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May 6, 2021

Ms. Jennifer Knoepfle
U.S. Environmental Protection Agency
77 West Jackson Boulevard
Chicago, Illinois 60604

Subject: 2010300074 - Winnebago County
Quality Assurance Project Plan, Revision 4
Southeast Rockford Groundwater Contamination Superfund Site
Source Area 7 Groundwater Management Zone Monitoring Activities
Rockford, Winnebago County, Illinois
Superfund/Technical

Dear Ms. Knoepfle:

On behalf of the Illinois Environment Protection Agency (Illinois EPA), CDM Smith Inc. (CDM Smith), is pleased to submit to U.S. Environmental Protection Agency (U.S. EPA) the above-referenced Quality Assurance Project Plan.

The QAPP was revised to incorporate the attached U.S. EPA comments dated April 14, 2021. Following each comment is a brief explanation, as necessary, describing how the comment was addressed.

If you have any questions or comments, please contact me at (312) 780-7737.

Sincerely,

John C. Grabs, P.G., PMP
Senior Project Manager
CDM Smith Inc.

attachment

cc: Brian Conrath, Illinois EPA

Response to Comments on Quality Assurance Project Plan Revision 3
Southeast Rockford Groundwater Contamination Site
Source Area 7 Groundwater Management Zone Monitoring Activities

General Comments

1. If the highlighting will be included in the final draft, define its meaning. If not, remove.

   ***Response**: Highlighting will be removed in the final draft.*

2. The cover letter is dated 2019.

   ***Response**: This will be corrected to reflect the actual resubmittal date.*

3. Throughout the document cite the new 2020 Statements of Work (SOWs) for the Contract Lab Program (CLP) rather than the retired 2016 versions (https://www.epa.gov/clp/superfund-clp-analytical-statements-work-sows). EPA is now scheduling under the new CLP SOW SFA01.1 (which consolidated the separate inorganic and organic SOWs).

   ***Response**: Incorporated in both QAPP and SAP (Appendix A to QAPP).*

4. The new CLP sampler's guide is also now active and supersedes the 2014 version. Upcoming CLP cases must follow this guidance. It is similar to the previous guide, and the volume requirements for some parameters were reduced. https://www.epa.gov/sites/production/files/2021-03/documents/samplers_guide_clp_guidance_for_field_samplers_november_2020.pdf

   ***Response**: The new sample's guide, including new sample volume requirements, were incorporated into both the QAPP and SAP.*

Specific Comments

1. **QAPP Worksheet #3 & #5: Project Organization and QAPP Distribution.** Define who is responsible for maintenance and distribution of the QAPP.

   ***Response**: Incorporated that CDM Smith is the responsible entity. Also changed the U.S. EPA RPM to Jennifer Knoepfle in all applicable locations.*

2. **QAPP Worksheet #9: Project Planning Session Summary.**

   a. How does planning in 2019 relate to this addendum submitted in December 2020?

      ***Response**: Text was added to clarify*

   b. Were there QAPP updates/addenda submitted between the 2019 meeting and this addendum?

      ***Response**: No, this was clarified in text.*

c. Was this meeting summary included in previous addenda or is this the appropriate place to document this meeting?

***Response**: Initial meeting summary was provided as was the addenda meeting summary in Revision 3. There were no other addenda.*

3. QAPP Worksheet #11: Project/Data Quality Objectives.

a. "US EPA Region 5" rather than "Region V"

***Response**: Done.*

b. "The scope of these activities will include one influent and one effluent sample collected monthly until the LTS is declared operational or functional by Illinois EPA and quarterly thereafter during system operation." Update this language to define monitoring within the timeline governed by this QAPP.

***Response**: This language appears in multiple locations within QAPP and SAP. It was updated in all locations.*

c. "The water level data are considered screening level and will be used to evaluate changes to groundwater flow." What does it mean to say that "water level data are considered screening level"?

***Response**: Text was changed to: "The water level data are checked in the field for anomalies against historical information and for transcription errors and will be used to evaluate changes to groundwater flow."*

4. **QAPP Worksheet #14 & #16: Project Tasks and Schedule**.

a. Field change notification: when and how is EPA informed of QAPP deviation?

***Response**: Text added stating; "Changes due to field conditions that necessitate a change or addenda to the QAPP will be submitted to U.S EPA and subject to the formal review and approval process."*

b. Are there deviations that would require prior approval by EPA?

***Response**: No, Illinois EPA is the technical lead and has authority to approve field changes.*

5. **QAPP Worksheet #22 Field Equipment Calibration, Maintenance, Testing, and Inspection.** YSI-600XL: How often is temp check performed against NIST-traceable thermometer?

***Response**: As noted in the YSI user manual, no calibration or maintenance of the temperature sensor is required.*

6. **QAPP Worksheet #25 Analytical Instrument and Equipment Maintenance, Testing, and Inspection**. Remove empty table if all the necessary information is contained in the text.

***Response**: The QAPP format is a fillable form and as such the table should not be deleted as it impacts the overall form format. A note below the table has been inserted indicating that the table is intentionally blank.*

7. **QAPP Worksheet #28: Analytical Quality Control and Corrective Action.** What corrective actions would be initiated by the data validator?

   ***Response**: The worksheet has been updated to reflect that the data validator would qualify data if warranted by the validation process.*

8. **QAPP Worksheet #29: Project Documents and Records**.

   a. What documents, if any, are transmitted to EPA?

      ***Response**: The table footnote has been updated to reflect that the QAPP and annual GMZ reports are submitted to U.S. EPA.*

   b. Include link with the language that states the validated EDDs will be uploaded to the R5 electronic data submission site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission (p. 67).

      ***Response**: The link for uploading validated EDDs to the Region 5 electronic data submission webpage has been inserted into the text.*

9. **Appendix B**.

   a. Which SOP addresses influent/effluent sampling?

      ***Response**: QAPP WS# 21 was updated to reflect that the tap water sampling SOP applies to the extraction well and influent and effluent sampling. Sections 5.2.2 and 5.3 of the SAP state this as well.*

   b. Field Measurement of Total Organic Vapors SOP is included; is it a project objective to conduct Total Organic Vapors monitoring?

      ***Response**: Organic vapor monitoring is not a project objective but is performed for health and safety purposes. The SAP states this objective.*

10. **QAPP Worksheets (p. 273)**. Remove any worksheets included here that were previously included in the addendum.

    ***Response**: These worksheets, specifically WS 12a, b, and c and 28 a, b, and c, pertain to analyses performed by ASB and are therefore included with the ASB analytical methods in Appendix B. The ASB-specific worksheets and analytical methods were placed in Appendix B based on discussions with and direction from Region 5 following its implementation of the Field and Analytical Services Teaming and Advisory Committee (FASTAC) tiered analytical process.*

Quality Assurance Project Plan
Final Draft

Southeast Rockford Groundwater Contamination Superfund Site Source Area 7 Groundwater Management Zone Monitoring Activities

Contract HWA-16303, Work Order #06

Prepared for:

Illinois Environmental Protection Agency

May 4, 2021

(Revision 4)

Prepared by:

CDM Smith

Table of Contents

QAPP Elements

QAPP Worksheet #1 & 2: Title and Approval Page

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet QAPP Worksheet #6: Communication Pathways

QAPP Worksheet #9: Project Planning Session Summary QAPP Worksheet #10: Conceptual Site Model

QAPP Worksheet #11: Project/Data Quality Objectives

QAPP Worksheet #12a: Measurement Performance Criteria Table-Inorganics

QAPP Worksheet #12b: Measurement Performance Criteria Table - VOC

QAPP Worksheet #12c: Measurement Performance Criteria Table -1,4-Dioxane

QAPP Worksheet #13: Secondary Data Uses and Limitations

QAPP Worksheet #14/16: Project Tasks & Schedule

QAPP Worksheet #15a: Groundwater Project Action Limits and Laboratory-Specific Detection/ Quantitation Limits

QAPP Worksheet #15b: Effluent Project Action Limits and Laboratory-Specific Detection/ Quantitation Limits

QAPP Worksheet #17: Sampling Design and Rationale QAPP Worksheet #18: Sampling Locations and Methods

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

QAPP Worksheet #20: Field QC Summary

QAPP Worksheet #21: Field SOPs

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

QAPP Worksheet #23: Analytical SOPs

QAPP Worksheet #24: Analytical Instrument Calibration

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

QAPP Worksheet #26 & 27: Sample Handling, Custody, and Disposal

QAPP Worksheet #28a: Analytical Quality Control and Corrective Action-VOC

QAPP Worksheet #28b: Analytical Quality Control and Corrective Action-1,4-Dioxane

QAPP Worksheet #28c: Analytical Quality Control and Corrective Action-Inorganics

QAPP Worksheet #29: Project Documents and Records

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action QAPP Worksheet #34: Data Verification and Validation Inputs QAPP Worksheet #35: Data Verification Procedures

QAPP Worksheet #36: Data Validation Procedures QAPP Worksheet #37: Data Usability Assessment

List of Figures

Figure 1 Area 7 Location

Figure 2 Monitoring Well Locations

Figure 3 Extraction Well Locations

Figure 4 Groundwater Management Zone Boundaries

Appendices

Appendix A – Sampling and Analysis Plan with CDM Smith Sampling Standard Operating Procedures

Appendix B – Analytical Services Branch Laboratory Information

Appendix C – Example Data Validation Forms

References

CDM Smith Inc. (CDM Smith). 1995. Southeast Rockford Final RI Report. January.

CDM Smith. 2000. Final Remedial Investigation Report for the Southeast Rockford Source Control Operable Unit. July.

CDM Smith. 2012. Area 7 Remedial Design Operations and Maintenance Plan. October.

Nationwide Environmental Services, Inc. 2017. Quality Assurance Project Plan Remedial Design/Remedial Action Southeast Rockford Groundwater Contamination Site. March.

U.S. Environmental Protection Agency (U.S. EPA). 2002. Superfund Record of Decision: Southeast Rockford Ground Water Contamination. June.

U.S. EPA. 2002. EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5. EPA/ December.

U.S. EPA. 2005. Uniform Federal Policy for Quality Assurance Project Plans. March.

U.S. EPA. 2009. Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use. Office of Solid Waste and Emergency Response No. 9200.1-85, EPA 540-R- 08-005. Office of Solid Waste and Emergency Response. January.

U.S. EPA. 2012. Uniform Federal Policy for Quality Assurance Project Plans Optimized Worksheets. March.

U.S. EPA. 2017. National Functional Guidelines for Organic Superfund Methods Data Review. EPA-540-R-2017-002. January.

EPA. 2017. National Functional Guidelines for Inorganic Superfund Methods Data Review. EPA-540-R-2017-001. January.

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QAPP Worksheet #1 & 2: Title and Approval Page

Site name: Southeast Rockford Groundwater Contamination Superfund Site (SERGC Superfund Site)

Project name: Area 7 Groundwater Monitoring Zone (GMZ) Monitoring Activities

Site location: Winnebago County, Illinois **Site number**: ILD981000417

Operable Unit: OU3

Document title: Quality Assurance Project Plan for Southeast Rockford Groundwater Contamination Superfund Site, Source Area 7 Groundwater Monitoring Zone Monitoring Activities

Lead Organization: Illinois Environmental Protection Agency (Illinois EPA)

Contractor Name: CDM Smith Inc. **Contract**: HWA-16303 **Work Order**: #06

Addendum Preparation date: May 4, 2021

Regulatory Program: Superfund aka Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), as amended

Approval entities: Illinois EPA and U.S. EPA

Dates and Titles of QAPPs prepared for previous site-specific work:

- Southeast Rockford Groundwater Contamination Superfund Site, Area 7:
  - Quality Assurance Project Plan, Indoor Air Sampling (IAS) Study. May 19, 2003.
  - Quality Assurance Project Plan Addendum Area 7 Phase II Pre-Remedial Design Field Study. March 14, 2006.
  - Quality Assurance Project Plan Addendum for the Indoor Air Sampling (IAS) Study and Source Area 7 Additional Phase II Pre-Remedial Design Field Activities. June 30, 2010.

Data Users: Illinois EPA, U.S. EPA, and CDM Smith

Prepared By: _____ Date: May 4, 2021
CDM Smith Project Scientist/Engineer

Prepared By: Jeniffer Oxford Date: May 5, 2021
CDM Smith Quality Assurance Reviewer

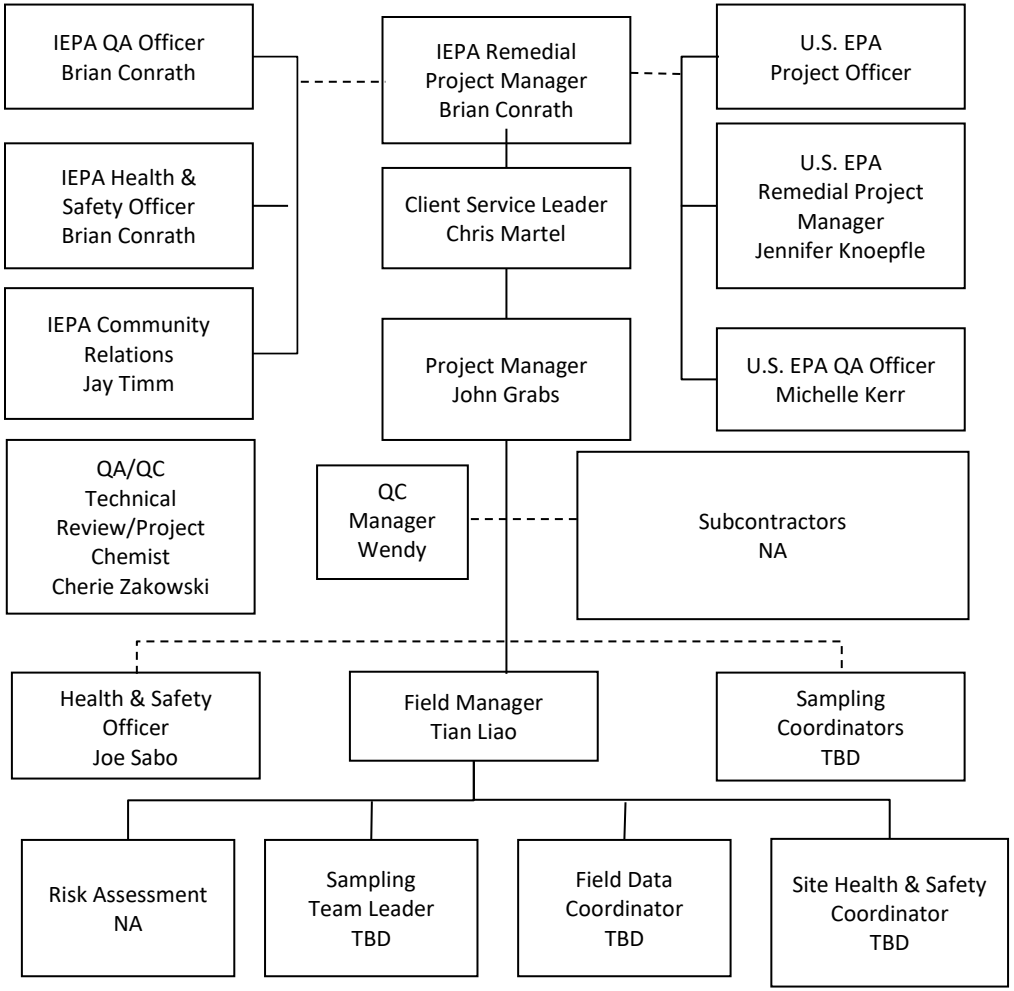
Prepared By: Brian A. Comath Date: 06 May 2021
Illinois EPA
Remedial Project Manager (RPM)

Prepared By: _____ Date: 13 May 2021
U.S. EPA Region 5
Remedial Project Manager (RPM)

Prepared By: _____ Date: 29 June 2021
U.S. EPA Region 5
Quality Assurance Reviewer

Prepared By: _____ Date: June 22, 2021
CDM Smith Project Manager

QAPP Worksheet #3 & 5: Project Organization and QAPP Distribution



Distribution List

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address	DCN
Jennifer Knoepfle	RPM	U.S. EPA	312-886-7153	Knoepfle.Jennifer@epa.gov	230674-PRD-01
Michelle Kerr	QAO	U.S. EPA	312-886-8961	Kerr. Michelle@epa.gov	230674-PRD -02
Brian Conrath	RPM	Illinois EPA	217-557-8155	Brian.Conrath@illinois.gov	230674-PRD -03
John Grabs	PM	CDM Smith	312-346-5000	grabsjc@cdmsmith.com	230674-PRD -04
Wendy Dewar	Project QC Manager	CDM Smith	847-975-5765	dewarwt@cdmsmith.com	230674-PRD -05
Tian Liao	Field Manager	CDM Smith	732-590-4680	liaot@cdmsmith.com	230674-PRD -06

Notes: CDM Smith Project QC Manager is responsible for maintenance and distribution of the QAPP.
DCN – Document Control Number
RPM – Remedial Project Manager
QAO– Quality Assurance Officer
QC - Quality Control
PM – Project Manager
QA – Quality Assurance
U.S. EPA – United States Environmental Protection Agency
Illinois EPA – Illinois Environmental Protection Agency

QAPP Worksheet #4, 7 & 8: Personnel Qualifications and Sign-off Sheet

ORGANIZATION: CDM Smith and Subcontractors

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Wendy Dewar	Project QC Manager, CDM Smith	B.S. Chemical Engineering, 34 years of experience		
John Grabs	Project Manager, CDM Smith	B. S. Geology, 30 years of experience	PG	
Chris Martel	Client Service Leader, CDM Smith	M.S. Environmental Engineering, 23 years of experience	PE, BCEE, LEED AP	
Tian Liao Field Sampling Staff TBD	Field Manager Project Engineers/Scientists	Various TBD	40-Hour OSHA HAZWOPER Training and Recurrent Annual 8- hour refreshers/CPR/First Aid	

ORGANIZATION: State and Federal Agencies

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Brian Conrath	Illinois EPA Remedial Project Manager	B.S. Geology, 19 years of experience		
Jennifer Knoepfle	U.S. EPA Remedial Project Manager	Ph.D, P.G.		
Michelle Kerr	U.S. EPA Quality Assurance Officer			

Personnel Responsibilities

Name	Title	Organizational Affiliation	Responsibilities
Brian Conrath	Remedial Project Manager	Illinois EPA	Overall responsibility for all phases of work, review and approval – Lead Agency
Jennifer Knoepfle	Remedial Project Manager	U.S. EPA Region 5	Funding Agency responsible for review and approval of QAPP
John Grabs	Project Manager	CDM Smith	The Project Manager is responsible for ensuring the quality of work performed for this project. The Project Manager interfaces directly with the Illinois EPA Program Manager and has overall responsibility and direction for task assignments.
Wendy Dewar	Project QC Manager	CDM Smith	The QA Manager reviews the project deliverables and has overall responsibility for project QA. The QA Manager will also perform a compliance check of all data received from the laboratory. The QA Manager is the main interface with the laboratory regarding project deliverables and QA/QC aspects of the analyses. The QA Manager also coordinates sample delivery and ensures that all analyses are performed, and results are delivered on time.
Joe Sabo	H&S Officer	CDM Smith	The H&S officer approves the HASP and provides guidance to field personnel on H&S issues.
Tian Liao	Field Manager	CDM Smith	Coordinate, direct, participate in and report site activities; ensures adherence to QAPP/SAP and communicates issues to the Project Manager and field team. Oversees subcontractors.
TBD	Laboratory Manager	U. S. EPA Contract Laboratory Program (CLP) or Analytical Services Branch (ASB) laboratory	The laboratory manager is responsible for the performance of requested analyses, project deliverables and QA/QC aspects of the analyses. The laboratory manager has overall responsibility to ensure that all analyses are performed according to the requested methods and results are delivered on time.
Wendy Dewar	QAPP Preparer	CDM Smith	The QAPP preparer is responsible for preparing the site-specific QAPP and is in close communication with the PM and Laboratory Manager regarding all aspects of project-specific requirements.

Notes:

PM – Project Manager
QA – Quality Assurance
QC – Quality Control
QAPP – Quality Assurance Project Plan
U.S. EPA – United States Environmental Protection Agency
SAP – Sampling and Analysis Plan
Illinois EPA – Illinois Environmental Protection Agency

QAPP Worksheet #6: Communication Pathways

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathways, etc.)
Project scope changes	CDM Smith PM	John Grabs	312-346-5000 grabsjc@cdmsmith.com	The PM will inform the Illinois EPA PM of any project scope changes by phone and email with 24 hours.
Management of required project tasks	CDM Smith PM	John Grabs	312-346-5000 grabsjc@cdmsmith.com	The CDM Smith PM will inform the appropriate CDM Smith project staff (field and non-field) of tasks to complete and the required completion date verbally prior to start of work and as needed during project execution. The CDM Smith project staff will communicate with the CDM Smith PM of task progress and resources/information required to complete tasks by email or phone daily or weekly depending on project phase.
Delays or changes to field work	CDM Smith Field Manager (FM)	Tian Liao	732-590-4680 tianl@cdmsmith.com	The FM will inform the CDM Smith PM of any delays or changes to field work by email or phone within 24 hours. If change is considered critical to the conduct of the project the FM will inform the PM by end of the workday. The CDM Smith PM will inform the Illinois EPA RPM by telephone or e-mail within 24 hours.
Daily field updates	CDM Smith FM	Tian Liao	732-590-4680 tianl@cdmsmith.com	The FM will inform the CDM Smith PM of daily field progress by email or phone. The CDM Smith PM will inform the Illinois EPA RPM of field work progress by telephone, email, or direct communication in the field daily or weekly depending on project phase.
Reporting of Laboratory Data Quality Issues	CDM Smith QC Manager	Wendy Dewar	847-975-5765 dewarwt@cdmsmith.com	The CDM Smith QCM or FM will inform the CDM Smith PM by email or phone of any issues related to data quality upon receipt of samples or during analyses. Such issues may include insufficient sample volume, sample shipping delays, temperature or preservations problems.
Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathways, etc.)

Communication Drivers	Organization	Name	Contact Information	Procedure (Timing, Pathways, etc.)
Analytical Corrective Actions	U.S. EPA CLP Or Region 5 Analytical Services Branch Laboratory	U.S. EPA CLP Or Region 5 Analytical Services Branch Laboratory	TBD	The need for corrective action will be determined by the project chemist and the lab QA Director. They will ensure the SOW/SOP requirements as detailed in this QAPP are met and all data will be reviewed for completeness and conformance with analytical guidelines prior to release to data users. Any data quality issues requiring corrective action will be reported to the CDM Smith QCM as soon as possible.
Recommendations to stop work and initiation of corrective actions	All	All	All	Any member of the project team (e.g., CDM Smith Field Staff, CDM Smith FM, CDM Smith QCM, Illinois EPA, etc.) has the authority to stop work and initiate corrective actions should there be a reason to do so. Examples include health and safety issues and problems with data quality. Whoever stops the work or initiates corrective actions will inform the CDM Smith FM and PM immediately verbally or by phone. The PM will ensure that the Illinois EPA RPM is informed of the stop work and corrective actions.
Distribution of analytical data	CDM Smith QCM U.S. EPA CLP or Region 5 Analytical Services Branch Laboratory	Wendy Dewar U.S. EPA Region 5 Sample management staff	847-975-5765 dewarwt@cdmsmith.com	CLP data will be distributed to CDM Smith QCM and other data users by U.S. EPA Region 5 after validation. Data will be provided in hard copy and digitally. Analytical Services Branch data will be distributed to CDM Smith electronically for validation by CDM Smith.
Approval of QAPP Amendments	Illinois EPA RPM U.S. EPA QAO	Brian Conrath Michelle Kerr	217-557-8155 312-886-8961	Approval of all QAPP amendments will be by the Illinois EPA RPM and U.S. EPA QAO in writing prior to the changes being implemented.

Notes:

Illinois EPA– Illinois Environmental Protection Agency
PM – Project Manager
FM – Field Manager
QA – Quality Assurance
TBD- To be Determined
U.S. EPA – United States Environmental Protection Agency
CLP – Contract Laboratory Program
QAO – Quality Assurance Officer
QCM – Quality Control Manager
QAPP – Quality Assurance Project Plan

QAPP Worksheet #9: Project Planning Session Summary

Date of planning session: March 11, 2019 through March 29, 2019, May 15, 2019 and October 2019.

Location: Various teleconferences, emails, and on-site discussions at Source Area 7.

Purpose: Develop scope of work for Area 7 GMZ monitoring based on historical and current groundwater monitoring information and requirements of the GMZ application. The information generated will be used to monitor compliance with the GMZ requirements and performance of the leachate treatment system (LTS).

Participants

Name	Organization	Title/Role	Email/Phone
John Grabs	CDM Smith	PM	312-780-7737 grabsjc@cdmsmith.com
Brian Conrath	Illinois EPA	RPM	217-557-8155 Brian.conrath@illinois.gov
Wendy Dewar	CDM Smith	Project QCM	312-346-5000 dewarwt@cdmsmith.com

Notes:

Illinois EPA – Illinois Environmental Protection Agency
Control Manager
PM – Project Manager
QAPP – Quality Assurance Project Plan QCM – Quality
SAP – Sampling and Analysis Plan
RPM – Remedial Project Manager

Consensus decisions made: Overall scope of work was agreed upon in March 2019. Sample analysis will be 21- day turnaround CLP level data analysis for target compound list (TCL) volatile organics in influent, effluent and groundwater from all wells and target analyte list (TAL) inorganics in groundwater from extraction wells and LTS influent and effluent samples. An additional request was made by U.S. EPA to add analysis for 1,4 dioxane for monitoring well and effluent samples to be performed by the U.S. EPA Region 5 Analytical Services Branch (ASB) laboratory. Based on historical groundwater data, some of the groundwater samples and the influent samples may be medium level concentration.

In May 2019, additional information regarding laboratory assignments was distributed by U.S. EPA Region 5 staff that impacts project planning. Based on this information, it is U. S. EPA Superfund policy that all analytical work go through a tiered approach of sample scheduling to reduce cost and maintain data quality. Depending on the information provided by the contractor on the Analytical Services Request (ASR) form, U.S. EPA Region 5 may first try to schedule analytical services with the ASB laboratory in Chicago. If the ASB laboratory cannot support the request, then the request will go to the Contract Laboratory Program (CLP). The sampling entity will be notified of their laboratory assignment as soon as it is procured. The November 15, 2019

QAPP was prepared assuming the analytical VOC and inorganic samples will go to the CLP as this has historically been the case. However, references have been added in relevant sections for procedures that may be unique to samples being sent to the ASB laboratory**. Appendix B** of this QAPP contains all relevant ASB information that would be needed if VOC and/or inorganic samples are scheduled to be sent to the ASB Laboratory for this project.

Samples collected for this project for 1,4-dioxane will be sent to and analyzed by the ASB laboratory as discussed in the October 2019 planning meeting.

Action Items

Action	Responsible Party	Due Date
QAPP/SAP preparation	CDM Smith QCM	July 2019
GMZ Application preparation	CDM Smith PM	September 2019

As required by U.S. EPA Quality Assurance policy that QAPPs be updated annually for ongoing projects, a second project planning/quality management meeting was conducted for this project in October 2020 via teleconference. The objectives of this meeting were to introduce new project personnel, define staff roles and responsibilities, discuss project changes and lessons learned since November 2019 and assign action items moving forward. The primary action item identified was preparation of this QAPP/SAP addendum to address the issues below:

- Update sampling standard operating procedures (SOPs)
- Revise groundwater sampling protocols due to field conditions
- Update analytical methods and laboratory options to be used for sample analysis
- Update sample validation requirements and protocols

Additionally, March 25, 2021, U.S. EPA issued information for their updated CLP SOWs, CLP Sampler's Guide and National Functional Guidelines for Superfund Data Review. This first QAPP addendum includes this new information.

The CDM Smith QCM is responsible for this action item; the due date for this first QAPP update/addendum is May 5, 2021.

QAPP Worksheet #10: Conceptual Site Model

Problem Definition and Background

Based on remedial investigations and site-specific risk assessment, Remedial Action Objectives (RAOs) were developed. The Area 7 RAOs provide a general description of what the remedial action will accomplish and are as follows:

- Prevent the public from ingestion of soil, and direct contact with soil containing contamination in excess of state or federal standards or that poses a threat to human health
- Prevent the public from inhalation of airborne contaminants in excess of state or federal standards or that pose a threat to human health
- Prevent the further migration of contamination from Area 7 that would result in degradation of site-wide groundwater or surface water to levels in excess of state or federal standards, or that pose a threat to human health or the environment

A number of potential remedial action alternatives for Area 7 were developed and evaluated based on RAOs, remediation goals, and comparative evaluation criteria. The detailed comparative analysis of Area 7 remedial alternatives is discussed in detail in the Record of Decision (ROD). The remedial action (RA) currently being implemented at Area 7 is a leachate treatment system (LTS) that includes leachate and soil vapor extraction and treatment and hydraulic containment within Area 7 to address contamination migrating from the area. Groundwater extraction wells have been installed for long-term hydraulic containment of the contaminated groundwater. A Groundwater Management Zone (GMZ) will be established as required by the ROD.

GMZ Monitoring in Area 7 will be performed to comply with GMZ application requirements and to monitor performance of the LTS in Area 7. **Figure 1** shows Source Area 7. **Figure 2** shows monitoring wells and **Figure 3** shows extraction wells to be sampled in Area 7 for the GMZ Monitoring activities. Additional hydrological data will also be gathered and used to optimize the contaminant removal and treatment system for Area 7.

Site Description

Area 7 is primarily a grassy area located at the eastern end of Balsam Lane. Area 7 contains Ekberg Park, an open field and a wooded area. Ekberg Park is owned and operated by the Rockford Park District and the remainder of Area 7 is owned by Glen Ekberg. The topography slopes gently to the north towards an intermittent creek. Ekberg Park consists of a playground, basketball court, and open space.

The open field and wooded areas are south of the park on a hillside and surface water drainage down the hillside flows north into the intermittent creek. Residences border areas to the west and southwest (downgradient) and are also located about 0.1 mile away to the east (upgradient).

Chlorinated solvents are the principal contaminants present at the site. Contaminants were released to the environment from improper disposal practices. Three primary volatile organic compound (VOC) source areas were identified during the investigations and are located at the northern and southern areas of Ekberg Park. Northern source areas contain the northwest side of the playground and southwest side of the tennis courts. The southern area includes the south side of the basketball courts. The depth to significant levels of contamination varies from 4 feet below ground surface (bgs) in the northern area to approximately 30 feet in some southern locations.

Contamination is present in soils both above and below the water table with non-aqueous phase liquid (NAPL) present at various locations. Groundwater samples collected from the aquifer in the overburden soils reveal a variety of types and classes of VOCs, with the majority being chlorinated solvents.

Groundwater flow in both the unconsolidated and bedrock aquifers is to the west northwest, with localized discharge of shallow groundwater to the intermittent creek. Depth to groundwater ranges from about 60 feet bgs at the south end of Area 7 to 25 feet bgs south of the park area, to 13 feet bgs within the park, to less than 2 feet bgs near the creek. Seasonally, the depth to groundwater is highly variable with 10-foot changes possible over the course of a year.

Previous Investigations

The Phase I Remedial Investigation (RI) for the Southeast Rockford Groundwater Contamination Site was conducted from May to October 1991 and consisted primarily of a site-wide soil gas survey, monitoring well installation, and groundwater sampling and analysis to investigate multiple suspected source areas. The investigation of a potential source in Area 7 was sparked by elevated concentrations of contaminants in the shallow monitoring well of a three well cluster immediately downgradient of Area 7. Phase I of the RI included review of historical aerial photographs, geophysical surveys and soil gas sampling. The geophysical surveys indicated several areas of subsurface anomalies which were subsequently investigated with soil gas sampling. Soil gas results indicated that high concentrations of VOCs, primarily trichloroethylene (TCE), perchloroethylene (PCE), and trichloroethane (TCA) were present in the subsurface.

During Phase II of the RI, additional work was conducted in Area 7 consisting of test-pit investigation, surface and subsurface soil sampling, and monitoring well installation.

The Phase II site-wide groundwater investigation conducted concurrently also indicated the same contaminant mix of TCA, PCE and TCE downgradient, confirming that subsurface in Area 7 was impacting site-wide groundwater. Residential air sampling was conducted near Area 7 in 1993 and 2003 to determine if soil and groundwater contamination was affecting indoor air quality in homes near the source. Indoor air evaluations indicated that migration pathways are generally inadequate or incomplete and do not result in indoor air concentrations at levels that present an unacceptable health risk.

The Remedial Investigation Report for the site-wide groundwater investigation and source area identification was completed by CDM Smith (CDM Smith 1995) and resulted in the signing of the

OU2 record of decision (ROD) which required additional extension of the City of Rockford municipal water system and selected natural attenuation, long-term groundwater monitoring, and source control measures that would be determined in the future under the Source Control Operable Unit (SCOU).

The SCOU RI and Focused Feasibility Study (FFS) reports were completed in July 2000. The SCOU FFS addressed contaminated soils, NAPL, and leachate considered to be principal threat wastes and the primary causes of groundwater contamination at the four primary source areas southeast Rockford. Alternatives developed in the SCOU FFS were separated into soil and leachate alternatives for each source area. In order to simplify the OU 3 ROD, technologies intended to contain and/or treat contaminated groundwater in the immediate vicinity of the primary source areas were considered leachate alternatives.

SCOU investigation results for Area 7 showed elevated concentrations of toluene, ethyl benzene, xylenes, and chlorinated VOCs in Area 7 soils. The previous RI identified subsurface contamination along drainage ditches in the southern portion of Area 7. The SCOU investigation confirmed that an additional hot-spot area of soil contamination exists in and to the west and northwest of Ekberg Park. Elevated levels of VOCs were also found in groundwater and in surface water of the intermittent creek located on the property. Shallow groundwater downgradient of Area 7 contains elevated concentrations of TCA as the primary contaminant and lesser concentrations of various other VOCs. NAPL were found in soil 11 feet below the groundwater table in a soil boring in the park. Additionally, Area 7 was found to have areas of highly contaminated silt and clay units indicating that NAPL has migrated into these fine- grained sediments.

Results of the groundwater investigation indicated that distribution and concentration of contaminants within groundwater monitoring wells were generally similar to results from previous sampling investigations. The highest concentrations of VOCs were observed within the unconsolidated aquifer at monitoring well location MW-134A (**Figure 2**). Results of the groundwater sampling from wells (MW135B, MW143, MW144, and MW145) in the southern portion of Area 7 also showed significant downgradient VOC contamination. These results also indicated that the VOC source extended further south than previously believed. Additional groundwater monitoring wells were installed in August 2010 to define the southern extent of VOC groundwater contamination.

In 2010, under an Explanation of Significant Differences (ESD) to the ROD, CDM Smith conducted a limited soil excavation northwest of the playground area to remove shallow low permeability source material. This limited action was completed in May 2011.

The remedial action (RA) currently being implemented at Area 7 includes leachate and soil vapor extraction and treatment and hydraulic containment within Area 7 to address contamination migrating from the area. Multiphase extraction wells have been installed in source areas to extract leachate, vapor and NAPL. The extracted stream is pumped to a treatment system consisting of liquid/vapor separators, oil/water separators, iron removal system, air stripper,

liquid phase carbon for contaminant treatment and vapor phase carbon for vapor effluent treatment. Treated effluent water is discharged to the intermittent creek located north of the treatment system. Groundwater extraction wells have been installed for long-term hydraulic containment of the groundwater along a north-south trending line at the edge of the contaminant plume. Extracted groundwater is pumped directly to the iron removal system since groundwater from these wells are not expected to contain NAPL. **Figure 4** shows the proposed GMZ boundary that will be established for the site to monitor the effectiveness of the contaminant treatment and containment system.

QAPP Worksheet #11: Project/Data Quality Objectives

Who will use the data? Illinois EPA, CDM Smith, and U.S. EPA Region 5.

What will the data be used for? The data will be used to comply with the groundwater monitoring requirements of the GMZ for Area 7 and to monitor effectiveness of the contaminant removal and treatment system. Additional hydrological data will also be used to optimize the extraction system performance.

What type(s) of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques): Beginning March 25, 2021, the U.S. EPA CLP is scheduling samples under their updated SOW which combines the organic and inorganic methods into one comprehensive SOW SFAM01.1. Details for this SOW may be found at: https://www.epa.gov/clp/epa-contract-laboratory-program-statement-work-superfund-analytical-methods-multi-media-multi-0.

The scope of these activities included one influent and one effluent sample collected quarterly during system operation. Influent and effluent samples will be analyzed for VOCs by Contract Laboratory Program (CLP) (21-day turnaround time) using SOW SFAM01.1 or equivalent or by ASB laboratory using SOP MS023 VOA in Water, Version 7. These samples will also be analyzed for total inorganics by CLP (21-day turnaround time) using SOW SFAM01.1 or equivalent or by ASB laboratory using SOP Metals003A ICP Metals – Thermo, Version 8. Effluent samples will also be analyzed for dissolved inorganics by CLP (21-day turnaround time) using SOW SFAM01.1 or equivalent or by ASB laboratory using SOP Metals003A ICP Metals – Thermo, Version 8 and 1,4-dioxane using ASB compound specific method SOP MS035, Version 2 (45-day turnaround time) or by CLP using SOW SFAM01.1 semi-volatile organic analysis (SVOA) modified by Method 3054.0. The CLP 1,4-dioxane method is an SVOA analysis by selective ion monitoring (SIM) at lower Contract Required Quantitation Limit (CRQL) for 1,4-dioxane only. Unless specified in the modification, all analyses, quality control, and reporting requirements specified in CLP SOW SFAM01.1 will be followed.

Groundwater sampling will be conducted quarterly for a period of at least 5 years. Groundwater samples collected from the monitoring and extraction wells will be analyzed for VOCs by CLP (21-day turnaround time) using SOW SFAM01.1 or equivalent or by ASB laboratory using SOP MS023 VOA in Water, Version 7. Samples from the monitoring wells will be analyzed for 1,4-dioxane using ASB compound specific method SOP MS035, Version 2 (45-day turnaround time) or by CLP using SOW SFAM01.1 for SVOA modified by Method 3054.0. Groundwater samples from the extraction wells will also be analyzed for inorganics by CLP (21-day turnaround time) using SOW SFAM01.1 or equivalent or by ASB laboratory using SOP Metals003A ICP Metals – Thermo, Version 8.

If VOC and/or inorganic samples for this project are sent to the Analytical Services Branch (ASB) laboratory, their standard operating procedures (SOPs) and parameter lists for VOCs and inorganics are provided in **Appendix B**. The U.S. EPA Region 5 ASB laboratory may also be used for the 1,4-dioxane analyses; the SOP is included in **Appendix B**.

How "good" does the data need to be in order to support the environmental decision? The VOC and 1,4-dioxane data will be definitive level data to support the RAOs and DQOs. The majority of the inorganic data parameters are considered informational with the exception of iron which is critical data as it is monitored for compliance with the discharge limits and as a component of treatment system fouling. The project RAOs are provided on Worksheet #10. The reporting limits need to be sufficient to compare results to Project Action Limits (PALs). PALs for groundwater are the remediation goals established in the ROD for Area 7 and for the additional parameter, 1,4-dioxane, the groundwater remediation goal is the groundwater quality standard for class I potable groundwater listed in Illinois Administrative code (IAC) 35 620.410. PALs for effluent are the discharge limits provided in the remediation contractor's operation and maintenance (O&M) plan which include limits established in the ROD as well as numerical standards per 35 IAC 302.208 and limits established by Illinois EPA based on Derived Water Quality Criteria, April 4, 2013. U.S. EPA-approved methods will be utilized (such as the most current contract laboratory statement of work) and must be followed by the laboratory. Refer to QAPP Work Sheets #15a, #15b and #37. Due to the differences in the list of parameters analyzed for VOCs by CLP versus ASB, eight VOC parameters with discharge limits that are analyzed by ASB laboratory are not analyzed by CLP. These parameters will be compared to the PALS for samples analyzed for VOCs by ASB and are noted on Worksheet 15b. Additionally, the prescribed field sampling and health and safety measures for the data gathering activities must be followed by the field staff to further ensure data gathered will meet the DQOs. The SAP is provided as **Appendix A** of this QAPP. The water level data are checked in the field for anomalies against historical information and for transcription errors and will be used to evaluate changes to groundwater flow. The health and safety plan and field procedures are presented in the SAP **Appendix A** and **Appendix B**, respectively.

How much data is needed? (number of samples for each analytical group, matrix, and concentration): The influent and final effluent from the LTS were sampled monthly for VOCs and total inorganics and the effluent was sampled for dissolved inorganics and 1,4-dioxane prior to the LTS being declared operational and functional. Beginning in December 2020 this sampling is conducted quarterly for the duration of system operation. Concentrations of VOCs in the influent are expected to be medium level as this sample is collected prior to treatment. Concentrations of VOCs, 1,4-dioxane and total and dissolved inorganics in the effluent are expected to be low as the samples will be collected at the end of the treatment process and prior to discharge.

Fourteen extraction wells will be sampled quarterly for VOCs and total inorganics for a total of 280 investigative samples over 5 years. Twenty-five monitoring wells that comprise the GMZ network will be sampled quarterly for VOCs and 1,4-dioxane for a total of 500 investigative samples. Most monitoring and extraction well samples are expected to be low to medium concentration because they are not located within hot spot areas. Based on historical data it is likely that VOC samples from multiple monitoring wells, including but not limited to MW-134A, MW-134B, MW-105A, MW-105B, MW-135A. MW-144, MW145, and MW-106A may be medium concentration during the early sampling events. Depending on system performance over time, sampling frequency and duration may be adjusted.

Worksheets #17 and #18 provide sampling rationale, a summary of the sampling and analysis network and specifies the sample matrix, the parameters to be measured, the number of samples to be collected, and the level of QC effort for each sample type. For VOC, inorganic and/or1,4-dioxane samples sent to the Analytical Services Branch (ASB) laboratory, analytical procedures and parameter lists are provided in the ASB method standard operating procedures (SOPs) in **Appendix B**.

Where, when, and how should the data be collected/generated? The sampling program began during the startup phase of the LTS. Influent and effluent were sampled monthly during LTS startup. A round of baseline water levels and groundwater sampling were conducted during startup in April 2019. Beginning in December 2020 after the start-up period, influent and effluent sampling will be conducted quarterly for the duration of LTS operation and groundwater sampling will be conducted quarterly for a period of 5 years. **Figures 2** and **3** show locations of monitoring and extraction wells to be sampled, respectively. Additional information is provided in Worksheets #17 and #18. Specific procedures for sample shipment are provided in Section 3 of the SAP; specific information on sampling locations is provided in Section; 4 and specific procedures for sample collection are provided in Section 5 of the SAP which is **Appendix A** of this QAPP.

Who will collect and generate the data? CDM Smith, under contract to Illinois EPA, will collect the influent, effluent and quarterly groundwater samples. The VOC, 1,4-dioxane and/or inorganic analysis may be performed by CLP, or any or all of these analyses may be assigned to be performed by the Region 5 ASB laboratory. The sample analysis is designed to provide data to monitor compliance with GMZ requirements and evaluate LTS performance. See **Appendix B** for ASB laboratory details.

How will the data be reported? CLP data will be reported with a Level IV data package electronically within 21 days of sample analysis. CLP data will be validated within 3 weeks of data package receipt by U.S. EPA ESAT data validation staff using the Stage 2B validation option. Data generated by Region 5 ASB laboratory will be reported within 45 days of sample analysis with a Level IV data package and will be validated by CDM Smith data validation staff using Stage 2B validation. The CDM Smith QC Manager will perform a compliance check of all data received from the CLP and ASB laboratories and evaluate the usability of the data to achieve the data quality objectives.

If the Stage 2B data validation identify data quality issues for either CLP or ASB data, the data validation staff and/or the CDM Smith QC Manager, in conjunction with the CDM Smith PM and Illinois EPA PM, may elevate the level of validation for the data to more fully evaluate the quality issues.

How will the data be archived? CDM Smith, under contract to Illinois EPA, will maintain a copy of all site-related data and files for a period of 10 years. Hard copy data will be maintained in secure files in CDM Smith Chicago office. Electronic data will be archived on CDM Smith's record storage system which is backed up in real time to CDM Smith's cloud storage. As necessary, hard copy records may be transferred to an offsite secure, controlled access records storage facility.

Notes:

Illinois EPA – Illinois Environmental Protection Agency
QAPP – quality assurance project plan
QC – quality control
U. S. EPA – United States Environmental Protection Agency
ESAT – Environmental Services Assistance Team
QA – quality assurance
VOC – volatile organic compound
TCL – Target compound list
ASB – Analytical Services Branch
SVOC – semi-volatile organic compound
SIM – selected ion monitoring
CRQL – Contract Required Quantitation Limit
SAP – Sampling and Analysis Plan
SOP – standard operation procedure
PAL – Project Action Limit

DQO for Area 7 GMZ Monitoring Activities

Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7
STATE THE PROBLEM	IDENTIFY THE DECISIONS	IDENTIFY INPUTS TO DECISIONS	DEFINE STUDY BOUNDARIES	DEVELOP DECISION RULES	SPECIFY LIMITS ON DECISION ERRORS	OPTIMIZE SAMPLING DESIGN
Need to collect quarterly groundwater monitoring data and water levels from 25 monitoring wells for VOCs and 1,4-dioxane to evaluate compliance with the GMZ established for Area 7 and evaluate leachate treatment system (LTS) performance.	Does groundwater VOC and 1,4-dioxane data indicate compliance with GMZ requirements? Does groundwater VOC and 1,4-dioxane data indicate changes in concentrations resulting from contaminant extraction and treatment?	Photoionization detector (PID) screening of groundwater to identify samples that may be medium concentration for VOCs. Quarterly water level measurements prior to sample collection. Validated VOC, inorganic and 1,4-dioxane data for	The boundary of the study includes the sampling port for the LTS influent located after the equalization tank and the sampling port for the effluent line from the LTS, the GMZ monitoring well network, the extraction well locations and the GMZ boundary.	If the quarterly groundwater results indicate non-compliance with GMZ requirements the GMZ boundaries may be modified or additional monitoring points may be required. If quarterly groundwater data indicate VOC or 1,4-dioxane concentration levels are decreasing	Professional judgment will be used to determine practical vs. statistical significance of test results. Quarterly water level measurements will be compared to historical levels obtained prior to the operation of the treatment system and with levels measured over time	For GMZ monitoring activities groundwater monitoring and extraction wells listed in the GMZ application will be sampled. Three wells are located upgradient and the remaining wells are located within the area of contamination or downgradient of the main areas of

Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7
STATE THE PROBLEM	IDENTIFY THE DECISIONS	IDENTIFY INPUTS TO DECISIONS	DEFINE STUDY BOUNDARIES	DEVELOP DECISION RULES	SPECIFY LIMITS ON DECISION ERRORS	OPTIMIZE SAMPLING DESIGN
Need to collect quarterly groundwater monitoring data for VOCs and inorganics from 14 extraction wells to evaluate and optimize LTS performance. Need to collect sample of effluent from the LTS immediately prior to discharge to confirm compliance with discharge limits. Need to collect sample of influent and effluent from LTS to calculate system performance efficiency and mass removal.	Does effluent and groundwater VOC, inorganic and 1,4-dioxane data indicate that the LTS is functioning as designed? Is the discharge from the LTS influencing the local hydrology? Does the effluent from the LTS meet discharge requirements? Does the calculated system efficiency and mass removal meet projected results?	groundwater samples collected during quarterly groundwater monitoring. Validated VOC, inorganic and 1,4-dioxane data for influent and effluent samples collected monthly during startup and quarterly after LTS is declared operational and functional.	See Figures 2, 3 and 4. Groundwater monitoring activities and water level measurements will be conducted quarterly for a period of 5 years. Influent and effluent sampling will be conducted monthly until the LTS is declared operational and functional and quarterly thereafter during LTS operation.	over time the LTS will be assumed to be operating as designed and monitoring frequency may be adjusted. If quarterly groundwater data indicate VOC and 1,4-dioxane concentration levels are stagnant or increasing over time the LTS operation will be evaluated and adjusted. If inorganic data indicate compounds that could foul the LTS, the treatment train will be modified. If water level data indicate impacts to the hydrology the effluent discharge process may be adjusted. If effluent data are consistent and below discharge limits the LTS will be assumed to be operating as designed. If LTS data are above discharge limits the LTS operation will be	during operation. The groundwater data will be evaluated over time and compared to the baseline data and remediation goals. The remediation goals are PALs provided on Worksheet #15a. Effluent data will be evaluated over time for consistency and compared to the discharge limits on Worksheet #15b.	contamination and downgradient of the extraction well system. Effluent sample will be collected from the sampling port located immediately before the LTS discharge point to evaluate fully treated leachate being discharged to the intermittent creek. Influent sample data will be used with effluent sample data to evaluate system performance over time and allow for system adjustment and/or optimization. Projected LTS efficiency/mass removal rate is 99-100% calculated by influent minus effluent divided by influent.

