquifer to bedrock 2. Downgradient of the Barrier Wall: variable direction with an overall net downward potential gradient from the surficial sand aquifer to bedrock.

No.	Ref. Page / Para.	COMMENT	RESPONSE
			The current CSM for flux between the surficial sand aquifer and bedrock is based on limited measurements, and additional information will be collected as part of this study. While assessing change in flux from the surficial sand aquifer to bedrock and back to the surficial aquifer after construction of the barrier wall is outside the scope of work for this study, information collected from the clustered wells as part of this work plan will provide information that can be used for this assessment if needed.
38	Page 26, § 4.4	Evaluation of geochemical tracers provides an important line of evidence for distinguishing between the sources of direct groundwater discharge or sediment dissolution. The relative concentrations of the proposed constituents in pore water, deep surface water and shallow surface water can be used to conduct end-member mixing calculations. Estimates of the fraction of pore water contribution to deep surface using relatively conservative constituents (e.g., potassium, ammonium) compared to a non-conservative constituent such as iron provide a line of evidence to assess the fractional contribution of sediment dissolution for observed arsenic concentrations in deep surface water. Likewise, estimates of pore water contribution to deep surface water helps constrain estimates of groundwater discharge based on observations of vertical gradient.	Agree, a discussion on geochemical tracers will be evaluated as discussed in Section 4.4.
		MassDEP Comments	
1	Section 1.3, Fourth Paragraph; and Figure 2	The conceptual site model should be expanded to note that groundwater from Shepley's Hill Landfill may migrate beneath the barrier wall through fractured bedrock to the east side of the wall and to Red Cove due to a strong downward hydraulic gradient induced along the west side the barrier wall. If arsenic concentrations in Red Cove sediment have rebounded significantly since the 2013 sediment removal action, then	See response to EPA Comment #37. As noted in the DQO section of the Work Plan, depending on the analysis of the data and the results gathered from this study, additional study/monitoring may be required and would be conducted under the revised LTMMP.

No.	Ref. Page / Para.	COMMENT	RESPONSE
		additional work may be necessary to determine if the potential contribution of arsenic via this pathway is significant.	
2	Section 2.2, First Paragraph	If the results from the planned work indicate that arsenic concentrations in Red Cove sediment have rebounded significantly since the 2013 sediment removal action, then sediment toxicity testing may be required to reassess ecological risks.	The following text was added to Section 1.0 to clarify: “Although sediment toxicity testing was recommended, collection of these data is not needed to assess the effectiveness of the barrier wall installation for the reduction of arsenic flux. Therefore, collection and testing of sediment for benthic toxicity are not included in this Work Plan.”
3	Table 2	Please confirm/correct the listing of iron-oxidizing bacteria analysis for sediment samples	Text was revised to clarify no iron-oxidizing bacteria analysis in sediment samples.
4	Section 3.2.2	The plan should note the rationale for selecting the top-of-water-column surface water samples for analysis of iron-oxidizing bacteria.	The text has been revised as suggested. Because iron oxidizing bacteria require a minimum level of oxygen to convert iron from ferrous to ferric and given the lack of water circulation (stagnant) in Red Cove, there is a reasonable likelihood that the dissolved oxygen levels at the GSI are too low to support a large population of iron oxidizing bacteria. It is more likely that a higher population count is present closer to the water surface. The plan currently calls for 14 samples collected for iron oxidizing bacteria. To establish if there is a difference within the water column, the text was revised to indicate that the 14 samples will be collected from 7 locations, with one top and one bottom sample at each location.
5	Section 3.5	To assess conditions between the barrier wall and Red Cove, groundwater samples should also be collected from monitoring well SHL-4. Available data indicate that shallow groundwater at this well location flows toward Red Cove (e.g., Figure E-1 of the 2020 Annual Report), and arsenic concentrations in samples collected from this well have increased since the barrier wall was installed (refer to chart in Appendix G of 2020 Annual Report), indicating a potential significant impact to groundwater attributable to the wall at this location.	Monitoring well SHL-4 has been added to the scope as a location of groundwater sampling. Soil core collection will also be completed within 10-feet of SHL-4 from two depths to provide information on the geochemical composition of the surficial sand aquifer at this location (see response to EPA Comment #6).