Step 1	Step 2	Step 3	Step 4	Step 5	Step 6	Step 7
STATE THE PROBLEM	IDENTIFY THE DECISIONS	IDENTIFY INPUTS TO DECISIONS	DEFINE STUDY BOUNDARIES	DEVELOP DECISION RULES	SPECIFY LIMITS ON DECISION ERRORS	OPTIMIZE SAMPLING DESIGN
				evaluated and adjusted. If the calculated system performance efficiency and/or mass removal rates are within the projected range the LTS will be assumed to be operating as designed. If the calculated efficiency and mass removal rate is not within the projected range the LTS operation will be evaluated and adjusted. Projected efficiency/mass removal rate is 99-100% calculated by influent minus effluent divided by influent.		

QAPP Worksheet #12a: Measurement Performance Criteria Table

Matrix	Water				
Analytical Group	TAL Inorganics				
Concentration Level	(Low/medium) ug/L				
Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)
See Worksheets #18 and #21	CLP SOW SFAM01.1 QAPP Worksheet #23 lists all applicable SOPs.	Precision	≤30% RPD	Field Duplicate	S & A
		Accuracy/Bias	No analyte > CRQL Exhibit C, Table 2	Equipment Blank; Preparation Blank	S & A A
		Precision	RPD ≤ 20; ±20% of true value;	Laboratory Duplicate Interference Check sample;	S & A A
		Accuracy/Bias	%R within 70-130%	LCS;	A
			%R within 75-125%	MS	
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare to historical data, calculate RPD at sampling point from previous event	S
		Sensitivity	See QAPP Worksheet #15b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assesses valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #12a in Appendix B

% R – Percent Recovery
≤ – Less than or equal to
MDL – Method Detection Limit
LCS – Laboratory Control Sample
CRQL – Contract Required Quantitation Limit
RPD – Relative Percent Difference
TAL – Target Analyte List
MS – Matrix Spike

QAPP Worksheet #12b: Measurement Performance Criteria Table

Matrix	Water				
Analytical Group	TCL CLP VOCs				
Concentration Level	Low /medium (µg/l)				
Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
See Worksheet #18 and #21	CLP SOW SFAM01.1 QAPP Worksheet #23 lists the applicable SOPs	Precision	≤30% RPD	Field Duplicate	S & A
		Accuracy/Bias	No analyte > CRQL	Method Blank Trip Blank	A S & A
		Precision	RPD per Table 11 SOW SFAM01.1 Exhibit D	MS/MSD	S & A
		Accuracy/Bias	Per Table 10;11 SOW SFAM01.1 Exhibit D	DMC; MS/MSD	A
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare data to historical data, calculate RPD at sampling point from previous event	S
		Sensitivity	See QAPP Worksheets #15a and b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assesses valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #12b in Appendix B

% – Percent
MS/MSD – Matrix Spike/ Matrix Spike Duplicate
≤ – Less than or equal to
RL – Reporting Limit
MDL – Method Detection Limit
RPD – Relative Percent difference
DMC – deuterated monitoring compound

QAPP Worksheet #12c: Measurement Performance Criteria Table

Matrix	Water				
Analytical Group	CLP SVOA SIM 1,4-Dioxane lower CRQL Method 3054.0				
Concentration Level	Low	(µg/l)			
Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
See Worksheet #18 and #21	CLP SOW SFAM01.1 with modified method 3054.0 SVOA SIM Analysis for 1,4-dioxane at lower CRQL QAPP Worksheet #23 lists the applicable SOPs	Precision	≤30% RPD	Field Duplicate	S & A
		Accuracy/Bias	No analyte ≥ CRQL	Method Blank	A
		Precision/Accuracy	RPD ≤ 30% R - 70%-130% ; R - 70%-130%	MS/MSD; LCS	S & A
		Accuracy/Bias	R 40-110%; 50-200%	DMC; Internal standard	A
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare data to historical data, calculate RPD at sampling point from previous event	S
		Sensitivity	See QAPP Worksheet #15a and b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assesses valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #12c in Appendix B

% – Percent
RL – Reporting Limit
MS/MSD – Matrix Spike/ Matrix Spike Duplicate
MDL – Method Detection Limit
≤ – Less than or equal to
RPD – Relative Percent difference
R - % Recovery
DMC – deuterated monitoring compound

SVOA – semi-volatile organic analysis SIM – Selected Ion Monitoring

Measurement performance criteria (MPC) for chemical analyses for each matrix and analytical parameter are summarized on the previous pages. The MPCs follow those defined in the referenced U.S. EPA method or laboratory standard operating procedures (SOPs). The quality of the data to be collected for this project will be verified through appropriate MPCs established for both sampling procedures and analytical methods. The sampling procedures and SOPs are provided in the SAP, Section 5 provided as **Appendix A** to this QAPP. Data quality indicators (DQIs), consisting of precision, accuracy, representativeness, comparability, completeness, and sensitivity, commonly referred to as PARCCS parameters are used to monitor data quality. The DQIs are defined as follows:

- Precision refers to the reproducibility of measurements. Precision is usually expressed as standard deviation, variance, percent difference, or range, in either absolute or relative terms.
- Accuracy/bias refers to the degree of agreement between an observed value (such as sample results) and an accepted reference value. A measurement is considered accurate and unbiased when the reported value agrees with the true value or known concentration of the spike or standard within acceptable limits.
- Representativeness describes the extent to which a sampling design adequately reflects the environmental conditions of a site. Representativeness is determined by appropriate program design, with consideration of elements such as proper well locations, drilling and installation procedures, operations process locations, and sampling locations. Variability within the context of representativeness is a measure of the degree to which data measure a variation at a sampling point when compared to previous or historical data.
- Comparability addresses the degree to which different methods or data agree or can be represented as similar. Comparability is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standard and comprehensive reporting formats.
- Completeness is a measure of the amount of valid data collected using a measurement system. Completeness is expressed as a percentage of the number of valid data generated versus the number of measurements that are specified in this QAPP.
- Sensitivity is the ability of a method or instrument to detect the target analytes at the level of interest with a stated level of confidence. Sensitivity can be measured by calculating the percent recovery of the analytes at the detection or reporting limit, which

is the minimum concentration of an analyte that can be routinely identified and quantified above the method detection limit by a laboratory.

The quality of the sampling procedures and laboratory results will be evaluated for compliance with project DQOs through a review of overall PARCCs, in accordance with procedures described in Worksheet #37 (Data Usability Assessment). The sampling procedures and SOPs are provided in the SAP, Section 5 provided as **Appendix A** to this QAPP. The results will be summarized in an overall data usability report.

QAPP Worksheet #13: Secondary Data Uses and Limitations

Data type	Source	Data uses relative to current project	Factors affecting the reliability of data and limitations on data use[1]
SERGC Sitewide RI Report	Southeast Rockford Final RI Report (CDM Smith 1995)	Well construction data, groundwater contours, contaminant concentrations in soil gas, soil and groundwater used to refine Area 7 remediation areas and potential concentrations	None
SERGC Source Area RI report	Final Remedial Investigation Report for the Southeast Rockford Source Control Operable Unit (CDM Smith 2000)	Well construction data, groundwater contours, contaminant concentrations in soil gas, soil and groundwater used to refine Area 7 remediation areas and potential concentrations	None
Area 7 groundwater monitoring data, soil gas and soil data	Source Area 7 Pre-Design Field Study Technical Memorandum (CDM Smith 2005)	Water level data, groundwater contaminant data, contaminate plume trends, source area data	None
Area 7 hydrogeological, groundwater and soil data	Area 7 Preliminary Remedial Design Report Source Area 7 Remedial Design (CDM Smith 2009)	Summary of historical and current data to define the known subsurface conditions at Area 7 and illustrate the data gaps. Provides all phases of pre-design investigation data and defines data needs in one document.	None
Project Action Limits	Southeast Rockford Groundwater Contamination EPA OU3 Record of Decision (Source Control Operable Unit) Operations & Maintenance Plan Bodine Environmental Services, Inc. Effluent Discharge Limits	Provides the project specific remediation goals for impacted media derived through the remedial investigation process. Provides the project specific discharge limits for treated effluent per ROD and Illinois EPA requirements and the system operation design parameters.	None

[1] All the above listed documents have been prepared or reviewed by CDM Smith and have undergone internal quality control reviews prior to submittal to Illinois EPA. All listed documents have been reviewed and approved by Illinois EPA and are part of the administrative record for the Site. All data presented in these documents were generated under an approved QAPP/FSP that document quality requirements for the data.

SERGC – Southeast Rockford Groundwater Contamination site
OU3 – Operable Unit 3
RI – Remedial Investigation

QAPP Worksheet #14/16: Project Tasks & Schedule

Sampling Tasks:

The scope of the influent and effluent sampling included monthly samples until the LTS was declared operational and functional and quarterly thereafter for the duration of LTS operation. Quarterly sampling began in December 2020. The influent sample will be collected from the sampling port located after the equalization tank, where the liquid streams from the multi-phase extraction wells and groundwater extraction wells are combined. The effluent sample will be collected from the effluent sample port located just prior to the LTS effluent discharge point. The influent and effluent samples will be analyzed for VOCs and total inorganics by U.S. EPA CLP or Region 5 ASB laboratory. The effluent samples will also be analyzed for dissolved inorganics and 1,4-dioxane by U.S. EPA CLP or Region 5 ASB laboratory.

The scope of the groundwater monitoring activities includes measuring groundwater levels in GMZ monitoring wells, collection of groundwater samples from 25 monitoring wells and 14 extractions wells quarterly for 5 years. Monitoring wells will be sampled for VOCs and extraction wells will be sampled for VOCs and inorganics to be analyzed by CLP or ASB. The monitoring wells will also be sampled for 1,4-dioxane to be analyzed by CLP or the ASB laboratory.

Additional information can be found on Worksheets #21, #17, #18 and in the Sampling and Analysis Plan (SAP) in **Appendix A**. If VOC, 1,4-dioxane and/or inorganic samples are scheduled to be analyzed by the Region 5 ASB laboratory relevant requirements and criteria are provided in **Appendix B**. The most current details for analytical scheduling including sample numbers and anticipated sample ship dates are provided to the RSCC for CLP samples and to the ASB laboratory using laboratory requests prior to each sampling event.

Analysis Tasks:

Groundwater samples from monitoring and extraction wells will be analyzed for VOCs by CLP (21-day turnaround time) using CLP SOW SFAM01.1 or equivalent or by U.S. EPA Region 5 ASB laboratory using SOP MS023 VOA in Water, Version 7. Monitoring wells will also be analyzed for 1,4-dioxane by CLP using SOW SFAM01.1 for SVOA modified by Method 3054.0 (21-day turnaround) or by ASB using method SOP MS035, Version 2 (45-day turnaround time). Data from these samples will be compared to project action limits as defined in the ROD; for 1,4-dioxane, the groundwater remediation goal is the groundwater quality standard for class I potable groundwater listed in Illinois Administrative code (IAC) 35 620.410. The project PALs and required reporting limits for groundwater are provided on Worksheet #15a in QAPP. Groundwater from wells will be field screened with a PID to determine samples that may be medium concentration.

Groundwater samples from extraction wells will also be analyzed for inorganics by CLP (21-day turnaround time) using SOW SFAM01.1 or equivalent or by the U.S. EPA Region 5 ASB laboratory using SOP Metals003A ICP Metals – Thermo, Version 8. Data from these samples will be reviewed to determine if concentrations of inorganics may interfere with operation of the treatment system.

Influent and effluent samples from the LTS will be analyzed for VOCs by CLP (21-day turnaround time) using CLP SOW SFAM01.1 (or equivalent) or by ASB using SOP MS023 VOA in Water (45-day turnaround time) and for total inorganics (21-day turnaround time) using CLP SOW SFAM01.1 (or equivalent) or the ASB laboratory using SOP Metals003A ICP Metals – Thermo (45-day turnaround time). Effluent samples will also be analyzed for dissolved inorganics (21-day turnaround time) using CLP SOW SFAM01.1 (or equivalent) or the ASB laboratory using SOP Metals003A ICP Metals – Thermo (45-day turnaround time) and 1,4-dioxane by CLP using SOW SFAM01.1 for SVOA modified by Method 3054.0 or by ASB using method SOP MS035 (45-day turnaround time). Data from the influent and effluent samples will be used to calculate LTS efficiency and mass removal rates. Data from the effluent samples will be compared to the discharge limits (see Worksheet #15b). Due to the differences in the list of parameters analyzed for VOCs by CLP versus ASB, eight VOC parameters with discharge limits that are analyzed by ASB laboratory are not analyzed by CLP. These parameters will be compared to the discharge limits for samples analyzed for VOCs by ASB and are noted on Worksheet 15b. Additional information is provided in Worksheets #23 and 24.

For samples scheduled to be analyzed by the U.S. EPA Region 5 ASB Laboratory relevant QAPP requirements are provided in **Appendix B**.

Quality Control Tasks:

1. Collect field duplicate samples for groundwater at a rate of one per 10 or fewer investigative samples.
2. Collect one duplicate effluent sample for the first sampling event each year. If upon data review the sample and duplicate agreement is not within the RPD provided in the QAPP, field procedures will be reviewed.
3. Collect extra volume for matrix spike, laboratory duplicate, and matrix spike/matrix spike duplicate at a rate of one per 20 or fewer samples.
4. Ship one VOC trip blank per cooler for all VOCs to CLP or ASB and 1,4-dioxane samples to ASB only.
5. Collect equipment blanks for monitoring wells only at a rate of one per 10 or fewer investigative samples.

6. Collect samples in compliance with the SAP, applicable SOPs in **Appendix A** and the *Contract Laboratory Program Guidance for Field Samplers, November 2020:* https://www.epa.gov/clp/contract-laboratory-program-guidance-field-samplers .
7. Check all COC forms against the sample bottle labels and ensure all listed sample bottles are contained in the cooler before sealing cooler for shipping.
8. Follow laboratory QC procedures using QC samples described in Worksheets #12a, #12b, 12c, #28a, #28b and #28c and methods.
9. Perform compliance check of all data received from the CLP/ASB laboratories.

For samples scheduled to be analyzed by the U.S. EPA Region 5 ASB Laboratory relevant QAPP information is provided in **Appendix B**.

Secondary Data:

Secondary data listed in Worksheet #13 were reviewed and used to help plan sample locations. Past data collection and analytical methodology are consistent with those listed in this QAPP.

Data Management Tasks:

Sample results and QC data from CLP will be delivered to CDM Smith as an electronic Level IV data deliverable in addition to hard-copied data package as per CLP SOW requirements. For samples analyzed by U.S. EPA Region 5 ASB, a level IV data package will be requested and delivered electronically. Electronic copies of all project deliverables, including graphics, are maintained by project number. Electronic files are routinely backed up and archived. The final project documents will be submitted to Illinois EPA, in hard copy and on CD ROM or other electronic means if requested.

Documentation and Records:

Information regarding sample collection will be recorded in field logbooks. Any changes made to the field logs will be initialed and dated. COCs, air bills, and sample logs will be prepared and retained for each sample. Sample documents for samples sent to the CLP will be created and tracked using the Scribe software tool. U.S. EPA CLP data will be provided as per CLP SOWs after Stage 2B validation by U.S. EPA Region 5 ESAT data validation staff within three weeks of receipt from CLP. A copy of all finalized documents and analytical data will be retained in the CDM Smith Project files. U.S. EPA Region 5 ASB data will be provided electronically within 45 days of sample receipt

prior to Stage 2B data validation by CDM Smith. CDM Smith will complete a Stage 2B validation for each data package within 30 days of receipt of the data package.

Field Change Notification (FCN) Forms:

If anticipated conditions are different from those encountered once the field work is under way, it may be necessary to implement a deviation from the approved QAPP. When such changes are required, the proposed change will be documented on a FCN Form by the CDM Smith FM and approved by CDM Smith's PM and the Illinois EPA PM. A copy of the FCN will be kept on site along with the approved QAPP. A copy of the FCN form will be distributed to the authorizing parties, the field staff, and the CDM Smith QC Manager in order to keep all staff informed of the change and to allow QA oversight of any changes. Electronic copies when available will be distributed immediately upon completion.

When significant field changes occur, the QAPP will be revised. Modifications will be carried out via revised pages to the QAPP. Minor changes will be made through formal memoranda from the CDM Smith PM to the Illinois EPA PM and will be included as addenda to the QAPP. The complete sign-off procedure will be followed if, in the judgment of the CDM Smith PM, major revisions to the QAPP are required. All revisions to the QAPP will be subject to CDM Smith's internal review process. All such changes will be approved by Illinois EPA prior to their implementation. Changes due to field conditions that necessitate a change or addenda to the QAPP will be submitted to U.S EPA and subject to the formal review and approval process.

Assessment/Audit Tasks:

Assessment of field activities will be carried out by the PM through frequent contact with the FM including daily updates on field work progress and review of the quarterly Field Progress Report. For each sampling event the PM will contact the FM daily by phone or email and will visit the site at least one time per event to ensure compliance with QAPP/SAP. No audits are planned by CDM Smith QA staff for these activities. Additional information is provided on Worksheets # 31, #32, and #33.

Data Review Tasks:

Laboratories will review all analytical data for completeness and quality. CLP data will be validated within three weeks of data package receipt by U.S. EPA ESAT data validation staff using the Stage 2B validation option and following the most recent National Functional Guidelines, Region 5 CLP validation SOPs and this QAPP. Data generated by U.S. EPA Region 5 ASB laboratory will be validated by CDM Smith following the most recent National Functional Guidelines, U.S. EPA Region 5 ASB laboratory analytical SOPs provided in **Appendix**

B and this QAPP. CDM Smith QC Manager (or designee) will also perform a compliance check of all data received from the laboratories and perform an assessment of data usability based on QAPP criteria. The Stage 2B validation option for VOC, inorganic and 1,4-dioxane data was selected for this project due to the fact that a significant quantity of historical data is available for Area 7 and the contaminant profiles are well documented. Additionally, all data packages submitted from the laboratories will be Level IV packages so if the Stage 2B validation indicates quality issues then the validation level can be elevated as approved by Illinois EPA. The water level data will be validated by double checking field measurements prior to recording in log book and checking the current water level information against historical and re-measuring any significant outliers. Data will be distributed to Illinois EPA, CDM Smith and other data users. See Worksheets #34 through #37 for additional information.

Notes: ESAT – Environmental Services Assistance Team
CDM Smith – CDM Smith Inc.
COC – Chain of Custody
PM – Project Manager
FM – Field Manager
QA – Quality Assurance
CLP – Contract Laboratory Program
SOW – Statement of Work
SVOC – Semi-Volatile Organic Compounds
QAPP – Quality Assurance Project Plan
QC – Quality Control
Illinois EPA – Illinois Environmental Protection Agency
SOP – standard operating procedure
U.S. EPA – United States Environmental Protection Agency
TCL VOC – Target Compound List Volatile Organic Compounds
ASB – Analytical Services Branch
SIM – selected ion monitoring

Activities	Organization	Dates (Month Day, Year)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
QAPP preparation	CDM Smith	March 2019	July 31, 2019	QAPP	August 30, 2019
Annual QAPP Review Including SOPs	CDM Smith	July 31, 2020 and annually thereafter	July 31, 2020 and annually thereafter	Letter or revised QAPP as appropriate	July 31, 2020 and annually thereafter
QAPP Revision	CDM Smith	December 2020	April 15, 2021	QAPP Addendum	May 4, 2021
GMZ Application Preparation	CDM Smith	September 1, 2020	January 15, 2021	Approved GMZ Application	January 30, 2021
Effluent monthly/quarterly sampling/analysis	CDM Smith CLP/ ASB	December 1, 2019	December 30, 2024	Analytical Data Packages, VOC, Inorganics and 1,4-dioxane	Validated analytical data due to CDM Smith 6 weeks from date of sampling Monthly/Quarterly for 5 years

Activities	Organization	Dates (Month Day, Year)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Quarterly Groundwater sampling/analysis	CDM Smith CLP/ ASB	December 1, 2019 Conducted quarterly for 5 years	December 30, 2024	Analytical Data Packages, VOC, Inorganics and 1,4-dioxane	Validated analytical data due to CDM Smith 6 weeks from date of sampling Quarterly for 5 years
Data validation/usability assessment	U.S. EPA ESAT Data Validation Staff CDM Smith data validation staff	21 days from date of sample collection for CLP 45 days from date of sample collection for ASB	Three weeks from date received from laboratory	Data validation report Data Usability Technical Memorandum	Quarterly for 5 years
Prepare GMZ Monitoring Report	CDM Smith	March 2021	April 2021	Annual GMZ Monitoring Report	Annually for 5 years
Address comments on Draft GMZ Monitoring Report	CDM Smith	March 2021 Prepared annually for 5 years	April 2026	Annual GMZ Monitoring Report Final	Annually for 5 years

Notes:

U.S. EPA – United States Environmental Protection Agency
CDM Smith – CDM Smith Inc.
GMZ – Groundwater Management Zone
ESAT – Environmental Services Assistance Team
CLP – Contract Laboratory Program
QAPP – Quality Assurance Project Plan
ASB – Analytical Services Branch

QAPP Worksheet #15a: Groundwater Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix	Groundwater			
Analytical Group	VOC			
Concentration Level	Trace/low/medium			
Analyte[3]	CAS Number	Project Action Limit[2] µg/L	CLP Method[1]/ASB Method[4] CRQLs/RL (low/medium) µg/L	CLP Method[1]/ ASB Method[4] MDLs µg/L
Vinyl chloride	75-01-4	2	0.50/2	NA/0.354
1,1-Dichloroethene	75-35-4	7	0.50/2	NA/0.676
1,2-Dichloroethane	107-06-2	5	0.50/2	NA/0.378
Cis-1,2-dichloroethene	156-59-2	70	0.50/2	NA/0.426
Trans-1,2-dichloroethene	10061-01-5	100	0.50/2	NA/0.476
Carbon Tetrachloride	56-23-5	5	0.50/2	NA/0.790
Ethylbenzene	100-41-4	700	0.50/2	NA/0.379
1,1,1-Trichloroethane	71-55-6	200	0.50/2	NA/0.706
1,1,2-Trichloroethane	79-00-5	5	0.50/2	NA/0.357
Trichloroethene	79-01-6	5	0.50/2	NA/0.470
Tetrachloroethene	127-18-4	5	0.50/2	NA/0.491
Toluene	108-88-3	1,000	0.50/2	NA/0.350
o-Xylene	95-47-6	10,000 (total)	0.50/2	NA/0.380

Matrix	Groundwater			
Analytical Group	VOC			
Concentration Level	Trace/low/medium			
Analyte[3]	CAS Number	Project Action Limit[2] µg/L	CLP Method[1]/ASB Method[4] CRQLs/RL (low/medium) µg/L	CLP Method[1]/ ASB Method[4] MDLs µg/L
m,p-Xylene	179601-23-1	10,000 (total)	0.50/4	NA/0.562
1,4-dioxane[4]	123-91-1	7.7	0.4/0.20	NA/0.15

Notes: For samples sent to the Analytical Services Branch laboratory see Appendix B for additional information

[1]CLP Method Analytical CRQLs are those documented in SOW SFAM01.1. EPA headquarters and SMO maintain laboratory MDLs which are not available to include above.

[2]The Project Action Limit is set at the Remediation Goal established in the ROD. There are no groundwater Project Action Limits for inorganics therefore they are not included in WS #15a.

[3]All groundwater samples will be analyzed for the full CLP TCL SOW/ASB volatile organic list of parameters. This table lists only those organic parameters with a PAL.

[4]1,4-dioxane will be analyzed by Region 5 ASB laboratory using SOP MS035, Version 2 or by CLP using SOW SFAM01.1 for SVOA modified by Method 3054.0. The PAL is the groundwater quality standard for class I potable groundwater listed in Illinois Administrative code (IAC) 35 620.410.

CAS – Chemical Abstract Service
MDL – method detection limit
CLP – Contract Laboratory Program
µg/L – microgram per liter
QAPP – Quality assurance project plan
CRQL – contract required quantitation limit
ASB – Analytical Services Branch
RL – Reporting Limit
VOC – Volatile Organic Compound
SVOA – Semi-Volatile Organic Analyte
NA – Not Available

QAPP Worksheet #15b: Effluent Project Action Limits and Laboratory-Specific Detection/Quantitation Limits

Matrix	Treated Effluent			
Analytical Group	VOC/1,4-dioxane[5]/Inorganics [3]			
Concentration Level	Trace			
Analyte[4]	CAS Number	Project Action Limit[2] µg/L	CLP Method[1]/ASB Method[5] CRQLs (low/medium)/RL[5] µg/L	CLP Method[1]/ASB Method[5] MDLs µg/L
Vinyl chloride	75-01-4	1700	0.50/2	NA/0.354
1,1-Dichloroethene	75-35-4	240	0.50/2	NA/0.676
1,1-Dichloroethane	75-34-3	2,000	0.50/2	NA/0.311
Cis-1,2-dichloroethene	156-59-2	1,100	0.50/2	NA/0.462
Trans-1,2-dichloroethene	10061-01-5	34,000	0.50/2	NA/0.476
Carbon Tetrachloride	56-23-5	280	0.50/2	NA/0.790
1,1,1-Trichloroethane	71-55-6	390	0.50/2	NA/0.706
1,1,2-Trichloroethane	79-00-5	4,400	0.50/2	NA/0.357
Trichloroethene	79-01-6	940	0.50/2	NA/0.470
Tetrachloroethene	127-18-4	150	0.50/2	NA/0.491
Chloroethane	75-00-3	1,000	0.50/2	NA/0.658
Benzene	71-43-2	310	0.50/2	NA/0.298
Ethylbenzene	100-41-4	14	0.50/2	NA/0.379
Toluene	108-88-3	600	0.50/2	NA/0.350
o-Xylene	95-47-6	360 (total)[4]	0.50/2	NA/0.380
m,p-Xylene	179601-23-1	360 (total)[4]	0.50/4	NA/0.562

Matrix	Treated Effluent			
Analytical Group	VOC/1,4-dioxane[5]/Inorganics [3]			
Concentration Level	Trace			
Analyte[4]	CAS Number	Project Action Limit[2] µg/L	CLP Method[1]/ASB Method[5] CRQLs (low/medium)/RL[5] µg/L	CLP Method[1]/ASB Method[5] MDLs µg/L
1,2-dichlorobenzene	95-50-1	170	0.50/2	NA/0.392
1,2-dichloroethane	107-06-2	4,500	0.50/2	NA/0.378
4-methyl-2-pentanone	108-10-1	1,400	5.0/5	NA/1.096
Styrene	100-42-5	200	0.50/2	NA/0.288
n-butylbenzene[6]	104-51-8	3.1	NA/2	NA/0.349
sec-butylbenzene[6]	135-98-8	6.6	NA/2	NA/0.373
p-isopropyltoluene[6]	99-87-6	6.8	NA/2	NA/0.369
naphthalene[6]	91-20-3	68	NA/2	NA/0.506
n-propylbenzene[6]	103-65-1	10	NA/2	NA/0.343
1,2,4-trimethylbenzene[6]	95-63-6	29	NA/2	NA/0.359
1,3,5-trimethylbenzene[6]	108-67-8	11	NA/2	NA/0.247
1,4-Dioxane[5]	123-91-1	36,000	0.4/0.205	NA/0.155
Dissolved Iron	7439-89-6	1,000	100/80	NA/21

Notes: For samples sent to the Analytical Services Branch laboratory see Appendix B for additional information

[1]CLP Method Analytical CRQLs are those documented in CLP SOW SFAM01.1. EPA headquarters and SMO maintain laboratory MDLs which are not available to include above.

[2]The Project Action Limit is set at the Discharge Limits provided in the ROD, the standards provided in 35 IAC 302.208 or Illinois EPA-Derived Water Quality Criteria, April 04, 2013.

. The PAL for system efficiency/mass removal rate is 99% calculated by influent minus effluent divided by influent and discussed on WS # 11, Step 7.

[3]All influent and effluent samples will be analyzed for the full CLP TCL/ASB volatile organic list and full CLP TAL/ASB inorganic list of parameters as per the SOWs. This table lists only those organic and inorganic parameters with a PAL.

[4]The effluent discharge limit for total xylenes is 360 µg/L.

[5]1,4-dioxane will be analyzed by Region 5 ASB laboratory using SOP MS035, Version 2 or by CLP using SOW SFAM01.1 for SVOA modified by Method 3054.0.

[6]Analyte is not included in the CLP SOW SFAM01.1 VOC analyte list. Data for these analytes will be compared to PALs only when reported by Region 5 ASB laboratory.

CAS – Chemical Abstract Service
µg/L – microgram per liter
ASB – Analytical Services Branch
MDL – method detection limit
QAPP – Quality assurance project plan
RL – Reporting Limit
CLP – Contract Laboratory Program
CRQL – contract required quantitation limit
NA – not available

QAPP Worksheet #17: Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach:

The goals of GMZ sampling activities are to comply with Area 7 GMZ monitoring requirements and to monitor progress and effectiveness of the contaminant extraction and treatment system. The sampling process was designed to address these goals.

Influent and effluent from the LTS will be sampled monthly until the LTS is declared operational and functional and quarterly thereafter for the duration of operation. The influent sample will be collected from the sampling port after the equalization tank where the incoming liquid streams are mixed prior to treatment. The effluent sample will be collected from the effluent sample port located just prior to the LTS effluent discharge point. The effluent sample for dissolved inorganics will be collected through a filter attached to sample tubing which will be attached to the sample port. The sampling frequency was designed to evaluate LTS performance more frequently during system startup and provide confirmation that the effluent discharged to the intermittent creek meets the discharge limits once the system is fully operational.

Area 7 GMZ monitoring wells and extraction wells (**Figures 2 and 3**) will be sampled quarterly for a period of five years. The sampling frequency will allow for any necessary adjustments to the GMZ configuration or to the contaminant extraction and treatment system to be accomplished in a timely manner.

Worksheet #18 provides the number of samples, matrices and analytical parameters.

Describe the sampling design and rationale in terms of what matrices will be sampled, what analytical groups will be analyzed and at what concentration levels, the sampling locations (including QC, critical, and background samples), the number of samples to be taken, and the sampling frequency:

Groundwater samples collected during Area 7 GMZ monitoring activities include quarterly rounds of sampling for a period of five years from 25 Area 7 GMZ monitoring wells and 14 hydraulic containment extraction wells. Monitoring wells include those in the approved GMZ application for Area 7, and extraction wells include those located in a north/south line along the border of the contaminant plume designed to control migration of leachate in a downgradient direction. Samples will be collected from monitoring wells with a low flow pump. Extraction well samples will be collected directly from the tap on waterlines that run from extraction wells to the treatment system. Some samples are expected to be at slightly higher concentrations initially during monitoring activities and the remaining samples are expected to be trace level.

If a monitoring well should become inaccessible for any sampling event, the situation will be assessed and if access can be restored quickly the well will be sampled for that event. If a well is

damaged or destroyed it will not be sampled for that particular event and will be repaired or replaced as necessary. Due to the frequency of sampling (quarterly) and the extended time frame over which sampling has been and will be conducted (five years minimum), this situation would not create a significant data gap. More detailed information regarding corrective actions to be taken for problems and issues is provided on Worksheet #31.

All groundwater samples will be analyzed for TCL VOCs by CLP or for VOCs by U.S. EPA Region 5 ASB Laboratory. The samples from the monitoring wells will also be analyzed for 1,4- dioxane by CLP or U.S. EPA Region 5 ASB laboratory. Data from these samples will be compared to PALs on Worksheet #15a. All groundwater PALs are above CLP SOW SFAM01.1 CRQLs and modified Method 3054.0 by CLP and the ASB RLs for VOCs and 1,4-dioxane. It is estimated that 500 investigative groundwater samples will be collected for VOCs and 1,4-dioxane from monitoring wells over the 5- year monitoring period. Approximately 280 investigative groundwater samples from the extraction wells will also be collected for VOCs and inorganics over the 5-year monitoring period. Equipment blanks and duplicates will be collected at a frequency of one per 10 investigative samples. Equipment blanks will be collected for monitoring wells only as it is not possible to run blank water through the taps for the extraction wells. Trip blanks will be included with each cooler shipped for VOC analysis and for 1,4-dioxane analyzed by the ASB laboratory. Laboratory duplicates, MS and MS/MSDs will be collected at a frequency of one per 20 investigative samples as per the EPA *Contract Laboratory Program Guidance for Field Samplers,* November 2020. For samples scheduled to be analyzed by the U.S. EPA Region 5 ASB laboratory, relevant information is provided in **Appendix B**.

Influent and effluent from the LTS will be sampled monthly until the LTS is declared operational and functional, and quarterly thereafter for the duration of LTS operation. The influent sample will be collected from the sampling port after the equalization tank where incoming liquid streams are mixed prior to treatment. The effluent sample will be collected from the effluent sample port located just prior to the LTS effluent discharge point. The effluent sample for dissolved inorganics will be collected through a filter attached to sample tubing which will be attached to the sample port. Influent and effluent samples will be analyzed for VOCs and total inorganics by U.S. EPA CLP or U.S. EPA Region 5 ASB laboratory. Effluent will also be analyzed for dissolved inorganics and 1,4- dioxane by CLP or U.S. EPA Region 5 ASB laboratory. The results will be compared to discharge limits provided on Worksheet 15b. The list of parameters with discharge limits was revised in December 2020 and approved by Illinois EPA to include parameters that have been detected in influent samples since LTS system start-up. The PALS were revised to reflect the most current surface water criteria. There are eight VOC parameters that are analyzed by ASB laboratory but not by CLP. These parameters will be compared to PALS for all samples analyzed for VOCs by ASB and are noted on Worksheet 15b. All effluent PALs are above CLP SOW SFAM01.1 CRQLs for VOCs and modified Method 3054.0 for 1,4-dioxane, the CLP SOW SFAM01.1 CRQLs and ASB RLs for VOCs, 1,4-dioxane and inorganics. For samples scheduled to be analyzed by the U.S. EPA Region 5 ASB laboratory, relevant information is provided in **Appendix B**.

Flow from influent and effluent sample ports will be kept as low as possible during sample collection. Influent samples are expected to be medium concentration as they are collected prior to treatment. Effluent samples are expected to be trace concentration and consistent in contaminant levels over the duration of sampling as the sample is collected at the end of the leachate treatment process prior to discharge. It is assumed that LTS startup will last 9 months and the overall system operation will continue for 5 years for a total of 29 influent and effluent samples. One field duplicate will be collected for effluent during the first sampling event each year and evaluated for acceptable relative percent difference (RPD). If sample and field duplicate agreement is outside the RPD defined in this QAPP, field procedures will be reviewed. Trip blanks will be included with each cooler shipped for VOC analysis and for 1,4-dioxane analyzed by the ASB laboratory. Equipment blanks will not be collected for the influent and effluent samples as it is not possible to run blank water through the sample ports. Laboratory duplicates, MS and MS/MSDs will be collected at a frequency of one per 20 investigative groundwater samples as per the *EPA Contract Laboratory Program Guidance for Field Samplers*. For influent and effluent sampling, the MS, laboratory duplicate, and MS/MSD QC samples will be associated with influent samples and collected for each event as these samples will contain contaminants of interest. For samples scheduled to be analyzed by the U.S. EPA Region 5 ASB laboratory, relevant information is provided in **Appendix B**.

Additional information regarding sample locations, collection and rationale is provided in the SAP which is **Appendix A** of this QAPP.

QAPP Worksheet #18: Sampling Locations and Methods

Sampling Location/ ID Number Well locations[3] on Figures 2 and 3	Matrix	Depth	Analytical Group	Concentration Level	Number of Samples (identify field[1] duplicates)	Sampling SOP2 Reference	Rationale for Sampling Location
Groundwater samples from monitoring wells: A7-MW106A-yymmdd	Water	Indicated in Well ID[3]	VOC; 1,4-dioxane	trace; low	Maximum 500 samples plus 50 field duplicates	CDM Smith SAP sections 3.1 through 3.6, 5, 6 and 7 and SOPs 1-6, 1-10, 1- 12, and 4-3.	Samples from monitoring wells as required by the approved GMZ application for Area 7.
Groundwater samples from extraction wells: A7-EWG003S-yymmdd	Water	Indicated in Well ID[3]	VOC; Inorganics	trace; medium/low	Maximum 280 plus 28 field duplicates	CDM Smith SAP sections 3.1 through 3.6, 5, 6 and 7 and SOP 1-9 & -10. Samples collected directly from waterline tap.	Samples from extraction wells to monitor and optimize the LTS.
Influent sample from LTS influent line: A7-IN-mmddyy; Effluent sample from LTS discharge line: A7-EFF-yymmdd	Water	NA	VOC; total Inorganics; 1,4-dioxane and dissolved inorganics for effluent only	Medium/trace; low; low	Estimated 29 plus 6 field duplicates	CDM Smith SAP sections 3.1 through 3.6, 5, 6 and 7 and SOP 1-9 & -10. Samples collected directly from in-line sampling port -dissolved inorganics field filtered	Influent and effluent samples to monitor and optimize the LTS. Effluent to confirm compliance with discharge limits.

Notes:

[1]MS/MSD, MS and laboratory duplicate samples will be collected at a frequency of 1 for every 20 samples. MS/MSD, MS and laboratory duplicates will be collected with extra volume as directed by the CLP Program Guidance for Field Samplers.

Field duplicates collected for every 10 groundwater samples. One field duplicate of effluent will be collected for the first sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD) defined in the QAPP, field procedures will be reviewed.

[2]Specify the appropriate letter or number from the Project Sampling SOP References table (Worksheet #21).

[3]See Table 4-1 in Appendix A for a list of monitoring and extraction wells with screened intervals.

ID – Identification
SAP – sampling and analysis plan
SOP – standard operating procedure
QAPP – quality assurance project plan
PID – photoionization detector

QAPP Worksheet #19 & 30: Sample Containers, Preservation, and Hold Times

Analyte/ Analyte Group	Matrix	Concentration Level	Method/SOP	Container(s) [1] (number, size & type per sample)	Preservation	Maximum Holding Time (preparation and analysis)	Data Package Turnaround
Volatile Organic Compounds	Water	Trace/Low /Medium	CLP ; SOW SFAM01.1 ASB SOP MS023	Four to CLP; 3 to ASB 40-ml amber glass vials with Teflon septum	Preserve with HCl to pH of 2 cool to 0 to 6 °C DO NOT FREEZE	14 days	CLP - 21 days; ASB – 45 days
Total and dissolved Inorganic Compounds	Water	Low/medium	CLP SOW SFAM01.1; ASB SOP Metals 003A-ICP	One 0.5-liter HDPE cylinder round bottle	Preserve to pH <2 with HNO_3 cool to 0 to 6 °C DO NOT FREEZE	6 months	CLP - 21 days; ASB – 45 days
1,4-Dioxane	Water	Low	CLP SOW SFAM01.1 for SVOA modified by Method 3054.0	CLP -Two 1-liter amber glass bottles;	CLP -No preservative; Cool to 0 to 6 ºC; DO NOT FREEZE	7 days to extraction; 40 days from extraction	CLP - 21 days; ASB – 45 days
			ASB SOP MS035	ASB -Two 250-ml amber glass bottles	ASB - Preserved with Na_2SO4 to pH <2 Cool to 0 to 6 ºC DO NOT FREEZE	28 days post extraction; if pH measured at lab is >2 holding time is reduced to 7 days	

Notes:

[1] Double volume is needed for aqueous inorganic MS and laboratory duplicates. Double volume is needed for aqueous CLP/ASB volatile organic MS/MSD samples and 1,4-dioxane to ASB. For 1,4-dioxane CLP MS/MSD analysis, five 1 – liter amber bottles are required.

ºC – Degrees Celsius
ml – milliliter
CLP SOW – Contract Laboratory Program Statement of Work
SOP – standard operating procedure
HDPE – high density polyethylene
L – liter
HCl – hydrochloric acid
Na_2SO4 – sodium bisulfate
HNO3 – Nitric acid
ASB – Analytical Services Branch
SVOA – semi-volatile organic analysis
SIM – selected ion monitoring
ICP – inductively coupled plasma
CRQL – Contract Required Quantitation

QAPP Worksheet #20: Field QC Summary

Matrix	Analytical Group	Concentration Level	Analytical and Preparation SOP Reference	No. of Sampling Locations[1]	No. of Field Duplicates[2,4]	MS/MSD, MS, laboratory duplicate[3] / No. of samples needing extra volume	No. of Equipment Blanks[5]	No. of Trip Blanks[6]	Total No. of Samples to Lab[2,4]
Groundwater	VOC	Trace/Low/ Medium	CLP SOW SFAM01.1; ASB SOP MS023 VOA	39	4	2	3	4	50
Groundwater	Inorganics	Low	CLP SOW SFAM01.1; ASB SOP Metals ICP	14	2	1	0	0	16
Groundwater	1,4-Dioxane	Low	CLP SOW SFAM01.1 SVOA modified by Method 3054.0; ASB SOP MS035	25	3	2	3	CLP – 0 ASB - 3	CLP – 31 ASB - 34
Influent; Effluent	VOC	Medium; Trace	CLP SOW SFA01. ; ASB MS023 VOA	2	1	1	0	1	4
Effluent	1,4-Dioxane	Low	CLP SOW SFAM01.1 SVOA modified by Method 3054.0; ASM SOP MS035	1	1	1	0	CLP – 0 ASB - 1	CLP – 2 ASB - 3
Effluent	Dissolved Inorganics	Medium; Low	CLP SOW SFAM01.1; ASB SOP Metals ICP	2	1	1	0	0	3
Influent; Effluent	Total Inorganics	Medium; Low	CLP SOW SFAM01.1; ASB SOP Metals ICP	2	1	1	0	0	3

Notes: If samples are sent to the Analytical Services Branch laboratory see Appendix B for laboratory SOPs

[1]This is the number of locations per sampling event. 25 monitoring wells and 14 extraction wells. 20 total quarterly groundwater sampling events are anticipated. 9 monthly and 20 quarterly effluent sampling events are anticipated.

[2]This is the total number of samples per groundwater sampling event. 20 total quarterly events are anticipated.

[3] Double volume is needed for aqueous CLP volatile organic and ASB 1,4-dioxane MS/MSD samples. For CLP 1,4-dioxane MS/MSD analysis, five 1 – liter amber bottles are required. Double sample volume will be collected for Inorganic MS and Inorganic laboratory duplicate.

[4]One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed.

[5] Equipment blanks will be collected for monitoring well samples only as it is not possible to run blank water through the sampling taps and ports for the extraction wells and influent and effluent sample points.

[6] One volatile organic trip blank will be shipped for analysis with each cooler of volatile organic and for 1,4-dioxane samples to ASB only. **No trip blank is required for 1,4-dioxane analyzed by CLP**

CLP SOW – Contract Laboratory Program Statement of Work

Laboratory Duplicate - this extra volume for inorganic samples does not count as another sample and is not included in the total number of samples.

MS – Matrix Spike – this extra volume for inorganic samples does not count as another sample and is not included in the total number of samples.

MS/MSD – Matrix spike/matrix spike duplicate – this extra sample volume for organic analysis does not count as another sample and is not included in the total number of samples.

QAPP – quality assurance project plan

SOP – standard operating procedure

SIM – selected ion monitoring

VOC – volatile organic compound

ASB – Analytical Services Branch

SVOA – semi-volatile organic analyte

QAPP Worksheet #21: Field SOPs

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)[1]	Comments
1-2	Sample Custody, Rev. 9, August 2020	CDM Smith	NA	Y	Scribe software will be used for CLP sample forms and customized labels. No sample tags will be used. ASB COCs will be used for samples going to ASB. – Use waterproof ink for sample bottle labels.
1-6	Groundwater Level Measurement, Rev. 10S, August 2020	CDM Smith	Water level indicator Electrical Tape	N	
1-12	Low Stress (Low Flow) Groundwater Sampling, Rev. 3, August 2020	CDM Smith	Grundfos pump, or equivalent, or peristaltic pump, in-line water quality meter	Y	
1-9	Tap Water Sampling, Rev. 8, August 2020	CDM Smith	NA	N	Applies to extraction well and influent/effluent tap sampling.
1-10	Field Measurement of Organic Vapors, Rev. 8, February 2020	CDM Smith	Multi-RAE or equivalent PID	N	
2-1	Packaging and Shipping Environmental Samples, Rev. 7, August 2020	CDM Smith	NA	Y	Methanol will not be used. Vermiculite will not be used. Laboratory provides bubblewrap for packing material
2-2	Guide to Handling Investigation-Derived Waste, Rev. 9, August 2020	CDM Smith	NA	Y	Purged groundwater will be collected in drums and transported to Area 7 treatment unit for treatment and disposal.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)[1]	Comments
4-1	Field Logbook Content and Control, Rev. 9, August 2020	CDM Smith	NA	Y	Logbook notes should include decontamination procedures used, descriptions of photos taken, problems encountered and notes of conversations with PM/ treatment plant staff. Details of samples collected including sample numbers and visual observations.
4-2	Photographic Documentation of Field Activities, Rev. 10, August 2020	CDM Smith	Digital Camera	N	
4-3	Well Development and Purging, Rev. 8, August 2020	CDM Smith	Pump, disposable tubing, electronic water-level meter, in-line water quality meter, turbidity meter	Y	No well development will be conducted.
4-5	Field Equipment Decontamination at Nonradioactive Sites, Rev.11, March 2020	CDM Smith	Pump, in-line meter	Y	An Alconox solution will be used to decontaminate field equipment
5-1	Control of Measurement and Test Equipment, Rev. 11, August 2020	CDM Smith	NA	Y	Leased or rented equipment only to be used.

[1]For SOPs modified for project (Y) please also see the Sampling and Analysis Plan in Appendix A

Notes: All SOPs are reviewed annually. No updates are necessary based on most recent review. Any updates required based on future reviews will be provided in an amended WS# 21.

Corrective action for problems and issues are discussed in Worksheet #31.

The activities for each sampling event will require a support vehicle that will store all equipment and supplies necessary for each sampling event.

NA – Not applicable

QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Multi-RAE plus PID Toxic Gas Monitor - 11.7 eV lamp	Calibration and checked at the beginning and end of day	As needed in field; semi-annually by supplier	Measure known concentration of Isobutylene (100 ppm calibration gas)	Upon receipt, successful operation	Calibrate am, check pm	± 10% of the calibrated value	Manually zero meter or service as necessary and recalibrate	Field Manager	Manufacturers specifications; SOP 1-10
YSI-600XL Flow through cell	Calibrate beginning of day and check calibration at the end of the day	Performed before shipment as needed	Measure with known NIST traceable buffers and conductivity calibration Solutions Temperature does not require check or calibration	Upon receipt, successful operation	Daily before each use	pH ±0.05 specific conductivity ±5 µS DO ±0.02 ppm Temp. ±0.30 °C	Recalibrate or service as necessary	Field Manager	Manufacturers specifications; SOP 1-10
Water Level Meter	NA	None	Check daily before each use	Check instruments are working	Check daily before each use	Pass/fail	Return to rental company for replacement	Field Manager	Manufacturers specifications; SOP 1-10
LaMotte Turbidity Meter	Accuracy/ calibration check Recalibrate or service as necessary at the beginning and end of each day	Return for replacement	Measure with standard solution	Upon receipt, successful operation	Calibrate before use and whenever anomaly suspected	Pass/fail	Replace battery or bulb or return for replacement	Field Manager	Manufacturers specifications; SOP 1-10

Notes: Spare parts and certified consumables are provided by supplier with provided equipment. All equipment, spare parts, and consumables are located on site in the site support vehicle with each piece of equipment. If additional parts or consumables are needed, they will be shipped directly from the equipment supplier; however; due to short duration of each sampling event this is not anticipated. Acceptance criteria for consumables includes review of certifications and expiration dates for calibration materials. Critical supplies and consumables for the U.S. EPA Region 5 ASB and CLP laboratories including supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials are not available to CDM Smith. U.S. EPA maintains this information.

[1]Specify the appropriate reference letter or number from the project sampling SOP references table (Worksheet #21).

QAPP – Quality assurance project plan
NIST – National Institute for Standards and Technology
DO – dissolved oxygen
SOP – standard operating procedure
µS – micro siemens
NA – not applicable eV – electron volt
ppm – parts per million

QAPP Worksheet #23: Analytical SOPs

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
				Purge and trap		
EPA Method 524.2 EPA Method 5030C EPA Method 8260C	CLP SOW SFAM01.1	Definitive	TCL VOCs in water	Gas Chromatogram/ Mass Spectrophotometer (GC/MS)	U.S. EPA CLP Laboratory	N
EPA Method 8260C Modified	SOP MS023: VOA in Water Version 7	Definitive	VOCs in water	Gas Chromatogram/ Mass Spectrophotometer (GC/MS)	US EPA Region 5 ASB Laboratory	N
SW-846 8270D	CLP SOW SFAM01.1 for SVOA modified by Method 3054.0	Definitive	1,4-dioxane in water	Gas Chromatogram/ Mass Spectrophotometer (GC/MS) Selected Ion Monitoring	U.S. EPA CLP Laboratory	N
EPA Method 522 EPA SW-846 8000D	ASB SOP MS035, Version 2	Definitive	1,4-dioxane in water	Gas Chromatogram/ Mass Spectrophotometer (GC/MS) Selected Ion Monitoring	Region 5 ASB Laboratory	N
EPA Method 200.7 EPA SW-846 3050B EPA SW-846 6010D	CLP SOW SFAM01.1	Definitive	TAL Inorganics in water (total and dissolved)	Inductively Coupled Plasma Atomic Emission Spectrometry	U.S. EPA CLP Laboratory	N
EPA Method 200.7 EPA SW-846 6010D	SOP Metals003A ICP Metals – Thermo, Version 8	Definitive	Inorganics in water (total and dissolved)	Inductively Coupled Plasma / Optical Emission Spectrometry (ICP-OES)	U.S. EPA Region 5 ASB Laboratory	N

Notes: If samples are sent to the Analytical Services Branch laboratory see Appendix B for additional information

QAPP – quality assurance project plan
SOP – standard operating procedure
VOC – volatile organic compound
CLP SOW – Contract Laboratory Program Statement of Work
ASB – Analytical Services Branch
SW-846 – Test Methods for Evaluating Solid Waste
EPA – U.S. Environmental Protection Agency

QAPP Worksheet #24: Analytical Instrument Calibration

Instrument	Calibration Procedure (SOP Reference)	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1,2]
GC/MS	Section 9.3- Initial Calibration Section 9.4-Initial Calibration Verification Section 9.5- Continuing Calibration Verification	9.3.2-Initial calibration 9.4.2-Initial Calibration Verification 9.5.2-Continuing Calibration	9.3.5-Initial Calibration 9.4.5-Initial Calibration Verification 9.5.5-Continuing Calibration	9.3.6-Initial Calibration 9.4.6-Initial Calibration Verification 9.5.6-Continuing Calibration	Laboratory Analyst	CLP SOW SFAM01.1 Exhibit D Trace Concentration of Volatile Organic Compounds Analysis Section 9
GC/MS	Section 9.4.1-Initial Calibration Section 9.4.2 -Continuing Calibration Verification	9.3.1.1-Initial calibration 9.4.2.1-Continuing Calibration	9.4.1.2-Initial Calibration 9.4.2.1.-Continuing Calibration	9.4.1.4-Initial Calibration 9.4.2.2-Continuing Calibration	Laboratory Analyst	U.S. EPA Region 5 ASB Laboratory SOP MS023 VOA in Water Version 7 Section 9.4
GC/MS SIM	Section II of Modified Method 3054.0 and as applicable; Section 9.3- Initial Calibration Section 9.4-Initial Calibration Verification Section 9.5- Continuing Calibration Verification	Section II of Modified Method 3054.0 and as applicable; 9.3.2-Initial calibration 9.4.2-Initial Calibration Verification 9.5.2-Continuing Calibration	Section II of Modified Method 3054.0 and as applicable; 9.3.5-Initial Calibration 9.4.5-Initial Calibration Verification 9.5.5-Continuing Calibration	Section II of Modified Method 3054.0 and as applicable; 9.3.6-Initial Calibration 9.4.6-Initial Calibration Verification 9.5.6-Continuing Calibration	Laboratory Analyst	CLP SOW SFAM01.1 for SVOA modified by Method 3054.0 Section II and CLP SOW SFAM01.1 Exhibit D SVOA Section 9
GC/MS	Section 9.4- Initial Calibration Section 9.4- Initial Calibration Verification Section 9.5- Continuing Calibration Verification	9.4.4-Initial calibration 9.4.6-Initial Calibration Verification 9.4.7-Continuing Calibration	10.7-Initial Calibration 10.7-Initial Calibration Verification 10.8-Continuing Calibration	9.4.5.1-Initial Calibration 9.4.6-Initial Calibration Verification 9.5.6-Continuing Calibration	Laboratory Analyst	Region 5 ASB Laboratory SOP MS035, Version 2 Low-Level 1,4- Dioxane in Water Sections 9 and 10

Instrument	Calibration Procedure (SOP Reference)	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference[1,2]
ICP-AES	Section 9.3-Instrument Calibration Section 9.4-Initial Calibration Section 9.5-Continuing Calibration	9.3.2-Instrument Calibration 9.4.2-Initial Calibration 9.5.2-Continuing Calibration	9.3.5-Instrument Calibration 9.4.5-Initial Calibration 9.5.5-Continuing Calibration	9.3.6-Instrument Calibration 9.4.6-Initial Calibration 9.5.6-Continuing Calibration	Laboratory Analyst	CLP SOW SFAM01.1 –Exhibit D Analytical Methods for Inductively Coupled Plasma-Atomic Emission Spectrometry Section 9
ICP-OES	Section 9.2.2.4 – Analytical Sequence	10.1.3-Instrument Calibration	10.2.1 – Instrument Audit Checks	10.2.2 -Instrument Audit Checks	Laboratory Analyst	SOP Metals003A ICP Metals – Thermo Sections 9 and 10

Notes: For samples sent to the Analytical Services Branch laboratory see Appendix B for additional information

[1]For the CLP laboratory all instrument calibration information is provided in the U.S. EPA CLP SOW SFAM01.1 for organics, inorganics, and SVOA modified by Method 3054.0 for 1,4-dioxane.

[2]For the U.S. EPA Region 5 ASB laboratory all instrument calibration is provided in ASB SOP MS035, Version 2; Low-Level 1,4-Dioxane in Water, SOP MS023 VOA in Water Version 7 and SOP Metals003A ICP Metals – Thermo Version 8

CLP SOW – Contract Laboratory Program Statement of Work

SOP – Standard operating procedure

GC/MS – Gas Chromatography/Mass Spectrometry

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy

ASB – Analytical Services Branch

SIM – selected ion monitoring

QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

The CLP and U.S. EPA Region 5 ASB laboratory will keep all maintenance, testing, and inspection records on file at the respective laboratory. The equipment and instrument maintenance, testing and inspection activities are documented in the current CLP SOW SFAM01.1 and in ASB SOPs in **Appendix B** and other laboratory procedures.

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Title/position responsible for corrective action	Reference

Notes: This table is left intentionally blank
ASB – Analytical Services Branch
SOP – Standard Operating Procedure

QAPP Worksheet #26 & #27: Sample Handling, Custody, and Disposal

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample collection and labelling (personnel/organization): CDM Smith FM and CDM Smith field staff.
Sample packaging (personnel/organization): CDM Smith FM and CDM Smith field staff.
Coordination of shipment (personnel/organization): CDM Smith PM or FM.
Type of shipment/carrier: Federal Express to CLP/ASB laboratories.
SAMPLE RECEIPT AND ANALYSIS
Sample receipt (personnel/organization): CLP/ASB laboratory sample custodian.
Sample custody and storage (personnel/organization): CLP/ASB laboratory sample receipt.
Sample preparation (personnel/organization): CLP/ASB laboratory personnel.
Sample determinative analysis (personnel/organization): CLP/ASB laboratory personnel
SAMPLE ARCHIVING
Field sample storage (number of days from sample collection): CLP/ASB samples will be shipped Federal Express same day as collected. The laboratories shall retain the samples in accordance with the CLP SOWs and ASB SOPs.
Sample extract/digestate storage (number of days from extraction/digestion): Refer to worksheet #19 for holding time requirements.
SAMPLE DISPOSAL
Personnel/organization: CLP/ASB laboratory personnel.
Number of days from analysis: In accordance with CLP SOW SFAM01.1, ASB SOP MS023 VOA in Water, ASB SOP Metals003A ICP Metals – Thermo, and ASB SOP MS035.

Notes: For samples sent to the Analytical Services Branch (ASB) laboratory their sample handling, custody and disposal requirements will apply. Appendix B includes the U.S. EPA Region 5 Analytical Service Request form.

FM – Field Manager
CLP SOW – Contract Laboratory Program State of work
ASB SOP – Analytical Services Branch Standard Operating Procedure
ICP -inductively coupled plasma

Collection, preservation, documentation, and shipping of water samples analyzed through the Contract Laboratory Program (CLP) will be carried out in accordance with *Contract Laboratory Program Guidance for Field Samplers* (OLEM 9240.0-51, USEPA 540-R-20-005). The sampling procedures including sample collection, packaging, shipment and delivery to the laboratory will follow all applicable guidelines in the field sampling guidance, the site-specific Sampling and Analysis Plan (**Appendix A**), and SOPs 1-2 and 2-1 as listed on Worksheet #21. Collection, preservation, documentation and shipping of samples analyzed through the U.S. EPA Region 5 ASB laboratory will be carried out in accordance with the *Contract Laboratory Program Guidance for Field Samplers,* as applicable, the site-specific Sampling and Analysis Plan (Appendix A), SOPs 1-2 and 2-1 as listed on Worksheet #21 and the U.S. EPA Region 5 Analytical Services Request Form.

QAPP Worksheet #28a: Analytical Quality Control and Corrective Action

Analytical Group: VOC
Analytical Method/SOP Reference: SOW SFAM01.1[1]
Matrix: Water (influent, effluent and groundwater)
Sampling SOP: See Worksheet #18, #21, and Appendix A

Concentration Level: Trace
Sampler's Name: TBD
Field Sampling Organization: CDM Smith
Analytical Organization: CLP Laboratory[1]
No. of Sample Locations: 40 wells sampled quarterly
1 effluent and 1 influent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples [2]	NA	Notify Site PM, qualify duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Method blank	1 per batch analyzed every 12 hours		Find source/rectify; Qualify results	Laboratory analyst/Data validator	Laboratory contamination	≤ CRQL
Surrogates	Added to every sample and standard		Check calculation / instruments - reanalyze samples. Failure allowances as per SOP; Qualify results	Laboratory analyst/Data validator	Laboratory accuracy/bias	Varies see SOP
MS/MSD	1 per 20 samples		Flag outliers; Qualify results	Laboratory analyst/Data validator	Precision Matrix interference/ laboratory accuracy	0-14% RPD 61-145% Recovery
Internal standards	Added to every sample and standard		Check calculation / instruments - reanalyze samples; Qualify results	Laboratory analyst/Data validator	Accuracy/bias	60-140% Recovery

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #28a in Appendix B

[1]See CLP SOW SFAM01.1 for analytical quality control and corrective actions

[2]One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD) defined in the QAPP, field procedures will be reviewed.

DQI – data quality indicator

QC – quality control

MS/MSD – matrix spike/matrix spike duplicate

RPD – relative percent difference

SOP – standard operation procedure

VOC – volatile organic compound

QAPP Worksheet #28b: Analytical Quality Control and Corrective Action

Analytical Group: 1,4-Dioxane
Analytical Method/SOP Reference: SOW SFAM01.1 for SVOA modified by Method 3054.0[1]
Matrix: Water (effluent and groundwater)
Sampling SOP: See Worksheet #18, #21, and Appendix A

Concentration Level: Low
Sampler's Name: TBD
Field Sampling Organization: CDM Smith
Analytical Organization: CLP Laboratory[1]
No. of Sample Locations: 25 wells sampled quarterly
1 effluent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples [2]	NA	Notify Site PM, qualify duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Method blank	1 per batch extracted/analyzed	< CRQL	Find source/rectify; Qualify results	Laboratory analyst/Data validator	Laboratory contamination	< CRQL
Internal Standard	Added to all ICAL, ICV and CCV calibration standards	50-200%	Check calculation / instruments - reanalyze samples. Rejection does not occur unless %R is <20%; Qualify results	Laboratory analyst/Data validator	Laboratory accuracy/bias	50-200% Rejection does not occur unless R is <20%
MS/MSD	1 per 20 samples	RPD ≤ 50% R - 15%-120%	Qualify outliers - Rejection does not occur unless R is <10%; Qualify results	Laboratory analyst/Data validator	Precision Matrix interference/ laboratory accuracy	RPD ≤ 30% R - 70%-130% Rejection does not occur unless R is <10%
LCS	1 per 20 samples	R - 15%-120%	Suspend analysis/rectify problem; reanalyze affected samples;	Laboratory analyst/Data validator	Laboratory accuracy	R - 70%-130% Rejection does not occur unless R is <10%

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
			Qualify outliers - Rejection does not occur unless R is <10%; Qualify results			
DMC	Added to every blank, sample, calibrations, LCSs and MSs	R 15-120%	Reanalyze samples/ re-extract and reanalyze; Qualify outliers- Rejection does not occur unless R is <10%; Qualify results	Laboratory analyst/Data validator	Laboratory Accuracy/bias	R 40-110% Rejection does not occur unless R is <10%

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #28a in Appendix B

[1]See Modified method 3054.0 SVOA SIM Analysis for Analyte 1,4-Dioxane at Lower CRQL, then CLP SOW SFAM01.1, then U.S. EPA National Functional Guidelines for Organic Superfund Methods Data Review (November 2020) for analytical quality control and corrective actions

[2]One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed, and field duplicates may be collected for subsequent sampling events until sample and duplicate agreement are within the defined RPD.

DQI – data quality indicator

QC – quality control

MS/MSD – matrix spike/matrix spike duplicate

RPD – relative percent difference

SOP – standard operation procedure

ASB – Analytical Services Branch

RL – reporting limit

DMC – Deuterated Monitoring Compound

LCS – Laboratory Control Sample

CRQL – Contract Required Quantitation Limit

R- % recovery

QAPP Worksheet #28c: Analytical Quality Control and Corrective Action

Analytical Group: TAL Metals
Analytical Method/SOP Reference: SOW SFAM01.1[1]
Matrix: Water (influent, effluent and groundwater)
Sampling SOP: See Worksheet #18, #21, and Appendix A

Concentration Level: Low/medium
Sampler's Name: TBD
Field Sampling Organization: CDM Smith
Analytical Organization: CLP Laboratory[1]
No. of Sample Locations: 14 wells sampled quarterly
1 effluent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples[2]	NA	Notify Site PM, qualify duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Preparation blank	1 per 20 samples or less	≤ CRQL	Suspend analysis/rectify problem; redigest and reanalyze affected samples; Qualify results	Laboratory analyst/Data validator	Laboratory contamination	≤ CRQL
ICS	Start of each run	±1 times CRQL or ±20% of established mean, whichever is greater	Check calculations and instruments; reanalyze affected samples; Qualify results		Laboratory accuracy/bias	See Method/ SOP QC Acceptance Limits
LCS	1 per 20 samples or less	70-130%R, 50-150%R for antimony and silver	Suspend analysis/rectify problem; reanalyze affected samples; Qualify results		Laboratory accuracy	
MS/Post digestion spike	1 per 20 samples or less	75-125% R	Flag outliers; Qualify results		Matrix interference	
Laboratory duplicate	1 per 20 samples or less	RPD ≤ 20%	Flag outliers; Qualify results		Laboratory precision	

Notes: If samples are sent to the Analytical Services Branch laboratory see Worksheet #2b8 in Appendix B

[1]See CLP SOW SFAM01.1 for analytical quality control and corrective actions

[2]One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed.

DQI – data quality indicator

QC – quality control

ICS – interference check sample

LCS – laboratory control sample

MS – matrix spike

SOP – standard operation procedure

RPD – relative percent difference

QAPP Worksheet #29: Project Documents and Records

Sample Collection Documents and Records[1]	On-site Analysis Documents and Records[1]	Off-site Analysis Documents and Records[2]	Data Assessment Documents and Records	Other
Logbook(s)	Logbook(s)	Sample receipt, custody, and tracking records	Corrective action reports[1,2]	Annual GMZ Reports[1, 6]
Regional CLP and ASB laboratory COC forms[5]	PID readings, in-line water quality readings	Preliminary analytical data reports	Analytical sample results[2]	Electronic data deliverables[2]
Photos	Field data summary	Final analytical data reports (Case narratives, sample results, QC summaries, raw data)	CLP and/or ASB laboratory certifications[2]	Subcontract documents (contracts, scopes of work, bids)[1]
Air bills	Equipment calibration logs	Laboratory electronic data deliverables	Superfund CLP National functional Guidelines for Data Review - most current	QAPP[4, 6]
GPS coordinates for wells	Well purging logs	Sample preparation logs	U.S. EPA Region 5 CLP Validation SOPs Organic Validation SOP 83074-8-33-601-SO-1143.R1 Inorganic Validation SOP 83074-8-33-607-SO-1138.R1	Non-conformance reports[4]
Custody seals	Photographs	Run logs	Current CLP SOWs and Modified 1,4-dioxane method 3054.0	--
Field change notification forms	Water level measurement logs	Equipment maintenance, testing, and inspection logs	ASB SOPs[2]	--
--	Field data collection logs	Instrument printouts (raw data)	QC audit reports [1,2]	--
--	Well construction logs	Quality control sample summary forms	Data validation reports[3]	--
--	--	Sample disposal records	Data package completeness checklist[3]	--
--	--	Corrective action reports	Data quality assessments[3]	--

Notes: For samples are sent to the Analytical Services Branch laboratory see Appendix B for laboratory information

[1]These documents and records are generated by the field team, verified by the field manager or project manager and stored in the CDM Smith Project files in Chicago.

[2]These documents and records are generated by the project laboratories (CLP or ASB), verified by the data validation staff, U.S. EPA Region 5 for CLP and CDM Smith for ASB, and stored at the respective laboratory and CDM Smith as part of the data packages.

[3]These documents and records are generated by U.S. EPA Region 5 data validation staff for CLP data, by CDM Smith for ASB data, verified by the CDM Smith QA Manager and stored in the CDM Smith project files in Chicago.

[4]These documents and records are generated by the CDM Smith QA Manager, verified by the project manager and stored in the CDM Smith project files in Chicago.

[5]Signed Regional COC forms will be sent to U. S. EPA Region 5 RSCC by e- mail.

[6] These documents will also be transmitted to U.S. EPA.

COC – Chain-of-custody
SOP – Standard Operating Procedure
ASB – Analytical Services Branch
CLP – Contract laboratory program
QAPP – Quality assurance project plan
QC – Quality control
QA – Quality assurance

Electronic analytical data from CLP and/or ASB laboratories will be a level IV data package that conforms to the requirements in the CLP SOW SFAM01.1 or equivalent; CLP Modified method 3054.0, ASB SOP MS023 VOA in Water, Version 7; ASB SOP Metals003A ICP Metals – Thermo, Version 8; and ASB SOP MS035, Version 2. All data will be provided electronically, and copies will be provided to Illinois EPA. Additionally, the validated electronic data deliverables (EDDs) and information files will be uploaded to the U.S. EPA Superfund electronic submission site: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission by CDM Smith.

The U.S. EPA Scribe program will be used for field documentation and generation of COC forms for all samples sent to the CLP. Preparation of sample labels/tags, TR/COC preparation, and sample packaging/shipping procedures for all samples will be performed in accordance with the *Contract Laboratory Program Guidance for Field Samplers* (OLEM 9240.0-51, USEPA 540-R- 020-005). See **Appendix B** for ASB field documentation requirements.

Record	VOCs	Metals	1,4-Dioxane
Narrative	X	X	X
COC	X	X	X
Summary Results	X	X	X
QC Results	X	X	X
Chromatograms	X		X
Tentatively Identified Compounds	X		

All hard copies including, but not limited to, analytical data, project reports, field logbooks, quality control and corrective action documentation will be archived in the project files in the CDM Smith Chicago office. All information in electronic format are stored in CDM Smith's electronic records storage system, ProjectWise. This system is backed up in real time on the CDM Smith cloud storage system. All files, hard copy and electronic are stored by CDM Smith for the time frame specified in the contract which is 10 years. Upon project close-out all files are transferred to Illinois EPA.

QAPP Worksheet #31, 32 & 33: Assessments and Corrective Action

Assessments

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Field progress report	CDM Smith FM	One assessment per quarterly field activities for a period of 5 years	At the conclusion of each quarterly GMZ monitoring event.	Summarizing email	Quarterly at conclusion of quarterly field activities report to CDM Smith PM/ Illinois EPA RPM
Field Audit	U.S. EPA RPM and/or QAO Illinois EPA RPM	One per year	TBD	Corrective Action Request	Verbal in 24 hours and written one week from completion of field audit to PM and FM
Data review and verification	U.S. EPA Region 5 ESAT chemist; CDM Smith Data validation staff	For all CLP data packages; For all ASB data packages	After data receipt during data review activities. Data turnaround 21 days for CLP, 45 days for ASB	E-mail or phone log of deficiencies	24 hours following data review to ASB Lab QA officer or RSCC
Data validation	U.S. EPA Region 5 ESAT chemist for CLP CDM Smith Data validation staff for ASB	One for each data package received	Immediately upon receipt of data. Validation due 3 weeks from receipt of data.	Data validation report	21 days from receipt of data
Corrective action reports	U.S. EPA CLP U.S. EPA Region 5 ASB CDM Smith QC Manager – Wendy Dewar	As required by corrective action (CA) request	Quarterly as needed. Summary annually.	Corrective action report	48 hours following receipt of CA request
Data usability assessment	CDM Smith QC Manager – Wendy Dewar, CDM Smith PM – John Grabs	For all field and CL/ ASB validated data	Quarterly within 2 weeks of data receipt.	Data Usability Section for Annual GMZ Report	Quarterly per sampling event, summary annually

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
GMZ Report	CDM Smith PM - John Grabs	Annually for a period of 5 years	2021 through 2025	Annual GMZ Report	Annually 2021 through 2025

Notes:
ASB – Analytical Services Branch
RSCC – U.S. EPA Regional Sample Control Coordinator
CA – corrective action
ESAT – Environmental Services Assistance Team
GMZ – Groundwater Management Zone
FM – Field Manager
QA – quality assurance
U.S. EPA – United States Environmental Protection Agency
RPM – Remedial Project Manager
QC – Quality Control
PM – Project Manager
QAPP – quality assurance project plan
CLP – Contract Laboratory Program
QAO – Quality Assurance Officer

Assessment Response and Corrective Action*

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for Monitoring Corrective Action Implementation
Field progress report	CDM Smith PM – John Grabs	Field notes/email	Upon receipt of notification for corrective action	CDM Smith FM	CDM Smith PM John Grabs and QC Manager Wendy Dewar
Field audit	CDM Smith PM and FM	Corrective action completion report/notification	Upon receipt of corrective action request or notification for corrective action	CDM Smith FM and/or PM	CDM Smith PM John Grabs and QC Manager Wendy Dewar
Data review and verification	U.S. EPA CLP U.S. EPA Region 5 ASB Laboratory	Corrective action reports and/or updated narratives/corrected data	24 hours from receipt of memorandum and before further analyses can be conducted.	CLP laboratory staff ASB Laboratory Staff	U.S. EPA Region 5 ESAT chemist CDM Smith Data validation staff
Data validation	U.S. EPA CLP U.S. EPA Region 5 ASB Laboratory	Corrective action reports and/or updated narratives/corrected data	48 hours from receipt of memorandum	CLP laboratory staff ASB Laboratory staff	U.S. EPA Region 5 ESAT chemist CDM Smith Data validation staff

Assessment Type	Responsibility for Responding to Assessment Findings	Assessment Response Documentation	Timeframe for Response	Responsibility for Implementing Corrective Action	Responsible for Monitoring Corrective Action Implementation
Corrective action reports	U.S. EPA CLP U.S. EPA Region 5 ASB CDM Smith QC Manager Wendy Dewar	Corrective action completion report/notification	As required by corrective action request	As specified on corrective action request	CDM Smith PM John Grabs CDM Smith QC Manager Wendy Dewar
Data usability assessment	CDM Smith PM – John Grabs CDM Smith QC Manager – Wendy Dewar	Corrective action request to assess impact on data usability and need for resample/analysis	As required by corrective action request	As specified on corrective action request	CDM Smith PM John Grabs CDM Smith QC Manager Wendy Dewar
GMZ Report	CDM Smith PM – John Grabs Illinois EPA RPM	Technical review committee comments	2 weeks from submittal of report	CDM Smith PM – John Grabs	CDM Smith QC Manager Wendy Dewar

* Corrective action is required when an item, condition, or situation detrimental to quality is identified. This may include deviation from prescribed methods, items exceeding predetermined acceptability limits, or failure to meet performance requirements or DQOs. Anyone finding a problem is responsible for reporting it. During routine activities, most corrective actions can be implemented immediately by the work project staff and documented in field notebooks. If the condition is not quickly corrected, the individual initiates a corrective action report (CAR) form and forwards it to the QC manager who assigns responsibility for the corrective action and the required timing for the response. The QC manager with the assistance of staff, is responsible for tracking, reviewing, accepting and verifying corrective action. The QC manager maintains a log of CARs with follow up actions and completion dates.

QAPP Worksheet #34: Data Verification and Validation Inputs

Item	Description	Verification (completeness)	Validation[1] (conformance to specifications)
Planning Documents/Records			
1	Approved QAPP	**X**	
2	Sampling and Analysis Plan /Field SOPs	**X**	
3	Laboratory SOPs/CLP SOWs	**X**	
Field Records			
4	Field logbooks	**X**	**X**
5	Equipment calibration records	**X**	**X**
6	Chain-of-Custody Forms	**X**	**X**
7	Sampling diagrams/surveys	**X**	**X**
8	Change orders/deviations	**X**	**X**
9	Field corrective action reports	**X**	**X**
Analytical Data Package			
16	Cover sheet (laboratory identifying information)	**X**	**X**
17	Case narrative	**X**	**X**
18	Internal laboratory chain-of-custody	**X**	**X**
19	Sample receipt records	**X**	**X**
20	Sample chronology (i.e. dates and times of receipt, preparation, & analysis)	**X**	**X**
23	RL/MDL establishment and verification	**X**	**X**
24	Standards Traceability	**X**	**X**
25	Instrument calibration records	**X**	**X**
26	Definition of laboratory qualifiers	**X**	**X**
27	Results reporting forms	**X**	**X**
28	QC sample results	**X**	**X**
29	Corrective action reports	**X**	**X**
30	Raw data	**X**	**X**
31	Electronic data deliverable	**X**	**X**

[1] All data will be validated at a Stage 2B option. If quality issues are identified the level of data validation may be elevated to further assess the quality issue. See WS# 36 for description of data validation stages.

QAPP Worksheet #35: Data Verification Procedures

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field logbook	QAPP, SOP 4-1, SAP	Verify that records are present and complete for each day of field activities. Verify that all planned samples, including field QC samples, were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that required field monitoring was performed and results are documented. These records include, but are not limited to, field screening data for organic vapors (PID readings) and water level measurements. Field sampling data requirements are described in Worksheet #11. Water level data will be checked against the baseline and historical water level results for reasonableness.	Daily – FM, Tian Liao At conclusion of quarterly field activities - PM, John Grabs
Chain-of-custody and shipping forms	QAPP, SOP 1-2, SAP	Verify the completeness of chain-of-custody records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required amount of sample material has been collected and that sufficient sample material is available for QC samples (e.g., MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors.	Daily - FM, Tian Liao At conclusion of quarterly field activities - Project Chemist
Laboratory deliverable	QAPP, CLP SOW SFAM01.1, CLP Modified SOW SFAM01.1 Method 3054.0, ASB SOP MS035, ASB SOP Metals003A ICP, ASB SOP MS023 ASB SOPs in **Appendix B**	Verify that the laboratory deliverable contains all records specified in the QAPP and lab SOPs. All laboratories are requested to provide Level IV data packages for all parameters. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the chain-of-custody to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present. Check for data transcription errors.	Before release - CLP laboratory QA Manager/ASB laboratory QA Manager Upon receipt – CDM Smith Chemist

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Corrective action reports	QAPP, SAP	Examine Corrective action reports. For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QCM, Wendy Dewar

CLP SOW – Contract Laboratory Program Statement of Work
SAP – Sampling and Analysis Plan
FM – Field Manager
PM – Project Manager
QAPP – Quality Assurance Project Plan
SOP– standard operating procedure
QCM – Quality Control Manager
ASB – Analytical Services Branch

QAPP Worksheet #36: Data Validation Procedures

Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (title and organizational affiliation)
Water	CLP TCL VOC; VOC by ASB	Low/medium	U.S. EPA NFG for Organic Superfund Methods Data Review, Region 5 Organic CLP Validation SOP[1], and CLP SOW SFAM01.1; U.S. EPA NFG for Organic Superfund Methods Data Review, ASB volatile organic SOP	U.S. EPA Region 5 ESAT chemist; CDM Smith Data Validation staff chemist
Water	1,4-dioxane by CLP SOW SFAM01.1 for SVOA modified by Method 3054.0; 1,4-dioxane by ASB	Low	CLP SOW SFAM01.1 for SVOA modified by Method 3054.0, Region 5 Organic CLP Validation SOP[1], CLP SOW SFAM01.1, and U.S. EPA NFG for Organic Superfund Methods Data Review; ASB 1,4-dioxane SOP, U.S. EPA CLP NFG for Organic Data	CDM Smith Data Validation staff chemist
Water	TAL Inorganics; Inorganics by ASB	Low/medium	U.S. EPA NFG for Organic Superfund Methods Data Review, Region 5 Inorganic CLP Validation SOP[1] and CLP SOW SFAM01.1; U.S. EPA NFG for Organic Superfund Methods Data Review and ASB inorganic SOP	U.S. EPA Region 5 ESAT chemist; CDM Smith Data Validation staff chemist

Notes: If data is generated by U. S. EPA Region 5 ASB Laboratory, the analytical SOPs can be found in Appendix B

[1] U. S. EPA Region 5 CLP Validation SOPs - Organic Validation SOP 83074-8-33-601-SO-1143.R1 and Inorganic Validation SOP 83074-8-33-607-SO-1138.R1

CLP SOW – Contract Laboratory Program Statement of Work
ESAT – Environmental Services Assistance Team
SIM – selected ion monitoring
SOP – standard operating procedure
ASB – Analytical Services Branch
VOC - Volatile organic compound
SOW – Statement of Work
NFG – National Functional Guidelines
U.S. EPA – United States Environmental Protection Agency
SVOA – Semi-volatile organic analysis

Data Verification/Validation Scope Overview

Laboratory results will be reviewed for compliance with data quality objectives (DQO). As required by Illinois EPA and U.S. EPA Region 5, all data will be validated based on the project needs. For the Area 7 GMZ monitoring, the Stage 2B validation option is planned because significant historical Stage 4 validated data exist for the site and data anomalies and/or quality issues will be easily identified based on the historical data. If data quality issues are identified or suspected, the level of data validation may be elevated to better define the quality issues and develop a corrective action if needed. Data validation and evaluation are discussed in the following subsections.

U.S. EPA Region 5 ESAT data validation staff will conduct a Stage 2B data validation for 100% of the CLP generated data as per the most current U.S. EPA's National Functional Guidelines, the current CLP SOWs, Region 5 Organic CLP Validation SOP 83074-8-33-601-SO-1143.R1, and Region 5 Inorganic CLP Validation SOP 83074-8-33-607-SO-1138.R1.

For project data generated by the U.S. EPA Region 5 ASB laboratory, a level IV data package will be requested with 45-day turnaround time. CDM Smith's data validation staff chemist will conduct a Stage 2B data validation for 100% of the data using the most current U.S. EPA's National Functional Guidelines (NFG) and the ASB analytical standard operating procedures provided in **Appendix B** as guidance. The current U.S. EPA NFG documents may be found here: https://www.epa.gov/clp/superfund-clp-national-functional-guidelines-data-review.

Example data validation forms used by CDM Smith for VOCs/1,4-dioxane and inorganics are provided in **Appendix C**.

Field Data Review

Field-generated information may include field logbooks, sampling logs, sample chain-of-custody forms, shipping documents, sampling observations, sample labels, and other miscellaneous field observations. All field measurements and or field log information will be entered in field logbooks and reviewed daily by the field manager or designee. The designee may be a qualified field geologist, engineer, environmental scientist, or technician. Verification of field results includes checking that results from field log books have been correctly transferred to data reports as well as checking to ensure all deviations from the approved sampling and analysis procedures are well documented. Field data review for water level data includes against the baseline and historical water level results for reasonableness.

Laboratory Data Review Requirements

U.S. EPA Region 5 ESAT staff will validate data generated by CLP analytical laboratories. CDM Smith data validation staff will validate data generated by the U. S. EPA Region 5 ASB laboratory.

Data validation will be performed according to the U.S. EPA CLP *National Functional Guidelines for Organic Superfund Methods Data Review (SFAM01.1)* (U.S. EPA 2020) and *National Functional Guidelines (NFG) for Inorganic Superfund Methods Data Review (SFAM01.0)* (U.S. EPA 2020), with method specific requirements superseding the NFG guidelines. Additional data validation guidelines for CLP data include the current CLP SOWs, CLP Modified method 3054.0 for 1,4-dioxane, U. S. EPA Region 5 Organic CLP Validation SOP 83074-8-33-601-SO-1143.R1 and U. S. EPA Region 5 Inorganic CLP Validation SOP 83074-8-33-607-SO-1138.R1. Additional guidelines for ASB data include the ASB laboratory SOPs in **Appendix B**.

All analytical data generated by the laboratories will be verified before submittal to CDM Smith. The internal data review process, which is multi-tiered, will include all aspects of data generation, reduction, and QC assessment. In each laboratory analytical section, the analyst performing the tests shall review 100 percent of definitive data. After the analyst's review has been completed,

100 percent of the data will be reviewed independently by a senior analyst or by the supervisor of the respective analytical section using the same criteria.

Elements for review or verification at each level must include, but not be restricted to, the following:

- Sample receipt procedures and conditions
- Sample preparation
- Appropriate SOPs and analytical methodologies
- Accuracy and completeness of analytical results
- Correct interpretation of all raw data, including all manual integrations
- Appropriate application of QC samples and compliance with established control limits
- Documentation completeness (for example, all anomalies in the preparation and analysis have been identified, appropriate corrective actions have been taken and documented in the case narrative[s], associated data have been appropriately qualified, and anomaly forms are complete)
- Accuracy and completeness of data deliverables (electronic)

Laboratory Data Evaluation

Calibration, QC, corrective actions, and qualification requirements for definitive data are shown in Worksheets #12a, #12b, #12c, #15a, #15b, #24, #28a, #28b and #28c. The laboratory may apply data qualifiers based on their review or add a note in the laboratory case narrative. Definitions of any data qualifiers applied by the laboratory must be defined in the case narrative. Data qualifiers are reviewed by the supervisor of respective analytical sections after the first and second level reviews of the laboratory data have been performed.

CDM Smith Data Review Guidelines

The laboratory assessment of the data quality will be reviewed for completeness and accuracy. Data review will be done manually and will include, but is not limited to, the following:

- Sampling documentation (such as the chain-of-custody form)
- Preservation summary and technical holding times
- Results of all analyses and analytes requested
- Use of the required sample preparation and analysis procedures

- The method detection and reporting limits will be evaluated against the project requirements
- The correctness of the concentration units
- Case narrative

Data Verification/Validation Guidelines

The data verification process builds on data review. For CLP/ASB data, data verification includes checking that results have been transferred correctly from laboratory data printouts to the laboratory report and to the staged electronic data deliverable (SEDD) format.

Project data will be reviewed and verified as part of the data assessment for this project. The review will be performed on an analytical batch basis by assessing QC samples and associated field sample results. Data verification guidelines have been developed in accordance with the method requirements, professional judgment, and general U.S. EPA National Functional Guidelines.

Summary data review and verification will be performed as follows:

- Chain-of-custody documentation
- Holding time
- QC sample frequencies
- Method and field blanks
- Laboratory control samples
- Surrogate spikes
- MS/MSD
- Initial and continuing calibration information
- Laboratory duplicate precision
- Case narrative review and other method-specific criteria

CLP data will be validated within three weeks of data package receipt by U.S. EPA Region 5 ESAT data validation staff using the Stage 2B validation option. ASB data will be validated within three weeks of data package receipt by CDM Smith Data validation staff using the Stage 2B validation option.

Data validation consists of examining the sample data package(s) against pre-determined standardized requirements. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, LCS/LCSDs, MS/MSDs, initial and continuing instrument calibration, and other reported information to determine the accuracy and completeness of the data package. During this process, the validator will verify that the analytical methodology was followed, and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information. Analytical results will then be qualified as necessary.

The manual verification/validation process includes data qualification for issues related to method blanks, laboratory control samples, MS/MSD samples, lab duplicates, surrogate recoveries, holding time, and reconciliation of dilutions and re-extractions. Data results and qualifiers, as well as the reason for each qualifier are entered into an electronic database and made available to the data users. During the validation process, appropriate laboratory qualifiers and validation qualifiers are combined to apply one final qualifier (if required) for each result.

The data validation stages are more fully described below:

- Stage 1 Validation/Verification: Verification and validation based only on completeness and compliance of sample receipt condition checks.
- Stage 2A Validation/Verification: Verification and validation based on completeness and compliance checks of sample receipt conditions and only sample-related QC results (e.g., method blanks, surrogates, LCSs, duplicates, internal standards, holding times, frequency of QC samples).
- Stage 2B Validation/Verification: Verification and validation based on completeness and compliance checks of sample receipt conditions and both sample-related and instrument-related QC results (e.g., method blanks, surrogates, LCSs, duplicates, internal standards, holding times, frequency of QC samples, initial calibrations, continuing calibration, calibration blanks, instrument tunes).
- Stage 3 Validation/Verification: Verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, and recalculation checks(e.g., method blanks, surrogates, LCSs, duplicates, internal standards, holding times, frequency of QC samples, initial calibrations, continuing calibration, calibration blanks, instrument tunes, instrument response data, recalculation of selected laboratory QC and sample results).
- Stage 4 Validation/Verification: Verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, and the review of actual instrument outputs (e.g., method blanks, surrogates, LCSs, duplicates, internal standards, holding times, frequency of QC samples, initial calibrations, continuing calibrations, calibration blanks, instrument

tunes, instrument response data, recalculation of selected laboratory QC and sample results, instrument output evaluated for sample results).

If, during the data review and verification process, a systematic problem or other major issue with the data is identified, the CDM Smith project chemist will contact the Region 5 ESAT data validation staff or the ASB laboratory's QA manager. The level of data validation may be elevated from Stage 2B to further assess the problem or issue. Additional evaluation of the data may be performed including an in-depth review of the raw data to verify accuracy followed by analysis and interpretation of the data in the context of the project objectives and end-use as part of the usability assessment. As is the normal data validation process, a data validation report will be prepared summarizing the findings and discussing their impact on the overall data usability. The data that may have needed more review than Stage 2B will be uploaded into the database with the appropriate qualifiers applied before data is released for project purposes. All data validation reports, including any data sets that required a more extensive data validation, will be incorporated into the final data evaluation summary report to be provided to the data users.

Qualification Conventions

The most common data qualifier definitions are summarized below:

- R – Rejected: The data are rejected because of deficiencies in meeting QC criteria and may not be used for decision making.
- J – Estimated: The analyte was positively identified; result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- UJ – The analyte was not detected; however, the result is estimated because of discrepancies in meeting certain analyte-specific QC criteria.
- U – Undetected: The analyte was analyzed for, but not detected or is qualified as nondetect because of blank contamination.

QAPP Worksheet #37: Data Usability Assessment

The data usability assessment is an evaluation based on results of data verification and validation in the context of the overall project decisions or objectives. The assessment determines whether project execution and resulting data meet the project DQOs. Both the sampling and analytical activities must be considered, with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following subsections summarize the processes to determine whether the collected data are of the right type, quality, and quantity to support the environmental decision making for the project and describes how data quality issues will be addressed and how any limitations of the use of the data will be handled.

Summary of Usability Assessment Processes

It is the responsibility of the laboratories to ensure that the data meet detection limits and laboratory QC limits listed in this QAPP. Analytical methods and associated detection and QC limits were selected by the data user to allow for a meaningful comparison to PALs. During the data verification assessment, non- conformances are documented, and data are qualified for use in decision making. The data are determined to be usable by the CDM Smith project chemist and PM based on requirements of this QAPP and project objectives. Data gaps will be present if a sample is not collected, a sample is not analyzed for the requested parameters, or the data are determined to be unusable. The need for further investigation will be determined on a case-by-case basis. All data are usable as qualified by the data validator, except rejected data. Estimated and/or biased results are usable. Outliers, if present, can be addressed on a case-by-case basis.

There is no generic formula for determining whether a result is an outlier. Potential outliers will be referred to a statistician and senior consultant, who will determine which formulas are appropriate for classifying data points in a statistically appropriate and defendable manner.

Evaluation Procedures to Assess Project-specific Overall Measurement Error

In-depth assessment occurs during the data verification process. The verification will assess conformance with the requirements of the methods, SOPs, and objectives of this QAPP. The findings of the data verification process will generate qualifiers applied to the data considered in context to assess overall usability of the data.

Personnel Responsible for Performing Usability Assessment

The data usability assessment will be performed by CDM Smith personnel.

- Cherie Zakowski, project scientist, CDM Smith
- John Grabs, PM, CDM Smith

- Tian Liao, FM, CDM Smith
- Wendy Dewar, QC Manager, CDM Smith

Usability Assessment Documentation

Analytical DQOs will be assessed through application of precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters to ensure that the data are of sufficient quality to meet the project objectives. The following subsections describe each of the PARCC parameters and how they will be assessed within this project. The data assessment results will be reported in the annual GMZ Report.

Precision

Precision is the degree of mutual agreement between individual measurements of the same property under similar conditions. Usually, combined field and laboratory precision is evaluated by collecting and analyzing field duplicates and then calculating the variance between the samples, typically RPD.

$$\text{RPD} = \frac{\text{A} - \text{B}}{\frac{(\text{A} + \text{B})}{2}} \times 100\%$$

Where:

A = First duplicate concentration

B = Second duplicate concentration

Field sampling precision is evaluated by analyzing field duplicate samples. Field duplicates will be collected and analyzed at a frequency of 10 percent of the investigative groundwater samples. For the effluent, one field duplicate will be collected for the first sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD) defined in the QAPP, field procedures will be reviewed, and field duplicates may be collected for subsequent sampling events until sample and duplicate agreement are within the defined RPD. Acceptance criteria for field sampling precision will be RPD of less than 30% for field duplicate samples, as presented in Worksheets #12a, #12b and #12c.

Laboratory analytical precision is evaluated by analyzing laboratory duplicates for inorganics or MSs and MSDs for organics. MS/MSD samples will be generated for all analytes when sufficient sample volume is provided.

Analytical results of each laboratory duplicate and/or MS/MSD pair will be used to calculate an RPD for evaluating precision. Acceptance criteria for laboratory analytical precision is presented in Worksheets #28a, b and c.

Accuracy/Bias

Accuracy reflects the total error associated with a measurement. Bias is defined as the difference between a measurement result and its unknown "true value". A measurement is considered accurate and unbiased when the reported value agrees with the true value or a known concentration of the spike or standard within acceptable limits. A program of sample spiking will be conducted to evaluate laboratory accuracy/bias. This program includes analysis of the MS and MSD samples, LCSs or blank spikes, surrogate standards, and method blanks. Surrogate standards, where applicable, are added to every sample analyzed for organic constituents. The results of the spiked samples are used to calculate the percent recovery for evaluating accuracy.

$$\text{Percent Recovery (\%R)} = \frac{S - C}{T} \times 100$$

Where:

S = Measured spike sample concentration

C = Sample concentration

T = True or actual concentration of the spike

Results that fall outside the accuracy/bias goals, which are presented on Worksheets #28a, b and c, will be further evaluated based on the results of other QC samples.

Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent the characteristics of a population, variations in a parameter at a sampling point, or an environmental condition that they are intended to represent. Representative data will be obtained for this project through adherence to the designed sampling program, selection of appropriate sampling locations and use of standardized sampling techniques. Representative data will also be obtained through proper collection and handling of samples to avoid interference and minimize contamination.

Representativeness of data will be ensured through the consistent application of established field and laboratory procedures. Equipment blanks (if appropriate) and laboratory blank samples will be evaluated for the presence of contaminants to aid in evaluating the representativeness of sample results. Data determined to be non-representative, by comparison with existing data, will be used only if accompanied by appropriate qualifiers and limits of uncertainty.

To evaluate the variability of the data within the context of representativeness, the most current sample results at each location will be compared to the most recent previous data for the location to evaluate the RPD for each sampling point. The project objectives ($\leq$ 30% RPD) will dictate the acceptable and/or desired limits on variability for sampling points.

Completeness

Completeness is a measure of the percentage of project-specific data that are valid. Valid data are obtained when samples are collected and analyzed in accordance with QC procedures outlined in the SAP. When all data validation is completed, the percent completeness value will be calculated by dividing the number of usable sample results by the total number of sample results planned for this investigation.

Completeness will also be assessed to determine if the samples that were planned for collection were collected. The total number of samples planned will be compared to the total number of samples collected.

% Completeness = X/Y x 100

Where: X = amount of valid data obtained

Y = amount of data expected to be obtained

Completeness will also be evaluated as part of the data quality assessment process. This evaluation will help determine whether any limitations are associated with the decisions to be made based on the data collected.

Comparability

Comparability expresses the confidence with which one data set can be compared with another. Comparability of data will be achieved by consistently following standard field and laboratory procedures and by using standard measurement units in reporting analytical data.

Sensitivity

The method detection limit (MDL) is defined as the smallest amount of concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99 percent). At the MDL, the false negative rate (Type II error) is 1 percent. The quantitation limit (QL) is at the lowest concentration that produces a quantitative result with specified limits of precision and bias. Analytical methods for this project have been selected so that the QL for each target analyte is below remediation goals listed in Worksheets #15a and #15b.

Data Quality Assessment

After data are validated, the project chemist will review and assess field and laboratory QC. The PARCC parameters will be assessed as described above. Data validation reports will be reviewed and assessed for meeting DQOs. The project chemist will review the data validation reports for any deviations and qualify data accordingly. The following data qualifiers will be used:

J – Result is estimated

U – Analyte is not detected at or above the stated QL/MRL/CRQL

R – Data are rejected

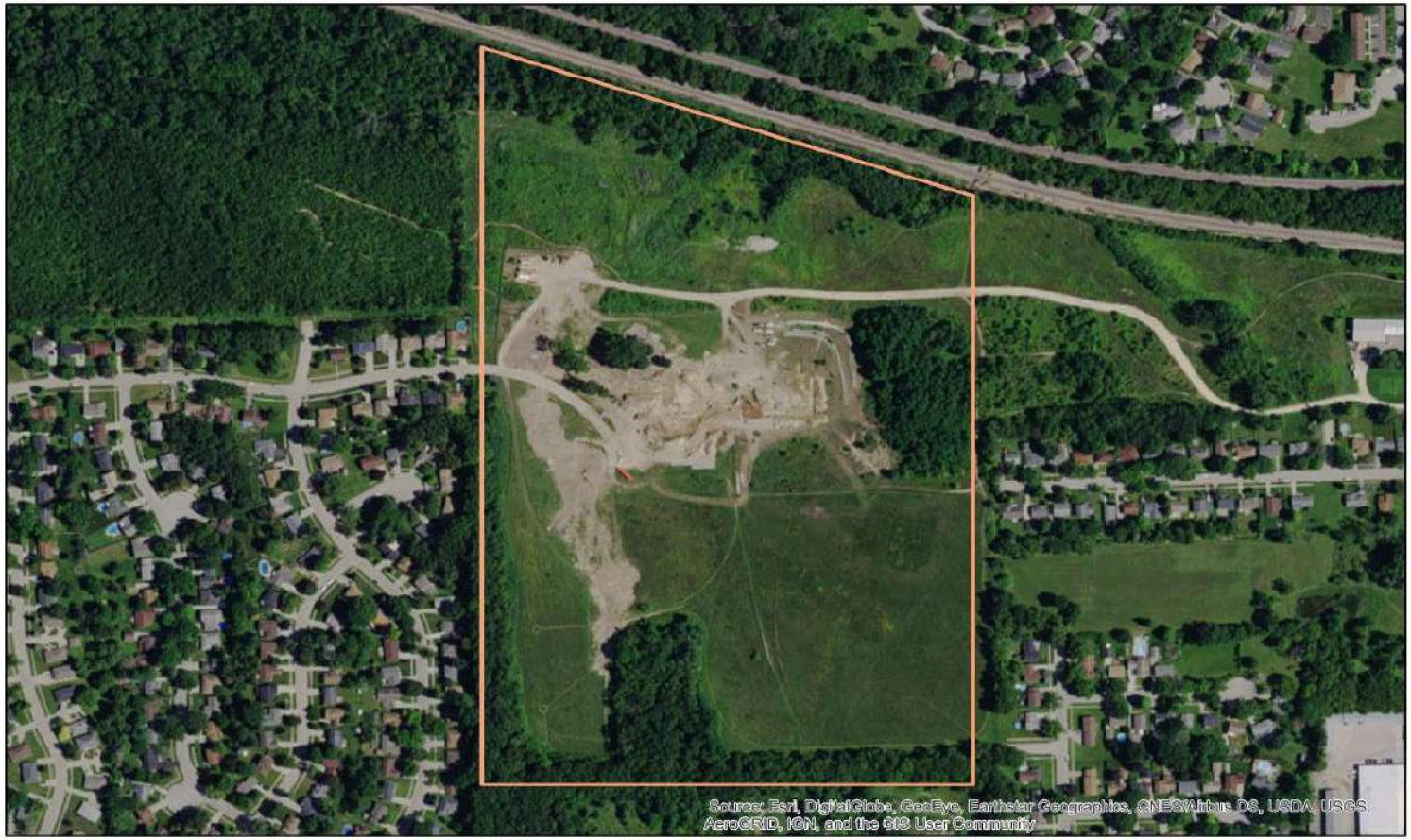
UJ – Analyte is not detected but there is uncertainty about the QL/MRL/CRQL

Data qualifiers are used to indicate uncertainties associated with the data. The assigned qualifiers will be entered into the validation code field in the database.