No.	Ref. Page / Para.	COMMENT	RESPONSE
6	Section 3.5	The relatively low potential contribution of arsenic from the vicinity of piezometers PZ-09 and PZ-10 – the saturated overburden is thin at these locations (e.g., Figure E-2 in the Annual Report) and low or non-detect concentrations were reported from these piezometers and nearby wells (2020 Annual Report) – suggests that PZ-06 be sampled instead of PZ-10.	PZ-06 has replaced PZ-10 as recommended.
7	Figure 3	Sediment samples should be redistributed to increase sample density in Red Cove, or additional samples should be collected from Red Cove to ensure that samples representative of conditions in Red Cove will be collected. Only three of the twenty proposed Red Cove sediment samples are located in Red Cove, where samples with the highest pre-removal arsenic concentrations were located and where sediment with the greatest potential for post-removal rebound is located, and seven of the proposed sample locations are outside of the removal area, where impacts were not deemed sufficient to warrant removal at the time of the removal action.	In recognition that the near shore area historically had higher arsenic concentrations, one of the bounding locations (the original RC-20) has been relocated to the near-shore Red Cove area at the midpoint between RC-01, RC-02, and RC-03 and the samples were renumbered. This change will provide 4 locations in the near shore area of Red Cove (RC-01 through RC-04) and a total of 13 locations within the former excavation area. The remaining (7) sample locations bound the excavation area. The bounding samples will verify if arsenic in the sediment has not increased in this area since excavation. Figure 3 and the text (Section 2.4.3) have been revised to reflect these changes.
8	Appendix A, Section 17.3	Please confirm/correct the statement indicating that the surface water samples will not be collected using a boat; sampling on foot could compromise sample representativeness by mobilizing sediment into the water column and disturbing flow within the water column.	All surface water samples will be collected using a boat. The text in Appendix A and in Section 3 was revised to clarify.
9		Please notify MassDEP in advance of sampling activities so that MassDEP can observe.	The Army will coordinate in advance of sampling activities with MassDEP.
		END OF COMMENTS	

ARMY'S 4/3/22 RESPONSES TO EPA FOLLOW-ON COMMENTS
ARMY'S 2/17/22 RESPONSES TO EPA'S 1/3/22 COMMENTS ON THE DRAFT BARRIER WALL PERFORMANCE MONITORING WORK PLAN

Unless otherwise noted below, Army's responses are acceptable.

PSC 2 – EPA appreciates (and accepts) the additional text added to Section 1. Although, EPA understands that sediment toxicity testing "is not needed to assess the effectiveness of the barrier wall installation for the reduction of arsenic flux", please indicate when/if sediment toxicity testing be conducted in the future to evaluate arsenic toxicity.

RESPONSE: The need for and timing of sediment toxicity testing will be discussed as part of the recommendations in this study.

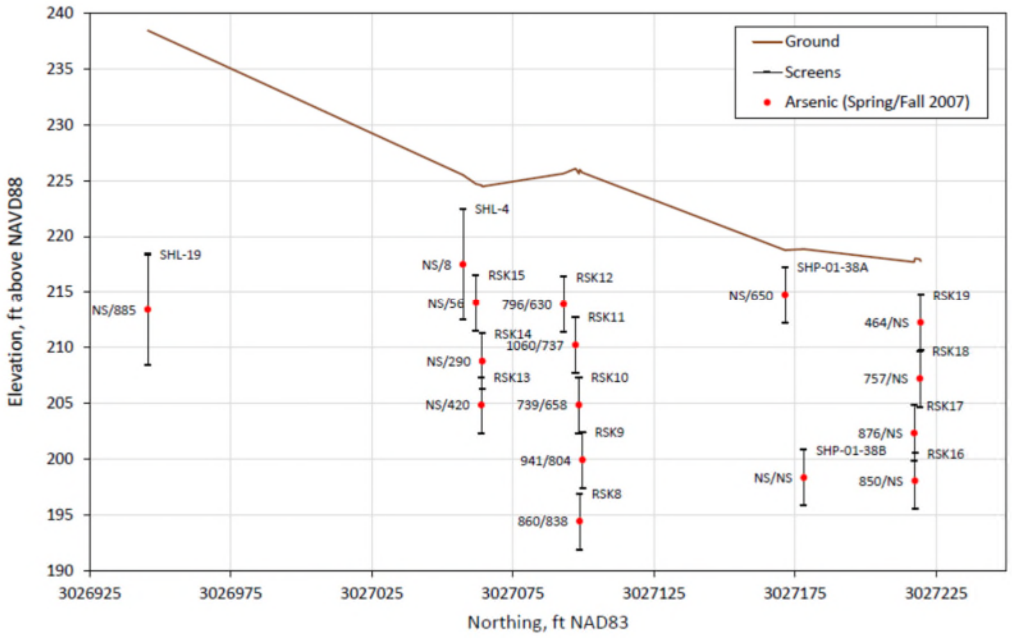
PSC 6 - The graphs below show measured arsenic concentrations before (2007) and after (2021) barrier wall installation for the three monitor well locations Army has targeted for the analysis of aquifer solids, along with concentrations for proximal RSK piezometers. For these graphs, monitoring well/piezometer location names are posted to the right of the top of screen, and arsenic concentrations are posted to the left of the middle of the screen (NS = not sampled). The Fall 2021 RSK piezometer data are from the sampling round we conducted during November 2021 with analyses conducted by the ORD lab in Ada, OK. The 2007 data was presented in the published EPA/ORD Final Report. The Fall 2021 data can be made available upon request.

RESPONSE: The Army requests that the EPA provide the data as soon as it is available.