The project chemist will prepare a data quality assessment report that will summarize the findings of the data assessment and discuss the usability of the data to be included in the report.

Data will be reported in tabular format to be included in the report. Electronic data from CLP or U. S. EPA Region 5 ASB will be transferred to the site database and stored with the project files. For analytical data generated by the ASB laboratory, please refer to **Appendix B** for pertinent information.

Figures



Site Boundary

0 175 350 700 Feet

N
W E
S

CDM Smith

Figure 1
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Monitoring Wells

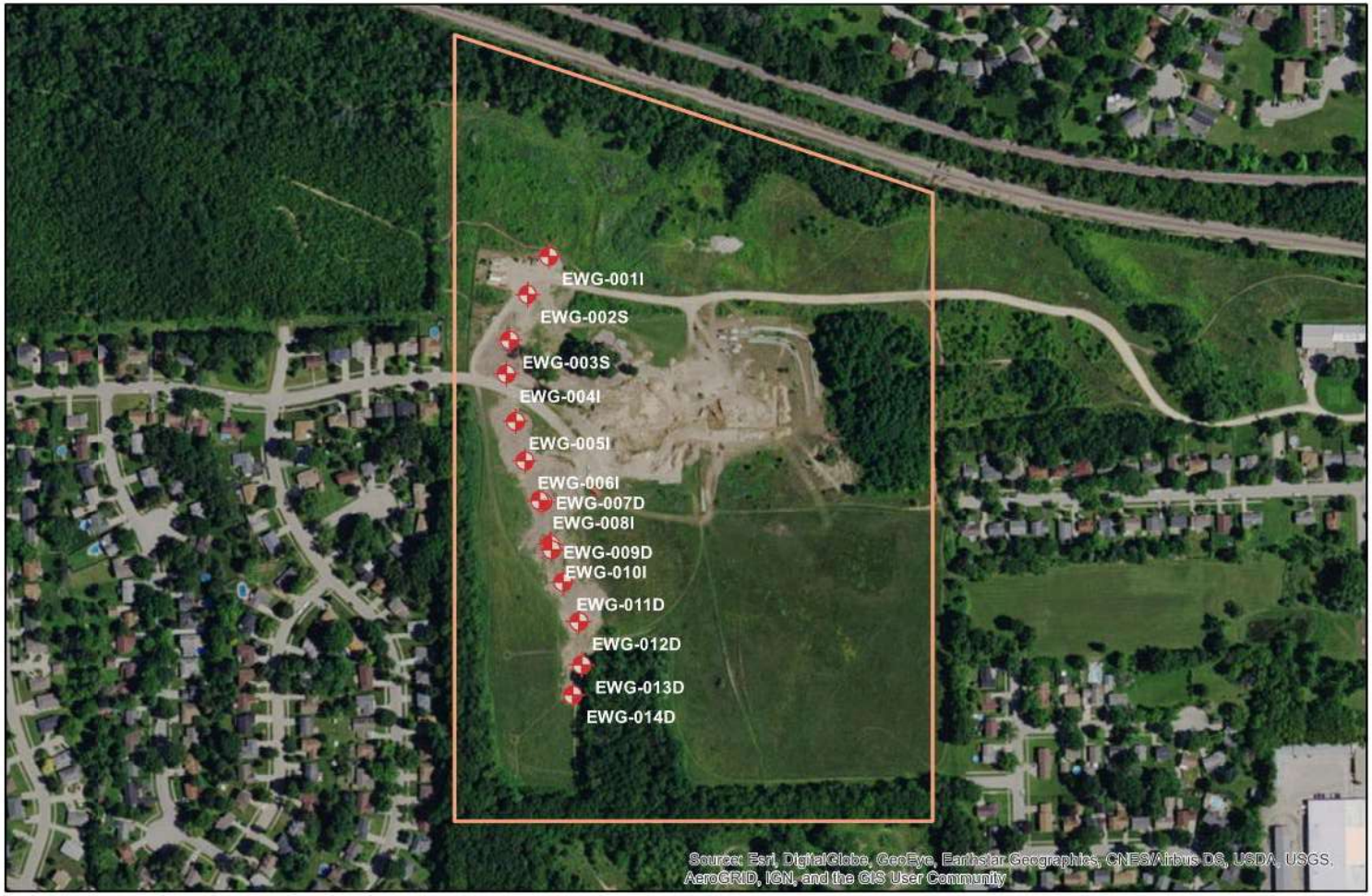
Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 2
Monitoring Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois



Extraction Wells

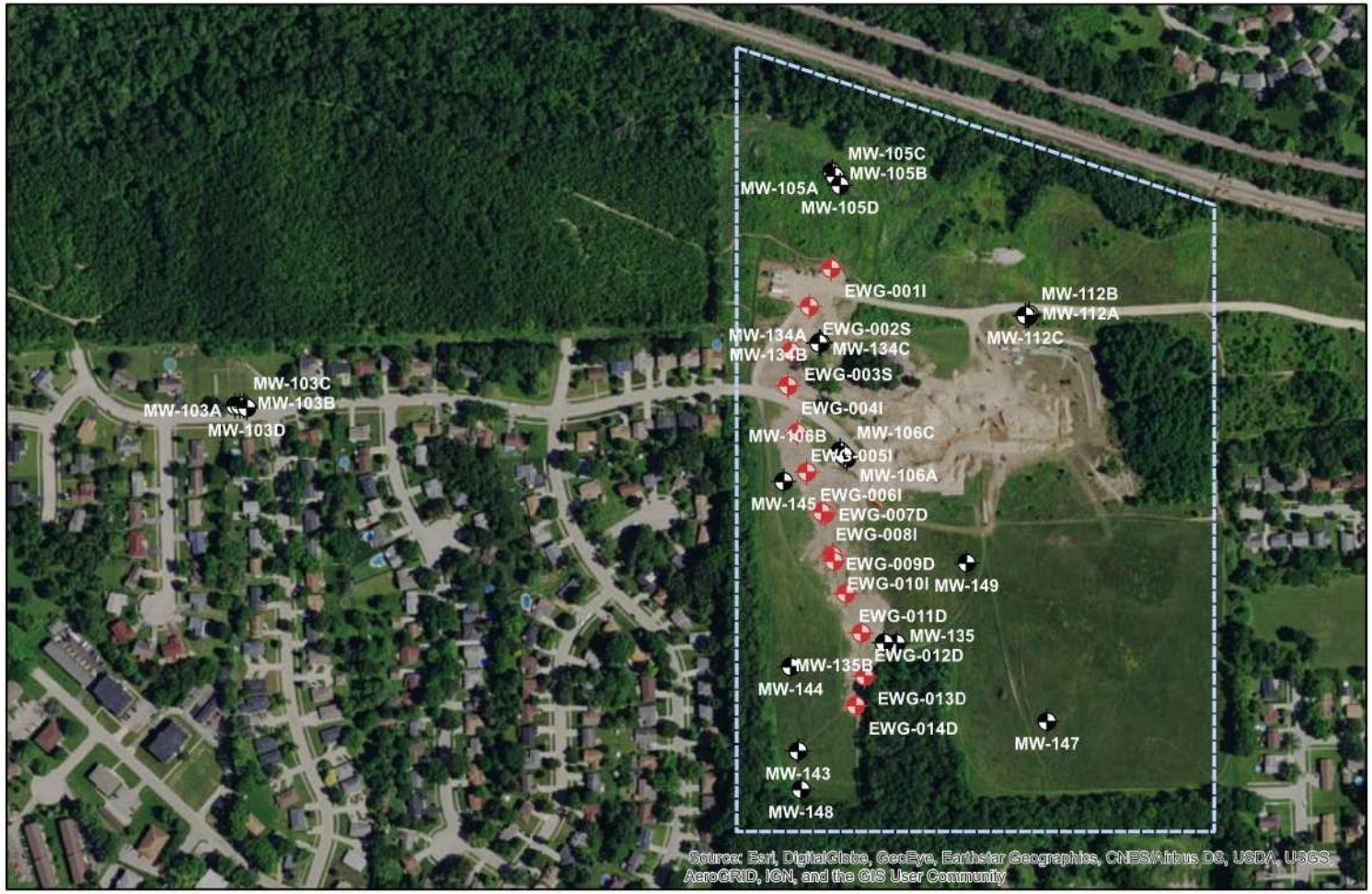
Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 3
Extraction Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois



Extraction Wells

Monitoring Wells

GMZ and Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 4
Groundwater Management Zone Boundary
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Appendix A

Sampling and Analysis Plan with CDM Smith Sampling Standard Operating Procedures

Sampling and Analysis Plan
Final Draft

Southeast Rockford Groundwater Contamination Superfund Site
Source Area 7
Groundwater Management Zone
Monitoring Activities

Contract HWA-16303, Work Order #06

Prepared for:

Illinois Environmental Protection Agency

May 4, 2021
Revision 4

Prepared by:

CDM Smith

Table of Contents

Section 1 Introduction 1-1
1.1 Objectives of Sampling Program 1-1
1.2 Sampling Team Responsibilities 1-2
1.2.1 Field Manager 1-2
1.2.2 Sampling Team Leader 1-2
1.2.3 Sampling Team Member(s) 1-2
1.2.4 Field Data Coordinator 1-2
1.2.5 Site Health and Safety Coordinator (SHSC) 1-2
1.2.6 Safety Technician 1-3
1.3 Scope of Sampling Activities 1-3

Section 2 Project Description 2-1
2.1 Area 7 Site Description 2-1
2.2 Previous Investigations 2-1
2.3 Remedial Action Objectives 2-3

Section 3 General Sampling Information 3-1
3.1 Sample Containers and Preservation 3-1
3.1.1 Liquid Samples 3-2
3.2 Sample Holding Times 3-3
3.3 Sample Packaging and Shipment 3-3
3.4 Chain-of-Custody Procedures 3-5
3.5 Documentation 3-5
3.5.1 Field Log Books 3-5
3.5.2 Sample Identification System 3-6
U.S. EPA Sample Number 3-6
3.6 Sample Documentation Forms 3-8

Section 4 Sampling Locations and Rationale 4-1

Section 5 Sampling Equipment and Procedures 5-1
5.1 Water Level Measurements 5-1
5.2 Groundwater Sampling 5-1
5.2.1 Monitoring Well Sampling 5-1
5.2.2 Extraction Well Sampling 5-2
5.3 LTS Influent and Effluent Sampling 5-2

Section 6 Decontamination Procedures 6-1

6.1 Personnel Decontamination 6-1
6.2 Equipment Decontamination 6-1
6.3 Sample Bottle Decontamination 6-1
6.4 Storage and Disposal of Investigation-Derived Waste 6-2

Section 7 Field Quality Control Procedures 7-1

7.1 Groundwater QC Samples 7-1
7.1.1 Field Duplicates 7-1
7.1.2 Equipment Blanks 7-1
7.1.3 Trip Blanks 7-1
7.1.4 Matrix Spike, Matrix Spike Duplicates, and Laboratory Duplicates 7-2

List of Figures

Figure 1. Area 7 Layout
Figure 2. Monitoring Well Locations
Figure 3. Extraction Well Locations
Figure 4. Ground Water Management Zone Boundaries

List of Tables

Table 1-1. Summary of Sampling and Analysis Program Southeast Rockford Area 7 GMZ Monitoring Activities 1-5
Table 3-1. Sample Volumes, Containers, and Preservation Techniques Low/ Medium Concentration 3-2
Table 4-1. Area 7 GMZ Groundwater Monitoring and Extraction Wells 4-2
Table 6-1. Standard Decontamination Protocol for Sampling Equipment 6-2

Appendices

Appendix A – Health and Safety Plan (HASP)

Appendix B - CDM Smith Technical SOPs

1-2 Sample Custody, Rev. 9, August 2020
1-6 Groundwater Level Measurement, Rev. 10S, August 2020
1-9 Tap Water Sampling Rev. 8, August 2020
1-10 Field Measurement of Organic Vapors, Rev. 8, February 2020
1-12 Low Stress (Low Flow) Groundwater Sampling, Rev. 3, August 2020
2-1 Packaging and Shipping Environmental Samples, Rev. 7, August 2020
2-2 Guide to Handling Investigation-Derived Waste, Rev. 9, August 2020
4-1 Field Logbook Content and Control, Rev. 9, August 2020
4-2 Photographic Documentation of Field Activities, Rev. 10, August 2020
4-3 Well Development and Purging, Rev. 8, August 2020
4-5 Field Equipment Decontamination, Rev. 11, March 2020
5-1 Control of Measurement and Test Equipment, Rev. 11, August 2020

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Section 1

Introduction

1.1 Objectives of Sampling Program

This Sampling and Analysis Plan (SAP) describes the field activities required to conduct groundwater monitoring for the Southeast Rockford Groundwater Contamination (SERGC) Superfund site, Source Area 7 (Area 7) groundwater management zone (GMZ) and to aid in evaluating and optimizing the efficiency of the leachate treatment system (LTS) currently operating at Area 7. Area 7 is shown in **Figure 1**. The objectives of the sampling program include:

- Provide LTS influent data for volatile organic compounds (VOCs) and effluent data for VOCs and 1,4 dioxane to evaluate LTS performance and confirm compliance with the effluent discharge limits.
- Compliance with the groundwater quality monitoring for Area 7 specified in the GMZ application.
- Assess water levels in Area 7 to determine the influence of LTS pumping and discharge on local hydrology.
- Obtain groundwater data over time to evaluate and optimize the LTS design and efficiency.

The following field activities will be completed to comply with the GMZ requirements, evaluate and improve performance of the LTS, and determine if groundwater extraction and/or discharge from the system is impacting groundwater flow patterns:

- Monitor for organic vapors during sampling for health and safety purposes.
- Collect one influent and one effluent sample from the LTS influent and effluent sampling ports quarterly during system operation. Influent will be analyzed for VOCs and total and dissolved inorganics and effluent will be analyzed for VOCs, 1,4 dioxane and inorganics.
- Collect groundwater samples for VOCs and 1,4 dioxane analysis on a quarterly basis for 5 years at the 25 monitoring wells shown in Figure 2.
- Collect groundwater samples for VOCs and inorganic analysis on a quarterly basis for 5 years at the 14 extraction wells located in a north-south trending as shown in Figure 3.
- Collect water levels prior to sample collection from all GMZ monitoring wells, manually, on a quarterly basis for 5 years.

Details of the sampling tasks are provided in Section 4.0 of this SAP. This addendum has been prepared to update the sampling program Standard Operating Procedures (SOPs), revise groundwater sampling protocols due to current field conditions and update analytical methods and laboratory options to be used for sample analysis.

1.2 Sampling Team Responsibilities

Field sampling will be performed by CDM Smith. The project organization chart is shown in the Southeast Rockford Area 7 GMZ Monitoring Activities Quality Assurance Project Plan (QAPP). Responsibilities of the sampling team are described below.

1.2.1 Field Manager

The Field Manager (FM) will be responsible for assigning the sampling team responsibilities (in conjunction with the Site Manager), as well as overseeing all field activities. The FM will coordinate mobilization and demobilization for the CDM Smith sampling team, as well as for any subcontractors. The FM will be responsible for keeping the Site Manager up to date on all sampling and subcontractor activities.

1.2.2 Sampling Team Leader

The Sampling Team Leader (STL) will be responsible for the sampling efforts, will assure the availability and maintenance of all sampling equipment and materials, and will maintain an adequate supply of shipping and packing materials. The STL will supervise the completion of all chain-of-custody records, the proper handling and shipping of the samples collected, be responsible for the accurate completion of field log books, and provide close coordination with the Field Data Coordinator (FDC) and the FM. The STL or FM will be present whenever samples are collected.

1.2.3 Sampling Team Member(s)

The Sampling Team Member(s) (STM) will perform field measurements, collect samples, prepare samples for shipping, and decontaminate sampling equipment as directed by the STL.

1.2.4 Field Data Coordinator

The FDC will remain in the support area and will accept custody of samples from the sampling team. The FDC will be responsible for the completion of all chain-of-custody and sample traffic control forms. The FDC will also be responsible for maintaining communications with on-site personnel and off-site laboratory personnel, as well as for logging all communications and site entries and departures.

1.2.5 Site Health and Safety Coordinator (SHSC)

The SHSC is responsible for daily supervision and documentation of all safety, decontamination, environmental monitoring and field medical monitoring activities. The SHSC is responsible for assuring that all field personnel comply with the provisions of the CDM Smith Health and Safety

Manual and site Health and Safety Plan (HSM/HASP). The SHSC has the authority to suspend site work if conditions become unsafe, if HSM/HASP requirements are not met, or if he/she determines that an upgraded level of protection may be required. The SHSC has the authority to restrict access to exclusion zones to personnel designated by CDM Smith or Illinois EPA. The SHSC is responsible for designating and marking restricted areas during various site activities and for re-designating these areas when it is appropriate to do so.

1.2.6 Safety Technician

The Safety Technician (a designated member of the sampling team) will assist with sampling, aid other sampling team members with the donning and doffing of protective clothing, decontamination of sample containers and equipment, and will be available to replenish miscellaneous supplies, such as ice, as needed. The Safety Technician will report directly to the SHSC in health and safety related duties and will assume the responsibilities of the SHSC in the event of their absence from the site or in an emergency.

1.3 Scope of Sampling Activities

The LTS sampling activities detailed by this plan includes the collection of an influent sample and effluent sample quarterly for the duration of system operation. The groundwater sampling activities include five years of quarterly sample collection and analysis. The sampling activities include the following:

- To evaluate LTS performance and confirm that the effluent discharged to the intermittent creek meets the discharge limits, an influent sample will be collected after the equalization tank and an effluent sample will be collected immediately prior to the LTS discharge location quarterly.
- To comply with the GMZ groundwater monitoring requirements, groundwater samples will be collected from the 25 monitoring wells shown on **Figure 1**.
- To evaluate and optimize the LTS effectiveness, groundwater samples will be collected from the 14 extractions wells that run from north to south shown on **Figure 2**.
- To evaluate potential impacts on the local groundwater flow regime, water levels will be measured prior to quarterly sampling for all monitoring wells sampled for GMZ monitoring. These results will be compared to the baseline water levels collected prior to start-up of the contaminant collection and treatment system and evaluated for changes over time.

Table 1-1 is a summary of the sampling and analysis program. The numbers and frequency of samples collected during the Area 7 GMZ Monitoring activities is estimated to be one quarterly influent and one quarterly effluent sample for the duration of LTS operation and 39 investigative groundwater samples collected quarterly for five years. However, the actual numbers and

frequency will depend on the results of confirmatory effluent samples and groundwater sampling activities.

Groundwater sampling may extend beyond five years total to comply with GMZ requirements and/or until the contaminant concentrations are below the remediation goals provided in the ROD and any other applicable standards such as the Illinois Groundwater Quality Standards for Class I Groundwater.

If a monitoring well should become inaccessible for any sampling event, the situation will be assessed and if access can be restored quickly the well will be sampled for that event. If a well is damaged or destroyed it will not be sampled for that particular event and will be repaired or replaced as necessary.

Table 1-1. Summary of Sampling and Analysis Program Southeast Rockford Area 7 GMZ Monitoring Activities

				QC Samples		
Sample Matrix	Field Parameters	Laboratory Parameters [7]	Investigative Samples[1]	Field Duplicates	Equipment Blank	Matrix Total[3,5]
Liquid Influent from LTS	Qualitative organic vapor screening with PID pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	CLP TCL Volatile Organics[2] SOW SFAM01.1, CLP TAL Total inorganics[4] SOW SFAM01.1; 21- day turn around; or ASB methods[7], 45-day turn-around	1	0	0	1
Liquid Effluent from LTS	Qualitative organic vapor screening with PID pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential Field filter for dissolved inorganics	CLP TCL Volatile Organics and CLP SVOC 1,4-dioxane only[2] SOW SFAM01.1, CLP TAL total and dissolved inorganics[4] SOW SFAM01.1; 21- day turn around; or ASB methods [7,8], 45-day turn-around	1	1 First annual sampling event [6]	0	2
Groundwater from Monitoring Wells	Qualitative organic vapor screening with PID pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	CLP TCL Volatile Organics and CLP SVOC 1,4-dioxane only[2] SOW SFAM01.1; 21-day turn around; or ASB methods[7,8]; 45-day turn-around	25	3	3	31
Groundwater from Extraction Wells	Qualitative organic vapor screening with PID pH, conductivity, temperature, turbidity, dissolved oxygen, redox potential	CLP TCL Volatile Organics[2] SOW SFAM01.1; CLP TAL total inorganics[4] SOW SFAM01.1; 21- day turn-around; or ASB methods[7], 45-day turn-around	14	2	0	16

1. Frequency of collection for all investigative and QC samples is 1. Samples will be collected quarterly for 5 years for a total of 20 rounds of samples.
2. Contract Laboratory Program SOW SFAM01.1 target compound list volatile compounds (CLP TCL VOCs) and CLP semi-volatile (SVOC) selective ion monitoring (SIM) analysis modified by method 3054.0 for 1,4-dioxane only.

3. The extra volume for samples to be collected for matrix spike (MS) and laboratory duplicates for inorganics, and matrix spike/matrix spike duplicate (MS/MSD) for organics is not included in the matrix total. The MS, laboratory duplicates and MS/MSDs will be collected at a frequency of one per group of 20 or fewer samples for inorganic and volatile organic analysis. Double volume is needed for aqueous CLP/ASB volatile organic MS/MSD samples and 1,4-dioxane to ASB. For CLP 1,4-dioxane MS/MSD analysis, five 1 – liter amber bottles are required. Double volume is needed for aqueous inorganic MS and laboratory duplicates. MS and MS/MSD will be collected for influent sample for every sampling event.

4. Contract Laboratory Program SOW SFAM01.1 target analyte list inorganics (CLP TAL inorganics).

5. One volatile organic trip blank will be shipped for analysis with each cooler of volatile organic and for 1,4-dioxane samples to ASB only. **No trip blank is required for 1,4-dioxane analyzed by CLP**. Trip blanks are not included in the matrix total.

6. One field duplicate of effluent will be collected for all parameters for the first sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD) defined in the QAPP, field procedures will be reviewed.

7. ASB laboratory parameters are specified in SOP MS023 VOA in Water Version 7 for volatile organics, and SOP Metals003A ICP Metals – Thermo Version 8 for inorganics provided in Appendix B of the QAPP.

8. For 1,4-dioxane samples shipped to and analyzed by ASB Laboratory, the parameter specific method SOP MS035 Version 2 will be used and a trip blank will be required.

Section 2

Project Description

2.1 Area 7 Site Description

Area 7 is primarily a grassy area located at the eastern end of Balsam Lane. Area 7 contains Ekberg Park, an open field and a wooded area. Ekberg Park is owned and operated by the Rockford Park District and the remainder of Area 7 is owned by Glen Ekberg. The park is currently closed due to the remedial action activities; however, the property is not secured by fencing or other means making public access possible. The topography slopes gently to the north towards an intermittent creek. Ekberg Park consists of a playground, basketball court, and open space. The open field and wooded areas are south of the park on a hillside and surface water drainage down the hillside flows north into the intermittent creek. Residences border the area to the west and southwest (downgradient) and are also located about 0.1 mile away to the east (upgradient).

Chlorinated solvents are the principal contaminants present at the site. Contaminants were released to the environment from improper disposal practices. Three primary volatile organic compound (VOC) source areas were identified during the investigations and are located at the northern and southern areas of Ekberg Park. Northern source areas contain the northwest side of the playground and southwest side of the tennis courts. The southern area includes the south side of the basketball courts. The depth to significant levels of contamination varies from 4 feet below ground surface (bgs) in the northern area to approximately 30 feet in some southern locations.

Contamination is present in the soils both above and below the water table with NAPL present at various locations. Groundwater samples collected from the aquifer in the overburden soil reveal a variety of different types and classes of VOCs, with the majority being chlorinated solvents.

Groundwater flow in both the unconsolidated and bedrock aquifers is to the west northwest, with localized discharge of shallow groundwater to the intermittent creek. Depth to groundwater ranges from about 60 feet bgs at the south end of Area 7 to 25 feet bgs south of the park area, to 13 feet bgs within the park, to less than 2 feet bgs near the creek. Seasonally, the depth to groundwater is highly variable with 10-foot changes possible over the course of a year.

2.2 Previous Investigations

The Phase I Remedial Investigation (RI) for the Southeast Rockford Groundwater Contamination Site was conducted from May to October of 1991 and consisted primarily of a site-wide soil gas survey, monitoring well installation, and groundwater sampling and analysis to investigate multiple suspected source areas. The investigation of a potential source in Area 7 was sparked by elevated concentrations of contaminants in the shallow monitoring well of a three well cluster immediately downgradient of Area 7. Phase I of the RI included review of historical aerial

photographs, geophysical surveys and soil gas sampling. The geophysical surveys indicated several areas of subsurface anomalies which were subsequently investigated with soil gas sampling. Soil gas results indicated that high concentrations of VOCs, primarily TCA, PCE and TCE were present in the subsurface. During Phase II of the RI, additional work was conducted in Area 7 consisting of test-pit investigation, surface and subsurface soil sampling, and monitoring well installation.

The Phase II site-wide groundwater investigation conducted concurrently also indicated the same contaminant mix of TCA, PCE and TCE downgradient, confirming that the subsurface in Area 7 was impacting site-wide groundwater. Residential air sampling was conducted near Area 7 in 1993 and 2003 to determine if the soil and groundwater contamination was affecting indoor air quality in homes near the source. The indoor air evaluations indicated that the migration pathways are generally inadequate or incomplete and do not result in indoor air concentrations at levels that present an unacceptable health risk.

The Remedial Investigation Report for the site-wide groundwater investigation and source area identification was completed by CDM Smith and resulted in the signing of the OU2 ROD which required additional extension of the City of Rockford municipal water system and selected natural attenuation, long-term groundwater monitoring, and source control measures that would be determined in the future under the SCOU.

The SCOU RI and Focused Feasibility Study (FFS) reports were completed in July 2000. The SCOU FFS addressed contaminated soils, non-aqueous phase liquids (NAPL), and leachate considered to be principal threat wastes and the primary causes of groundwater contamination at the four primary source areas southeast Rockford. Alternatives developed in the SCOU FFS were separated into soil and leachate alternatives for each source area. In order to simplify the OU 3 ROD, technologies intended to contain and/or treat contaminated groundwater in the immediate vicinity of the primary source areas were considered leachate alternatives.

According to the SCOU investigation results for Area 7, elevated concentrations of toluene, ethyl benzene, xylenes, and chlorinated volatile organic compounds were found in Area 7 soils. The previous remedial investigation identified subsurface contamination along the drainage ditches in the southern portion of Area 7. The SCOU investigation confirmed that an additional hot-spot area of soil contamination exists in and to the west and northwest of Ekberg Park.

Elevated levels of VOCs were also found in the groundwater and in the surface water of the intermittent creek located on the property. Shallow groundwater downgradient of Area 7 contains elevated concentrations of TCA as the primary contaminant and lesser concentrations of various other VOCs. Non-aqueous phase liquids (NAPL) were found in soil 11 feet below the groundwater table in a soil boring in the park. Additionally, Area 7 was found to have areas of highly contaminated silt and clay units indicating that NAPL has migrated into these fine-grained sediments.

The results of the groundwater investigation indicated that the distribution and concentration of contaminants within groundwater monitoring wells were generally similar to results from previous sampling investigations. The highest concentrations of VOCs were observed within the unconsolidated aquifer at monitoring well location MW-134A (Figure 2). The results of the groundwater sampling from wells (MW135B, MW143, MW144, and MW145) in the southern portion of Area 7 also showed significant downgradient VOC contamination. These results also indicated that the VOC source extended further to the south than previously believed. Additional groundwater monitoring wells were installed in August 2010 to define the southern extent of VOC groundwater contamination.

In 2010, under an Explanation of Significant Differences (ESD) to the ROD, CDM Smith conducted a limited soil excavation northwest of the playground area to remove shallow, low-permeability source material. This limited action was completed in May 2011.

2.3 Remedial Action Objectives

Based on remedial investigations and site-specific risk assessment, Remedial Action Objectives (RAOs) were developed. The Area 7 RAOs provide a general description of what the remedial action will accomplish and are as follows:

- Prevent the public from ingestion of soil, and direct contact with soil containing contamination in excess of state or federal standards or that poses a threat to human health
- Prevent the public from inhalation of airborne contaminants in excess of state or federal standards or that pose a threat to human health
- Prevent the further migration of contamination from Area 7 that would result in degradation of site-wide groundwater or surface water to levels in excess of state or federal standards, or that pose a threat to human health or the environment

A number of potential remedial action alternatives for Area 7 were developed and evaluated based on RAOs, remediation goals, and comparative evaluation criteria. The detailed comparative analysis of Area 7 remedial alternatives is discussed in detail in the Record of Decision (ROD).

The remedial action (RA) currently being implemented at Area 7 includes leachate and soil vapor extraction and treatment and hydraulic containment within Area 7 to address contamination migrating from the area. Multiphase extraction wells have been installed in the source areas to extract leachate, vapor, and NAPL. The extracted vapor/liquid stream is pumped to a treatment system consisting of liquid/vapor separators, oil/water separators, iron removal system, air stripper, liquid phase carbon for leachate treatment and vapor phase carbon for vapor effluent treatment. Treated effluent water is discharged to the intermittent creek located north of the treatment system. Groundwater extraction wells have been installed for long-term hydraulic containment of the contaminated groundwater. The extracted groundwater is pumped directly to

the iron removal system because groundwater from these wells is not expected to contain NAPL. **Figure 3** shows the location of Area 7 with the GMZ boundary that has been established for the site to monitor the effectiveness of the LTS.

This SAP as amended includes the sampling activities required to comply with the GMZ groundwater monitoring requirements and improve performance of the contaminant containment and treatment system.

Section 3

General Sampling Information

Collection, preservation, documentation, and shipping of samples analyzed through the U.S. EPA CLP or U.S. EPA Region 5 ASB laboratory will be carried *out in accordance with Contract Laboratory Program Guidance for Field Samplers,* November 2020 (OLEM 9240.0-51, USEPA 540-R-20-005) which can be found at https://www.epa.gov/clp/contract-laboratory-program-guidance-field-samplers and the ASB Request Form included in **Appendix B** of the QAPP. Site-specific sampling information is included in the following sections. However, the sampling procedures will follow all applicable guidelines in the field sampling guidance and CDM Smith SOPs and are not limited to the information provided in Section 3.

To initiate a request for sample analysis, the FM will transmit a U.S. EPA Region 5 Analytical Service Request Form (ARF) to the Regional Sampling Control Center Coordinator (RSCC) and ASB Sample Coordinator 2 to 3 weeks in advance unless there is an emergency situation where as much advance notice as possible will be provided. The RSCC and ASB Sample Coordinator will determine laboratory assignments in accordance with the U.S. EPA Region 5 Field and Analytical Services Teaming and Advisory Committee process and will notify the FM of the assignments. Depending on the laboratory assignments, the FM will subsequently coordinate with the RSCC for samples being submitted to CLP for analysis and with the ASB Sample Coordinator for samples being submitted to ASB for analysis. The FM will notify the RSCC and ASB Sample coordinator of any last-minute changes in the sampling schedule. An example ARF form is provided in **Appendix B** of the QAPP.

3.1 Sample Containers and Preservation

All groundwater samples and the influent and effluent samples for volatile organics, 1,4-dioxane and inorganics will be collected in decontaminated, pre-preserved sample bottles and vials provided by a commercial vendor. At the LTS influent and effluent and monitoring and extraction well locations, concentrations of contaminants are anticipated to be either low or medium concentrations as designated by the U.S. EPA CLP. Sampling, handling, and shipping of the samples will be performed in accordance with these anticipated concentrations. CDM Smith SOP 2-1, Packaging and Shipping Environmental Samples will also be used for reference.

A summary of sample containers, their size and construction material, sample matrix, and holding times is given in **Table 3-1**. Internal laboratory QC requirements for analytical samples are discussed in Worksheets #28a, #28b, and #28c in the Area 7 GMZ Monitoring Activities QAPP. If VOC, 1,4-dioxane and/or inorganic samples are to be analyzed by ASB, this information is provided in **Appendix B** of the QAPP.

The sample containers and their preservation will be as follows:

Table 3-1. Sample Volumes, Containers, and Preservation Techniques Low/ Medium Concentration

Analysis	Container	Preservative	Maximum Holding Time	Volume of Sample[1]	Matrix
Volatile Organic Compounds CLP SOW SFAM01.1; ASB SOP MS023	Four to CLP; 3 to ASB 40-ml amber glass vials with Teflon septum	Preserve with HCl to pH of 2; Cool to 0 to 6° C Do not freeze	14 days	Fill completely (no air bubbles)	Liquid
1,4-dioxane ASB SOP MS035	Two 250-ml amber glass bottles	ASB only Preserved with Na_2SO4 to pH <2; Cool to 0 to 6° C Do not freeze	28 days post extraction; if pH measured at lab is >2 holding time is reduced to 7 days	Fill to shoulder of bottle	Liquid
1,4-dioxane CLP SOW SFAM01.1 SVOA modified by method 3054.0	Two 1-liter amber glass bottles	CLP only No preservative; Cool to 0 to 6° C Do not freeze	7 days to extraction; 40 days from extraction		
Total and dissolved metals Inorganic Compounds CLP SOW SFAM01.1; ASB SOP Metals003A ICP Metals - Thermo	One 0.5-liter high density polyethylene round bottle	Preserve to pH<2 with HNO3; Cool to 0 to 6° C Do not freeze	6 months	Fill to shoulder of bottle	Liquid

[1] Double volume is needed for aqueous inorganic MS and laboratory duplicates. Double volume is needed for aqueous CLP/ASB volatile organic MS/MSD samples and 1,4-dioxane to ASB. For 1,4-dioxane CLP MS/MSD analysis, five 1-liter amber bottles are required.

VOCs – volatile organic compounds
HCl – hydrochloric acid
HNO^3 – nitric acid
ASB – Analytical Services Branch
SVOA – Semi-volatile organic analysis
CRQL – Contract Required Quantitation Limit
CLP SOW – Contract Laboratory Program Statement of Work
Na_2SO4 – sodium bisulfate
ml - milliliter
SOP – Standard Operating Procedure
SIM – Selective Ion Monitoring
ICP – Inductively Coupled Plasma

3.1.1 Liquid Samples

- **Four** 40-ml amber glass VOA vials (pre-preserved with hydrochloric acid) will be collected at all locations sampled for CLP volatile organic analysis; **Three** 40-ml amber glass VOA vials (pre-preserved with hydrochloric acid) will be collected at all locations sampled for ASB volatile organic analysis.

- One 0.5-liter high density poly bottle (pre-preserved with nitric acid) will be collected at each location sampled for analysis of total CLP/ASB inorganics.

- One 0.5-liter high density poly bottle (pre-preserved with nitric acid) will be collected at each location sampled for analysis of dissolved CLP/ASB inorganics.
- Two 250-ml amber glass bottles (pre-preserved sodium bisulfate) will be collected at each location sampled for ASB analysis of 1,4-dioxane.
- Two 1-liter amber glass bottles will be collected at each location sampled for CLP analysis of 1,4-dioxane. No preservative is required.
- Sample collection will be performed in accordance with Contract Laboratory Program Guidance for Field Samplers, November 2020 (OLEM 9240.0-5.1, EPA-540-R-020-0105) : https://www.epa.gov/clp/contract-laboratory-program-guidance-field-samplers, ARF Request Form and CDM Smith SOPs 1-6, 1-9, 1-10, and 1-12, and this SAP.
- At sample collection points where field duplicate samples will be collected, a double-volume sample will be collected but will be labeled and submitted to the CLP or U.S. EPA Region 5 ASB laboratory for analysis as separate samples.
- At sample collection points where matrix spike/matrix spike duplicates (MS/MSD) samples for CLP/ASB volatile organic analysis and ASB 1,4-dioxane will be collected, double the sample volume will be collected.
- At sample collection points where matrix spike/matrix spike duplicates (MS/MSD) samples for CLP 1,4-dioxane will be collected, five 1-liter amber bottles will be collected.
- At sample collection points where matrix spikes or laboratory duplicate samples for inorganic analysis will be collected, double the sample volume will be collected.
- All water samples will be cooled to ≤6°C in an iced cooler following individual sample collection.

3.2 Sample Holding Times

The sample holding times for all samples are listed on **Table 3-1**. To expedite sample analysis, the groundwater samples will be shipped overnight to the laboratories via Federal Express on the day of sample collection. The ASB holding times are also provided in **Appendix B** of the QAPP.

3.3 Sample Packaging and Shipment

Following sampling, the sample bottle exteriors will be decontaminated near the sampling location or rinsed with potable or distilled water prior to shipment. The FM will help the FDC prepare documentation (Scribe software for CLP samples) and package sample bottles for shipment according to the following procedures:

- Check for proper sample preservation; tighten sample bottle caps securely; mark liquid levels if bottles are partially full.
- Ensure traffic report labels (CLP samples) and/or sample labels are securely attached to the sample container; place each container in a zip-top baggie, ensuring that labels can be read.
- For samples to the CLP in accordance with the U.S. EPA Region 5 January 11, 2018 Memorandum from Tim Prendiville, Chief, Science and Quality Assurance Section, sample tags are no longer required.
- Place containers in a cooler lined with absorbent material; maintain at 0° C to 6° C with cold packs or ice sealed in plastic bags, do not freeze; fill remaining space in cooler with additional packing material.
- Place completed chain-of-custody forms (COC) and/or traffic reports (CLP samples) in a zip-lock baggie and tape to inside of cooler lid.
- Close cooler and seal with strapping tape; if cooler has a drain port, seal it with tape; place one custody seal across closure at front of cooler and across hinge area at back of cooler, or rear side corner.
- Affix air bill with shipper's and consignee's addresses to top of cooler; place "This End Up" labels appropriately. Restricted article air bills will be used in shipping medium-concentration samples.

Collected and packaged samples will then be shipped or delivered to the designated laboratory.

For all samples shipped to the ASB and CLP laboratories, the address to which the cooler is to be returned must be included with the COC. Additionally, ASB cannot accept Saturday sample deliveries. The ASB requirements are provided on page 5 of the ARF in **Appendix B** of the QAPP.

Chain-of-Custody forms will be reviewed against the samples packed in each cooler prior to shipment. Chain-of-Custody forms will be sent with the samples to the laboratory, while copies are retained for the Sampling Trip Report and the project files. They will be internally reviewed upon completion of activities and verified against field logs, and laboratory report. For sample sent to CLP laboratories, Scribe will be used to prepare Chain-of-Custody forms and upload the xml file to the Sample Management Office (SMO) portal website on the evening of the sample ship date. This will allow U.S. EPA to retrieve sample shipping information. Hardcopies will be kept with the sample and data packages and PDF copies will be emailed to the RSCC. The shipping information to be provided must include:

1. Samplers' names and phone numbers where they can be reached

2. Case numbers
3. Name of laboratory(ies)
4. Date of shipment
5. Carrier and air bill number
6. Number, concentration and matrices of samples shipped
7. Information regarding changes and delays pertaining to the activity

CDM Smith SOP 2-1, Packaging and Shipping Environmental Samples will also be used for reference. See **Appendix B** of the QAPP for U.S. EPA Region 5 ASB laboratory specific procedures. A copy of the ASB laboratory COC is provided in **Appendix B** of the QAPP. Scribe will not be used to generate the COC for ASB laboratory.

3.4 Chain-of-Custody Procedures

Chain-of-custody will be maintained throughout the sample preparation procedure as described in the Southeast Rockford Area 7 GMZ Monitoring Activities QAPP. Preparation of sample labels and Traffic Report/Chain-of-Custody Form (TR/COC) preparation, and sample packaging/shipping procedures for all samples will be performed in accordance with the U.S. EPA document *Contract Laboratory Program Guidance for Field Samplers*, November 2020 (OLEM 9240.0-5.1, EPA-540-R-020-0105) and the ASB laboratory. Care will be taken to ship only the laboratory copy of the COC to the laboratory and the Regional copy of the COC will be emailed to the Region. CDM Smith SOP 1-2, Sample Custody will also be used for reference. See **Appendix B** of the QAPP for ASB laboratory procedures and example COC form.

3.5 Documentation

This section outlines the documentation required for all field activities, sample collection, handling and shipment to be conducted during the Area 7 GMZ Monitoring Activities.

3.5.1 Field Log Books

Field log books will provide the means of recording pertinent data collected during the performance of the field activities. As such, entries will be described in as much detail as possible so that site personnel can reconstruct a particular situation without reliance on memory.

Field log books will be bound, field survey books. Log books will be assigned to field personnel and will be stored in the document control center when not in use. Each log book will be identified by the project-specific document number.

The title page of each notebook will contain:

- Person or Organization to whom the book is assigned
- Book Number
- Project Name
- Start Date
- End Date

Entries into the log book will contain a variety of information. At the beginning of each entry, the date, start time, weather, name of all team members present, level of personal protection being used, and the signature of the person making the entry will be recorded. The names of visitors to the site, field sampling or investigation team personnel and the purpose of their visit will be recorded in the field log book. At the end of each day's activity, the log will be closed with the time and signature of the person making the last entry (log-closed line). The log-closed lines and the following log-open lines will be placed so that no unauthorized entries can be made between entries.

Measurements made and samples collected will be recorded. All entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, dated, and initialed by the person making the correction.

Wherever a sample is collected or a measurement is made, a detailed description of the location of the station, which shall include compass and distance measurements, shall be recorded. The number of the photographs taken of the station with a brief description including and the direction faced will be noted. All equipment used to make measurements will be identified, along with the date of calibration. CDM Smith SOP 4-2, Photographic Documentation of Field Activities, will also be used for reference.

The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample location identifiers will be assigned prior to sample collection. Duplicates, which will receive a separate sample number, will be noted under sample description. CDM Smith SOP 4-1, Field Logbook Content and Control will also be used for reference.

3.5.2 Sample Identification System

U.S. EPA Sample Number

Each sample being sent to the CLP for analysis must have a U.S. EPA case number and a sample number, regardless of the laboratory to which it is going. The RSCC will assign a case number for the sampling event. A new case number will be assigned approximately every two weeks. The RSCC will also provide a series of sample numbers to be used during the sampling events. The sample numbers typically consist of the letters "E" (U.S. EPA code for Region 5 organic samples)

or "ME" (U.S. EPA code for Region 5 inorganic samples) followed by four to six numerical digits. A sample number will be automatically assigned to each sample by the Scribe program. Sample numbers will only be required for the samples submitted to a CLP laboratory.

Station Name/ Location Identification

In addition to the sample number, each groundwater, influent and effluent sample will also be assigned a station name and station location in accordance with SCRIBE requirements. The station name ID assigned to each sample collected in Area 7 will be "A7". Station locations will also be used for samples sent to the ASB laboratory for analysis.

The first two letters of the station location ID identify the sample matrix. The additional alpha/number portion of the location ID will correspond to the sampling location designations. Sample matrices will be recorded using the following code:

SAMPLE MATRIX/LOCATION ID	CODE
Influent from LTS	IN
Effluent from LTS	EF
Groundwater from extraction well	EWG
Groundwater from monitoring well	MW

Groundwater, influent and effluent samples collected during the Area 7 GMZ monitoring activities sampling will have up to 16 alphanumeric characters. After the Area 7 code, the next set of letters and numbers will correspond to the influent, effluent or monitoring or extraction well location within the source area. Monitoring wells that are nested with wells at different depths will have an additional letter designation. The letter suffix will document what depth the sample was collected from within the well nest. The letter "A" will represent the shallowest sample, "B" a sample from the second depth interval, "C" the third sample depth, etc. Extraction wells use "S", "I", and "D" to designate relative depth. The final set of numbers will designate the date on which the sample was collected.

An example of a groundwater sample collected from a monitoring well on June 27, 2019 is as follows:

A7-MW106B-190627

This identifier denotes a groundwater sample that would have been collected in Area 7 from monitoring well MW-106B which is the second sampling interval for this well nest location.

For all sample matrices, field duplicate, field blank and trip blank samples will be given a unique sample designation that does not allow the laboratory to identify the sample as a QC sample. The investigative and corresponding field QC sample identifiers will be cross referenced in the field log book so that the correlation can be made when data is being tabulated and evaluated.

Sample designations will be recorded in the sample field book, on the chain-of-custody forms, the traffic reports, the sample identification record form, and on the sample labels affixed to the sample containers.

3.6 Sample Documentation Forms

TR/COC forms for CLP samples will be generated using U.S. EPA's Scribe software. Preparation of the forms will be performed in accordance with the Scribe operating instructions and the U.S. EPA guidance Contract Laboratory Program Guidance for Field Samplers, November 2020 (OLEM 9240.0-5.1, EPA-540-R-020-0105).

TR/COC forms will be assigned a number by the Scribe program. In the event that a document is voided, it will not be destroyed; instead, voided sample documents will be saved and returned to the RSCC. The other copies will be emailed in PDF format to the RSCC immediately following sampling shipment.

A. Traffic Report/Chain-of-Custody Form

    1. One form per shipping container (cooler) will be used
    2. Separate forms will be used for organic and inorganic samples.
    3. Carrier service will not need to sign form if custody seals remain intact
    4. Will be used for all samples

B. Chain-of-Custody Seals

    1. Two seals per shipping container will be used to secure the lid and provide evidence that samples have not been tampered with
    2. Seals will be covered with clear tape
    3. Seal numbers will be recorded on Chain-of-Custody Form
    4. Seals will be used for all sample shipping containers
    5. The STL or FM will sign and date each custody seal

The TR/COC will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the TR/COC will be retained for the field files.

The U.S. EPA Region 5 ASB sample shipping requirements and example forms are provided in **Appendix B** of the QAPP. These requirements and forms will be used any samples shipped to the ASB laboratory. For samples shipped to the ASB and CLP laboratories, the address to which the cooler is to be returned must be included with the COC. Additionally, ASB laboratories cannot accept Saturday sample deliveries.

The sample handling technician will maintain lists cross-referencing site sample numbers, field QC samples, analyses to be performed, custody seal number, shippers' air bill numbers, and consigned laboratories in a bound logbook using black ink.

Section 4

Sampling Locations and Rationale

4.1 Area 7 GMZ Monitoring

The overall objective of this project is to monitor and manage the remediation of contaminants released from the Area 7 portion of SERGC in accordance with the ROD, the RD specifications, and the GMZ application. In addition to performing groundwater monitoring consistent with the GMZ application, CDM Smith will collect data to evaluate and improve performance of the LTS. Once the data has been evaluated, CDM Smith will coordinate with the remediation contractor, Bodine, to reconfigure operating parameters as necessary to improve system performance and achieve performance standards as rapidly as possible.

The field investigation activities to be performed are described in the following subsections.

4.1.1 Water Level Survey

Water levels will be measured in the GMZ monitoring wells shown in **Figure 2** and listed in **Table 4-1** prior to start-up of the contaminant collection and treatment system to develop baseline groundwater level data and prepare a baseline groundwater contour map.

Water levels will be measured prior to sampling for all quarterly sampling events of the GMZ monitoring activities. This data will be used to evaluate hydrological impacts of the LTS pumping and discharge on the local groundwater regime in Area 7.

4.1.2 Groundwater Sampling

In order to comply with the GMZ application, quarterly groundwater sampling will be performed for a period of at least five years. Groundwater samples for laboratory analysis will be collected from the Area 7 GMZ monitoring wells and from the extraction wells installed as the hydraulic containment component of the LTS. Groundwater quality data from these wells will be compared to results from the baseline sampling event and will be evaluated for changes over time to determine the adequacy of leachate controls implemented at the site. Based on historical data it is likely that VOC samples from multiple monitoring wells, including, but not limited to MW-134A, MW-134B, MW-105A, MW-105B, MW-135A. MW-144, MW145, and MW-106A may be medium concentration during the early sampling events. The data will also be evaluated to determine if the frequency of monitoring can be reduced to semiannually depending on consistency in groundwater concentrations and approval of Illinois EPA.

The monitoring and extraction well locations are shown in **Figures 2 and 3**, respectively. All monitoring wells will be sampled to provide a synoptic round of water level measurements and groundwater quality conditions over time. All groundwater samples will be analyzed for TCL VOCs by U.S. EPA CLP or VOCs by U.S. EPA Region 5 ASB laboratory using SOP MS023. The list of

VOCs analyzed by ASB laboratory is provided in the method SOP in **Appendix B** of the QAPP. The 25 monitoring wells will also be analyzed for 1,4-dioxane by U.S. EPA Region 5 ASB laboratory using SOP MS035 or by CLP using CLP SOW SFAM01.1 SVOA SIM modified by Method 3054.0.

Groundwater samples from the 14 extraction wells will also be analyzed for total TAL inorganics by U.S. EPA CLP or by U.S. EPA Region 5 ASB laboratory using SOP Metals003A ICP Metals – Thermo to evaluate any potential impacts of inorganic compounds on the LTS operation.

4.1.3 LTS Influent and Effluent Sampling

To evaluate LTS performance and provide confirmation that the effluent discharged to the intermittent creek met the discharge limits, the influent and effluent was sampled monthly for the first year of operation after the LTS was declared operational and functional. Quarterly sampling began in December 2020 and will continue for the duration of LTS operation. The influent sample is collected from the sampling port located after the equalization tank, where the liquid streams from the multi-phase extraction wells and groundwater extraction wells are combined. The samples from this location are expected to be medium concentration. The effluent sample is collected from the effluent sample port located after the second set of bag filters, just prior to the LTS effluent discharge point. The sample for dissolved metals is collected through a filter attached to sample tubing which will be attached to the sample port. The influent and effluent samples will be analyzed for VOCs and total inorganics by U.S. EPA CLP or U.S. EPA Region 5 ASB laboratory. The effluent samples will also be analyzed for dissolved inorganics and 1,4-dioxane by U.S, EPA CLP or U.S. EPA Region 5 ASB laboratory. The effluent results will be compared to the discharge limits provided in the QAPP. The data from the influent and effluent samples will be used to calculate the treatment system performance efficiency and contaminant mass removal.

Table 4-1. Area 7 GMZ Groundwater Monitoring and Extraction Wells

Well ID	Screened Interval (bgs)	Total Depth (bgs)
MW-103A	31 - 41	41
MW-103B	65 - 75	75
MW-103C	97.9 - 107.9	107.9
MW-103D	190.5 - 200.5	200.5
MW-105A	12 - 22	22
MW-105B	44 - 54	54
MW-105C	85 - 95	95
MW-105D	146.5 - 156.5	156.5
MW-106A	30.5 - 40.5	40.5
MW-106B	76.4 - 86.4	86.4

Well ID	Screened Interval (bgs)	Total Depth (bgs)
MW-106C	109.4 - 119.4	119.4
MW-112A	25 - 35	35
MW-112B	85 - 95	95
MW-112C	290 - 300	300
MW-134A	23 - 28	28
MW-134B	40 - 45	45
MW-134C	58 - 63	63
MW-135A	24 - 34	34
MW-135B	70 - 80	80
MW-143	65 - 75	75
MW-144	75 - 85	85
MW-145	36 - 46	46
MW-147	74 - 84	84
MW-148	89 - 99	99
MW-149	65 - 76	75
EWG-001I	46 - 61	66.5
EWG-002S	30 - 40	45.5
EWG-003S	28 - 38	43.5
EWG-004I	36 - 56	61.5
EWG-005I	29 - 49	54.5
EWG-006I	32 - 42	47.5
EWG-007I	30 - 60	65.5
EWG-008D	73 - 93	98.5
EWG-009D	73 - 93	93.5
EWG-010I	38 - 58	63.5
EWG-011D	46-51 & 66-96	96.5
EWG-012D	Org35 - 75	80.5
EWG-013D	60 - 90	95.5
EWG-014D	60 - 90	90.5

Notes:

bgs = below ground surface
MW – groundwater monitoring well
EWG – groundwater extraction well

Section 5

Sampling Equipment and Procedures

5.1 Water Level Measurements

Water level measurements were collected from all GMZ monitoring wells prior to LTS start-up in April 2019 and sampled quarterly thereafter for a period of five years. Water levels will be measured manually with downhole water level monitoring equipment as per the procedures in CDM Smith SOPs 1-6, Groundwater Level Measurement and SOP 5-1, Control of Measurement and Test Equipment and procedures in this SAP.

All monitoring equipment will be tested in the field according to the manufacturer's instructions prior to use. The following steps will be followed when taking water level measurements using electrical tapes:

1. Before lowering the probe into the well, the circuitry shall be checked by dipping the probe in tap water and checking to ensure that the signaling device responds to probe submergence. The probe shall then be lowered slowly into the well until contact with the water surface is indicated by an electronic "beep" and/or light. The electrical tape reading is made at the measuring point.

2. Take a second and third check reading to verify the measurement before completely withdrawing the tape from the well.

3. Decontaminate the electrical tape before proceeding to the next well to minimize cross contamination.

5.2 Groundwater Sampling

Groundwater samples will be collected from the Area 7 GMZ monitoring wells and from the north-south line of hydraulic containment extraction well as shown in **Figures 2 and 3**. The wells were sampled in April 2019 to provide a synoptic round of water level and groundwater quality measurements. The groundwater sampling will be conducted quarterly for a period of five years for a total of twenty rounds of sampling. For samples scheduled to be analyzed by the ASB laboratory, pertinent information is provided in **Appendix B** of the QAPP.

5.2.1 Monitoring Well Sampling

For the Area 7 GMZ monitoring wells, each monitoring well will be purged using a submersible pump and pump controller or peristaltic pump capable of operating at low-flow purging rates (500 ml/min or less). Use of a peristaltic pump is necessary at some sampling locations because bent or otherwise obstructed monitoring well riser prevent the insertion of a submersible pump. The flow rate will be low enough to ensure that the purge water is not visibly agitated, and the turbidity is minimized. The wells requiring use of a peristaltic pump include but may not be

limited to MW-105 A, C, and D, MW-106C and MW-112A. Narrow-diameter bladder pumps will be used for damaged wells with water depths that exceed the suction-lift ability of a peristaltic pump which may include MW105D and MW-106C. For all wells being sampled, field measurements of pH, temperature, specific conductance, dissolved oxygen (DO), turbidity, and oxygen reduction potential (ORP) will be taken using a multi-parameter probe. The probe will be contained within a flow-through cell, and purged water will be passed through this cell. Parameter readings will be recorded at five- minute intervals. Purging will continue until the following conditions are met:

- Field measurements have stabilized (pH ± 0.25 standard units, dissolved oxygen 10 percent, specific conductance ± 50 µmhos/cm, turbidity less than 5 NTUs or 10 percent, temperature ± 0.5^{o} C).
- A maximum of three well volumes have been purged from the well.

New or dedicated pump tubing will be used for each discrete sampling location. The groundwater sample will be collected directly from the pump discharge tubing into pre-preserved sample containers. All groundwater samples from GMZ monitoring wells will be analyzed for low/medium concentration VOCs and 1,4-dioxane by U.S. EPA CLP or U.S. EPA Region 5 ASB laboratory. Based on historical data it is likely that VOC samples from multiple monitoring wells including but not limited to MW-134A, MW-134B, MW-105A, MW-105B, MW-135A. MW-144, MW145, and MW-106A may be medium concentration during the early sampling events.

U.S. EPA CLP field sampling protocols, chain-of-custody and shipping procedures will be used for all sample collection. CDM Smith SOPs 1-12, Low Stress (Low Flow) Groundwater Sampling, SOP 4-3. Well Development and Purging and SOP 1-10, Field Measurement of Organic Vapors will also be used for reference.

5.2.2 Extraction Well Sampling

The extraction wells will be sampled from a tap on the waterlines that run to the LTS. Flow from the tap will be kept as low as possible during sample collection. Samples from the extraction wells will be analyzed for total inorganics and VOCs by U.S. EPA CLP or U.S. EPA Region 5 ASB laboratory. U.S. EPA CLP field sampling protocols, chain-of- custody and shipping procedures will be used for all sample collection. CDM Smith SOP 1-10, Field Measurement of Organic Vapors and CDM Smith SOP 1-9, Tap Water Sampling will also be used for reference.

5.3 LTS Influent and Effluent Sampling

The influent sample will be collected from the sampling port after the equalization tank where the incoming liquid streams are mixed prior to treatment. The effluent will be sampled directly from the sample port on the LTS discharge line located after the second set of bag filters and immediately prior to discharge into the intermittent creek. The sample for dissolved metals will be collected through a filter attached to disposable sample tubing which will be attached to the

sample port. Influent and effluent samples will be analyzed by CLP or ASB for total inorganics, and VOCs. The effluent samples will also be analyzed for dissolved inorganics and 1,4-dioxane by CLP or U.S. EPA Region 5 ASB laboratory. U.S. EPA CLP field sampling protocols, chain-of-custody and shipping procedures will be used for all sample collection. CDM Smith SOP 1-10, Field Measurement of Organic Vapors and CDM Smith SOP 1-9, Tap Water Sampling will also be used for reference. For samples scheduled to be analyzed by the ASB laboratory, pertinent information is provided in **Appendix B** of the QAPP.

Section 6

Decontamination Procedures

Procedures to be followed to decontaminate equipment and personnel are fully described in the Area 7 GMZ Monitoring Activities HASP provided in **Appendix A** of this SAP and CDM Smith SOP 4-5, Field Equipment Decontamination. The procedures are summarized below.

6.1 Personnel Decontamination

Personnel decontamination stations will be set up near the LTS treatment building. Personnel will become thoroughly familiar with the decontamination procedure before work begins in exclusion zones. The exclusion zone will be defined as an area 25 feet surrounding the groundwater collection points. The decontamination procedure is as follows:

- Place equipment and/or samples in segregated equipment drop-areas
- Remove disposable outer booties (when used)
- Remove chemical resistant outer gloves
- Remove hard hat, goggles/safety glasses/face shield
- Remove inner disposable gloves
- Wash hands and face with water and hand soap

6.2 Equipment Decontamination

All reusable, non-dedicated sampling equipment will be decontaminated prior to use. Between samples, the groundwater pump will be decontaminated. Water level measurement equipment will be decontaminated by scrubbing the equipment in a low-sudsing detergent solution, rinsing the equipment in tap water, and air drying the equipment.

All reusable, non-dedicated equipment will be decontaminated between samples, and before removal from the site. The procedure is given in **Table 6-1**.

6.3 Sample Bottle Decontamination

Sample bottles for shipment to the laboratories will be decontaminated by immersing the bottle up to the neck in soap (Alconox or equivalent) and water solution and then rinsing with potable or distilled water. Solvents will not be used to wash sample bottles.

Table 6-1. Standard Decontamination Protocol for Sampling Equipment

STEP 1:	Scrub equipment thoroughly with soft-bristled brushes in a low-sudsing detergent solution.
STEP 2:	Rinse equipment with tap water by submerging and/or spraying.
STEP 3:	Rinse equipment with distilled water by spraying until dripping.
STEP 4:	Place equipment on plastic or aluminum foil and allow to air dry for 5 to ten minutes.
STEP 5:	Wrap equipment in plastic or aluminum foil for handling and/or storage until next use.

NOTE: In order to avoid analytical problems caused by solvent use in decontamination, solvents will not be used for decontamination. Only distilled water shall be used for rinsing equipment. An exception will be made if upon visual observation or high organic vapor readings it is determined that a zone of highly contaminated material is encountered. In such an event, isopropyl alcohol will be used before step 1 above.

6.4 Storage and Disposal of Investigation-Derived Waste

The sampling activities are expected to produce liquid investigation-derived waste (IDW). Plastic and personal protective equipment (PPE) such as plastic sheeting, tubing, disposable sampling equipment, gloves, and overboots, will also be generated during sampling activities. The activities, the anticipated type, and the planned handling of the wastes are summarized below.

- Groundwater purging and sampling: LIQUIDS: Contain purge water in 55-gallon DOT approved drums for transport and disposal at Source Area 7 groundwater treatment system.
- PPE. Dispose of as common refuse.

At the end of each day, liquid IDW will be transported to the Source Area 7 groundwater treatment system for treatment and disposal. Solid IDW will be moved to a secure waste storage area designated by Illinois EPA. It is estimated that 4 drums will be required for the purge and decontamination water per sampling event. The procedures described in CDM Smith SOP 2-2, Guide to Handling Investigation-Derived Waste will also be used as a guideline for IDW handling.

Illinois EPA will be responsible for signing all manifests for hazardous waste transport and disposal.

Section 7

Field Quality Control Procedures

To ensure the level of data quality required for Superfund related sampling events, the following Quality Control (QC) procedures will be performed. QC sample requirements are summarized in **Table 1-1**.

7.1 Groundwater QC Samples

In order to reduce bias in the laboratory measurement systems, field QC samples (with the exception of MS and MSDs) will be labeled as separate sample locations and will not contain any prefixes or suffixes that identify the sample as a QC sample. The field QC samples will be documented as such and cross referenced in the field notebook to allow for appropriate data assessment.

7.1.1 Field Duplicates

Duplicate samples will be collected as follows:

- One duplicate effluent sample will be collected for the first sampling event each year. If upon data review the sample and duplicate agreement is not within the RPD provided in the QAPP, field procedures will be reviewed and adjusted if necessary.
- One duplicate groundwater sample for every 10 or fewer investigative samples collected in the field.

7.1.2 Equipment Blanks

One equipment blank water sample will be prepared for every ten or fewer investigative groundwater samples from monitoring wells. Equipment blanks will be prepared by pouring distilled water over/through the sampling pump and into the sample bottles, at the same volume as the groundwater samples. Equipment blanks for all monitoring well parameters will be prepared to check the adequacy of the field decontamination procedures. The equipment blank sample locations will be recorded in the field log book.

7.1.3 Trip Blanks

Trip blanks for VOC analysis will be included in each sample shipment that includes liquids for volatile organic analysis. For VOCs, the trip blank will consist of four 40-ml VOA vials for CLP and three 40-ml VOA vials for ASB, filled with reagent-grade distilled water and preserved with HCL to a pH less than 2. The trip blank will be prepared in the office or laboratory, transported to the field, and shipped with the other samples to the CLP or ASB laboratory without being opened. For 1,4- dioxane samples trip blanks will be prepared for samples being analyzed by the U.S. EPA Region 5 ASB laboratory only. The trip blanks will be prepared by the U.S. EPA Region 5 ASB laboratory, transported to the field and shipped with the other samples to the ASB laboratory. For

1,4-dioxane samples scheduled to be analyzed by the CLP, no trip blank will be prepared or included with the sample shipment as the CLP analysis method is a semi-volatile method and does not require trip blanks for field QC.

7.1.4 Matrix Spike, Matrix Spike Duplicates, and Laboratory Duplicates

All samples designated as matrix spike/matrix spike duplicate (MS/MSD) samples for CLP/ASB VOC and ASB 1,4-dioxane analysis will be collected at double sample volume. For CLP 1,4-dioxane MS/MSD analysis, five 1 – liter amber bottles are required. All samples designated as matrix spike samples for inorganic matrix spikes and laboratory duplicates will be collected at double the sample volume. MS/MSD, MS and laboratory duplicate samples will be indicated by noting it on the TR/COC forms.

Groundwater samples collected for VOC and 1,4-dioxane MS/MSD, inorganic MS and laboratory duplicate analysis will be collected at a frequency of one per 20 samples. MS/MSD samples will be selected from locations that are known or suspected to be contaminated, if possible, as per the EPA guidance *Contract Laboratory Program Guidance for Field Samplers*. The influent samples collected for inorganics and VOCs will have an inorganic MS and VOC MS/MSD collected for all sampling events as this location will have a greater level of contamination than the effluent sample location. The effluent samples will have an MS/MSD collected for 1,4-dioxane for all sampling events.

Figures

Site Boundary

0 175 350 700 Feet

N
W E
S

CDM Smith

Figure 1
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Monitoring Wells

Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 2
Monitoring Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Extraction Wells

Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 3
Extraction Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Extraction Wells

Monitoring Wells

GMZ and Site Boundary

0 175 350 700 Feet

N W E S

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Figure 4
Groundwater Management Zone Boundary
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Appendix A

Health and Safety Plan (HASP)

Appendix B

CDM Smith Technical SOPs

Figures

Site Boundary

0 175 350 700 Feet

N
W E
S

CDM Smith

Figure 1
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Monitoring Wells

Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 2
Monitoring Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Extraction Wells

Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 3
Extraction Well Locations
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Extraction Wells

Monitoring Wells

GMZ and Site Boundary

0 175 350 700 Feet

N W E S

CDM Smith

Figure 4
Groundwater Management Zone Boundary
SERGC Superfund Site - Source Area 7
Rockford, Winnebago County, Illinois

Appendix A

Health and Safety Plan (HASP)

HEALTH AND SAFETY PLAN FORM

CDM Smith Health and Safety Program

This document is for the exclusive use of CDM Smith and its subcontractors

CDM Smith

PROJECT NAME	Area 7 SE Rockford Superfund	**PROJECT#**	1681-239358	**REGION**	Central
SITE ADDRESS	11th Street to Alpine Road (West to East)	**CLIENT ORGANIZATION**		Illinois EPA	
	Harrison Ave. to Sawyer Road Ext. (North to South)	**CLIENT CONTACT**		Brian Conrath	
	Rockford, IL	**CLIENT CONTACT PHONE #**		(217) 557-8155	

(x) AMENDMENT TO EXISTING APPROVED H&SP?

(x) H&SP AMENDMENT NUMBER? 3 **(x) DATE OF PREVIOUS H&SP APPROVAL** 3/20/2020

OBJECTIVES OF FIELD WORK:
(e.g. collect surface soil samples):

1. Groundwater sampling at existing monitoring wells.
2. Field Data Coordinator (FDC) will be in charge of sample management, labels, ensuring all samples are intact and accounted for, run errands for field personnel if needed, as well as helping out sampling teams if they are experiencing issues.
3. Conducting RA oversight of the process contractor: submittal review, field oversight, progess meeting attendance, request for information processing, change order review, inspections and testing, application for payment review, varying stages of completion review/inspection, and initial start-up/shake-down of system operation.
4. CDM Smith will oversee some activities in areas known to not be contaminated. For example, inspection of electrical or mechanical connections in vaults or in building.
5.

SITE TYPE: *Check as many as applicable*

Active	(X)	Landfill	( )	Unknown	( )
Inactive	( )	Uncontrolled	( )	Military	( )
Secure	( )	Industrial	( )	Other (specify)	(X)
Unsecure	(X)	Recovery	( )	Remediation site	
Enclosed space	( )	Well Field	( )		

All requirements described in the CDM Smith Health and Safety Manual are incorporated in this health and safety plan by reference.

SEE PAGE 10 FOR PROJECT-SPECIFIC COVID-19 SAFETY AWARENESS PROCEDURES

PERSONNEL AND RESPONSIBILITIES

NAMES OF WORK CREW MEMBERS	Company / Division / Office	Current Training & Medical?	Project or Site Responsibilities	Tasks On Site?
John Grabs	CDM-TSU-Chicago	Yes	Project Manager	1,3-4
Tian Liao	CDM-TSU-Edison	Yes	Field Task Manager; Site H&S	1-2
Justin McCane	CDM-TSU-Louisville	Yes	FIeld Personnel	1
Matt Garbarino	CDM-TSU-Lexington	Yes	Field Personnel	1
Nick Revetta	CDM-TSU-Columbus	Yes	Field Personnel	1
EDI Personnel	EDI-Chicago	Yes	Field Personnel	1

BACKGROUND REVIEW: (X) Complete ( ) Incomplete

SITE MAP: *Show Exclusion, Contamination Reduction, and Support Zones. Indicate Evacuation and Reassembly Points*

HEALTH AND SAFETY PLAN FORM
CDM Smith Health and Safety Program

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HISTORY: *Summarize conditions that relate to hazard. Include citizen complaints, spills, previous investigations or agency actions, known injuries, etc.*

Groundwater contamination by Volatile Organic Compounds (VOCs) was originally discovered by the City of Rockford in 1981. Subsequent investigations performed between 1981 and 1997 revealed dissolved and non-aqueous phase VOC contamination in groundwater, as well as VOC contamination in soil and soil gas. The VOCs of concern are primarily chlorinated solvents (primarily 1,1,1-Trichloroethane), although benzene, toluene, ethylbenzene, and xylene (BTEX) are also a potential concern.

Continued groundwater monitoring is necessary to define the nature and extent of the contamination.

WASTE TYPES: ( ) Liquid ( ) Solid ( ) Sludge ( ) Gas ( ) Unknown (**X**) Other, specify: Potential LNAPL

WASTE CHARACTERISTICS: *Check as many as applicable.*

( ) Corrosive (**X**) Flammable ( ) Radioactive
( ) Toxic (**X**) Volatile ( ) Reactive
( ) Inert Gas ( ) Unknown

() Other: _____

WORK ZONES: Exclusion Zone (EZ) - 20 feet around sampling point during groundwater sampling.
Contamination Reduction Zone (CRZ) - Adjacent to the EZ.
Support Zone - Field vehicles.

The EZ will be strictly limited to CDM personel, their subcontractors, and persons approved by the CDM site supervisor. All persons in the EZ must wear required PPE, have current OSHA safety training and be under a medical monitoring program. No untrained or unapproved visitors will be allowed in the EZ.

HAZARDS OF CONCERN: *Check as many as applicable.*

(X) Heat Stress CDMS Guideline ( ) Noise CDMS Guideline
(X) Cold Stress CDMS Guideline ( ) Inorganic Chemicals
(X) Explosive/Flammable (X) Organic Chemicals
( ) Oxygen Deficient (X) Motorized Traffic
( ) Radiological ( ) Heavy Machinery
(X) Biological - ticks, biting insects (X) Slips & Falls CDMS Guideline
(X) Other: COVID-19 (See attached COVID-19 Safety Guidelines)
( ) Other: _____

FACILITY'S PAST AND PRESENT DISPOSAL METHODS AND PRACTICES:

Various locations: Disposal methods and practices appear to be typical of a residential/light industrial area. It is hypothesized that materials were trucked to the area and dumped directly onto the ground surface and in areas of natural depressions. Areas are serviced by a city water and sewer system.

This plan incorporates CDM Smith's procedure for: *(Click on the relevant topics to download the hazard guideline. Delete irrelevant topics.)*

Housekeeping
Traffic and Work Zone Safety
Tools and Power Equipment
Manual Material Handling
Hazardous Waste Site Controls
Flammable and Combustible Liquids
Hazardous Waste Site Decontamination

16.2 Housekeeping

These guidelines are for the establishment and administration of a clean and orderly work environment at field project sites. A continuous housekeeping program strongly tends to prevent accidents. A clean and orderly work environment can be achieved and maintained through ongoing housekeeping efforts undertaken by personnel at all levels. Project managers shall initiate participation in housekeeping activities and good work habits, not only at the end of a work assignment but throughout the evolution of the project.

- To achieve these benefits, the team shall plan the location of equipment and storage facilities to allow the easy flow of personnel, equipment, materials, fire hazards, and to prevent the obstruction of evacuation, fire fighting, or rescue activities.
- Store materials in a manner that facilitates access of material handling equipment and personnel handling limitations. Lack of sufficient workspace and storage capacity leads to the potential for accidents and decreases efficiency.
- Avoid storage of flammable liquids, such as paints and thinners, unless they are required for specific project needs. If needed, such storage shall be within a metal storage cabinet that has been labeled and approved for the storage of flammable liquids.
- Continuously maintain work areas in a neat and orderly manner.
- Containers should be provided for the collection of waste, trash, and other nonhazardous refuse. Investigation-derived waste and other waste materials that are potentially hazardous should be stored and labeled in accordance with project-specific procedures that meet regulatory and client requirements.
- Deploy leads, hoses, and extension cords so they do not present tripping hazards and are not subject to contact with moisture or physical stress. Where possible, they should be hung overhead with nonconductive material and kept away from walkways, doors, stairs, and ladders.
- Protect protruding rebar and anchor bolts and conspicuously mark them.
- Clean small spills that create slip hazards and/or flammability hazards immediately and do not leave them unattended.
- Keep walkways, aisles, stairways, and passageways in a clear and unobstructed condition.
- Prohibit eating and drinking in work areas where there is potential exposure to toxic or hazardous materials. Smoking is limited to designated smoking areas where there is no such exposure.

16.3 Manual Material Handling

CDM Smith employees should follow the work practices outlined below when lifting and carrying heavy objects.

- Test any load they are required to lift and compare its weight, volume, and shape to their lifting abilities. Employees shall not attempt to lift beyond their capacity.
- Obtain assistance in lifting heavy objects. Back belts or back braces may be used if desired; however, many ergonomists do not believe that these devices create a benefit or provide protection.
- When two or more persons are involved in a manual lift, one person should provide direction of the lift.
- When two or more persons are carrying an object, each employee, if possible, should face the direction in which the object is being carried.
- When two or more persons carry a heavy object that is to be lowered or dropped, there shall be a prearranged signal for releasing the load.
- The right way to lift is easiest and safest. Crouch or squat with the feet close to the object to be lifted, secure good footing, take a firm grip, bend the knees, keep the back vertical, and lift by bending at the knees and using the leg and thigh muscles. Exercise caution when lifting or pulling in an awkward position.
- Employees should avoid twisting or excessive bending when lifting or setting down loads.
- When moving a load horizontally, employees should push the load rather than pull.
- For tasks that require repetitive lifting, the load should be positioned to limit bending and twisting. The use of lift tables, pallets, and mechanical devices should be considered.
- When gripping, grasping, or lifting an object such as a pipe or board, the whole hand and all the fingers should be used. Gripping, grasping, and lifting with just the thumb and index finger should be avoided.

16.12 Tools and Power Equipment

16.12.1 Hand Tools

CDM Smith employees who have a need to use basic hand tools should use the following work practices:

- All tools used on CDM Smith projects, regardless of ownership, shall be of an approved type and maintained in good condition. Tools are subject to inspection at any time. The project manager has the authority and responsibility to condemn unserviceable tools, regardless of ownership.
- Tag defective tools to prevent their use or removal from the job site.
- Use the proper tool for the job performed.
- Do not use hammers with metal handles, screwdrivers, knives with metal continuing through the handle, and metallic measuring tapes on or near energized electrical circuits or equipment.
- Do not throw tools from place to place or from person to person. Tools that must be raised or lowered from one elevation to another shall be placed in tool buckets or firmly attached to hand lines.
- Do not place tools unsecured on elevated places.
- Dress, repair, or replace all impact tools such as chisels, punches, drift pins, etc., that become mushroomed or cracked before further use.
- Use suitable holders or tongs, not the hands, to hold chisels, drills, punches, ground rods, or pipes that are struck by another employee.
- Do not use shims to make a wrench fit.
- Do not use wrenches with sprung or damaged jaws.
- Do not use pipe or other means to extend a wrench handle for added leverage unless the wrench was designed for such use.
- Use tools only for the purposes for which they have been designed.
- Store and handle tools with sharp edges so that they will not cause injury or damage. They shall not be carried in pockets.
- Use eye protection when using or working around impact type tools (e.g., hammer, chisel, ax, hatchet, etc.).
- Replace wooden handles that are loose, cracked, or splintered. The handle shall not be taped, glued, or lashed with wire.

- Keep all cutting tools such as saws, wood chisels, knives, or axes in suitable guards or in special compartments.
- When using such tools as screwdrivers and wrenches, avoid using your wrists in a bent, flexed, extended, or twisted position for long periods of time. Employees should maintain their wrists in a neutral or straight position.
- Do not leave tools lying around where they may cause a person to trip or stumble.
- When working on or above open grating, use a canvas or other suitable covering to cover the grating to prevent tools or parts from dropping to a lower level where others are present, or barricade or guard the danger area.
- Do not depend on the insulation on hand tools to protect users from shock.

16.12.2 Electric Tools

CDM Smith employees who have a need to use electric power tools should use the following work practices:

- The non-current carrying metal parts of portable electric tools such as drills, saws, and grinders shall be effectively grounded when connected to a power source unless the tool is an approved double-insulated type or the tool is connected to the power supply by means of an isolating transformer or other isolated power supply, such as a 24-volt DC system.
- All power tools shall be examined before use to ensure general serviceability and the presence of all applicable safety devices. The electric cord and components shall be given a thorough examination for cracks, exposed wires, or other defects.
- Power tools shall be used only within their capability and shall be operated in accordance with the manufacturers' instructions.
- The use of eye protection is required when using or working around power tools.
- Operators should take care to use appropriate hand positions on cutting tools such as saws, drills, or grinders to avoid hand injury.
- All tools shall be kept in good repair and disconnected from the power source while repairs are being made.
- Electrical tools shall not be used where there is a hazard of flammable vapors, gases, or dusts until that hazard is firmly under control.
- GFCI should be used with all electric power tools.
- All guards and safety interlocks with which the tools were purchased shall be in place and in working order.

- Any tool that is identified as defective should be tagged "not for use," and set aside for repair and/or discarded.
- Do not wear loose or frayed clothing while operating power tools and equipment. Hair should not stick out from hard hats.
- Do not use electrical cords to transport, suspend, hoist, or lower tools.
- Do not allow power cords to lie in water.
- Disconnect rotating tools from the power source before adjusting, servicing, or cleaning them. Follow the lockout procedure described in Section 16.5.
- Do not modify tools.

16.12.3 Pneumatic Tools

CDM Smith employees that use pneumatic power tools should use the following work practices:

- Compressed air and compressed air tools shall be used with caution.
- Pneumatic tools shall never be pointed at another person.
- Pneumatic hose connections should be secured by some positive means to prevent them from becoming accidentally disconnected. Chicago fittings have wire holes to allow such security.
- Pneumatic power tools shall be secured to the hose by some positive means to prevent the tool from becoming accidentally disconnected.
- Safety clips or retainers shall be securely installed and maintained on pneumatic impact tools to prevent attachments from being accidentally expelled.
- Compressed air shall not be used for cleaning purposes except when reduced to less than 30 psi and then only with effective chip guarding and PPE.
- Compressed air shall not be used to blow dust or dirt from clothing (or skin).
- The manufacturer's safe operating pressure for hoses, pipes, valves, filters, and other fittings shall not be exceeded.
- The use of hoses for hoisting or lowering tools shall not be permitted.
- All compressed air hoses exceeding 30 psi shall have a safety device at the source of supply or branch line to reduce pressure in case of hose failure or disengagement of a connection.

- Before making adjustments or changing air tools, the air shall be shut off at the air supply valve ahead of the hose. The hose shall be bled at the tool before breaking the connection. Disconnection at the quick-change connectors is one way to meet this goal.
- Eye protection is required when using or working around pneumatic tools.
- Use hearing protection if noise exposure is a concern (i.e., if it is too loud to conduct a normal conversation).
- Pneumatic tools shall be operated only by persons trained in their use.
- A pneumatic tool used where it may contact exposed live electrical parts shall have a nonconductive hose and an accumulator to collect moisture.
- Employees shall not use any part of their bodies to locate or attempt to stop an air leak.
- All guards and safety interlocks must be in place and functional.

16.12.4 Engine-Powered Tools

CDM Smith employees that use engine-powered tools should use the following work practices:

- Stop the engine and allow it to cool before refueling, servicing, or maintenance.
- Use care in refueling. Clean up any small spills of fuel or oil immediately.
- The use of eye protection is required when using or working around engine-powered tools.
- Use hearing protection if noise exposure is a concern (i.e., if it is too loud to conduct a normal conversation).
- If possible, disconnect the spark plug before performing an adjustment, maintenance, or service.
- Use tools in well ventilated areas to eliminate any accumulation of fumes.
- Do not use tools in a flammable or explosive atmosphere.
- Equip engines with spark-arresting mufflers.
- Avoid contact with hot engine components.
- All guards and safety interlocks should be in place and functional.

16.17 Flammable and Combustible Liquids

Work with flammable or combustible liquids exposes the employees to fire, explosion, and toxicity hazards. They should implement the following controls.

16.17.1 Storage and Handling

- Only approved containers and portable tanks should be used for the storage and handling of flammable and combustible liquids.
  - Approved safety cans shall be used for the handling and use of flammable liquids in quantities greater than 1 gallon.
  - For quantities of 1 gallon or less, only the original container or approved safety cans shall be used for storage, use, and handling of flammable/combustible liquids.
  - The requirements for shipping these liquids exceeds those described here. If flammable or combustible liquids must be shipped, the individual offering the material for shipment must have completed DOT Hazardous Material Training. Contact your HSM for information on DOT training.
- Flammable or combustible liquids shall not be stored near exits, stairways, or pathways that people normally use for safe passage.
- No more than 25 gallons of flammable/combustible liquids shall be stored in a room outside of a storage cabinet or tank approved for the purpose.
- Quantities of flammable and combustible liquids in excess of 25 gallons shall be stored in an acceptable or approved cabinet meeting the requirements of 29 CFR 1926.152(b)(2)(i).
- Cabinets shall be labeled in conspicuous lettering, “Flammable - Keep Fire Away.”
- Not more than 60 gallons of flammable or 120 gallons of combustible liquids shall be stored in any one storage cabinet. Not more than three cabinets may be located in a single storage area.

16.17.2 Outdoor Storage

- For storage of flammable and combustible liquids outdoors, containers (not more than 60 gallons each) shall not exceed 1,100 gallons in any one pile or area. Five feet of clearance shall separate piles or groups of containers. These containers shall remain at least 20 feet from any other building or structure.
- Within 200 feet of each pile of containers, there shall be a 12-foot wide access way to permit approach of fire control apparatus.

- The storage area shall be graded in a manner to divert possible spills away from buildings or other exposures, or shall be surrounded by a curb or earth dike at least 12 inches high. Provisions shall be made for the controlled draining of accumulations of groundwater or rainwater, or spills of flammable or combustible liquids when curbs or dikes are used.

- At least one portable fire extinguisher, having a rating of not less than 20 pounds, shall be located not less than 25 feet or more than 75 feet from any flammable or combustible liquid storage area located outdoors.

- Precautions shall be taken to prevent the ignition of flammable/combustible vapors. Sources of ignition include, but are not limited to: open flames; lightning; smoking; cutting and welding; hot surfaces; frictional heat; static, electrical, and mechanical sparks; spontaneous ignition, including heat-producing chemical reactions; and radiant heat.

16.17.3 Dispensing Flammable and Combustible Liquids

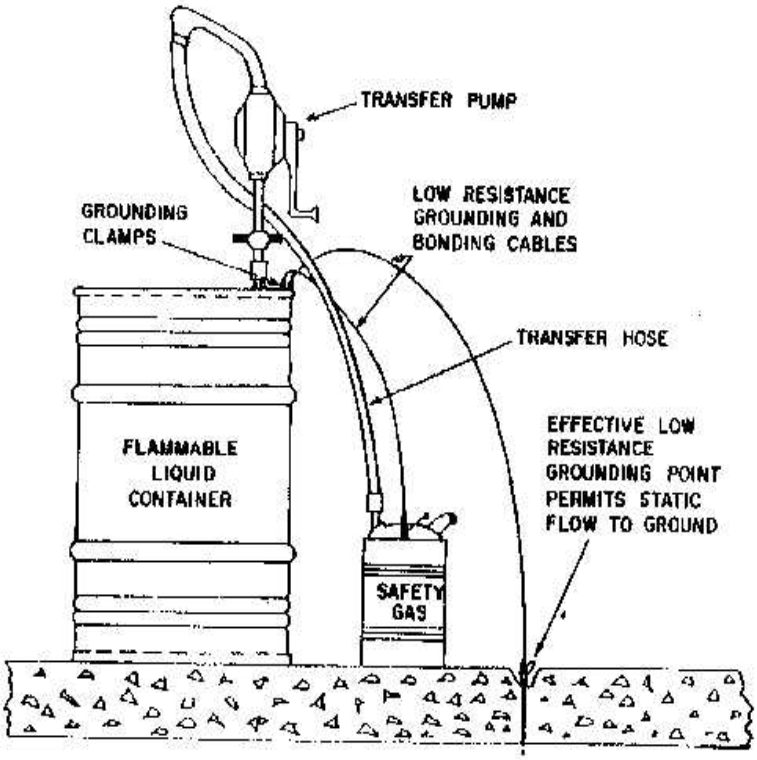
- Areas where flammable or combustible liquids are dispensed at one time, in quantities greater than 5 gallons from one tank or container to another tank or container, shall be separated from other operations by a distance of 25 feet or by construction having a fire resistance of at least 1 hour. Adequate natural or mechanical ventilation shall be provided to maintain the concentration of flammable/combustible vapor at or below 10 percent of the LEL.

- Static electricity is generated by the contact and separation of dissimilar material, such as when fluid flows through a pipe or from an orifice into a tank. If the accumulation of static charge is sufficient, a static spark may occur. Transfer of flammable/combustible liquids from one container to another should be done only when containers are electrically bonded and grounded to prevent such accumulation of static charge (Figure 16-3).

- The management of flammable and combustible liquids is much more complicated than is indicated by the length of this section, which reviews only those issues appropriate to the incidental use of these materials.

- Storage and handling of the mobile and combustible liquids should comply with the requirements of National Fire Code No. 30 (see http://www.nfpa.org/Codes/NFPA_Codes_and_Standards/List_of_NFPA_documents/NFPA_30.asp – you will need a password from the CDM Smith Infocenter).

Figure 16-1
Typical Grounding System



16.20 Hazardous Waste Site Controls

Work sites designated as hazardous waste sites must control access to the work area to only authorized personnel and conform to general work practices expected at hazardous waste site operations as required by the OSHA Standard for Hazardous Waste Operations, 29 CFR 1910.120. The following concepts should be reflected in the HSP for the project.

16.20.1 Access Control

Controlled access to hazardous waste site work areas is required to protect personnel working on the site as well as to limit the potential for transporting contaminants off site. Depending on the size of the work site, hazards and contaminants present, and complexity of the work, access control may range from verbally cautioning nonauthorized personnel to stay away from the work area, to a program including site security, signs, or formal sign-in and sign-out procedures. Details of site-specific access control procedures should be included in the site-specific HSP. Some general work practices for access control are noted below:

For small-scale site investigations that are short-term projects (i.e., days, not weeks or months), identify a work area to the work crew and keep persons not associated with the job site out of the work area. If the site is in an area where nonauthorized persons are likely to be encountered, traffic cones, caution tape, and signs identifying the area as a controlled access area may be used.

For more extensive projects where work may be done for weeks or longer, the team should deploy more extensive access controls. They should:

- Set up physical barriers and hire security personnel to prevent nonauthorized persons from entering the work site.
- Keep the number of personnel and equipment on site to the minimum required to do the project effectively and safely.
- Establish work zones within the site (Section 16.20.2).
- Establish controlled access points to be used by authorized personnel.
- Track the entry and exit of personnel through a check-in, checkout system.
- Establish a formal decontamination corridor from exclusion zones.

16.20.2 Work Zones

Field project managers working under HSPs for hazardous waste operations are required to establish work zones to prevent or reduce the spread of site contaminants to noncontaminated areas on or off site. Movement between zones should be restricted to those that need access to a specific area, and entry and exit between zones should be through designated access control points. A description of the three work-zone system for hazardous wastes is provided below.

Exclusion Zone – The exclusion zone should include any area where contamination is known or suspected. Areas of air, water, or soil that are contaminated with hazardous materials (biohazards, radioactive materials, chemicals) should be included in the exclusion zone. The zone should be well known to site workers. On smaller projects, this can be a verbal identification to site workers, such as "a 20-foot radius around the drill rig." On larger projects, or in areas that may be encountered by observers or the general public, the zone may need to be defined with caution tape, traffic cones, or in some instances, fencing and barriers. The need will be site-specific and the specific method should be identified in the site-specific HSP. Some work practices that should be followed in the exclusion zone include:

- Employees in the exclusion zone must wear the PPE designated in the site HSP for tasks executed within the zone.
- No eating, drinking, chewing gum or tobacco, smoking, application of cosmetics, including application of lip balm, sunscreen, or insect repellant is allowed in the exclusion zone.
- Sitting or kneeling in areas of high concentrations of contaminants should be avoided.
- If any PPE becomes defective, the employee should leave the work area via the designated egress area, decontaminate as needed, and replace the defective PPE before returning to work in the exclusion zone.
- Prescription drugs should not be used within the exclusion zone unless approved by CDM Smith's medical consultant. The use of illegal drugs or consumption of alcohol is prohibited.
- When leaving the exclusion zone, employees should exit via the designated access/ egress point(s) and follow decontamination procedures described in the site HSP.

Contaminant Reduction Zone – A contaminant reduction zone (CRZ) is established to provide a transition between the exclusion zone and the support zone. The CRZ is set up at the access control points of the exclusion zone and will vary in size depending on the complexity of activities that need to occur within the zone. For small site investigations, the CRZ may simply be a designated area near containers set up to collect used disposable PPE and some soap and water. For larger projects, the CRZ may include specific decontamination points and be staffed by personnel specifically designated to participate in the decontamination of personnel and equipment exiting the exclusion zone. Depending on the site contaminants, level of contamination, and decontamination procedures, personnel in the CRZ may be required to wear protective clothing, gloves, or respirators. The specific requirements will be outlined in the site HSP. The CRZ should be placed in an area that is not contaminated at the boundary of the exclusion zone.

Support Zone – The support zone is established near the entrance to the site and is far enough from the exclusion zone and CRZ that specialized protective clothing or respirators are not used. The use of normal field PPE such as hard hats, safety glasses,

and safety work boots is expected except for areas such as office trailers, break and lunch areas, or other areas designated as having no known or anticipated hazards. Operational support activities and equipment storage and maintenance areas are located in the support zone. No equipment or personnel should go from the exclusion zone to the support zone without passing through the CRZ and being decontaminated in accordance with the site HSP.

Mobile Work Zone – For those projects that involve brief periods of work in multiple locations, a specific area may be designated as the exclusion zone for the duration of the work performed in that area. The exclusion zone can be terminated (provided there are no ongoing hazards or potential exposures to contaminants) and moved to the next area of work. For example, during soil borings or well installation, the exclusion zone can be defined as, "1.5 times the mast height" of the drill rig. Once the boring has been closed, or well installed and secured, and all drill cuttings have been secured, the area can be opened up and a new exclusion zone established around the next boring location.

16.20.3 Considerations when Establishing Work Zones

Work zones should be large enough to perform tasks within the zone safely, with no exposure to hazards to personnel outside the zone, but they should also be small enough to be able to secure and control access. Some considerations in establishing work zones include:

- Physical and topographical features of the site
- Dimensions of the contaminated area
- Weather
- Physical, chemical, and toxicological characteristics of contaminants and chemicals used in the zone
- Potential for exposure to site contaminants
- Known and estimated concentrations of contaminants
- Air dispersion of contaminants
- Fire and explosion potential
- Planned operations and space needed to perform the work safely
- Surrounding areas
- Decontamination procedures
- History of job site

16.20.4 General Hazardous Waste Site Work Practices

- **Buddy System** - Work should be scheduled so that no person works unobserved within the exclusion zone at any time. Each worker within the exclusion zone should maintain visual contact with at least one other worker on the site. All site personnel should remain aware of each other and monitor each other's condition.

- **Eating, drinking, chewing gum or tobacco, and smoking** are prohibited within the contaminant reduction and exclusion zones. (**Exception for heat stress:** Squirt bottles of water, Gatorade, or other fluids may be consumed via squirt bottles in the contaminant reduction zone with the approval of the HSM. Open bottles, cups, etc. should not be permitted.)

- Sitting or kneeling should be avoided in areas of known or suspected areas of contamination.
- Hands and face should be thoroughly washed when leaving the work area.
- Defective PPE should be repaired or replaced immediately.

Sections 5, 6, 7, 9, and 11 of this manual are particularly applicable to H&S at hazardous waste sites.

16.22 Traffic and Work Zone Safety

These guidelines apply whenever CDM Smith employees or subcontractors work in areas exposed to vehicular traffic on public streets or highways.

- Where vehicular traffic hazards exist because of work at locations near public streets or roads, a system of traffic and work zone controls should be developed to mitigate the hazard. The system should meet the requirements of Part 6 of the Manual of Uniform Traffic Control Devices (MUTCD) published by the Federal Highway Administration, or the applicable state version of the MUTCD.

- In general, when the MUTCD allows the use of traffic safety direction devices, such as cones, CDM Smith will supplement those direction devices with a physical barrier, such as a truck.

- All traffic control systems on public roads must be coordinated with local traffic control officials as required by applicable law.

- Periodically evaluate effectiveness of temporary traffic control setups by walking or riding the job area looking for evidence of poor controls and near misses such as swerving traffic, motorists braking quickly, skid marks, blind spots, etc.

- Give motorists plenty of advanced warning of upcoming work zones.

- All employees working within designated work zones or near vehicular traffic should wear high-visibility clothing such as orange, yellow, or yellow-green shirts, jackets, or vests. During wet or inclement weather, similarly colored rainwear should be worn.

- During night work, between the hours of sunset and sunrise, high-visibility clothing should incorporate reflective striping or fabric and be visible at a distance of 1,000 feet. This clothing should meet ANSI standard #107 for High Visibility Safety Apparel.

- All employees working near traffic and vehicles must maintain situational awareness at all times. Stay mindful that warning signs and cones inform drivers to take action but that some drivers may not pay attention, and vehicles may still enter the work zone.

HEALTH AND SAFETY PLAN FORM **CDM Smith Health and Safety Program**	*This document is for the exclusive use of CDM Smith and its subcontractors*	CDM Smith

DESCRIPTION AND FEATURES: *Include principal operations and unusual features (containers, buildings, dikes, power lines, hillslopes, rivers, etc.)*

Source Area 7 is primarily a grassy area located at the eastern end of Balsam Lane. It contains Eckberg Park and an open area containing some woodlands. The park includes a basketball court, tennis court and a playground. The open field and wooded ares are located south of the park on a hillside, which slopes to the north. Two small valleys merge at the base of the hill where surface water drains to an intermittent creek which runs along the north side of Area 7. Residences border the area to the west and southwest (downgradient) and distantly to the east (upgradient). Parts of Area 7 were once used as gravel pits, which were subsequently filled with waste.