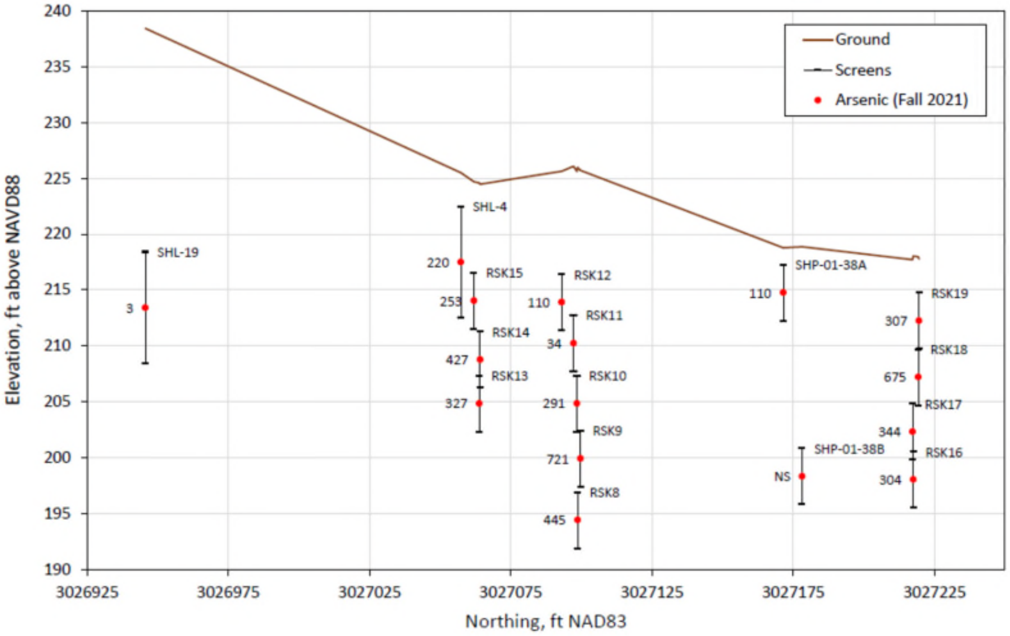
PSC 32 – As noted in the original comment, EPA requests that a conference call be convened, *prior to the commencement of field work*, to discuss ORD's intentions regarding the receipt and analysis of split sediment samples. A brief one-page description of ORD's intentions with regards to the sediment split samples prior to the call, if desired.

RESPONSE: Prior to field mobilization, a coordination call to discuss the split samples will be scheduled.

SHL BARRIER WALL AQUIFER SOLIDS SPRING/FALL 2007 GW



SHL BARRIER WALL AQUIFER SOLIDS FALL 2021 GW



Responses to the 3/2/22 Comments on the 2/17/22 Army's responses to DEP Comments (RTCs):

RTC 1: MassDEP accepts the response to this comment with the understanding that the Army recognizes its conceptual model may be incomplete because it does not account for potential migration of groundwater from the landfill to Red Cove through bedrock beneath the barrier wall.

Response to RTC 1: Understood, the conceptual model will be adjusted appropriately as new information is obtained, including information collected as part of this study.

RTC 7: Relocation of one sample from outside the removal area to Red Cove will provide more data representative of conditions in Red Cove; however, even with this adjustment, the proposed decision rules (Section 2.5) and proposed analysis of sample results (Section 4.2) will not provide meaningful results. In particular, the use of the 95% UCL is inappropriate because the proposed sample locations are not representative of a single environmental condition (i.e., a single population with an underlying statistical distribution). Instead, samples will be collected from at least two distinct settings: the removal area (where the 270 mg/kg RG was applied during the removal action) and the area outside the removal area, and reasonably three distinct settings: a hot spot in Red Cove, the remainder of the removal area, and outside the removal area. Consequently, MassDEP may not accept conclusions based on the proposed rules or proposed analysis of sample data.

Response to RTC 7: The work plan currently includes the collection of samples from 4 locations within the 0.6-acre area in question. This is a subset of the 14 samples proposed within the larger excavation area (2.8 acres) depicted in orange on Figure 3 of the work plan. References to the 95% UCL have been removed from the work plan.

As discussed as part of DQO 1, data do not exist to document the current conditions at the site. The presence or absence of multiple data populations cannot be established without current data, and the evaluation of the data will include using multiple lines of evidence to determine the presence or absence of multiple data populations. The text in Section 4.2 of the work plan was revised as follows: "Data evaluations will include the development of population statistics for comparison to applicable standards and project goals. Although there is no single statistical test to determine if a sampling of data is from a single statistical population, there are several lines of evidence that can be indicative of a single population. These lines of evidence include a single statistical distribution, a linear probability plot, a low coefficient of variation, and relatively few statistical outliers. Concentrations will be presented graphically to show the horizontal and vertical distribution in Red Cove and relational correlations will be developed between media (surface water, sediment, porewater, and groundwater)."