SURROUNDING POPULATION: ( ) Residential ( ) Industrial ( ) Commercial ( ) Rural ( ) Urban OTHER:

HAZARDOUS MATERIAL SUMMARY: ***Highlight or bold waste types and estimate amounts by category.***

CHEMICALS: *Amount/Units:*	**SOLIDS:** *Amount/Units:*	**SLUDGES:** *Amount/Units:*	**SOLVENTS:** *Amount/Units:*	**OILS:** *Amount/Units:*	**OTHER:** *Amount/Units:*
Acids	Flyash	Paints	Ketones	Oily Wastes	Laboratory
Pickling Liquors	Mill or Mine Tailings	Pigments	Aromatics	Gasoline	Pharmaceutical
Caustics	Asbestos	Metals Sludges	**Hydrocarbons**	Diesel Oil	Hospital
Pesticides	Ferrous Smelter	POTW Sludge	Alcohols	Lubricants	Radiological
Dyes or Inks	Non-Ferrous Smelter	Distillation Bottoms	**Halogenated (chloro, bromo)**	**Polynuclear Aromatics**	Municipal
Cyanides	Metals	Aluminum	Esters	PCBs	Construction
Phenols	Dioxins		Ethers	Heating Oil	Munitions
Halogens					
Other - *specify*	Other - *specify*	Other - *specify*	Other - *specify*	Other - *specify*	Other - *specify*

HEALTH AND SAFETY PLAN FORM
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KNOWN CONTAMINANTS	HIGHEST OBSERVED CONCENTRATION	PEL/TLV *ppm or mg/m3 (specify)*	IDLH *ppm or mg/m3 (specify)*	Warning Concentration *(in ppm)*	SYMPTOMS & EFFECTS OF ACUTE EXPOSURE	PHOTO IONIZATION POTENTIAL
1,1,1-Trichloroethane	360 mg/kg (S)	350 ppm	700 ppm	400 ppm	Headache, CNS depression, loss of balance, eye irritation	11
Tetrachloroethylene	260 mg/kg (S)	25 ppm	150 ppm	47 ppm	Irritated eyes, nose, throat, flushed face & neck, dizziness	9.32
Trichloroethylene	130 mg/kg (S)	50 ppm	1,000 ppm	82 ppm	Vertigo, visual disturbance, headache, drowsiness	9.45
Vinyl chloride	14 mg/L (GW)	1 ppm	Carc.	NA	Weakness, stomach pain, cancer	10
1,1-Dichloroethane	0.24 mg/kg(S)	100 ppm	3,000 ppm	120 ppm	Skin irritation, drowsiness	11.1
1,1 Dichloroethylene	0.69 mg/L (GW)	1 ppm	>500 ppm	1.1 ppm	No acute effects	<11.0
1,2 Dichloroethylene	49 mg/kg (S)	200 ppm	1,000 ppm	1.1 ppm	Irritated eyes, CNS depression	10
Benzene	0.032 mg/L (GW)	0.5 ppm	500 ppm	61 ppm	Eye & nose irritation, headache, giddiness, nausea, fatigue	9.25
Ethyl benzene	31 mg/kg (S)	100 ppm	800 ppm	200 ppm	Eye & nose irritation, headache, narcosis	8.76
Toluene (skin)	23 mg/kg (S)	50 ppm	500 ppm	1.7 ppm	Fatigue, confusion, euphoria, dizziness, headache, tears	8.82
Xylene	210 mg/kg (S)	100 ppm	900 ppm	5 ppm	Eye, nose & throat irritation, drowsiness, nausea, incoordination	8.44
Chloroform	.028 mg/l (GW)	2 ppm	500 ppm	192 ppm	Mental dullness, headaches, anesthesia, dizziness	11.4

NA = Not Available **NE = None Established** **U = Unknown**

Verify your access to an MSDS for each chemical you will use at the site.

S = Soil SW = Surface Water T = Tailings W = Waste TK = Tanks SD = Sediment
A = Air GW = Ground Water SL = Sludge D = Drums L = Lagoons OFF = Off-Site

HEALTH AND SAFETY PLAN FORM
CDM Smith Health and Safety Program

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	SPECIFIC TASK DESCRIPTIONS	Disturbing the Waste?	TASK - SPECIFIC HAZARDS	HAZARD & SCHEDULE
1	Groundwater monitoring and sampling at existing monitoring wells using low-flow submersible pump	**Intrusive**	Low-flow submersible pumps, slips, trips, falls and potential contact with contaminated groundwater	**Low Hazard** 7/1/19 to 6/30/22
2	RA oversight of various tasks performed by O&M contractor	**Intrusive**	Contact with potentially contaminated groundwater/soils, or noise from heavy machinery	**Low Hazard** 7/1/19 to 6/30/22
3	RA oversight of various activities in areas known to not be contaminated	**Non-Hazardous**	Slips, trips, falls; motorized machinery; electrical hazards; noise; vehicle operation	**Low Hazard** 7/1/19 to 6/30/22
4		**Pick from the list**		**Pick from the list**
5		**Pick from the list**		**Pick from the list**
6		**Pick from the list**		**Pick from the list**

SPECIALIZED TRAINING REQUIRED:

SPECIAL MEDICAL SURVEILLANCE REQUIREMENTS:

OVERALL HAZARD EVALUATION: ( ) High ( ) Medium (**X**) Low ( ) Unknown *(Where tasks have different hazards, evaluate each.)*

JUSTIFICATION: Intrusive work with low hazard and medium levels of contamination

FIRE/EXPLOSION POTENTIAL: ( ) High ( ) Medium (**X**) Low ( ) Unknown

HEALTH AND SAFETY PLAN FORM

CDM Smith Health and Safety Program

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CDM Smith

PROTECTIVE EQUIPMENT: *Specify by task. Indicate type and/or material, as necessary. Group tasks if possible. Use copies of this sheet if needed.*

BLOCK A

TASKS: 1 - 2 - 3 - 4
LEVEL: D - Modified
(X) Primary ( ) Contingency

Respiratory: (X) Not needed
( ) SCBA, Airline:
( ) APR:
( ) Cartridge:
( ) Escape Mask:
( ) Other:

Head and Eye: ( ) Not needed
(XX) Safety Glasses:
( ) Face Shield:
( ) Goggles:
( ) Hard Hat:
( ) Other:

Boots: ( ) Not needed
(XX) Steel-Toe ( ) Steel Shank
( ) Rubber **(XX) Leather**
( ) Overboots:

Prot. Clothing: ( ) Not needed
( ) Encapsulated Suit:
( ) Splash Suit
( ) Apron:
( ) Tyvek Coverall or
( ) Saranex Coverall
( ) Cloth Coverall:
(XX) Other: work clothes

Gloves: ( ) Not needed
( ) Undergloves:
(XX) Gloves: nitrile
( ) Overgloves:

Other: specify below
(X) Tick Spray
(X) **Hand Sanitizer/Disinfecting Wipes**
(X) Hearing Protection
(XX) Sun Screen

BLOCK B

TASKS: 1
LEVEL: D - Modified
( ) Primary (X) Contingency

Respiratory: ( ) Not needed
( ) SCBA, Airline:
( ) APR
( ) Cart
( ) Esca
( ) Othe

Head and Eye: ( ) Not needed
( ) Safety Glasses:
( ) Face Shield:
( ) Goggles:
( ) Hard Hat:
( ) Oth

Boo ed
( ) Shar
( ) Rubber ( ) Leather
( ) Overboots: Latex

Prot. Clothing: ( ) Not needed
( ) Encapsulated Suit:
( ) h S
( ) n
( ) k C all or
( ) nex erall
( ) Co
( ) Other:

Gloves: ( ) Not needed
( ) Undergloves:
( ) Gloves:
( ) Overgloves:

Other: specify below
( ) Tick S
( ) Floatation Device
( ) Hearing Protection
( ) Sun Screen

Exit Area

BLOCK C

TASKS: 1 - 2 - 3 - 4 - 5 - 6 - 7 - 8 - 9 - 10
LEVEL: A - B - C - D - Modified
( ) Primary ( ) Contingency

Respiratory: ( ) Not needed
( ) SCBA, Airline:
( ) APR:
( ) Cartridge:
( ) Escape Mask:
( ) Other:

Head and Eye: ( ) Not needed
( ) Safety Glasses:
( ) Face Shield:
( ) Goggles:
( ) Hard Hat:
( ) Other:

Boots: ( ) Not needed
( ) Steel-Toe ( ) Steel Shank
( ) Rubber ( ) Leather
( ) Overboots:

Prot. Clothing: ( ) Not needed
( ) Encapsulated Suit:
( ) Splash Suit
( ) Apron:
( ) Tyvek Coverall
( ) Saranex Coverall
( ) Cloth Coverall:
( ) Other:

Gloves: ( ) Not needed
( ) Undergloves:
( ) Gloves:
( ) Overgloves:

Other: specify below
( ) Tick Spray
( ) Flotation Device If Over Water
( ) Hearing Protection
( ) Sun Screen

BLOCK D

TASKS: 1 - 2 - 3 - 4 - 5 - 6 - 7 - 8 - 9 - 10
LEVEL: A - B - C - D - Modified
( ) Primary ( ) Contingency

Respiratory: ( ) Not needed
( ) SCBA, Airline:
( ) APR:
( ) Cartridge:
( ) Escape Mask:
( ) Other:

Head and Eye: ( ) Not needed
( ) Safety Glasses:
( ) Face Shield:
( ) Goggles:
( ) Hard Hat:
( ) Other:

Boots: ( ) Not needed
( ) Steel-Toe ( ) Steel Shank
( ) Rubber ( ) Leather
( ) Overboots:

Prot. Clothing: ( ) Not needed
( ) Encapsulated Suit:
( ) Splash Suit
( ) Apron:
( ) Tyvek Coverall
( ) Saranex Coverall
( ) Cloth Coverall:
( ) Other:

Gloves: ( ) Not needed
( ) Undergloves:
( ) Gloves:
( ) Overgloves:

Other: specify below
( ) Tick Spray
( ) Floatation Device
( ) Hearing Protection
( ) Sun Screen

This health and safety plan form constitutes hazard analysis per 29 CFR 1910.132

HEALTH AND SAFETY PLAN FORM **CDM Smith Health and Safety Program**	*This document is for the exclusive use of CDM Smith and its subcontractors*	CDM Smith

MONITORING EQUIPMENT: *Specify by task. Indicate type as necessary. Attach additional sheets if needed.*

INSTRUMENT	TASK	ACTION GUIDELINES		COMMENTS
Combustible Gas Indicator		0-10% LEL 10-25% LEL >25% LEL 21.0% O2 <21.0% O2 <19.5% O2	*No explosion hazard* *Potential explosion hazard; notify SHSC* *Explosion hazard; interrupt task/evacuate* *Oxygen normal* *Oxygen deficient; notify SHSC* *Interrupt task/evacuate*	(X) Not Needed
Radiation Survey Meter		3 x Background: >2mR/hr:	*Notify HSM* *Establish REZ*	(X) Not Needed
Photoionization Detector 10.6 eV Lamp Type ____	*Specify:* 1 & 2	*0 to 1 ppm: Level D.* *1 to 15 ppm: Level D, use detector tubes (see below)* *>15 ppm: Leave area. Call HSM*		(X) Not Needed
Flame Ionization Detector Type_____	*Specify:*			(X) Not Needed
Single Gas Type_____ Type Benzene	*Specify:* 1 & 2	*<0.5 ppm: Level D* *>0.5 ppm: Leave area. Call HSM*		(X) Not Needed
Respirable Dust Monitor Type visual	*Specify:* 1 & 2	*If team observes visible concentrations of airborne dust or dry, windy conditions that dust, team will leave area.* *<0.5 ppm: Level D* *>0.5 ppm: Leave area. Call HSM*		(X) Not Needed
Other Type_____ Type_____	*Specify:*			(X) Not Needed

DECONTAMINATION PROCEDURES

ATTACH SITE MAP INDICATING EXCLUSION, DECONTAMINATION, & SUPPORT ZONES AS PAGE TWO

Personnel Decontamination	**Sampling Equipment Decontamination**	**Heavy Equipment Decontamination**
Summarize below or attach diagram;	*Summarize below or attach diagram;*	*Summarize below or attach diagram;*
Team members will remove their protective clothing in the following order (as applicable): Equipment Drop Outer Glove Removal Hard Hat Removal Inner Glove Removal Hand and Face wash ( ) Not Needed	Bag all disposable sampling and PPE equipment. Wash/rinse the outside of sample containers in soapy/clean water. Wash all non-disposable sampling equipment in Alconox solution or equivalent with designated brush. Follow with tap water and distilled water rinse. ( ) Not Needed	Wash all non-disposable sampling equipment in Alconox solution or equivalent with designated brush. Perfrom this task on a location that allows capture of contaminated water. ( ) Not Needed
Containment and Disposal Method	**Containment and Disposal Method**	**Containment and Disposal Method**
Disposal as general refuse.	Decon water containerized in poly tote on site. IDW disposal as general refuse.	Decon water containerized in poly tote on site. IDW disposal as general refuse.

HAZARDOUS MATERIALS TO BE BROUGHT ONSITE

Preservatives		*Decontamination*		*Calibration*	
(X) Hydrochloric Acid	( ) Zinc Acetate	(X) Alconox ™	( ) Hexane	(X) 100 ppm isobutylene	( ) Hydrogen Sulfide
( ) Nitric Acid	( ) Ascorbic Acid	( ) Liquinox ™	( ) Isopropanol	( ) Methane	( ) Carbon Monoxide
( ) Sulfuric Acid	( ) Acetic Acid	( ) Acetone	( ) Nitric Acid	( ) Pentane	( ) pH Standards
( ) Sodium Hydroxide	( ) Other:	( ) Methanol	( ) Other:	( ) Hyrogen	( ) Conductivity Std
All preservatives are sm. Quantities		( ) Mineral Spirits		( ) Propane	( ) Other:

HEALTH AND SAFETY PLAN FORM

CDM Smith Health and Safety Program

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CDM Smith

EMERGENCY CONTACTS

Water Supply
EPA Release Report #: 800 / 424 - 8802
CDM Smith 24-Hour Emergency #: PSG 732 / 539 - 8128
Facility Management
Other (specify)
CHEMTREC Emergency #: 800 / 424 - 9300

SAFETY NARRATIVE: *Summarize below*

CDM Smith staff may chose to wear more protection than that directed by this plan.
No hazard is anticipated to personnel outside of the exclusion zone.
Without regard to instruments, CDM Smith personnel will leave the area and may then consider upgrade of their level of protection if they experience dizziness, nausea, or any other irritative symptoms caused by their environment.
SHSC will designate contamination reduction (decon) zone, support zone, and evacuation routes prior to each task.

COVID-19 Safety Awareness General Guidelines

1. All personnel must maintain 5 to 6 feet of distance between one another in order to avoid physical contact with one another as best you can
2. Every person needs to bring their own vehicle, there will be no ridesharing
3. Avoid touching your face, to prevent this, try and wear gloves not only while engaged during sampling actvitities, but during non sampling activities as well
4. Wipe down surfaces that are in constant use by personnel with disinfecting wipes
5. Occupation of the Area 7 treatment building office is limited to a maximum of two people
6. **See attached for additional and more specific COVID-19 safety guidlelines**

HEALTH AND SAFETY PLAN APPROVALS (H&S Mgr must sign each plan)

Prepared by John Grabs Date 9/23/2020

HSM Signature Joseph Sabo

Joe Sabo CSP/CHST

EMERGENCY CONTACTS	NAME	PHONE
Health and Safety Manager	Joe Sabo	857-500-3636
Site Safety Coordinator	Andrew Schamber	317-450-2565
Client Contact	Brian Conrath	(217) 557-8155
Other *(specify)*		
Environmental Agency		
State Spill Number	Illinois	(800) 292 - 4706
Fire Department		911
Police Department		911
State Police		911
Health Department		
Poison Control Center	Nationwide	800 / 222 - 1222
Occupational Physician	Dr. Jerry Berke	800 / 350 - 4511
All One First Aid/Non-Emergency		800 / 350 - 4511

MEDICAL EMERGENCY		PHONE
Hospital Name:	Swedish American Hospital	(815) 968-4400
Hospital Address	1309 2nd Avenue, Rockford, IL	
Name of Contact at Hospital:		
Name of 24-Hour Ambulance:		911
Route to Hospital:		

See attached hospital map.

Distance to Hospital 5.3 miles See map

HEALTH AND SAFETY PLAN SIGNATURE FORM

All site personnel must sign this form indicating receipt of the H&SP. Keep this original on site. It becomes part of the permanent project files. Send a copy to the Health and Safety Manager (HSM).

SITE NAME/NUMBER: Area 7 SE Rockford Superfund

DIVISION/LOCATION: 3750 Balsam Lane Rockford, IL

CERTIFICATION:

I understand, and agree to comply with, the provisions of the above referenced H&SP for work activities on this project. I agree to report any injuries, illnesses or exposure incidents to the site Health and Safety Coordinator (SHSC). I agree to inform the SHSC about any drugs (legal and illegal) that I take within three days of site work.

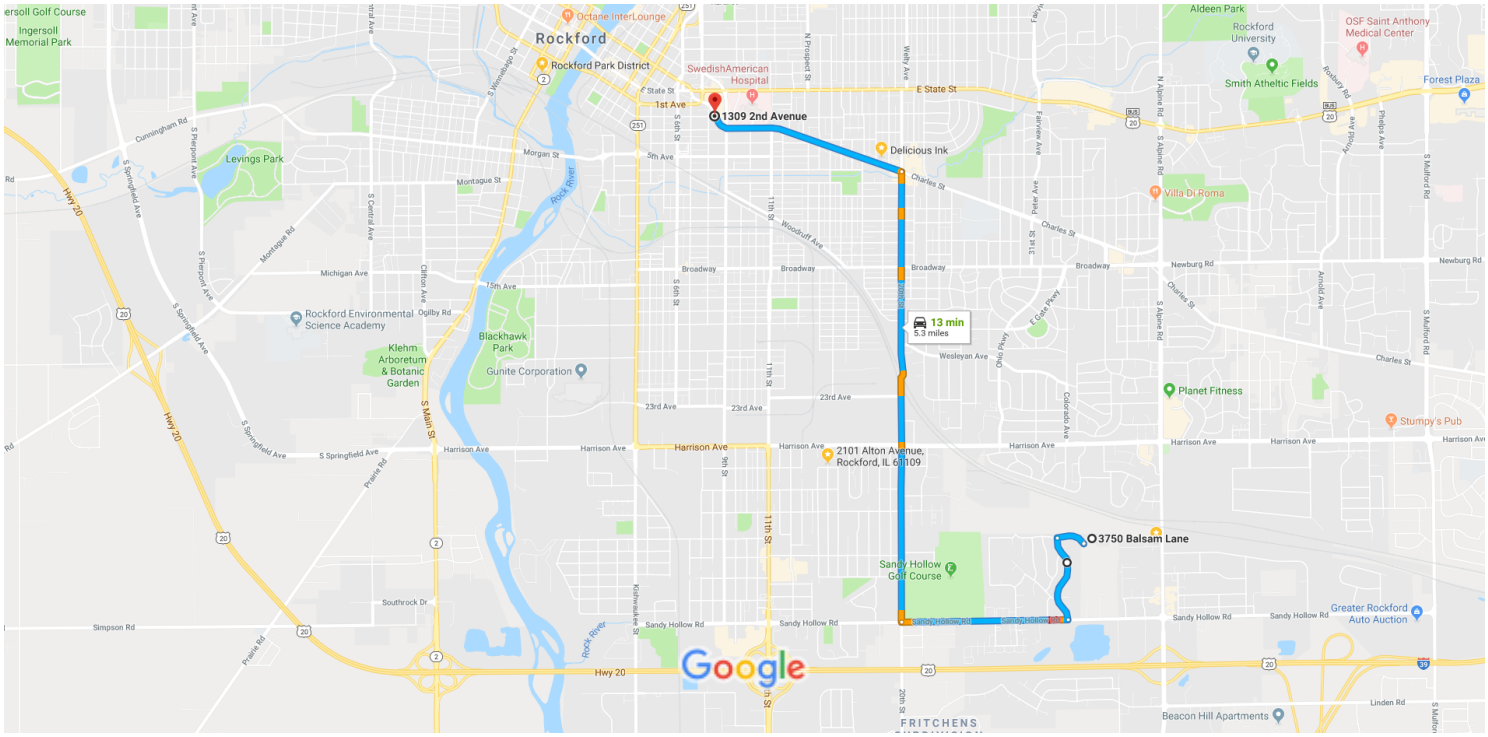
PRINTED NAME	SIGNATURE	DATE

Google Maps

3750 Balsam Lane, Rockford, IL to 1309 2nd Avenue, Rockford, IL

Drive 5.3 miles, 13 min

Hospital Route from Area 7



Map data ©2019 Google 2000 ft

3750 Balsam Ln

Rockford, IL 61109

1. Head northwest on Balsam Ln toward Bavarian Ln

   0.2 mi

2. Turn left onto Bavarian Ln

   0.5 mi

3. Turn right onto Sandy Hollow Rd

   0.9 mi

4. Turn right onto 20th St

   Pass by Subway Restaurants (on the right in 2.5 mi)

   2.5 mi

5. Turn left onto Charles St

   1.1 mi

1309 2nd Ave

Rockford, IL 61104

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan

CDM Smith

Memorandum

Date: *July 22, 2020*
Subject: *Updated Field Guidance*

COVID-19 Prevention Guidance for Field Activities

Per Tim Wall's memorandum on 3/16/20, Working Safely during the Coronavirus (COVID-19) Outbreak Update, we have new firm-wide policies for how to best respond to this outbreak, establish continuity of operations, and protect personnel.

This document is intended to provide basic guidance to field and project teams that have operations outside of a CDM Smith office other than CCI construction sites. Included are measures on how to best protect employees and minimize potential exposure to COVID-19.

Planning

All projects involving field work should have an H&S plan to address specific hazards associated with that project. Since potential exposure to this virus is a new hazard, those H&S plans will need to be modified at the project level to address their specific COVID-19 exposures. These modifications will need to be communicated to personnel ASAP. The practices below must be evaluated and included in any greater planning activities and project-specific H&S plans. For non-routine exposure scenarios contact your H&S Manager for assistance in working out appropriate precautions. All new H&S Plans will need to incorporate the guidance in this memo.

COVID-19 Practices to Minimize Exposure

COVID-19 exposure is most directly associated with close contact with an infected individual. There are also less direct means of contact that are not as fully understood such as contact with contaminated surfaces, droplets, and residues. To minimize exposure, it is imperative that field staff exercise the precautions below

When not to report for Site Work

Have you had exposure to or contact with someone diagnosed with COVID-19? Close contact means having been within 6 feet of that person for an extended time or being exposed to their cough or sneeze.

- You have a fever, a cough, difficulty breathing or have lost your sense of taste or smell.
- Has a Public Health Official informed you that you were potentially exposed to COVID-19?
- Do you share a household with a public health professional who might have been exposed?

What to do if you or someone gets sick while on site:

- If you become ill while on travel or at work, you should self-isolate in your hotel room and contact Allone Health to determine if you should visit a medical facility or local medical provider. From there, notify your supervisor, team lead, or HR representative of your symptoms and any recommendations from Allone Health. For COVID-19 like symptoms, medical providers are requesting that you call first before seeking treatment.

- In general, an employee that has been sick can return to the site once their healthcare provider has cleared them to return for duty or if they have been symptom free without the use of medication for 72 hours. If they test positive for COVID-19, typically they would need to test negative two consecutive times before being allowed to return to work.

Best Practices

- Maintain social distancing. Stay a minimum of 6 feet away from other people. **This is the most important action to limit exposure.**
- Minimize contact with others. Do not shake hands (use non-contact greetings).
- Increase the frequency of hand washing, for a minimum of 20 seconds. Use hand sanitizer as you can.
- The use of a loose-fitting face covering or face mask is required when social distancing of 6 feet or more is not possible. Face masks may also be required by certain local governments and/or clients.
- CDM Smith Equipment Center has a limited supply of work gloves, hand sanitizer and surgical style pleated face masks that meet CDC, local government and client requirements for face masks. Limited amounts are available for staff involved with field work. Contact the Equipment Center to request any PPE.
- Staff may also make face masks themselves. See https://www.ecommunity.com/giveppe/homemade-mask-instructions or https://www.youtube.com/watch?v=j8aYEBtUQ9E&feature=youtu.be
    - Please note that use of a cloth or surgical style face mask is not adequate protection alone, physical distancing, hand washing, and disinfection of common surfaces needs to be incorporated into your daily activities Face masks should not be used where the use of N95 or ½ respirators have been approved.
- No sharing of PPE without first disinfecting the equipment.
- Do not use common coffee pots or water coolers. Bring your own and use individual water bottles.
- Minimize time in shared office spaces, trailers etc. Maximize physical distancing.
- Avoid touching your face, in particular your mouth, eyes, and nose.
- Regularly disinfect common surfaces, several times per day if possible. If not available, the surface can be cleaned with soap and water or a diluted solution of bleach (https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/disinfecting-your-home.html)
- Plan work and meetings to minimize the density of people in one area.
- Organize virtual meetings as opposed to in-person meetings where possible.
- At the beginning of your work day, discuss with any CDM Smith, subcontractor or Client team members the precautions that are to be taken to minimize exposure.

- Disinfectants to consider (https://www.epa.gov/pesticide-registration/list-n-disinfectants-use-against-sars-cov-2)or alternative disinfectants as follows:
    - isopropyl alcohol (aka isopropanol aka IPA) min. 70%-recommended sit time 5 minutes,
    - household hydrogen peroxide, min. 3% (Note: opened/expired H_20_2 will likely be less than 3%, start with unopened/unexpired bottle) – recommended sit time 2 minutes (test as it may bleach fabrics),
    - quaternary ammonium, recommended sit time 5-10 minutes dependent on the mixture. (via spray bottle) *,
    - 10% bleach (1 part 5% household bleach to 9 parts water); recommended sit time up to 10 minutes depending on the label (spray bottle) *

The physical distancing, personal hygiene, and use of protective equipment guidance above are the most effective means to minimize exposures to COVID-19

Appendix B

CDM Smith Technical SOPs

Sample Custody	SOP 1-2 Revision: 9 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Cherie Zakowski

1.0 Objective

Because of the evidentiary nature of the samples, possession of samples collected during environmental investigations should be traceable from the time the samples are collected until their derived data are incorporated into reports and/or introduced as evidence in legal proceedings. Sample custody procedures are followed to maintain and document sample possessions. All paperwork associated with the sample custody procedures will be retained in CDM Smith files unless the client requests that it be transferred to them for use in legal proceedings or at contract completion.

Note: Sample custody documentation requirements vary with the specific EPA region or client. This technical standard operating procedure (SOP) is intended to present basic sample custody requirements, along with common options. Specific sample custody requirements shall be presented in the project-specific quality assurance (QA) project plan or project-specific modification or clarification form.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Sample - A sample is a material to be analyzed that is contained in single or multiple containers representing a unique sample identification number.

Sample Custody - A sample is under custody if:

1. It is in your possession
2. It is in your view, after being in your possession
3. It was in your possession and you locked it up
4. It is in a designated secure area

Chain-of-Custody Record - A chain-of-custody record is a form used to document the transfer of custody of samples from one individual to another.

Custody Seal - A custody seal is a tape-like seal that is part of the chain-of-custody process and is used to detect tampering with samples after they have been packed for shipping.

Sample Label - A sample label is an adhesive label placed on sample containers to designate a sample identification number and other sampling information.

Sample Tag - A sample tag is attached to a sample container with string to designate a sample identification number and other sampling information. Tags may be used when it is difficult to physically place adhesive labels on the container (e.g., in the case of small air sampling tubes). Check with your EPA regional Contract Laboratory Program (CLP) coordinator as not all regions require sample tags.

3.0 General Responsibilities

Project Manager - The project manager is responsible for ensuring that field personnel are trained in the use of this SOP and related SOPs.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events. The FTL is also responsible for coordinating with the subcontractor laboratory to ensure that adequate

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information is recorded on custody records. The FTL determines whether proper custody procedures were followed during the fieldwork.

Sampler - The sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

Field Sample Custodian - The field sample custodian, when designated by the FTL, is responsible for accepting custody of samples from the sampler(s) and properly packing and shipping the samples to the laboratory assigned to perform the analyses. A field sample custodian is typically designated only for large and complex field efforts.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP).

4.0 Required Supplies

- Chain-of-custody records (applicable client or CDM Smith forms)
- Sample labels and/or tags
- Scribe software (if required)
- Custody seals
- Clear tape
- Computer
- Printer and paper

5.0 Procedures

5.1 Chain-of-Custody Record

This procedure establishes a method to maintain custody of samples through a chain-of-custody record. This procedure will be followed for all samples collected or split samples until they are accepted at the laboratory.

Field Custody

1. Collect only the number of samples needed to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations before the actual fieldwork. As few people as possible shall handle samples.

2. Complete sample labels or tags for each sample using waterproof ink.

3. Maintain personal custody of the samples (in your possession) at all times until custody is transferred for sample shipment or directly to the analytical laboratory.

Transfer of Custody and Shipment

1. Complete a chain-of-custody record for all samples (see Figure 1 for an example of a chain-of-custody record. Similar forms may be used when requested by the client). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the sample custodian in the appropriate laboratory.

   - The date/time will be the same for both signatures when custody is transferred directly to another person. When samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures. Common carriers are not required to sign the chain-of-custody record.
   - In all cases, it should be readily apparent that the person who received custody is the same person who relinquished custody to the next custodian.
   - If samples are left unattended or a person refuses to sign, this should be documented and explained on the chain-of-custody record.

   Note: If a field sample custodian has been designated, he/she may initiate the chain-of-custody record, sign, and date as the relinquisher. The individual sampler(s) should sign in the appropriate block but does (do) not need to sign and date as a relinquisher (refer to Figure 1).

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2. Package samples properly for shipment and dispatch to the appropriate laboratory for analysis. Each shipment should be accompanied by a separate chain-of-custody record. If a shipment consists of multiple coolers, a chain-of-custody record shall be filled out for each cooler documenting only samples contained in that particular cooler.

3. The original record will accompany the shipment, and the copies will be retained by the FTL and, if applicable, distributed to the appropriate sample coordinators. Freight bills will also be retained by the FTL as part of the permanent documentation. The shipping number from the freight bill shall be recorded on the applicable chain-of-custody record and field logbook in accordance with SOP 4-1, Field Logbook Content and Control.

Procedure for Completing CDM Smith Example Chain-of-Custody Record
The following procedure is to be used to fill out the CDM Smith chain-of-custody record. The record provided herein (Figure 1) is an example chain-of-custody record. If another type of custody record (i.e., provided by the EPA Contract Laboratory Program [CLP] or a subcontract laboratory or generated by Scribe) is used to track the custody of samples, the custody record shall be filled out in its entirety.

1. Record the project number.
2. Record FTL for the project (if a field sample custodian has been designated, also record this name in the “Remarks” box).
3. Record the name and address of the laboratory to which samples are being shipped.
4. Enter the project name/location or code number.
5. Record the overnight courier’s airbill number.
6. Record the sample location number.
7. Record the sample number.
8. Note preservatives added to the sample.
9. Note media type (matrix) of the sample.
10. Note the sample type (grab or composite).
11. Enter the date of sample collection.
12. Enter the time of sample collection in military time (24 hour clock).
13. When required by the client, enter the names or initials of the samplers next to the sample location number of the sample they collected.
14. List parameters for analysis and the number of containers submitted for each analysis.
15. Enter appropriate designation for laboratory quality control (e.g., matrix spike/matrix spike duplicate [MS/MSD], matrix spike/duplicate [MS/D]), or other remarks (e.g., sample depth).
16. Sign the chain-of-custody record(s) in the space provided. All samplers must sign each record.
17. If sample tags are used, record the sample tag number in the “Remarks” column.
18. The originator checks information entered in Items 1 through 16 and then signs the top left “Relinquished by” box, prints his/her name, and enters the current date and time (military).
19. Send the top two copies (usually white and yellow) with the samples to the laboratory; retain the third copy (usually pink) for the project files. Retain additional copies for the project file or distribute as required to the appropriate sample coordinators.
20. The laboratory sample custodian receiving the sample shipment checks the sample label information against the chain-of-custody record. Sample condition is checked and anything unusual is noted under “Remarks” on the chain-of-custody record. The laboratory custodian receiving custody signs in the adjacent “Received by” box and keeps the copy. The white copy is returned to CDM Smith.

5.2 Sample Labels and Tags

Unless the client directs otherwise, sample labels or tags will be used for all samples collected or accepted for CDM Smith projects.

1. Complete one label or tag with the information required by the client for each sample container collected. A typical label or tag would be completed as follows (see Figure 2 for example of sample tag; labels are completed with the equivalent information):

   - Record the project code (i.e., project or task number).
   - Enter the station number (sample number or EPA CLP identification number) if applicable.

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- Record the date to indicate the month, day, and year of sample collection.
- Enter the time (military) of sample collection.
- Place a check to indicate composite or grab sample.
- Record the station (sample) location.
- Sign in the space provided.
- Place a check next to "yes" or "no" to indicate if a preservative was added.
- Place a check under "Analyses" next to the parameters for which the sample is to be analyzed. If the desired analysis is not listed, write it in the empty slot. Note: Do not write in the box for "laboratory sample number."
- Place or write additional relevant information under "Remarks."
- If perfluoroalkyl substances (PFAS) samples are collected, ball point pens should be used on any labels or custody seals. Sharpies (or equivalent) may be used to label sample bottles in the staging area, but markers should not be used in the immediate sampling environment.

2. Place adhesive labels directly on the sample containers. Place clear tape over the label to protect from moisture.

3. Securely attach sample tags to the sample bottle if required. On 2.27 liter (80 oz.) amber bottles, the tag string may be looped through the ring-style handle and tied. On all other containers, it is recommended that the string be looped around the neck of the bottle, then twisted, and relooped around the neck until the slack in the string is removed. In some instances, when the tag cannot be physically attached to the sample container, it is acceptable to simply place the sample tag in the zip lock bag with a sample container.

4. Double-check that the information recorded on the sample label or tag is consistent with the information recorded on the chain-of-custody record.

5.3 Custody Seals

Two custody seals should be placed on opposite corners of all shipping containers (e.g., cooler) before shipment. The seals shall be signed and dated by the shipper.

Custody seals may also be required to be placed on individual sample bottles. Check with the client or refer to EPA regional guidelines for direction. In these instances, the custody seal is placed over, or in some cases around, the lid or cap of the sample container.

If custody seals are required on individual sample bottles and PFAS samples are collected, ball point pens should be used on any custody seals. Sharpies (or equivalent) may be used in the staging area, but markers should not be used in the immediate sampling environment.

5.4 Sample Shipping

SOP 2-1, *Packaging and Shipping Environmental Samples* defines the requirements for packaging and shipping environmental samples.

6.0 Restrictions/Limitations

Check with the EPA region or client for specific guidelines. If no specific guidelines are identified, this procedure shall be followed.

For EPA CLP sampling events, combined chain-of-custody/traffic report forms generated with Scribe or other EPA-specific records may be used. Refer to regional guidelines for completing these forms.

The EPA Scribe software may be used to customize sample labels and custody records when directed by the client or the CDM Smith project manager.

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The evolving regulatory standards for PFAS should be considered in the implementation of SOP 1-2 as well as the development of project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans, and laboratory statements of work used to guide the collection of samples for PFAS analysis.

A consideration of the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, and reporting limits are all impacted with the inclusion of sampling for PFAS analysis.

Before implementing this SOP for collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to this SOP should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith regulatory updates, and state and federal regulatory limit updates can be found on the PFAS InfoCenter: https://cdmsmith.libguides.com/PFAS_InfoCenter

7.0 References

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U. S. Environmental Protection Agency. 2015. Scribe v3.10. . https://response.epa.gov/site/site_profile.aspx?site_id=ScribeGIS

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_____. Revised March 1992 or current revision. *National Enforcement Investigations Center, Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p.85.

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Figure 1
Example CDM Smith Chain-of-Custody Record

125 Maiden Lane, 5th Floor
New York, NY 10038
(212) 785-9123
Fax: (212) 785-6114

CHAIN OF CUSTODY RECORD

PROJECT ID.	FIELD TEAM LEADER	LABORATORY AND ADDRESS	DATE SHIPPED
PROJECT NAME/LOCATION		LAB CONTRACT:	AIRBILL NO.

MEDIA TYPE	PRESERVATIVES	SAMPLE TYPE
1. Surface Water	1. HCl, pH <2	G = Grab
2. Groundwater	2. HNO3, pH <2	C = Composite
3. Leachate	3. NaOH, pH >12	
4. Field QC	4. H2SO4, pH <2	
5. Soil/Sediment	5. Zinc Acetate, pH >9	
6. Oil	6. Ice Only	
7. Waste	7. Not Preserved	
8. Other _____	8. Other _____	

SAMPLE LOCATION NO.	LABORATORY SAMPLE NUMBER	PRESER-VATIVES ADDED	MEDIA TYPE	SAMPLE TYPE	20__ DATE	TIME SAMPLED	ANALYSES (List no. of containers submitted)								REMARKS (Note if MS/MSD)
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															

SAMPLER SIGNATURES:

RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME
(SIGN)		(SIGN)		(SIGN)		(SIGN)	
RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME	RELINQUISHED BY: (PRINT)	DATE/TIME	RECEIVED BY: (PRINT)	DATE/TIME
(SIGN)		(SIGN)		(SIGN)		(SIGN)	

COMMENTS:

DISTRIBUTION: White and yellow copies accompany sample shipment to laboratory; yellow copy retained by laboratory. Pink copy retained by samplers.

1/98

Note: If requested by the client, different chain-of-custody records may be used. Copies of the template for this record may be obtained from the Chantilly Graphics Department.

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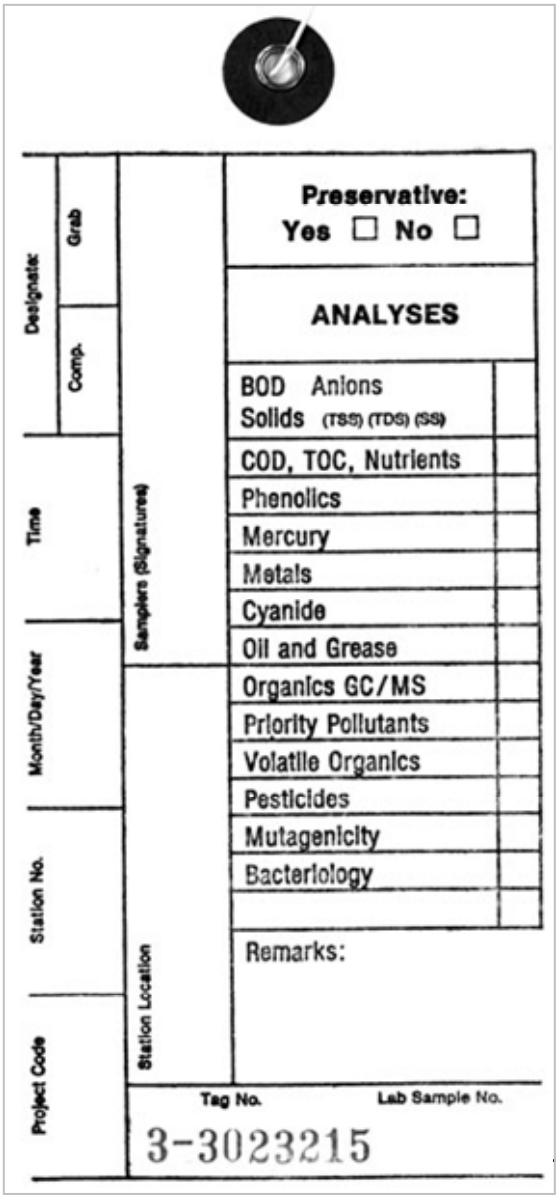
Figure 2
Example Sample Tag



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Groundwater Level Measurement	SOP 1-6 Revision10S August 2020

Approved: Ernest Ashley **Technical Review:** Nicholas Castonguay, P.G.

1.0 Objective

Groundwater level measurements are fundamental in the evaluation of groundwater resources, characterization of sites, and groundwater remediation. Groundwater level measurements are conducted during site investigation activities including groundwater gauging, groundwater sampling, and aquifer testing. This technical standard operating procedure (SOP) defines the techniques and requirements for obtaining depth to groundwater (or groundwater level) measurements.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Water Level Indicator - A portable device for measuring the depth from a fixed point (which could be below, at, or above the ground surface) to groundwater inside a well, borehole, or other underground opening

Measurement Point - An easily located and clearly defined mark at the top of a well from which all water level measurements from that particular well are made. The measurement point shall be as permanent as possible to provide consistency in measurements.

Electrical Tape - A graduated plastic tape onto which a water-sensitive electrode is connected that will electronically signal the presence of water (as a result of circuit closure)

Immiscible Fluids - Two or more fluid substances that will not mix and, therefore, will exist together in a layered form. The fluid with the highest density will exist as the bottom layer, the fluid with the lowest density will exist as the top layer, and any other fluid layers will be distributed relative to their respective densities.

Discharge - The removal/release of water from the zone of saturation

Recharge - The addition of water to the zone of saturation

Static Water Level - The level of water in a well, borehole, or other underground opening that is not influenced by discharge or recharge

Well Casing - A steel, stainless steel, or polyvinyl chloride (PVC) pipe that extends into a borehole and is connected to the well screen or sealed at the bedrock surface in open-hole wells. The upper portion (approximately 3 to 4 feet) of the well casing is normally enclosed by an outer steel protective casing.

Protective Casing - A steel cylinder or square protective sleeve extending approximately 3 to 5 feet into the ground, surrounding the well casing. For flush-mounted wells, the protective casing will extend only high enough so that the well and protective casing can be enclosed by a road box or equivalent vault. In above-grade wells, the protective casing will extend above the ground surface approximately 2 to 3 feet. The protective casing protects the well casing.

Pressure Transducer - A type of measurement device that converts pressure-induced mechanical changes into an electrical signal

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

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2.3 Discussion

The most common uses of static groundwater water level data are to: determine the elevation of groundwater, the direction of groundwater flow (horizontally and vertically), identify areas of recharge and discharge, evaluate the effects of manmade and natural stresses on the groundwater system, define the hydraulic characteristics of aquifers, and evaluate surface water body-aquifer relationships. Specific uses for water level data may include to:

- Determine ambient daily, precipitation influenced and seasonal fluctuations in groundwater levels
- Determine the change in water level due to tidal influences
- Determine the change in water level due to distribution and rate of regional groundwater withdrawal or recharge
- Show the relationship of groundwater to surface water
- Estimate the amount, source, and area of recharge and discharge
- Determine rate and direction of groundwater movement

Static water level measurements shall be obtained from each well before purging, sampling, or other disturbance of the water table.

3.0 General Responsibilities

Project Manager - The project manager is responsible for ensuring that measurements are conducted in accordance with this procedure and any other SOP pertaining to site activities related to obtaining groundwater level measurements.

Field Team Leader - The field team leader is responsible for ensuring that field personnel obtain water level measurements in accordance with this and other relevant procedures.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field sampling plan or site/quality assurance project plan (QAPP).

4.0 Required Equipment

4.1 General

- Site plans, including well construction information, and health & safety plan
- Field logbook
- Waterproof ink pens
- Decontamination equipment and supplies, including rinse bottles, deionized water, scrub brush and absorbent pads.
- Five-gallon buckets
- Water level meter
- Engineering ruler
- Photoionization detector
- Pressure transducer and transducer cable
- Portable computer with transducer software
- Pressure transducer communication cable

4.2 Measuring Devices

The equipment required to obtain water level measurements is dependent on the type of procedure chosen. Measurements may be made with a number of different devices and procedures. Measurements are taken relevant to a permanent measurement point on the well riser or well casing.

Electrical tapes are preferred over other devices such as steel tape because of the electrical tape's simplicity and ability to make measurements quickly. Many types of electrical instruments have been devised for measuring water levels; most operate on the principle that a circuit is completed when two electrodes are immersed in water. Examples of electrical tapes that are frequently used include the Solinst® and Heron® electronic water level indicators. These instruments are powered by batteries that should be checked before mobilization to the field.

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Electrical tapes are coiled on a hand-cranked reel unit that contains the batteries and a signaling device that indicates when the circuit is closed (i.e., when the probe reaches the water). Electrodes are generally contained in a weighted probe that keeps the tape taut in addition to providing some shielding of the electrodes against false indications as the probe is lowered into the hole. The electrical tapes are marked with 0.01-foot increments. Exercise caution while using electrical tapes when the water contains elevated amounts of dissolved solids. Under these conditions, the signaling device will remain activated after the probe is removed from the water. When the water being measured contains very low amounts of dissolved solids, it is possible for the probe to extend several inches below the water level before activating the signaling device or may not signal at all. Both of these conditions are related to the conductivity of the water and in some cases may be compensated for by the sensitivity control, if the device has this option. In groundwater with high conductivity, the sensitivity control may need to be turned down. In groundwater with low conductivity, the sensitivity control may need to be turned up to obtain a proper depth to groundwater measurement.

Pressure transducers and associated equipment are used to measure water levels in groundwater monitoring wells at user-specified intervals and record the measurements in the computer memory for later retrieval. A pressure transducer installed at a fixed depth in a groundwater well senses the change in pressure against a membrane. The pressure changes occur in response to changes in the height, and thus in the weight of the water column in the well above the transducer. Pressure transducers typically record temperature, pressure, and the converted depth to water or groundwater elevation. Two types of pressure transducers are typically used, vented and non-vented. The vented transducers have an air tube that is open to the atmosphere above the water level which automatically compensates for barometric pressure changes. The non-vented transducers are completely submerged with no open tube to the atmosphere. When using non-vented transducers, a barometric pressure transducer is also needed above ground to record barometric pressure readings to allow for water level corrections. An example of a pressure transducer that is frequently used includes the In-Situ Level Troll 700® Piezoresistive Titanium vented transducer. Depending on the range of groundwater levels, the appropriate pressure in pounds per square inch gauge (psig) for the instrument will need to be selected as shown below.

Level Troll 700 vented psig vs maximum water level

- 5 psig: 11.5 feet
- 15 psig: 35 feet
- 30 psig: 69 feet
- 100psig: 231 feet
- 300 psig: 692 feet
- 500 psig: 1153 feet

5.0 Procedures

5.1 Preparation

The following steps should be taken when preparing to obtain water level measurements:

1. Assign a designated field logbook to record all field events and measurements according to SOP 4-1. Document any and all deviations from SOPs and site-specific plans in the field logbook and include rationale for the changes.

2. Always exercise caution to prevent inappropriate or contaminated materials from entering any groundwater well.

3. Standing upwind from the well, open the groundwater well. Monitor the well with a photoionization detector, flame ionization detector, or equivalent vapor analyzer as soon as the cap is opened, as dictated by the site-specific health and safety plan.

For comparability, water level measurements shall always be referenced to the same vertical (elevation) datum marker, such as a United States Geological Survey (USGS) vertical and horizontal control point monument. The elevations calculated from the measurement of static water levels shall be referenced to mean sea level unless otherwise specified in the site-specific plans.

The measurement point should be as permanent as possible, clearly defined, marked, and easily located. Frequently, the top of the PVC riser is designated as the measurement point. However, since the top of the riser is seldom smooth and horizontal, one particular

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point on the riser pipe shall be designated and clearly marked. This can be accomplished by marking a point on the top of the riser pipe with a notch or indelible pen. To avoid spilling liquids into the well, paints or other liquid marking materials shall not be used.

5.2 Water Level Measurement Using Electrical Water Level Indicators

The following steps should be followed when taking water level measurements using electrical tapes:

1. Before lowering the probe into the well, the circuitry shall be checked by dipping the probe in tap water and checking to ensure that the signaling device responds to probe submergence. The probe shall then be lowered slowly into the well until contact with the water surface is indicated. The electrical tape reading is made at the measuring point. Take a second and third check reading to verify the measurement before completely withdrawing the tape from the well. All three measurements shall be recorded in the field logbook.

2. Independent electrical tape measurements of static water levels using the tape shall agree within 0.01 foot for depths of less than about 200 feet. At greater depths, independent measurements may not be this close. For a depth of about 500 feet, the maximum difference of independent measurement using the same tape shall be within 0.1 foot.

3. Decontaminate the electrical tape according to SOP 4-5 before proceeding to the next well to minimize cross contamination.

It may be necessary to check the electrical tape length with a graduated steel tape after the line has been used for a long period of time (at least annually) or after it has been pulled hard in attempting to free the line. Some electrical tapes, especially the single line wire, are subject to becoming permanently stretched.

5.3 Water-Level Measurement Using Pressure Transducers

The following steps should be followed when using pressure transducers:

1. Before departing to the site, confirm transducers and cables are functioning properly and can connect to the transducer software (e.g., Win-Situ®) on the portable computer via the communication cable (e.g., Troll communication cable).

2. Obtain a water level measurement prior to installing the pressure transducer. Once the static water level is confirmed, slowly lower the pressure transducer and associated cable to the desired depth in the well below the expected range of water level fluctuation. Do not allow the cable to rub on the outside edge of metal casing. As noted above, do not submerge the transducer to a water depth pressure greater than the pressure rating of the transducer. Secure the transducer cable to the top of the well casing using a well dock and PVC sleeve or an eye bolt with a small carabiner.

3. Monitor the water level in the well to ensure stabilization due to displacing water from the transducer installation. The water level is considered stable when the level is longer trending in one direction. After stabilization, connect the communication cable from the pressure transducer to the portable computer to program the pressure transducer to the measured depth to water (reference level). During programming, select the testing type (e.g. long-term monitoring) and the frequency of measurements (e.g., every 10 minutes) including other required input parameters. Confirm the depth of the probe is equal to the desired probe installation depth in the well (depth to water + probe depth below water). Once the pressure transducer log has started, confirm the pressure transducer readings are recording and are equal to the water level readings in the well via manual measurement.

4. Disconnect the transducer cable from the communications device, install the cable desiccant and confirm the cable is secure inside the well casing or well road-box. When conducting both short and long-term monitoring, it is important to conduct periodic visits to obtain water level measurements, complete data downloads and confirm the transducer is functioning properly. It is good practice to manually read the water level when downloading the data as transducers can drift slightly over time. If drift does occur, this can be corrected via re-programming the depth to water reference during the site visit.

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5.4 Other Water Level Measurement Methods

Although the methods cited above (electrical water level indicator and pressure transducers) for measuring water levels predominates in the environmental sector, there are a number of other methods available that may be well suited for a particular purpose.

5.4.1 Ultrasonic Method

The ultrasonic method electronically measures the time it takes a sound wave to reach and reflect off the water surface and return to the ground surface. These instruments contain electronic microprocessors, capable of performing this measurement many times each second. The actual depth to water, as calculated by the microprocessor, is an average of many individual readings.

5.4.2 Pressure Gauge Method

This method, also called the air-line submergence method, uses a pressure gauge and is the preferred method for obtaining water level measurements in pumping wells. An air line constructed of semi-rigid tubing is inserted into the well below the water table. The tube end at the surface is connected to an air tank or compressor and pressure gauge. Filtered air is then forced through the tube and the resultant pressure is read in pounds per square inch (psi). This reading is converted to feet of water in the column and subtracted from the total tube length to give depth to water. Readings are then converted to groundwater elevation. Results are plotted on a field logging form. Calibration records and the exact procedures used should be maintained.

5.4.3 Acoustic Probe Method

The acoustic probe is an electronic device containing two electrodes and a battery-powered transducer. The probe is attached to a tape. The probe is lowered into the well until a sound is detected, indicating the electrodes in the probe have contacted the water surface. This method is similar to the electrical probe method discussed in Section 5.2.

5.5 Procedural Considerations For PFAS

Development of PFAS awareness in guidance and incorporation into specific SOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. For example, Federal drinking water MCL for Arsenic is 10,000 ppt where the EPA health advisory level is a combined 70 ppt for PFOA and PFOS.
Procedural modifications to field activities when sampling for PFAS analysis involve the concerns listed below. Practice good housekeeping, document these extra steps in detail, and maintain vigilance to adhere to clearly defined exclusion zones:

Good practices

- Use good safety planning and institute exclusion zones, contamination reduction zones, and support zones (no visitors within 30 feet of sampling).
- Do not eat in or near sampling areas (food packaging may contain PFAS).
- Be vigilant about PPE donning and doffing practices.
- Wash hands before and after eating .
- Wear powderless nitrile gloves and change them frequently.
- Develop a project-specific checklist for review with field personnel.
    - It should be project-specific.
    - Completed prior to beginning PFAS sampling event, or when there are staffing changes, or when sample media change and associated sampling/field equipment changes
    - Consider appropriate sampling/field work sequences for each matrix
    - Sampling/field work sequences
    - PFAS sampling/field work should occur first on sites where other contaminants will also be sampled.
    - CDM Smith PFAS Sampling Guidance Team can assist with development of your project-specific checklist.

Avoid

- **Polytetrafluoroethylene (PTFE),low density polyethylene (LDPE)**, sticky notes, waterproof field book, aluminum foil
- Field filtering of water samples because of glass fiber filters

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- Avoid use of markers, if possible. Use only regular ink pens during sampling. If needed, write over regular ink with marker after the sample containers are sealed.
- Consult materials checklists for equipment concerns

6.0 Restrictions/Limitations

6.1 Groundwater and Miscible Fluids

It may not be possible to obtain an accurate reading where water is rapidly dripping or flowing into a well, either from the top of the well or from fractures.

The effect of the water flowing into the well may interfere with an electronic water level measuring device, resulting in a false water level measurement. If water levels should be recorded in wells completed in aquifers that are recharging or discharging, the electronic water level indicator is the preferred measuring device but shall be used with awareness of possible false measurements. To minimize the effects of "splashing," a 1-inch drop pipe (decontaminated for environmental wells) may be lowered into the pumping well into which the water level indicator is then inserted. This will minimize the effect of "splashing" until the probe contacts the groundwater. It will also protect the probe from becoming tangled in pump wiring or well spacers associated with downhole equipment such as submersible pumps. It is recommended that pressure transducers be used when water levels need to be recorded at frequent intervals during aquifer testing (recharging or discharging) in combination with water level indicators.

6.2 Immiscible Fluids

For wells containing immiscible contaminants including light non-aqueous phase liquid (LNAPL) and dense non-aqueous phase liquid (DNAPL), field personnel will need to use special procedures for the measurement of fluid levels. The procedure to follow will depend on whether layers are light immiscibles that form lenses floating on the top of the water table (LNAPL), or dense immiscibles that sink through the aquifer and form lenses over less permeable layers (DNAPL).

In the case of LNAPL and DNAPL, measurements of immiscible fluid and water levels can be accomplished by using an oil/water interface probe manufactured by companies such as Solinst®, Geotech® and Heron®.

Techniques have been specially developed to measure fluid levels in wells containing immiscible fluids, particularly petroleum products. An oil/water interface probe can be used to detect the presence of conducting and nonconducting fluids. Thus, if a well is contaminated with low density, nonconducting immiscible fluids such as gasoline, the probe will first detect the surface of the gasoline, but it will not register electrical conduction (continuous signal). However, when the probe is lowered deeper to contact water, it will detect electrical conduction (beeping signal). Normally, a variation in an audible signal indicates the difference between phases. The variation in the audible signal associated with the detection of differing phase liquids will allow the user to obtain a groundwater depth and dense immiscible thickness measurement.

The oil/water interface probe is less effective with heavier and less refined petroleum products because the product tends to stick to the tape or probe, giving a greater product thickness measurement than is present. In instances where an accurate product thickness measurement cannot be completed, a bottom filling bailer can be used to provide an estimate of the thickness of LNAPL or DNAPL in a groundwater well. Note the diameter of the bailer opening is typical narrower than the diameter of the bailer sidewalls resulting in an underestimate of the thickness of the NAPL layer. Another method of measuring thickness of a separate phase layer is by using tubing which can be sealed at the top to form a vacuum after insertion through the layer, similar to a Coliwasa sampler or "thief". Note that water levels obtained in this situation are not suitable for determining hydraulic gradients without further interpretation. To use such data to determine hydraulic gradients, the difference in density between the light immiscible phase and water has to be considered.

6.3 Conducting Groundwater Level Measurement at Per- and Polyfluoroalkyl Substances (PFAS) Sites

The evolving regulatory standards for PFAS should be considered when implementing groundwater level gauging as well as when developing project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data

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management plans, accident prevention plans, health and safety plans, and laboratory statements of work used to guide the collection of samples for PFAS analysis.

Consider PFAS-specific sampling requirements during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), the selection of action levels, and reporting limits are all impacted with the inclusion of sampling for PFAS analysis.

Before implementing groundwater level monitoring during the collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to groundwater level measurement should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith regulatory updates, and state and federal regulatory limit updates, can be found on the PFAS InfoCenter:

https://cdmsmith.libguides.com/PFAS_InfoCenter

Procedural modifications for groundwater gauging and other related field activities at PFAS sites is attached. A check list containing common materials and sampling equipment that contain PFAS compounds can be found here:

https://www.yammer.com/cdmsmith.com/#/files/214861635584

7.0 References

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Tap Water Sampling	SOP 1-9 Revision: 8 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Robert Alexander

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the requirements for collecting tap water samples for the purpose of assessing water quality. General guidelines for purging the water supply system before sample collection are also provided. Depending on the objective of the sampling event as defined in the site-specific sampling plan, the water source may be from a private or public potable water supply, such as a groundwater well or a surface water reservoir.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Holding Tank - An in-house water reservoir that provides a limited reserve water supply and equalizes water pressure throughout the plumbing system. Most domestic well holding tanks have a storage capacity of approximately 30 gallons.

Onsite Water Supply - A source of potable water located on the property to be sampled. The water source could be a groundwater aquifer (i.e., a residential groundwater well) or a surface water body (i.e., a water intake from a lake).

Potable Water - Water considered safe for human consumption

Tap Water Samples - Samples of water collected from a faucet or spigot at a residence, business, or industrial plant. Usually, samples are collected from the tap(s) nearest the water supply source or area of interest along the distribution system.

Water Filter - A device used to remove suspended particulate matter and/or various compounds from the water source. One type of common filter is a water softener that uses a calcium-salt filter to remove calcium and magnesium ions from potable water to reduce the hardness.

2.2 Associated Procedures

- SOP 1-2, *Sample Custody*
- SOP 2-1, *Packaging and Shipping Environmental Samples*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-3, *Well Development and Purging*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

2.3 Discussion

Tap water sampling may be conducted in residential, commercial, or industrial settings. Consequently, sampling personnel will interface with the general public (i.e., homeowners, business owners, or concerned citizens) and must present themselves in the utmost professional manner. Permission to access the property must be obtained before conducting the tap water sampling event; the client should be consulted as to the proper notification procedures. At the time of the sampling, it is recommended that a letter of introduction be presented to the property owner or representative, explaining the purpose of the tap water sampling and indicating the name of the person and phone number to contact if the property owner has questions. At no time should the sampling team enter a home or business without the approval of the property owner; the property owner or representative must be present to enter a building.

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Generally, water supply sources and distribution systems can be categorized into two types:

- Onsite water supplies such as private, groundwater wells or surface water intakes for single residences, businesses, or industrial plants with limited distribution systems

- Large distribution systems from public or municipal groundwater or surface water supplies with extensive distribution systems for multiple users

The site-specific sampling plan or quality assurance project plan (QAPP) should describe the source of the potable water supply, the water distribution system, and other site-specific factors that may affect the water quality (well construction details, local hydrogeology, the presence of filters or holding tanks within the distribution system, pipe age, and composition, etc.). It is preferable to collect the samples from a tap located prior to a filtering device or a holding tank so that contaminants will be less likely to have been removed or allowed to settle out. The sampling objectives and sampling requirements, including analytical parameters, preservatives, and sample handling procedures must also be specified. Depending on the water source and distribution system, the site-specific sampling plan should describe the requirements for purging the system before collecting the tap water sample and for disposing of the purged water.

The procedures described in this SOP provide guidelines to obtain representative tap water samples from water supplies/distribution systems ranging from small onsite water supplies to large multiuser distribution systems.

3.0 General Responsibilities

Project Manager - Translates project requirements into technical direction of project. Sets technical criteria, reviews, and approves technical progress. Ensures that all participating personnel have proper training.

Field Team Leader - The field team leader is responsible for ensuring that sampling efforts are conducted in accordance with this procedure and any associated SOPs.

Sampling Personnel - Field team members are responsible for conducting tap water sampling events in accordance with this procedure, all associated SOPs, and requirements as described in the site-specific plans.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the site-specific sampling plan or QAPP.

4.0 Required Equipment

All or part of the equipment listed may be required at any specific site, depending on the plan(s) for that site.

- Site-specific plans including letter(s) of introduction
- Field logbook and indelible black ink pens and markers
- Forms and other documentation for sample shipment
- Sample containers, labels, and preservatives, as required
- Insulated cooler and waterproof sealing tape
- Ice bags or “blue ice”
- Plastic zip-top bags
- 5-gallon bucket and stopwatch
- Temperature, conductivity, pH, dissolved oxygen, and turbidity meters (with clean beakers or other appropriate containers), as required by the site-specific plans
- Photoionization detector (PID) and/or other monitoring/screening instruments as required by the site-specific health and safety plan or sampling plan
- Decontamination supplies, as required by SOP 4-5

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- Personal protective equipment (PPE), as required by the site-specific health and safety plan
- Latex, nitrile, or other appropriate gloves

*** If sampling for Per- and Polyfluoroalkyl Substances (PFAS)**, the potential for cross-contamination from common materials must be evaluated. Commonly used sampling materials and equipment to avoid (or verify composition) include:

- **PTFE, low density polyethylene (LDPE)**, **Teflon**, sticky notes, waterproof field book, aluminum foil, plastic sheeting
- Field filtering of water samples because of **glass** fiber filters
- Avoid use of markers, if possible, during sampling - use only regular ink pens. If needed, write over regular ink with marker after the sample containers are sealed.

Consult materials checklists for other equipment concerns. A check list containing common materials and sampling equipment that contain PFAS compounds is attached, and an up-to-date list will be maintained here:
https://www.yammer.com/cdmsmith.com/#/files/214861635584

5.0 Procedures

1. Obtain the name(s) of the resident(s) or water supply owner/operator, the exact mailing address, and telephone numbers. This information is required to obtain access to the property to be sampled and to submit a letter of introduction to the owner/representative.

2. Determine the location of the tap to be sampled based on its proximity to the water source. It is preferable that the tap being sampled be prior to any holding or pressure tanks, filters, water softeners, or other treatment devices that may be present.

3. If the sample must be collected at a point in the water line beyond a pressurization or holding tank, a sufficient volume of water shall be purged to provide a complete exchange of fresh water into the tank and at the location where the sample is collected. If the sample is collected from a tap or spigot located just before a storage tank, spigots located inside the building or structure shall be turned on to prevent any backflow from the storage tank to the sample tap or spigot. It is generally advisable to open as many taps as possible during the purge, to ensure a rapid and complete exchange of water in the tanks.

4. Samples collected to determine if system related variables (e.g., transmission pipes, water coolers/heaters, holding/ pressurization tanks) are contributing to the quality of potable water shall be collected after a specific time interval (e.g., weekend, holiday). Sample collection shall consist of an initial flush, a sample after several minutes, and another sample after the system has been purged.

5. Devices such as hoses, filters, or aerators attached to the tap may harbor a bacterial population and therefore shall be removed before sampling.

6. Potable water samples which have been chlorinated must be treated with sodium thiosulfate to dechlorinate the sample prior to other preservation and shipping for analysis.

7. Sample containers shall not be rinsed before use when sampling for bacterial content, and precautions shall be taken to avoid splashing drops of water from the ground or sink into either the bottle or cap.

8. Samples of the raw water supply and the treated water after chlorination shall be collected when sampling at a water treatment plant.

9. In the logbook, record the location and describe the general condition of the tap selected for sampling. The rationale used in selecting the tap sampling location, including any discussions with the property owner, shall also be recorded. Provide a sketch of the water supply/distribution system noting the location of any filters or holding tanks and the water supply source (i.e., an onsite groundwater well or surface water intake or a water service line from a public water main). If an onsite water supply is present, observe and record the surrounding site features that may provide potential sources of contamination to the water supply.

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10. Don the appropriate personal protective clothing as dictated by the site-specific health and safety plan. Latex or nitrile gloves shall be changed between sampling locations to avoid possible cross contamination of the tap water samples.

11. Before sample collection, the supply system shall be purged by turning the cold water tap on. The following general guidelines shall be followed to determine when the system is adequately purged (refer to the site-specific sampling plans for any other requirements):

    - **Onsite Water Supply**. A minimum of three standing volumes of water (i.e., the static volume of water in the well and holding tank, if present) shall be purged. Obtain water temperature, conductivity, and pH measurements after each volume of water is purged. If the standing volume of water in the supply system is unknown, the tap shall be allowed to run for a minimum of 15 minutes and temperature, conductivity, and pH measurements, or other parameters as specified by the project plan, shall be collected at approximately 3- to 5-minute intervals. (In general, well construction details and holding tank volumes shall be obtained before conducting the sampling event to estimate the standing volume of the water supply system.) The system is considered adequately purged when the temperature, conductivity, and pH stabilize within 10 percent for three consecutive readings. If these parameters do not stabilize within 15 minutes, then purging shall be discontinued and tap water samples may be collected as discussed in Section 6.0.

    - **Large Distribution Systems**. Because it is impractical to purge the entire volume of standing water in a large distribution network, a tap shall be run for a minimum of 5 minutes, which shall be adequate to purge the water service line. Obtain temperature, conductivity, and pH measurements at approximately 1-minute intervals. The system is considered adequately purged when the temperature, conductivity, and pH readings, or other parameters as specified by the project plan, stabilize within 10 percent for three consecutive readings. If these parameters do not stabilize within 5 minutes, then purging shall be discontinued and tap water samples may be collected as discussed in Section 6.0. During purging, a 5-gallon bucket and stopwatch may be used to estimate the flow rate if required by the site-specific plans. Dispose of the purged water according to the site-specific plans. Record the temperature/conductivity/pH readings, or other parameters as specified by the project plan, the volume of water purged, the flow rate if measured, and the method of disposal in the field logbook.

12. After purging the supply system, collect the samples directly from the tap (i.e., if a hose was used for purging, the hose shall be disconnected before sampling). Any fittings on the end of the faucet that might introduce air into the sample (i.e., a fine mesh screen that is commonly screwed onto the faucet) shall be removed before sample collection also.

13. Obtain a smooth-flowing water stream at moderate pressure with no splashing. Samples for volatile organic compound (VOC) analyses shall be collected using a reduced flow rate (see below). Hold the sample bottle in one hand and the cap in the other; do not touch the inside of the cap; do not allow the faucet to touch the inside of the bottle; do not allow splashing water from the ground or sink to enter the bottle or cap. VOC samples shall be filled first, followed by other organic analyses, inorganic analyses, and then other water quality parameters. Refer to the site-specific plans for the required sample parameters, preservatives, and sample handling procedures. The following general guidelines shall be followed when collecting samples:

    - **VOC**. Reduce the flow rate to a minimum to reduce aeration of the VOC sample. Use a pre-preserved “test” vial to determine the appropriate amount of hydrochloric acid (HCl) needed to reduce the pH of the sample to less than 2. Dispose of this test vial after the appropriate amount of HCl is determined. Add the required amount of HCl to the sample vials and then fill the vials with the sample water. Quickly replace the cap and check for air bubbles. If air bubbles are present, the vial will be discarded, and a new vial will be filled as detailed above.

    - **Semivolatile Organic Compounds (SVOCs), Pesticides, and Polychlorinated Biphenyls (PCBs)**. Generally, aqueous samples for SVOCs and pesticides/PCBs require no preservative. Sample containers may be filled directly from the tap.

    - **Total (unfiltered) Metals**. Generally, tap water samples are not collected for filtered (dissolved) metals because risk assessment data needs require total metals analyses (check the site-specific plans to determine filtering requirements). The sample container

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for total metals may be filled directly from the tap. Nitric acid (HNO_3) shall then be added to the filled container to preserve the sample to a pH less than 2.

- **Other Sample Parameters**. Other water quality parameters, such as cyanide dissolved oxygen, hardness, nitrate/nitrite, etc., shall be collected and preserved as required by the site-specific sampling plans.

14. Label all sample containers as required and place them in a cooler with ice. Record all appropriate data in the field logbook and on the chain-of-custody forms.

5.1 Procedural Considerations for PFAS

The evolving regulatory standards for PFAS must be considered in the implementation of tap water sampling as well as the development of project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans and laboratory statements of work used to guide the collection of samples for PFAS analysis.

A consideration of the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, reporting limits are all impacted with the inclusion of sampling for PFAS analysis.

Before implementing this tap water sampling TSOP for collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to surface water sampling should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith Regulatory Updates can be located at the INFOCENTER. Specific State and Federal Regulatory Limit updates can be found here: https://cdmsmith.libguides.com/PFAS_InfoCenter

A check list containing common materials and sampling equipment that contain PFAS compounds is attached and can also be found here: https://www.yammer.com/cdmsmith.com/#/files/214861635584

Procedural modifications to field activities when sampling for PFAS analysis involve the concerns listed below. Good Housekeeping, detailed documentation of these extra steps and vigilance about clearly defined exclusion zones that are strictly adhered to:

Good practices

- Utilize good safety planning and institute Exclusion Zones, Contamination Reduction Zones, and Support Zones (No visitors within 30 feet of sampling)
- Do not eat in or near sampling areas (food packaging may contain PFAS)
- Be vigilant about PPE donning and doffing practices
- Wash hands before and after eating
- Wear powderless nitrile gloves and change them frequently
- Only open sample container during sample collection and never set the sample container lid down
- Keep hands away from the container opening when sampling and keep lid protected
- Develop a Project Specific Checklist for review with field personnel
    - It must be project specific
    - Completed prior to beginning PFAS sampling event, or
    - Whenever there are staffing changes, or
    - Whenever sample media change and associated sampling/field equipment changes
    - Consider appropriate sampling/field work sequences for each matrix
    - Sampling/field work sequences
    - PFAS sampling/field work should occur first on sites where other contaminants will also be sampled
    - CDM Smith PFAS Sampling Guidance Team can assist with development of your project specific checklist

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6.0 Restrictions/Limitations

To protect the sample from contamination on the exterior of a tap, a tap shall not be chosen for sampling if any of the following conditions exist:

- A leaky tap allowing water to flow out from around the stem of the valve handle and down the outside of the faucet.
- A tap located too close to the bottom of the sink or the ground surface.
- A tap that allows water to run up on the outside of the lip.
- A tap that does not deliver a steady stream of water. A temporary fluctuation in line pressure may cause sheets of microbial growth, lodged in some pipe sections or faucet connections, to break loose.

Careful sampling for VOC analysis, or for any other compound(s) that may be degraded by aeration, is necessary to minimize sample disturbance and, hence, analyte loss.

7.0 References

U. S. Environmental Protection Agency. 2019. Region 4. The Field Branches Quality System and Technical Procedures, Potable Water Supply Sampling. ASBPROC-305-R4. June.

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Attachment – Materials of concern when sampling for tap water for PFAS compounds

Item	Good to Use	Needs Verification (2)(3)	Should Avoid (1)(2)	Comments
Field Clothing or PPE				
Clothing or boots containing "water resistance" or "stain-treated" fabrics				
Cloths washed with fabric softeners				Fabric softeners may contain PFAS.
New and unwashed clothing				Fabric treatment may contain PFAS.
Uncoated Tyvek				USEPA PFAS sampling guidance from Region 2 prohibits use of Tyvek.
Coated Tyvek				
PVC or wax-coated fabrics				
Neoprene				
Synthetic and natural fibers (preferably cotton)				
Steel-toed boots made with polyurethane and PVC				If it is not possible to find PFAS free steel-toed footwear, PFAS-free over boots may be worn. The over boots must be put on and the hands washed after putting the over boots on prior to the beginning of the sampling activities. Over boots may only be removed in the staging area and after the sampling activities have been completed.
Well laundered clothes				Several times from time of purchase
Well washed cotton coveralls				Washed several times
Personnel Hygiene and Protective Skin Products				
Sunscreens				Good to use: Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, and baby sunscreens that are "free" or "natural."
Insect Repellents				Good to use: Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics.
International Brands of sunscreens and insect repellents				Must be evaluated on a case-by-case basis.
Field Sampling Items				
Waterproof field paper or books				Use loose plain paper.
Post-it notes				
Aluminum foil				
Brand-name markers				Sharpie may be used to label sample bottles in the staging area, but markers should not be used in the immediate sampling environment.
Off-brand markers				

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Item	Good to Use	Needs Verification (2)(3)	Should Avoid (1)(2)	Comments
Ball point pens				
Plastic clipboards				NJ DEP sampling guidance, use metal clipboard.
Plastic table cover				
Sampling Equipment				
Item containing high-density polyethylene (HDPE)				
Item containing polypropylene				PP sample bottles must be used for drinking-water samples in accordance with USEPA method 537.1.1.
Item containing polyurethane				
Item containing Polyvinyl chloride (PVC)				
Item containing silicon				
Item made of stainless steel				
Alconox®				
Citronex®				
Liquinox®				
Powderless nitrile gloves				
HDPE Hydrasleeves or sonic core bags				
Neoprene				
Crisco® or other vegetable-based greases for lubricating parts				
Item containing PTFE				Items or equipment that contain PTFE parts that will be in direct contact with sampling media.
Item containing Teflon®				Field sampling items or equipment that contains Teflon® and that will be in direct contact with the sampling media.
Item containing fluoropolymer				
Low-density polyethylene (LDPE)				Items or equipment that contains LDPE parts and that will be in direct contact with the sampling media.
Viton® O-rings				Viton® O-rings used in pressure washers used for sampling equipment decontamination.
Glass sample containers				
Field filter				Field filtration should be avoided regardless of filter types.
Decon 90				
Items containing fluorosurfactants				
Teflon-bearing plumber's tape				

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Item	Good to Use	Needs Verification (2)(3)	Should Avoid (1)(2)	Comments
Blue (or chemical) ice				Later data (unpublished) suggest no cross contamination from blue ice. The category may be changed after data are published.
Water ice				Double bag in polyethylene bags
Internal valves and equipment parts for sampling or decon				
Methanol or other solvents				
LDPE plastic bags (e.g., Ziploc® bags)				For larger biota sampling, Ziploc bags may be used, but collecting an equipment blank is recommended because these bags may be made of LDPE.
Drilling fluids				
LDPE sonic core sample bags				Manufactured by Boart Longyear and Hole Products.
Equipment with moving parts that may be lubricated with PFAS containing lubricants or greases				
Rental equipment				Must be verified to have no PFAS-bearing parts prior to use.
Others				
Food wrappers				Field personnel must wash hands after having food wrapped with grease-repelling paper.

(1) If an alternative is not available for an item that may contain PFAS, the item should be pre-tested for PFAS before use in the field.
(2) This mostly refers to the immediate sampling environment, particularly, the item is in contact with environmental media to be sampled.
(3) There are no standard operation procedures on how an item can be verified; please contact PFAS experts for advice on the best practice of testing a potential PFAS containing item. PFAS experts at CDM Smith may be contacted through the Emerging Contaminants Group.

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Field Measurement of Total Organic Vapors	SOP 1-10 Revision: 8 Date: February 2020

Approved: Ernest Ashley **Technical Review:** Nicholas Castonguay, P.G.

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to define the techniques and the requirements for the measurement of total organic vapors in the field.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Photoionization Detector (PID) - A field portable, hand-held instrument that measures the concentration of gaseous organic compounds through the photoionization of organic vapors

Flame Ionization Detector (FID) - A field portable, hand-held instrument that measures the concentration of gaseous organic compounds through the flame ionization of organic vapors

Colorimetric Indicator Tubes - A field portable, hand-held pump that measures the concentration of specific organic gaseous compounds in air through a glass tube containing a granular carrier solid that has been saturated with a specific chemical reagent (i.e. benzene). The indicator tubes and pumps are typically referred to by their brand names such as Dräger Tubes®.

Gas Chromatograph - A field portable, hand-held instrument that measures the concentration of specific volatile organic compounds (VOCs) and is capable of analyzing groundwater, soil, and air samples. The gas chromatographs are typically referred by their brand names such as the FROG-5000™, which is identified as a gas chromatograph and photoionization detector (GC/PID).

2.2 Associated Procedures

- SOP 1-4, *Subsurface Soil Sampling*
- SOP 1-5, *Groundwater Sampling Using Bailers*
- SOP 1-6, *Water Level Measurement*
- SOP 1-8, *Volatile Organic Compound Air Sampling Using USEPA Method TO-15 with SUMMA Canister*
- SOP 1-12, *Low-Stress (Low-Flow) Groundwater Sampling*
- SOP 3-1, *Geoprobe® Sampling*
- SOP 3-5, *Lithologic Logging*
- SOP 4-3, *Well Development and Purging*

2.3 Discussion

The measurement of organic vapors is a required step during numerous field and site characterization activities. The primary purpose of such measurements is health and safety monitoring to determine if the breathing zone in a work area is acceptable or if personal protective equipment such as a respirator or a supplied air device is necessary for field personnel. At the perimeter of a work area, measurements can be taken as part of a perimeter air monitoring plan to document protection of a surrounding community. In addition to health and safety monitoring, total organic vapor measurement is also used in conjunction with sampling activities, including screening subsurface soil samples, soil vapor and indoor air sampling, and groundwater sampling, where measurements are useful for establishing approximate contaminant levels or ranges.

The two types of instruments most commonly used to measure total organic vapors are PIDs and FIDs. Both instruments first ionize the gaseous compound and then measure the response, which is proportional to the concentration. For site specific contaminants of concern (COCs) such as benzene, colorimetric tubes can be used to determine air concentrations in the breathing zone primarily for

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health and safety purposes. For multi-media organic vapor measurement, the GC/PID can be used to determine specific VOC concentrations primarily for site characterization purposes.

2.3.1 PID Operation

The PID is preferred when the compound of interest is an aromatic or halogenated volatile organic compound (VOC). The PID ionizes the sampled vapors using an ultraviolet lamp that emits light energy at a specific electron voltage (eV - labeled on the lamp). The ultraviolet lamp produces photons that are absorbed by the sampled vapor molecule. The molecule becomes excited, producing a positively charged ion and emitting an electron. The number of electrons emitted is proportional to the concentration of the sampled gases. Every organic compound has a specific ionization potential in electron volts. The energy emitted by the lamp should be higher than the ionization potential of the compound for the compound to become ionized and emit an electron. If the ionization potential of the compound is higher than the eV of the lamp, there will be no response on the instrument. Therefore, the ionization potential of the known or suspected compounds shall be checked against the energy of the ultraviolet lamp to verify that the energy provided by the lamp is greater. The manufacturer's manual should be consulted to determine the appropriate ultraviolent lamp to be used for the known or suspected compounds. Additionally, manufacturer's manuals shall be consulted to obtain the appropriate correction factors for known or suspected contaminants.

Water vapor in the vapor sample can interfere with the PID detector and cause the instrument to stop responding or cause the zero baseline to drift. This can occur using the PID on a rainy day or when sampling headspace samples that have been in the sun. If moisture interference is suspected, the calibration gas shall be used to check the instrument response by inserting the gas as a check sample, not by recalibrating. If the response is lower than the gas level, then the probe and the ionization chamber should be dried out before reusing the instrument.

The sampling probe shall not be inserted directly into soil samples or dusty areas, as the instrument vacuum will pull dirt into the ionization chamber. Under particularly dirty or dusty conditions, the lamp may become covered with a layer of dust. If dirty conditions are encountered, or if the instrument response seems to have decreased, then the lamp should be cleaned. The instrument manual provides instructions on how to remove the instrument cover to access the lamp, and how to clean the screen in the ionization chamber and the surface of the lamp.

The instrument manual may provide instruction on use of disposable dust and/or moisture filters for minimizing effects from dust and/or moisture. The ultraviolet lamp in the PID is sensitive to shock, especially when using the higher eV lamps. Therefore, they should be handled and transported carefully.

2.3.2 FID Operation

The FID is preferred when sampling for petroleum hydrocarbons and other organic compounds such as methane (landfill gases). It responds well to aromatic hydrocarbons but is not as convenient to use as the PID. The FID allows measurement of a wide variety of compounds, but in general its sensitivity is not as high as the PID for compounds where the PID is applicable. The FID is virtually unaffected by ambient levels of water vapor.

The FID ionizes the vapor sample by burning it in a hydrogen/air flame and measuring the response beyond what is caused by the hydrogen alone. This instrument requires a hydrogen supply, contained in a small tank in the instrument. This hydrogen, including the gas in the instrument tank, is considered a flammable gas and appropriate requirements should be adhered to when shipping. The instrument shall be emptied of hydrogen before shipping. Federal Express Hazardous Material shipping manifests must be completed when shipping the gas.

The hydrogen gas in the FID combustion chamber is ignited by pressing a red button on the side of the instrument, which sends electrical current to a small resistance coil igniter in the combustion chamber. This igniter is very sensitive, and if the red button is pressed for longer than 5 seconds, the coil will burn out and the instrument will be unusable unless another igniter is available. If the instrument will not light, check the electrical connections and switches for proper settings. Check that the pump is pumping and allow fresh air to flow through the combustion chamber for several minutes before lighting. Check to see if the exhaust port of the combustion chamber is dirty.

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2.3.3 Colorimetric Indicator Tube Operation (DrägerTubes®and Pump)

Colorimetric indicator tubes such as DrägerTubes® and pumps can provide real time VOC and other organic gaseous compound concentrations in ambient air. The colorimetric tube is preferred when monitoring for site specific COCs in the breathing zone. The pump is compatible with over 500 different short-term gas detection tubes and is suitable for site characterization and industrial hygiene purposes. The Dräger Tubes® are capable of analyzing VOCs in the air with results reporting in parts per million (ppm) concentrations. The glass colorimetric indicator tubes typically consist of a glass tube containing a granular solid that has been saturated with a specific chemical compound (i.e., benzene). As contaminated air is pumped through the glass tube, the contaminant reacts with the reagent on the granular solid to produce a color stain or change in color, which is proportional to the amount of contamination in the air. The air is drawn through the DrägerTubes® by a hand pump or a battery-powered pump that controls the volume of air sampled. The number of pump strokes (i.e. the volume of air) required to reach this full stain is compared to a chart to determine the concentration of the contaminant in the air. A set number of pump strokes (volumes) of air will be drawn through the tube and the length of stain is compared to a calibration scale, often printed on the tube, to determine the concentration. For these tubes, a high concentration would cause a deeper or darker color change after a set number of pump strokes. It is critical that the operator of the tube be familiar with the manufacturer's directions and knows which mode of operation is used.

2.3.4 Gas Chromatograph Operation (FROG-5000™)

A portable field gas chromatograph such as the FROG-5000™ can provide real time soil, groundwater, and air VOC concentrations that can aid in the characterization of contaminated sites. The FROG-5000™ chemical analysis system is a field portable gas chromatograph/photoionization detector (GC/PID) capable of analyzing samples in minutes. The FROG-5000™ is vendor calibrated for VOCs of interest and is ready to analyze samples upon arrival in the field. The FROG-5000™ quantitatively determines VOC concentrations down to single part per billion (ppb) or at high part per million (ppm) concentrations depending on its configuration. During sample analysis (thermal desorption), the VOCs are passed into a miniature GC column where the mixed VOC analytes are each separated. The PID then determines the resolved VOCs eluting from the GC and outputs the concentration. A real-time readout and associated laptop connected to the FROG-5000™ allows the field analyst to monitor analysis of each sample by observing the development of the gas chromatogram in real-time.

3.0 Responsibilities

Project Manager - The project manager is responsible for ensuring that field activities are conducted in accordance with this procedure and any other SOPs pertaining to the specific activity.

Field Team Leader - The field team leader is responsible for ensuring that field personnel conduct field activities in accordance with this and other relevant procedures. The field team leader is responsible for ensuring the appropriate field equipment is used for site specific or contaminants of concern.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project-specific quality assurance plan.

4.0 Required Equipment

- Site-specific plans (i.e., scope of work)
- Health and safety plan
- Field logbook
- Waterproof black ink pen
- Personal protective clothing and equipment
- Photoionization detector
- Flame ionization detector
- Dräger Tubes® for required COCs and pump
- Calibration gases in a range appropriate for the expected use

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- 0.5 liter (16-ounce) or "Mason" type glass jar or Ziploc-type plastic bags
- Hydrogen canister and fill valve and hose (if using FID for a period of more than 1 day)

5.0 Procedures

5.1 Direct Reading Measurement (PID and FID)

1. Connect the measurement probe to the instrument and make necessary operational checks (e.g., battery check, etc.) as outlined in the manufacturer's manual.

2. Calibrate the instrument following the applicable manufacturer's manual.

3. Make sure the instrument is reading zero and all function and range switches are set appropriately.

4. Insert the end of the probe directly into the atmosphere to be measured (e.g., breathing zone, monitoring well casing, split spoon, etc.) and read the total organic vapor concentration in parts per million (ppm) from the instrument display. Apply the appropriate correction factor if necessary. Record the highest instrument response.

5. Immediately document the reading in the field logbook or on the appropriate field form.

5.2 Headspace Measurement (PID and FID)

1. Connect the measurement probe to the instrument and make necessary operational checks (e.g., battery check, fan check, etc.) as outlined in the manufacturer's manual.

2. Calibrate the instrument following the appropriate manufacturer's manual.

3. Make sure the instrument is reading zero and all function and range switches are set appropriately.

4. Fill a clean glass jar or Ziploc-type plastic bag approximately half-full of the sample to be measured. For a jar, quickly cover the top of the jar with one or two sheets of clean aluminum foil and apply cap to seal the jar. For a bag, quickly seal the bag minimizing volume of air in the bag.

5. Allow headspace to develop for approximately 10 minutes. It is generally preferable to shake the sealed jar for 10 to 15 seconds at the beginning and end of headspace development. For a bag, kneed the bag to break apart the sample and maximize sample surface area.

Note: When the ambient temperature is below 0°C (32°F), the headspace development and subsequent measurement shall occur within a heated vehicle or building.

6. For a jar, remove the jar cap and quickly puncture the foil and insert the instrument probe to a point approximately one-half of the headspace depth. Do not let the probe contact the soil. For a bag, quickly puncture the bag wall and insert the probe, wrapping the bag wall around the probe stem to minimize loss of vapors. If using a PID and there is condensation on the inside of the jar or bag, only leave the probe in the jar or bag long enough to obtain a reading. Remove the probe and allow fresh air to flow through the instrument to avoid excess water vapor to build up.

7. Read the total organic vapor concentration in ppm from the instrument display. Apply the appropriate correction factor if necessary. Record the highest instrument response.

8. Immediately record the reading in the field logbook or on the appropriate field form.

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5.3 Colorimetric Indicator Tube Measurement (Dräger Tubes® and Pump)

1. Sample pumps should be checked for leaks to ensure that the appropriate volume of air is drawn through the tube. The pump should be allowed to fully complete every pump stroke. Incomplete strokes or leaks in the system will cause less than the appropriate volume of sample air to pass through the tube, potentially resulting in a lower reading than is actually present. Perform a pump leak test by inserting an unopened tube into the hand pump socket. Squeeze the pump completely and release. If the stroke indicator has not appeared after 15 minutes, then your pump is leak-proof. If the stroke indicator does appear, then try another pump or contact the field team leader for direction. Otherwise, remove the tube and reset the counter to zero and prepare for use.
2. Open both ends of the tube using the integrated ceramic blade on the bottom of the pump or use the tube opener. When using the opener on the pump, make sure to keep the tube pressed against the ceramic edge and turn the tube to score the glass. When using the tube opener, use the instructions on the side of the pump box. Insert the tube into the pump with the black arrow facing inwards. Squeeze the pump to create one stroke. The number of strokes required will be indicated on your chosen tube pack and instructions.

3. The tube with the hand pump may be operated as follows:

   A. The pump may be operated until the length of the stain reaches a certain preset point. The number of pump strokes (i.e. the volume of air) required to reach this full stain is compared to a chart to determine the concentration of the contaminant in the air. In this case, a high concentration of contaminant in the air would require fewer strokes to reach full stain.

   B. A set number of pump strokes (volumes) of air will be drawn through the tube and the length of stain is compared to a calibration scale, often printed on the tube, to determine the concentration. For a set number of pump strokes, a high concentration would cause a longer stain.

   C. A predetermined number of pump strokes of air are drawn through the tube and the degree or tint of the color change is compared to a chart to determine the concentration. For these tubes, a high concentration would cause a deeper or darker color change after a set number of pump strokes. It is critical that the operator of the tube be familiar with the manufacturer's directions and knows which mode of operation is used.

4. Read the total organic vapor concentration and record in the field logbook. If breathing zone air concentrations exceed contaminant thresholds, keep a safe distance from the work zone, contact the field team leader, and upgrade PPE (i.e. level D to C), if required and available. If upgraded PPE is not available, contact the field team leader to discuss plans for executing work when the appropriate PPE is available.

5.4 Gas Chromatograph Operation (FROG-5000™)

1. Before conducting field work, confirm the FROG-5000™ is functioning properly and can connect to the appropriate software on the portable computer.

2. Before sampling, make sure the instrument settings are correct for the application. Typically, the equipment rental company can provide training, calibrate, and set up project specific analytical parameters to analyze for so that the unit is ready to go. Run a blank before attempting sample analysis, a blank run should produce a clean baseline.

3. For soil, water and air sampling, refer to the FROG-5000™ user manual since there are numerous steps to prepare and analyze the samples. Before conducting field work, review the user manual to ensure proper and efficient use of the software and instruments in a field setting. The user manual is located here: http://www.defiant-tech.com/pdfs/FROG-5000-Manual-2017-Rev3.pdf.

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5.5 Procedural Considerations For PFAS

Development of PFAS awareness in guidance and incorporation into specific SOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. (For example, Federal drinking water MCL for Arsenic is 10,000 ppt where the EPA health advisory level is a combined 70 ppt for PFOA and PFOS.)
Procedural modifications to field activities when sampling for PFAS analysis involve the concerns listed below. Good housekeeping, detailed documentation of these extra steps, and vigilance about clearly defined exclusion zones that are strictly adhered to shall be followed:

PFAS Sampling Materials Checklist

- Use good safety planning and institute exclusion zones, contamination reduction zones, and support zones (no visitors within 30 feet of sampling).
- Do not eat in or near sampling areas (food packaging may contain PFAS).
- Be vigilant about PPE donning and doffing practices.
- Wash hands before and after eating.
- Wear powderless nitrile gloves and change them frequently.
- Only open sample container during sample collection and never set the sample container lid down.
- Keep hands away from the container opening when sampling and keep lid protected.
- Develop a project specific-checklist for review with field personnel.
    - It should be project-specific.
    - Completed prior to beginning PFAS sampling event or whenever there are staffing changes or whenever sample media change and associated sampling/field equipment changes
    - Consider appropriate sampling/field work sequences for each matrix
    - Sampling/field work sequences
    - PFAS sampling/field work should occur first on sites where other contaminants will also be sampled.
    - CDM Smith PFAS Sampling Guidance Team can assist with development of your project-specific checklist.

Avoid

- **PTFE, LDPE**, sticky notes, waterproof field book, aluminum foil
- Field filtering of water samples because of **glass** fiber filters
    - Avoid use of markers, if possible, during sampling; use only regular ink pens. If needed, write over regular ink with marker after the sample containers are sealed.
- Consult materials checklists for equipment concerns.

6.0 Restrictions/Limitations

The two PID and FID methods outlined above are the most commonly used for field measurement of total organic vapors but do not apply to all circumstances. Consult project- or program-specific procedures and guidelines for deviations. Both the PID and FID provide quantitative measurement of total organic vapors, but generally neither instrument is compound-specific. The typical reading range of the PID is 0 to 10,000 ppm, and the typical reading range of the FID is 0 to 50,000 ppm. The FID will measure methane while the PID will not. ***Note***: The presence of methane will cause erratic PID measurements. In methane rich environments, toxic organic vapors shall be monitored with an FID. If desired, a charcoal filter can be placed temporarily on the FID inlet probe, which will trap all organic vapors except methane. The filtered (methane only) reading can be subtracted from unfiltered (total organic vapors) to provide an estimate of non-methane organic vapors. The reading accuracy of both instruments can be affected by ambient temperature, barometric pressure, humidity, lithology, etc.

The colorimetric Dräger Tubes® method is commonly used to determine the concentration of site specific COCs primarily for industrial hygiene purposes. The tubes' accuracy may be affected by temperature, humidity and atmospheric pressure. Colder temperatures will typically slow down the reaction and high temperatures may affect the rate of chemical reaction. If tubes are to be used in cold weather, they should be stored in a warm place and carried next to the body. Where temperature will significantly affect the performance of the tube, the manufacturer will include compensation factors in the instructions. Regarding limitations, tubes can have poor accuracy with errors ranging from 25% to 50%. The manufacturers generally provide accuracy information with the

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instructions. The tubes have a specific shelf-life which may cause the degradation of the reagent. Chemical reagents will deteriorate over time, even if the tube is not opened and exposed to air. High temperatures may cause degradation of the reagent. The manufacturer will stamp an expiration date on each pack of tubes. Storing the tubes in a refrigerator may maintain or extend the shelf life, but expired tubes should not be used.

The GC/PID such as the FROG-5000™ is typically used for site characterization and can also be used for industrial hygiene purposes. The FROG-5000™ quantitatively determines soil, groundwater and air VOC concentrations down to single part per billion (ppb) concentrations or at high part per million (ppm) concentrations depending on its configuration. The chemicals commonly detected by the FROG-5000™ include benzene, toluene, ethylbenzene, xylenes, vinyl chloride, trans-1,2-dichloroethene, cis-1,2-dichloroethene, trichloroethene, tetrachloroethene, chlorinated alkenes, and naphthalene. Please refer to the user manual for restrictions/limitations: http://www.defiant-tech.com/pdfs/FROG-5000-Manual-2017-Rev3.pdf.

6.1 Conducting Total Organic Vapor Monitoring at Per- and Polyfluoroalkyl Substances (PFAS) Sites

At sites where sample collection for PFAS analysis will occur, the evolving regulatory standards for PFAS should be considered in the implementation of total organic vapor monitoring as well as the development of project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans, and laboratory statements of work used to guide the collection of samples for PFAS analysis.

A consideration of the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels reporting limits are all impacted with the inclusion of sampling for PFAS analysist.

Before implementing total organic vapor monitoring during the collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to field measurements for total organic vapors should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith regulatory updates, as well as state and federal regulatory limit updates can be found on the PFAS InfoCenter: https://cdmsmith.libguides.com/PFAS_InfoCenter

Procedural modifications for groundwater gauging and other related field activities at PFAS sites is attached. A checklist containing common materials and sampling equipment that contain PFAS compounds can be found here: https://www.yammer.com/cdmsmith.com/#/files/214861635584

7.0 References

American Society for Testing & Materials (ASTM). D4490-96 (Re-Approved 2016), Standard Practice For Measuring the Concentration of Toxic Gasses or Vapors Using Detector Tubes.

Defiant Technologies Inc, FROG-5000™. Chemical Analysis User's Manual, 2018 (Rev.3). http://www.defiant-tech.com/pdfs/FROG-5000-Manual-2017-Rev3.pdf.

Department of Defense. Environmental Field Sampling Handbook, Revision 1. April 2013 or current revision.

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Low- Stress (Low-Flow) Groundwater Sampling	SOP 1-12 Revision: 3 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Nicholas Castonguay, P.G.

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define the procedural requirements for low-flow (minimal drawdown) groundwater sampling.

2.0 Background

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Low-flow groundwater sampling is a method of collecting samples from a well that, unlike traditional purging methods, does not require the removal of large volumes of water from the well. The objective of low-flow groundwater sampling is to collect samples with minimal alterations to water chemistry through pumping the well at a rate low enough to minimize drawdown and to avoid disturbance in the well. Low-flow groundwater sampling refers to the velocity that water enters the pump intake, and that is imparted to the formation pore water in the immediate vicinity of the well screen. It does not necessarily refer to the flow rate of water discharged at the surface of the well, which can be affected by flow regulators or restrictions.

Water level drawdown provides the best indication of the stress imparted by a given flow-rate for a given hydrogeological situation. The objective of low-flow groundwater sampling is to pump the well in a manner that minimizes stress (drawdown) to the system. Minimal drawdown should be stabilized so that the water to be sampled is representative of the formation surrounding the screened interval and is not from the stagnant water column above the screened interval. Minimal drawdown is achieved to the extent practical taking site sampling objectives into account. Typically flow rates on the order of 0.1 to 0.5 liter per minute (L/min) are used. However, achieving flow rates of 0.1 to 0.5 L/min can be dependent on site-specific hydrogeology. Some very coarse-grained sand formations have successfully been sampled via low-flow techniques at flow rates up to 1 L/min. The effectiveness of using low-flow purging is intimately linked with proper well screen location, well screen length, and well construction and development techniques.

Low-flow groundwater sampling can be used to collect samples for all categories of aqueous-phase contaminants and naturally occurring analytes, including volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), and other organic compounds including metals and other inorganics, pesticides, polychlorinated biphenyls (PCBs), radionuclides, microbiological constituents, and per- and polyfluoroalkyl substances (PFAS). Low-flow groundwater sampling techniques are particularly well-suited in applications where it is desirable to sample aqueous-phase constituents that may sorb or partition to particulate matter. It is not applicable to sampling wells that contain either light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs).

A variety of sampling devices are available for low-flow groundwater sampling, including peristaltic pumps, bladder pumps, electrical submersible pumps, and gas-driven pumps. Pump type should be selected based on known site conditions, including well depth, well diameter, water level, and anticipated well volume, as well as sampling objectives. Note that peristaltic pumps (suction pumps) cannot be used under conditions where the water table is greater than 25 feet below ground surface. Additionally, in most instances, peristaltic pumps may not be used for collecting VOC samples because they create a vacuum that potentially contributes bias to sampling for VOC's via low flow techniques. Bailers, too, are generally inappropriate devices for low-flow sampling. Gas-driven pumps are generally not advisable for VOC or SVOC sample collection due to the potential for sample contamination.

Dedicated pumps (those that are permanently installed in the well, e.g., bladder pumps) are preferred over portable pumps because they eliminate disturbance to the water column in the well during pump insertion, thus providing lower turbidity values, shorter purge times, and lower purge volumes to achieve stabilized indicator parameter measurements. However, portable pumps can be used if care is taken to minimize disturbance to the water column during pump insertion, and if adequate time is allowed following pump insertion and prior to pump operation for any particulates agitated in the water column to settle. Both dedicated and portable pumps should be easily adjustable and should operate reliably at lower flow rates. All pumps typically have some limitations that should be evaluated with respect to site-specific considerations and data quality objectives on a case-by-case basis.

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Water quality indicator field parameters should be continuously monitored during low-flow purging using a flow-through cell, or in-line parameter monitoring techniques. Continuous indicator parameter monitoring is a critical component to low-flow groundwater sampling. Water quality indicator parameters include temperature, pH, oxidation-reduction potential (ORP), specific conductivity, dissolved oxygen (DO), and turbidity. The flow-through cell enables continuous collection of real-time parameters during low-flow purging. Stabilization is achieved after all parameters fall within established limits for three successive readings as discussed in Section 5. Stabilization of low-flow parameters is further discussed in Section 5.0 (Procedure) of this SOP.

Advantages of low-flow groundwater sampling are:
- Improved sample quality (e.g., less turbid and more representative of the aquifer)
- Potentially reduced purging and sampling times
- Reduced purge water volume

2.1 Associated Procedures
- SOP 1-6, *Water Level Measurement*
- SOP 2-2, *Guide to Handing of Investigation Derived Waste*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 General Responsibilities
Project Manager - The project manager is responsible for ensuring that field personnel are trained in the use of this procedure and for verifying that well development and purging are carried out in accordance with this procedure.

Field Team Leader - The field team leader is responsible for complying with this procedure as well as reading/reviewing field plans and coordinating assigned tasks before conducting the field work.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment
- Site plans and well construction information
- Field logbook and health & safety plan
- Waterproof ink pens
- Pump (including peristaltic pumps, bladder pumps, electrical submersible pumps, and gas-driven pumps as discussed in Section 2.0 of this SOP).
- Appropriate controller for selected pump type.
- For bladder pumps: Compressor and controller for the system (compressed non-reactive gas may also be used in lieu of a compressor).
- Power source (e.g., battery or generator), as required.
- Pump tubing (typically polyethylene with Teflon® lining). Note that portable bladder pumps require combination tubing (for air and water); therefore, the correct tubing sizes for the portable bladder pump should be verified. Additionally, peristaltic pumps require flexible tubing (silicone or Tygon) tubing for the pump head. Polyethylene tubing with Teflon® should not be used when sampling for per- and polyfluoroalkyl substances (PFAS). It is recommended that high-density polyethylene tubing be used instead.
- Electronic water level meter or oil-water interface probe (according to SOP 1-6)
- Water quality meter (e.g., YSI 600 or YSI Professional Series) with a closed flow-through cell for continuous in-line measurement of temperature, pH, conductivity, ORP, and DO prior to sample collection.
- Turbidity meter (reporting in nephelometric turbidity units [NTUs])
- Standards for calibration and field check, as needed, of water quality and turbidity meters (as determined by anticipated field conditions).
- Volume measuring device to determine flow (e.g., graduated cylinder).

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- Stop watch
- Tape measure
- Engineering ruler
- Personal protective equipment as specified in the site-specific health and safety plan.
- Polyethylene sheeting
- Sample containers, including packaging supplies and all associated paperwork (e.g., chain of custody forms) as required in the sampling plan and per SOP 2-1, packaging and shipping environmental samples.
- Decontamination supplies, as required, according to SOP 4-5.
- Disposal drums (e.g., 55-gallon Department of Transportation-approved) or other purge water storage container, if required by the site-specific sampling plan.
- Photoionization detector (PID)/organic vapor monitor (OVM) or equivalent as specified in site-specific health and safety plan.

Note: All sampling devices (bladders, pumps, and tubing) should be constructed of stainless steel, polyethylene, Teflon®, glass, or similar non-reactive materials. Procedural modifications for low-flow groundwater sampling at PFAS sites are included in Section 6 and attached.

5.0 Procedure

The following steps should be followed for low-flow groundwater sampling activities:

1. Review site-specific health and safety plan, and site-specific project and sampling plans before initiating sampling activities.

2. Review available existing data for site to evaluate approach to sampling site wells. Prepare to sample site wells in the order of least contaminated to most contaminated. Additionally, existing site data should be reviewed to determine anticipated hydrogeologic conditions and well completion details.

3. Prior to sampling, all sampling devices and monitoring equipment shall be calibrated according to manufacturer's recommendations and the site-specific sampling plan. Calibration of pH should be performed with at least two buffers that bracket the expected pH range. DO calibrations should be corrected for local barometric pressure readings and altitude.

4. Put on personal protective clothing and equipment as specified in the site-specific health and safety plan.

5. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any. If no reference mark exists, create a reference mark on the well riser using a permanent marker or equivalent. Record the location of the reference mark in the field notes.

6. Monitor the air space at the wellhead for VOCs using a PID/OVM or equivalent immediately upon removal of the well plug and as according to health and safety requirements.

7. Determine the depth to static water level in accordance with SOP 1-6, taking precautions to minimize disturbance of the stagnant water column above the screened interval during water level measurement. Well depth should be obtained from review of the well completion logs, from previous work or after sampling is complete. Insertion of a water level measuring device to the bottom of the well casing will result in resuspension of settled solids from the formation surrounding the screened interval, thus requiring longer purging times for turbidity and other field parameter equilibration.

8. Dedicated sampling devices (those permanently installed in the well) capable of pumping and sampling are preferred over any other type of device. Any portable sampling device should be slowly and carefully lowered to the middle of the screened interval or slightly above the middle to minimize excessive mixing of the stagnant water in the casing above the screen with the screened interval zone water, and to minimize resuspension of solids collected at the bottom of the well or in the surrounding formation within the screened interval.

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9. New polyethylene tubing shall be used for each sample when using non-dedicated sampling equipment. Prepare the pump and tubing for insertion into the well. Lower the pump intake down into the well casing. Connect the flow-through cell in-line with the pump effluent tubing.

10. Generally, the pump intake should be placed in the mid-point of the screened interval. This provides consistency between sampling rounds. However, if the geology of the screened interval consists of heterogeneous materials with layers of contrasting hydraulic conductivity, the pump intake should be positioned adjacent to the zone of highest hydraulic conductivity (as determined via review of the existing site hydrogeologic conditions/well completion logs). Also, the sampling plan should be consulted to determine if particular zones (e.g., known zones of contamination) are targeted for sampling per DQOs). When conducting low flow sampling to evaluate vapor intrusion, the pump intake should be collected at or near the water table (i.e., 0-2 feet below the water table) to ensure groundwater samples are representative of the shallowest portion of the aquifer.

11. To achieve low-flow purging conditions, the purge rate should generally not exceed 0.5 L/min. Adjust the pump control to stabilize the flow rate, and therefore minimize drawdown (less than 0.3 foot during purging activities). The water level in the well should be measured throughout the purging process to monitor drawdown. Flow rate can be measured from the discharge tube using a volumetric measuring device (e.g., a graduated cylinder) and a stopwatch. (Note: determine flow rate by measuring volume in 0.5-minute or 1-minute increments.)

12. Record water level measurements, and field parameters including pH, temperature, specific conductivity, oxidation reduction potential (ORP), DO, turbidity, and flow rate every three to five minutes during the purging process. Record all measurements and observations in the logbook or on a groundwater purging and sampling form (**Attachment 1**). Purging shall continue until the field parameters have stabilized. Parameters are considered stable when three consecutive readings are within the limits of the criteria defined in Table 5.1 and/or in accordance with the site-specific sampling plan. Turbidity ideally should stabilize below 10 NTU prior to sample collection, particularly if groundwater samples are to be collected for metals or PCB analyses.

TABLE 5.1 Stabilization of Water Quality Indicator Parameters

Parameter	Units	Stabilization Criteria
Water Level	Feet/meters	< 0.3 foot (< 0.1 meter)
Temperature	°F/°C	± 3 percent, or ±1.8 degrees Fahrenheit (°F) /±1 degree Celsius (°C)
pH	(n/a)	± 10 percent, or ±0.1 standards units (SU)
Specific Conductivity	µm/cm	±3 percent (microsiemens per centimeter, or µm/cm)
ORP	mV	±10 millivolts (mV)
Dissolved Oxygen	mg/L	±10 percent for values greater than 0.5 mg/L, or 0.2 milligram per liter (mg/L) - whichever is greater. If three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized.
Turbidity	NTU	± 1 Nephelometric Turbidity Unit (NTU) (±10 percent for turbidity if greater than 5 NTU) If three Turbidity values are less than 5 NTU, consider the values as stabilized
Flow Rate	L/min	0.1 to 0.5 Liters per minute (L/min) (< 1 L/min), specific flow rates and sampling rates to be identified in the sampling plan if project/contract required.

13. In low recharge aquifers, the following steps shall be followed:
    (1) If the initial water level is less than 10 feet above the top of the well screen, then purge the well until dry and allow sufficient recharge to collect samples.
    (2) If the initial water level in the well is greater than 10 feet above the top of the screen, then care shall be taken to prevent the dewatering of the screened interval during purging of the well.
        (2a) Continue purging until the water level is between 1 foot (0.3 meter) and 5 feet (1.5 meters) above the top of the screened interval.
        (2b) Allow the well to recharge, then continue purging until at least one full initial well volume has been purged.
    (3) Record all data, measurements, and observations in the logbook.

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14. After field parameters have stabilized, disconnect the flow through cell, and collect groundwater samples directly from the discharge tubing into an appropriate sample container. If using a peristaltic pump to collect VOC samples, refer to item 16 of this SOP for the correct procedure for sampling VOCs with a peristaltic pump. If an in-line, flow-through cell is used to continuously monitor indicator parameters, it should be disconnected or bypassed during sample collection. During sample collection, maintain the pump rate at the same rate used during purging (unless specified in the sampling plan). The pump rate used during sample collection may need to be lowered to minimize aeration, bubble formation, or turbulent flow of water into sample bottles, or to prevent sample preservatives from being washed out of the sample container. Note: Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOC and dissolved gases in the groundwater samples.

15. Groundwater sampling (including the collection of all required quality assurance/quality control samples specified in the sampling plan) shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment used for purging. Sampling should occur in a progression from the least to the most contaminated well, if this is known. Generally, volatile (e.g., solvents and fuel constituents) and gas sensitive (e.g., Fe^{2+}, CH_4, H_2S/HS^-, and alkalinity) analytes should be sampled first. The sequence in which samples for most inorganic parameters are collected is immaterial unless filtered (dissolved) samples are required. Filtered samples should be collected last and in-line filters should be used. After all unfiltered samples have been collected, a 0.45 micron (µm) in-line filter shall be inserted in the discharge line for collection of filtered samples, as required.

16. VOC samples should not be collected directly from the discharge end of a peristaltic pump. After field parameters have stabilized, and all other samples have been collected as required, stop the pump and simultaneously pinch the discharge end of the tubing shut. Disconnect the flow-through cell. Remove the tubing from the well and fill the VOC sample containers from the influent end of the sample tubing, (the end of the tubing that was located down-well during purging activities). The flow rate when filling sample vials may be controlled by setting the peristaltic pump in reverse.

17. Place all samples in a cooler with ice or ice packs to comply with project, laboratory, and/or regulatory requirements. Do not use "blue ice" packs with samples to be analyzed for PFAS.

18. After sampling activities have been completed, remove the portable pump assembly from the well, if used, and decontaminate all non-disposable components. Re-install and secure the well gripper plug and cover. Clean up the work area; containerize and/or dispose of purge water as required by the site-specific sampling plan, and dispose of tubing and all other disposable sampling equipment as investigation derived waste (IDW) after each use as described in the site-specific sampling plan.

5.1 Conducting Low Flow Groundwater Sampling at Per- and Polyfluoroalkyl Substances (PFAS) Sites

The evolving regulatory standards for PFAS should be considered in the implementation of low-flow groundwater sampling as well as the development of project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans, and laboratory statements of work used to guide the collection of samples for PFAS analysis.

A consideration of the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, reporting limits are all impacted with the inclusion of sampling for PFAS analysist.

Before implementing low-flow groundwater sampling for collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to low-flow groundwater sampling should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith regulatory updates can be located at the INFOCENTER. Specific state and federal regulatory limit updates can be found here:

https://cdmsmith.libguides.com/PFAS_InfoCenter

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Procedural modifications for low-flow groundwater sampling and other related field activities at PFAS sites are provide below. A check list containing common materials and sampling equipment that may contain PFAS compounds is attached and also can be found here:

https://www.yammer.com/cdmsmith.com/#/files/214861635584

Procedural Considerations For PFAS Sampling

Development of PFAS awareness in guidance and incorporation into specific TSOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. (For example, Federal drinking water MCL for Arsenic is 10,000 ppt where the EPA health advisory level is a combined 70 ppt for PFOA and PFOS.) Procedural modifications to field activities when sampling for PFAS analysis involve the concerns listed below. Good housekeeping, detailed documentation of these extra steps and vigilance about clearly defined exclusion zones should be strictly adhered to.

Good practices

- Utilize good safety planning and institute exclusion zones, contamination reduction zones, and support zones (no visitors within 30 feet of sampling).
- Do not eat in or near sampling areas (food packaging may contain PFAS).
- Be vigilant about PPE donning and doffing practices.
- Wash hands before and after eating.
- Wear powderless nitrile gloves and change them frequently.
- Only open sample container during sample collection and never set the sample container lid down.
- Keep hands away from the container opening when sampling and keep lid protected.
- Develop a project-specific checklist for review with field personnel.
    - It should be project-specific
    - Completed prior to beginning PFAS sampling event, whenever there are staffing changes, and whenever sample media change and associated sampling/field equipment changes
    - Consider appropriate sampling/field work sequences for each matrix
    - Sampling/field work sequences
    - PFAS sampling/field work should occur first on sites where other contaminants will also be sampled.
    - CDM Smith PFAS Sampling Guidance Team can assist with development of your project-specific checklist.

Avoid

- **PTFE, LDPE**, sticky notes, waterproof field book, aluminum foil
- Field filtering of water samples because of glass fiber filters
    - Avoid use of markers, if possible, during sampling use only regular ink pens. If needed, write over regular ink with marker after the sample containers are sealed.
- Consult materials checklists for equipment concerns.

6.0 Restrictions/Limitations

Only grounded electrical devices should be used for low-flow sampling activities. If a gasoline-powered electrical source is used, place portable power sources (e.g., generators) 50 feet (15 meters) or farther from the wellhead to prevent potential contamination of samples. Additionally, it should be clearly noted in the field notes or on the Groundwater Sampling Log (Attachment 1) if a well has been pumped dry and allowed to recharge prior to sample collection, as low-flow sampling data is no longer applicable.

7.0 References

ASTM D6452-99(2012)e1, Standard Guide for Purging Methods for Wells Used for Groundwater Quality Investigations, ASTM International, West Conshohocken, PA, 2012.

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U. S. Environmental Protection Agency. May 2002. *Groundwater Sampling Guidelines for Superfund and RCRA Project Managers.* Ground Water Forum Issue Paper, EPA 542-S-02-001, OSWER, Technology Innovative Office, Washington, D.C.

_____. Region 4. April 2017. The Field Branches Quality System and Technical Procedures, Operating Procedure Groundwater Sampling. SESDPROC-301-R4.

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Attachment 1

EXAMPLE LOW-FLOW GROUNDWATER SAMPLING PURGING DATA SHEET

Site Name: _____ **Date:** _____

OVM: **FID** ☐ **PID** ☐ **In Casing (ppm): (Initial)** _____ **(Vented to)** _____

Well ID: _____

Purging/Sampling Device: _____

Initial Static Water Level (feet btoc): _____

Analytical Parameters: _____

Final Water Level (feet btoc): _____

QC Samples Collected: _____

Purge Start Time: _____

Sample Number: _____

Sample Time: _____

Samplers' Signatures: _____

Time	Water Level (ft btoc)	Temperature (ºC)	pH (SU)	Conductivity (µs/cm)	Dissolved Oxygen (mg/L)	ORP (mV)	Turbidity (NTU)	Flow Rate (mL/min)	Comments (e.g., depth of pump intake, screened interval)

Parameter	Units	Stabilization Criterion
Water Level	Feet/meters	< 0.3 foot (< 0.1 meter)
Temperature	°F/°C	± 3 percent, or ±1.8 degrees Fahrenheit (°F) /±1 degree Celsius (°C)
pH	(n/a)	± 10 percent, or ±0.1 standards units (SU)
Specific Conductivity	µm/cm	±3 percent (microsiemens per centimeter, or µm/cm)
ORP	mV	±10 millivolts (mV)
Dissolved Oxygen	mg/L	±10 percent, or 0.2 milligram per liter (mg/L) - whichever is greater If three DO values values are less than 0.5 mg/L, consider the values as stabilized.
Turbidity	NTU	± 1 Nephelometric Turbidity Unit (NTU) (±10 percent for turbidity if greater than 5 NTU), If three turbidity values are less than 5 NTU, consider the values as stabilized.
Flow Rate	L/min	0.1 to 0.5 Liters per minute (L/min) (< 1 L/min)

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Attachment

PFAS Sampling Materials Checklist

Development of PFAS sampling guidance TSOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the parts per trillion (ppt) range. The levels of awareness for PFAS cross contamination vary by the opportunities of introducing PFAS into the environment media under investigation.

1. Critical: Items in direct contact of environmental media under investigation. These can include, but not limited to, sample containers, sampling parts and equipment, drilling equipment, well construction items and materials, parts and equipment for hydrogeological testing, in-situ treatment parts and equipment.
2. Very Important: PPE, personal hygiene that are used by sampling personnel.
3. Important: Items used in coolers for shipping and transporting PFAS samples.
4. Less important/awareness level concern: Activities in the staging area away from immediate PFAS investigation area.

Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Field Clothing or PPE				
Clothing or boots containing "water resistance" or "stain-treated" fabrics			X	
Cloths washed with fabric softeners			X	Fabric softeners may contain PFAS.
New and unwashed clothing			X	fabric treatment may contain PFAS
Uncoated Tyvek		X		USEPA PFAS sampling guidance from Region 2 prohibits use of Tyvek.
Coated Tyvek			X	
PVC or wax-coated fabrics	X			
Neoprene	X			
Synthetic and natural fibers (preferably cotton)	X			
Steel-toed boots made with polyurethane and PVC	X			If it is not possible to find PFAS free steel-toed footwear, PFAS-free over boots may be worn. The over boots must be put on and the hands washed after putting the over boots on prior to the beginning of the sampling activities. Over boots may only be removed in the staging area and after the sampling activities have been completed.
Well-laundered clothes	X			several times from time of purchase.
Well-washed cotton coveralls	X			washed several times.

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Personnel Hygiene and Protective Skin Products				
Sunscreens		X		Good to use: Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, and baby sunscreens that are “free” or “natural.
Insect Repellents		X		Good to use: Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics.
International brands of sunscreens and insect repellents		X		Must be evaluated on a case-by-case basis.
Field Sampling Items				
Waterproof field paper or books			X	Use loose plain paper.
Post-it notes			X	
Aluminum foil			X	
Brand-name markers			X	Sharpie may be used to label sample bottles in the staging area, but markers should not be used in the immediate sampling environment.
Off-brand markers		X		
Ball point pens	X			
plastic clipboards			X	NJ DEP sampling guidance, use metal clipboard.
Plastic table cover			X	
Sampling Equipment				
Item containing high-density polyethylene (HDPE)	X			
Item containing polypropylene	X			PP sample bottles must be used for drinking-water samples in accordance with USEPA method 537.1.1.
Item containing polyurethane	X			
Item containing Polyvinyl chloride (PVC)	X			
Item containing silicon	X			
Item made of stainless steel	X			
Alconox®	X			
Citronex®	X			
Liquinox®	X			
Powderless nitrile gloves	X			

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
HDPE Hydrasleeves or sonic core bags	X			
Neoprene	X			
Crisco® or other vegetable-based greases for lubricating parts	X			
Item containing PTFE			X	Items or equipment that contains PTFE parts that will be in direct contact with sampling media.
Item containing Teflon®			X	Field sampling items or equipment that contains Teflon® and that will be in direct contact with the sampling media.
Item containing fluoropolymer			X	
Low-density polyethylene (LDPE)			X	Items or equipment that contains LDPE parts and that will be in direct contact with the sampling media.
Viton® O-rings			X	Viton® O-rings used in pressure washers used for sampling equipment decontamination.
Glass sample containers			X	
Field filter			X	Field filtration should be avoided regardless of filter types.
Decon 90			X	
Items containing fluorosurfactants			X	
Teflon-bearing plumber's tape			X	
Blue (or chemical) ice			X	Later data (unpublished) suggest no cross contamination from blue ice. The category may be changed after data are published.
Water ice	X			Double bag in polyethylene bags.
Internal valves and equipment parts for sampling or decon		X		
Methanol or other solvents		X		
LDPE plastic bags (e.g., Ziploc® bags)		X		For larger biota sampling, Ziploc bags may be used, but collecting an equipment blank is recommended because these bags may be made of LDPE.
Drilling fluids		X		
LDPE sonic core sample bags		X		Manufactured by Boart Longyear and Hole Products.

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Equipment with moving parts that may be lubricated with PFAS containing lubricants or greases		X		
Rental equipment		X		Must be verified to have no PFAS-bearing parts prior to use.
Others				
Food wrappers			X	Field personnel must wash hands after having food wrapped with grease repelling paper.

(1) If an item that may contain PFAS but alternative is not available, the item should be tested for PFAS before use.
(2) This mostly refers to the immediate sampling environment, particularly, the item is in contact with environmental media to be sampled.
(3) There are no standard operation procedures on how an item can be verified. Please contact PFAS experts for advice on the best practice for testing an item for potential PFAS contamination.

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Packaging and Shipping Environmental Samples	SOP 2-1 Revision: 7 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Mary Lou Fox

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to outline requirements for packaging and shipping environmental samples. Additionally, Sections 2.0 through 7.0 outline requirements for packaging and shipping regulated environmental samples under the U.S. Department of Transportation (USDOT) Hazardous Materials Regulations, the International Air Transportation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations for shipment by air and applies only to domestic shipments. This SOP does not cover requirements for packaging and shipping equipment (including data loggers and self-contained breathing apparatus [SCBAs] or bulk chemicals that are regulated under the DOT, IATA, and ICAO.

1.1 Packaging and Shipping of All Samples

This SOP applies to packaging and shipping all environmental samples. If the sample is preserved or radioactive, the following sections may also be applicable and therefore, should be reviewed for additional requirements.

Section 2.0 - Packaging and Shipping Samples Preserved with Methanol
Section 3.0 - Packaging and Shipping Samples Preserved with Sodium Hydroxide
Section 4.0 - Packaging and Shipping Samples Preserved with Hydrochloric Acid
Section 5.0 - Packaging and Shipping Samples Preserved with Nitric Acid
Section 6.0 - Packaging and Shipping Samples Preserved with Sulfuric Acid
Section 7.0 - Packaging and Shipping Limited-Quantity Radioactive Samples

Note: This SOP does not address shipment of hazardous materials. Shipping of hazardous materials are strictly prohibited unless you have received training that meets CDM Smith, DOT, and IATA requirements. Check with CDM Smith University for training courses.

1.2 Background

1.2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Environmental Sample - An aliquot of air, water, plant material, sediment, or soil that represents the contaminant levels on a site. Samples of potential contaminant sources, like tanks, lagoons, or non-aqueous phase liquids are normally not considered "environmental" for this purpose. This procedure applies only to environmental samples that contain less than reportable quantities for any foreseeable hazardous constituents, according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Custody Seal - A custody seal is a narrow adhesive-backed seal that is applied to individual sample containers and/or the container (i.e., cooler) before shipping offsite. Custody seals indicate that sample integrity has not been compromised during transport from the field to the analytical laboratory.

Inside Container - The container (normally made of glass or plastic) that actually contacts the shipped material. Its purpose is to keep the sample from mixing with the ambient environment.

Outside Container - The container (normally made of metal or plastic) that the transporter contacts. Its purpose is to protect the inside container.

Secondary Containment - The outside container provides secondary containment if the inside container breaks (i.e., plastic overpackaging if liquid sample is collected in glass).

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Allowed Quantity - Allowed quantities, below which USDOT, IATA, ICAO regulations do not apply, limit the mass or volume of a hazardous material in the inside and outside containers. Allowed quantity limits are very low and most regulated shipments are made under excepted or limited quantities (see descriptions below).

Excepted Quantity – Excepted quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

Limited Quantity - Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions. The limited quantity will be a higher amount than the excepted quantity for a given substance.

Performance Testing - Performance testing refers to required testing of outer packaging, such as drop and stacking tests.

Qualified Shipper - A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials. Required trainings include Hazardous Materials Shipping (HAZ 003) for ground shipping of hazardous materials and Hazardous Transportation/Shipping Air (IATA THZ 203) and hold resulting training certificates not more than three years old.

1.2.2 Associated Procedures

- SOP 1-2, *Sample Custody*

1.2.3 Discussion

Proper packaging and shipping is necessary to ensure the protection of the integrity of environmental samples shipped for analysis. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places both CDM Smith and the individual employee at risk of serious fines. The analytical holding times for the samples must not be exceeded. The samples shall be packed in time to be shipped for overnight delivery. Prior arrangements with the laboratory should be made before sending samples for weekend delivery.

1.3 Required Equipment

- Coolers with return address of the appropriate CDM Smith office
- Heavy-duty plastic garbage bags (contractor bags, 3.0 millimeter thickness)
- Plastic zip-type bags, small and large
- Clear tape
- Nylon reinforced strapping tape
- Duct tape
- Kitty litter/pine bedding (or an equivalent nonflammable material that is inert and absorbent)*
- Bubble wrap (optional)
- Ice
- Custody seals
- Completed chain-of-custody record or contract laboratory program (CLP) custody records, if applicable
- Completed bill of lading
- “This End Up” and other directional arrow labels

*Check for any client-specific or laboratory requirements related to the use of absorbent packaging materials.

1.4 Packaging Environmental Samples

The following steps are required when packing sample bottles and jars for shipment:

1. Verify the samples undergoing shipment meet the definition of “environmental sample” and are not a hazardous material as defined by USDOT. Professional judgment and/or consultation with qualified persons such as the appropriate health and safety coordinator or the health and safety manager shall be observed.

2. Select a sturdy cooler in good condition. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Ensure the handles used for carrying the cooler are in good condition. Also, tape the drain plug from the inside and outside of the cooler. Line the cooler with a large heavy-duty plastic garbage bag.

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3. Be sure the caps on all bottles are tightly sealed and will not leak; check to see that labels and chain-of-custody records are completed properly (SOP 1-2, Sample Custody).

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Up to three volatile organic analyte (VOA) vials may be packed in one bag. Binding the vials together with a rubber band on the outside of the bag or separating them so that they do not contact each other will reduce the risk of breakage. VOA vials may be packaged in foam containers designed for packaging them as well. Bottles may be wrapped in bubble wrap. Optionally, place three to six VOA vials in a quart metal can and then fill the can with kitty litter/pine bedding or equivalent. Note: Include trip blanks in coolers containing VOA samples.
5. Place 2 to 4 inches of an absorbent material into a cooler that has been lined with a garbage bag, and then place the bottles and cans in the bag with sufficient space to allow for the addition of packing material between the bottles and cans. It is preferable to place sample bottles and jars (especially glass) into the cooler vertically. Glass containers are less likely to break when packed vertically rather than horizontally, and sample bottles are less likely to leak when upright in the cooler.

6. While placing sample containers into the cooler, conduct an inventory of the contents of the shipping cooler against the chain-of-custody record. The chain-of-custody with the cooler shall reflect only those samples within the cooler.

7. Put ice in large plastic zip-top bags (double bagging the zip-tops is preferred) and properly seal. Place the ice bags on top of and/or between the samples. Several bags of ice are required (dependent on outdoor temperature, staging time, etc.) to maintain the cooler temperature at approximately 4° $\pm$ 2° Celsius (C) if the analytical method requires cooling. Large volumes of soil samples may require an interim cooling step to ensure that the shipping ice will be sufficient for the samples to reach the laboratory in satisfactory condition. Fill all remaining space between the bottles or cans with packing material. Securely fasten the top of the large garbage bag with fiber or duct tape.

8. When approved by the client/ laboratory, an alternate method of packing ice to keep sample cold during shipment is to line the cooler with a contractor bag and then put a layer of ice in the bottom of the bag. Place an inner contractor bag inside the outer contractor bag and the ice layer and place samples inside the inner bag. If a temperature blank is being used, place the temperature blank inside the inner bag with the samples. Then pour ice between the inner and outer contractor bags around the inner bag containing the samples. When all samples and a temperature blank, if applicable, are in the inner bag, twist the inner bag closed, tape the twisted closure, and place a custody seal around the taped closure. Cover the custody seal with clear tape. The custody seal around the inner bag closure maintains the custody seal for the samples, should the cooler open for any reason during shipment. Twist the outer bag closed and seal it with tape, as well.

9. Place the completed chain-of-custody record or the CLP traffic report form (if applicable) for the laboratory into a plastic zip-top bag, seal the bag, tape the bag to the inner side of the cooler lid, and close the cooler.

10. Secure the cooler lid with nylon reinforced strapping tape by wrapping each end of the cooler a minimum of two times. Attach completed chain-of-custody seals across the opening of the cooler on opposite sides. The custody seals should be affixed to the cooler with half of the seal on the strapping tape, so that the cooler cannot be opened without breaking the seal. Complete two more wraps around with fiber tape and place clear tape over the custody seals.

11. Mark the shipping container lid with a **"THIS END UP"** and appropriate upward position arrow labels. These upward position arrows should also be affixed to the cooler. Place a label containing the name and address of the shipper (CDM Smith) to the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Aircraft, Flammable Solids, etc.) are not permitted on the outside of containers used to transport environmental samples and should not be used. The name and address of the laboratory shall be placed on the container. When shipping by common courier, the bill of lading shall be completed and attached to the lid of the shipping container.

12. Per- and polyfluoroalkyl substances (PFAS) samples (collected in high density polyethylene containers), once they are securely closed and labeled, are to be shipped as "other environmental samples" with water ice to chill the samples below 6° Celsius. A PFAS sample container should not be opened after being sealed following collection. If, for any reason, the container is opened at this

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point, great care should be taken to avoid cross-contamination as materials allowed for the shipment of the sealed containers may be sources of PFAS.

2.0 Packaging and Shipping Samples Preserved with Methanol

2.1 Containers

1. The maximum volume of methanol in a sample container is 30 milliliters (ml) per sample vial and 500 mls total for the cooler for shipment as an excepted quantity.

2. The sample container should not be full of methanol.

2.2 Responsibility

To legally ship a dangerous good such as methanol, applicable training is required. It is the responsibility of the qualified shipper to:

1. Ensure that the samples undergoing shipment contain no other contaminant that meets the definition of "hazardous material" as defined by the USDOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

   Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

2.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

1. Inner packing (may consist of glass or plastic jars)

2. Outer packaging (for excepted or limited quantities) should be an insulated cooler that has passed the ICAO drop test and is in good condition.

3. Survey documentation (if shipping from Department of Energy [DOE] or radiological sites)

4. "Dangerous Goods" labels completed appropriately for methanol for excepted quantities, or appropriate hazard class labels (i.e. flammable liquid") for limited quantities

5. Orientation labels

6. Consignor/consignee labels

2.4 Packaging Samples Preserved with Methanol

The following steps are required when packaging excepted or limited-quantity sample shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

   All sample containers will be properly labeled and the label protected with waterproof tape before sampling. No taping of labels should be done for VOC samples that are collected with EPA method 5035 in pre-weighed vials, as the mass of the soil sample is obtained by the difference between the received sample weight (soil + methanol + vial) and the weight of the vial and methanol that was pre-weighed. At a minimum the label should contain:

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- Project name
- Project number
- Date and time of sample collection
- Sample location
- Sample identification number
- Collector's initials
- Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

2. Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.

3. Place the bubble-wrapped container into a 3.0-mil zip-type bag, removing trapped air.

4. Place wrapped containers inside a polyethylene bottle filled with an absorbent; seal the bottle. (Maximum of four VOA vials will fit inside a 500-ml wide-mouth polyethylene bottle.)

5. Total volume of methanol per shipping container should not exceed 500 ml.

6. Line the bottom of the cooler with absorbent material to absorb any leakage that may occur.

7. Line the cooler with a contractor grade garbage bag.

8. Pack the samples, with bottles placed upright, appropriately inside the garbage bag to prevent movement during shipment.

9. Place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

10. Seal the garbage bag by tying or taping.

11. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any excepted or limited-quantity shipment of dangerous goods.

12. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

13. If the shipment is from a DOE or other facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

14. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

15. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

16. For a limited quantity, mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Methanol Mixture
    UN1230
    LTD. QTY.

17. **For an excepted quantity, there must be a "dangerous goods in excepted quantities" label on the cooler. The label must have the hazard class for methanol on it below the "E". Methanol is flammable (class 3).**

18. Place a label on the top or front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a "flammable liquid" label to the outside of the cooler for limited quantities.

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20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown below in Figure 1.

    Note: No marking or labeling can be obscured by strapping or duct tape.

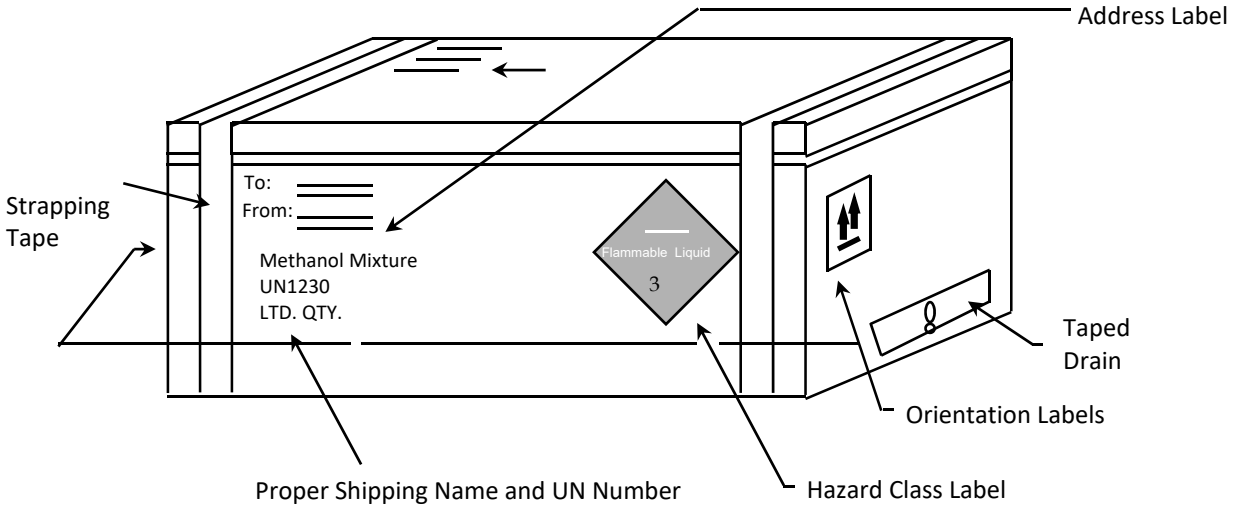
    Note: Place inner packaging of dangerous goods into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 millirem/hour (mrem/h) on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Excepted/Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods airbill.

Figure 1
Example of Cooler Label/Marking Locations for Limited Quantities



3.0 Packaging and Shipping Samples Preserved with Sodium Hydroxide

3.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sodium Hydroxide Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
NaOH	30%	>12	0.08%	'--	.25	0.5	1	2

20 drops = 1 ml

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3.2 Responsibility

It is the responsibility of the qualified shipper to determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

3.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Outer packaging (for limited quantities) should be an insulated cooler that has passed the ICAO drop test and is in good condition.
- Inner packings may consist of glass or plastic jars no larger than 1 pint.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

3.4 Packaging Samples Preserved with Sodium Hydroxide

Samples containing sodium hydroxide (NaOH) as a preservative that exceed the excepted concentration of 0.08 percent (2 ml of a 30 percent NaOH solution per liter) may be shipped as a limited quantity per packing instruction Y819 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are required when packaging limited-quantity samples shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label should contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. If the containers are breakable; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 3.0-mil zip-type bag, removing trapped air.

5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.

6. The total volume of sample in each cooler should not exceed 1 liter.

7. Place sufficient amount of an absorbent in the bottom of the cooler to absorb any leakage that may occur.

8. Line the cooler with a contractor grade garbage bag .

9. Pack the samples, with bottles placed upright, appropriately inside the garbage bag to prevent movement during shipment.

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10. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

11. Seal the garbage bag by tying or taping.

12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

14. If the shipment is from a DOE or other radiological facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Sodium Hydroxide Solution
    UN1824
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a "corrosive" label to the outside of the cooler.

20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown above in Figure 1.

    Note: **Samples meeting the allowed concentration of 0.08 percent NaOH by weight may be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.**

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: Place the inner packaging of dangerous goods into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods Air Bill.

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4.0 Packaging and Shipping Samples Preserved with Hydrochloric Acid

4.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Hydrochloric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*		
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**
HCl	2N	<2	0.04%	.2	.5	1

20 drops = 1 ml

4.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site; therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

4.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3.

- Inner packing may consist of glass or plastic jars no larger than 1 pint.
- Outer packaging (for limited quantities) should be an insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

4.4 Packaging Samples Preserved with Hydrochloric Acid

The following steps are to be followed when packaging limited-quantity sample shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label should contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. Wrap each container (40-ml VOA vials) in bubble wrap (secure with waterproof tape) to prevent breakage.

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4. Place the bubble-wrapped container into a 3.0-mil zip-type bag, removing trapped air.
5. Place wrapped containers inside a polyethylene bottle filled with an absorbent; seal the bottle. (No more than four VOA vials will fit inside a 500-ml, wide-mouth polyethylene bottle.)
6. Total volume of sample inside each cooler should not exceed 1 liter.
7. Line the bottom of the cooler with sufficient absorbent material to absorb any leakage that may occur.
8. Line the cooler with a contractor-grade garbage bag.
9. Pack the samples, with bottle placed upright, appropriately inside the garbage bag to prevent movement during shipment.
10. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.
11. Seal the garbage bag by tying or taping.
12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.
13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.
14. If the shipment is from a DOE or other radiological facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.
15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.
16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.
17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below) .

    Hydrochloric Acid Solution
    UN1789
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.
19. Affix a “corrosive” label to the outside of the cooler.
20. Affix package orientation labels on two opposite sides of the cooler.
21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: **Samples containing less than the allowed concentration of 0.04 percent HCl by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.**

    Note: No marking or labeling can be obscured by strapping or duct tape.

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Note: Place the inner packaging of dangerous goods inside the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler is to be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey should be documented, and the results reviewed by the qualified shipper.
23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Excepted/Limited-Quantity (Appendix A).
24. Complete a Dangerous Goods airbill.

5.0 Packaging and Shipping Samples Preserved with Nitric Acid

5.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Nitric Acid Preservatives

Preservative		***Desired in Final Sample***		***Quantity of Preservative (ml) for Specified Container***				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
HNO_3	6N	<2	0.15%		2	4	5	8

5 drops = 1 mg/L

5.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Determine the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by DOT.
2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site; therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

5.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) should be an insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or radiological sites)
- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

5.4 Packaging Samples Preserved with Nitric Acid

Samples containing nitric acid (HNO_3)as a preservative that exceed the excepted concentration of 0.15 percent HNO_3 will be shipped as a limited quantity per packing instruction Y807 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are to be followed when packaging limited-quantity sample shipments:

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1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label should contain:
   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form

3. If the containers are breakable; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 3.0-mil zip-type bag, removing trapped air.

5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.

6. Line the bottom of the cooler with sufficient absorbent material to absorb any leakage that may occur.

7. Line the cooler with a contractor grade garbage.

8. Pack the samples, with bottles placed upright, appropriately inside the garbage bag to prevent movement during shipment.

9. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

10. Seal the garbage bag by tying or taping.

11. The maximum volume of preserved solution in the cooler should not exceed 500 ml.

12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

14. If the shipment is from a DOE or other radiological facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below) for limited quantities.

    Nitric Acid Solution (with less than 20 percent)
    UN2031
    Ltd. Qty.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a "corrosive" label to the outside of the cooler.

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20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown above in Figure 1.

    Note: **Samples meeting the allowed concentration of 0.15 percent HNO3 by weight will be shipped as nonregulated or nonhazardous following the procedure in Section 1.4.**

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: **Place** the inner packaging of dangerous goods inside the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods airbill.

6.0 Packaging and Shipping Samples Preserved with Sulfuric Acid

6.1 Containers

The inner packaging container (and amount of preservative) that may be used for these shipments includes:

Excepted Quantities of Sulfuric Acid Preservatives

Preservative		*Desired in Final Sample*		*Quantity of Preservative (ml) for Specified Container*				
		pH	**Conc.**	**40 ml**	**125 ml**	**250 ml**	**500 ml**	**1 L**
H_2SO_4	37N	<2	0.35%	.1	.25	0.5	1	2

5 drops = 1 ml

6.2 Responsibility

It is the responsibility of the qualified shipper to:

1. Ensure the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by the DOT.

2. Determine the amount of preservative in each sample so that accurate determination of quantities can be made.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

6.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Inner packings may consist of glass or plastic jars no larger than 100 ml.
- Outer packaging (for limited quantities) should be an insulated cooler that has passed the ICAO drop test.
- Survey documentation (if shipping from DOE or other radiological sites)

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- Class 8 corrosive labels
- Orientation labels
- Consignor/consignee labels

6.4 Packaging of Samples Preserved with Sulfuric Acid

Samples containing sulfuric acid (H_2SO_4) as a preservative that exceed the excepted concentration of 0.35 percent will be shipped as a limited quantity per packing instruction Y809 of the IATA/ICAO Dangerous Goods Regulations.

The following steps are required when packaging limited-quantity samples shipments:

1. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

2. All sample containers will be properly labeled and the label protected with waterproof tape before sampling.

   At a minimum the label should contain:

   - Project name
   - Project number
   - Date and time of sample collection
   - Sample location
   - Sample identification number
   - Collector's initials
   - Preservative (note amount of preservative used in miscellaneous section of the chain-of-custody form)

3. Wrap each glass container in bubble wrap (secure with waterproof tape) to prevent breakage.

4. Place the bubble-wrapped container into a 3.0-mil zip-type bag, removing trapped air.

5. Place glass containers inside a polyethylene bottle filled with an absorbent; seal the bottle.

6. Line the bottom of the cooler with sufficient absorbent material, or other approved material, to absorb any leakage that may occur.

7. Line the cooler with a contractor grade garbage bag cooler.

8. Pack the samples appropriately inside the garbage bag (bottles placed upright) to prevent movement during shipment.

9. Place sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

10. Seal the garbage bag by tying or taping.

11. The maximum volume of preserved solution in the cooler should not exceed 500 ml.

12. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

13. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

14. If the shipment is from a DOE or other radiological facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

15. Wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

16. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

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17. Mark the outside of the cooler with the proper shipping name of the contents, corresponding UN number, and LTD. QTY. (as shown below).

    Sulfuric Acid Solution
    UN2796
    LTD. QTY.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code for both shipper and recipient.

19. Affix a "corrosive" label to the outside of the cooler.

20. Affix package orientation labels on two opposite sides of the cooler.

21. Secure the marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of cooler labeling/marking locations is shown in Figure 1.

    Note: **Samples containing less than the allowed concentration of 0.35 percent H_2SO_4 by weight will be shipped as nonregulated or nonhazardous in accordance with the procedure described in Section 1.4.**

    Note: No marking or labeling can be obscured by strapping or duct tape.

    Note: **Place** the inner packaging of dangerous goods into the designated cooler for shipment. Other nonregulated environmental samples may be added to the cooler for shipment.

22. When shipping from a DOE facility, the cooler will be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

23. Complete the Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Limited-Quantity (Appendix A).

24. Complete a Dangerous Goods airbill.

7.0 Packaging and Shipping Limited-Quantity Radioactive Samples

7.1 Containers

The inner packaging containers that may be used for these shipments include:

1. Any size sample container

7.2 Description/Responsibilities

The qualified shipper should ensure that the samples undergoing shipment contain no other contaminant that meets the definition of hazardous material as defined by the DOT.

The qualified shipper will ship all samples that meet the Class 7 definition of radioactive materials and meet the activity requirements specified in Table 4 and 7 of 49 CFR 173.425, as Radioactive Materials in Limited Quantity. The qualified shipper should verify that all packages and contents meet the requirements of 49 CFR 173.421, *Limited Quantities of Radioactive Materials*.

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Packaging used for shipping is required to meet the general requirements for packaging and packages specified in 49 CFR 173.24 and the general design requirements provided in 173.410. These standards state that a package must be capable of withstanding the effects of any acceleration, vibration, or vibration resonance that may arise under normal transport conditions, without any deterioration in the effectiveness of the closing devices on the various receptacles or in the integrity of the package as a whole and without loosening or unintentionally releasing the nuts, bolts, or other securing devices, even after repeated use.

If the shipment is from a DOE facility, radiological screenings will be completed on all samples taken. The qualified shipper should review the results of each screening (alpha, beta, and gamma speciation). Samples will not be shipped offsite until the radiological screening has been performed.

The total activity for each package will not exceed the relevant limits listed in Table 4 and 7 of 49 CFR 173.425. The A_2 value of the material will be calculated based on all radionuclides found during previous investigations (if any) in the area from which the samples are derived. The A_2 values to be used will be the most restrictive of all potential radionuclides as listed in 49 CFR 173.435.

The radiation level at any point on the external surface of the package bearing the sample(s) will not exceed 0.005 millisievert per hour (mSv/h) (0.5 mrem/hour). These will be verified by dose and activity monitoring before shipment of the package.

The removable radioactive surface contamination on the external surface of the package will not exceed the limits specified in 49 CFR 173.443(a). CDM Smith will apply the DOE-established free release criteria for removable surface contamination of less than 20 disintegrations per minute (dpm)/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma). It shall be noted that these values are more conservative than the DOT requirements for removable surface contamination.

The qualified shipper should verify that the outside of the inner packaging is marked “Radioactive.”

The qualified shipper should verify that the excepted packages prepared for shipment under the provisions of 49 CFR 173.421 have a notice enclosed, or shown on the outside of the package, that reads, **“This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910.”**

> ***Note:*** Responsibilities may vary from site to site; therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific QAPP.

7.3 Additional Required Equipment

The following equipment is needed in addition to the required equipment listed in Section 1.3:

- Survey documentation/radiation screening results (if shipping from DOE or radiological sites)
- Orientation labels
- Excepted quantities label
- Consignor/consignee labels

7.4 Packaging of Limited-Quantity Radioactive Samples

The following steps are required when packaging limited-quantity sample shipments:

1. The cooler is to be surveyed by a qualified radiation control technician to ensure that radiation flux on exterior surfaces does not exceed 0.5 mrem/h on all sides. This survey will be documented and the results reviewed by the qualified shipper.

2. Tape any interior opening in the cooler (drain plug) from the inside to ensure control of interior contents. Also, tape the drain plug from the outside of the cooler.

3. All sample containers will be properly labeled and the label protected with waterproof tape before sampling. At a minimum the label should contain:

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- Project name
- Project number
- Date and time of sample collection
- Sample location
- Sample identification number
- Collector's initials

4. If the containers are breakable; wrap each container in bubble wrap (secure with waterproof tape) to prevent breakage.

5. Line the bottom of the cooler with sufficient absorbent material, or other approved packaging material, to absorb any leakage that may occur.

6. Line the cooler with a contractor grade garbage bag .

7. Pack the samples, with bottles placed upright, appropriately inside the garbage bag to prevent movement during shipment.

8. If required, place a sufficient amount of double-bagged ice around the samples to maintain the required temperature during shipment.

9. Seal the garbage bag by tying or taping.

10. Place a label marked "radioactive" on the outside of the sealed bag.

11. Enclose a notice that includes the name of the consignor or consignee and the following statement: ***"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."***

12. Note that both the DOT and IATA apply different limits to the quantity in the inside packing and in the outside packing.

13. The maximum weight of the package shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

14. Secure the chain-of-custody form (placed inside a zip-type bag) to the interior of the cooler lid.

15. If the shipment is from a DOE or other radiological facility, place the results of the radiation screen and cooler/sample survey with the chain-of-custody.

16. If a cooler is used, wrap strapping tape or duct tape around both ends of the cooler and around the cooler lid.

17. Affix custody seals to opposite sides of the cooler lid. Cover the custody seals with clear waterproof tape.

18. Place a label on the front of the cooler with the company name, contact name, phone number, full street address, and state with zip code, for both shipper and recipient.

19. Affix package orientation labels on two opposite sides of the cooler/package.

20. Affix a completed "excepted quantities" label to the side of the cooler/package.

21. Secure any marking and labels to the surface of the cooler with clear waterproof tape to prevent accidental removal during shipment. An example of the cooler labeling/marking is shown below in Figure 2.

    Note: No marking or labeling can be obscured by strapping or duct tape.

22. Complete the Shipment Quality Assurance Checklist (Appendix B).

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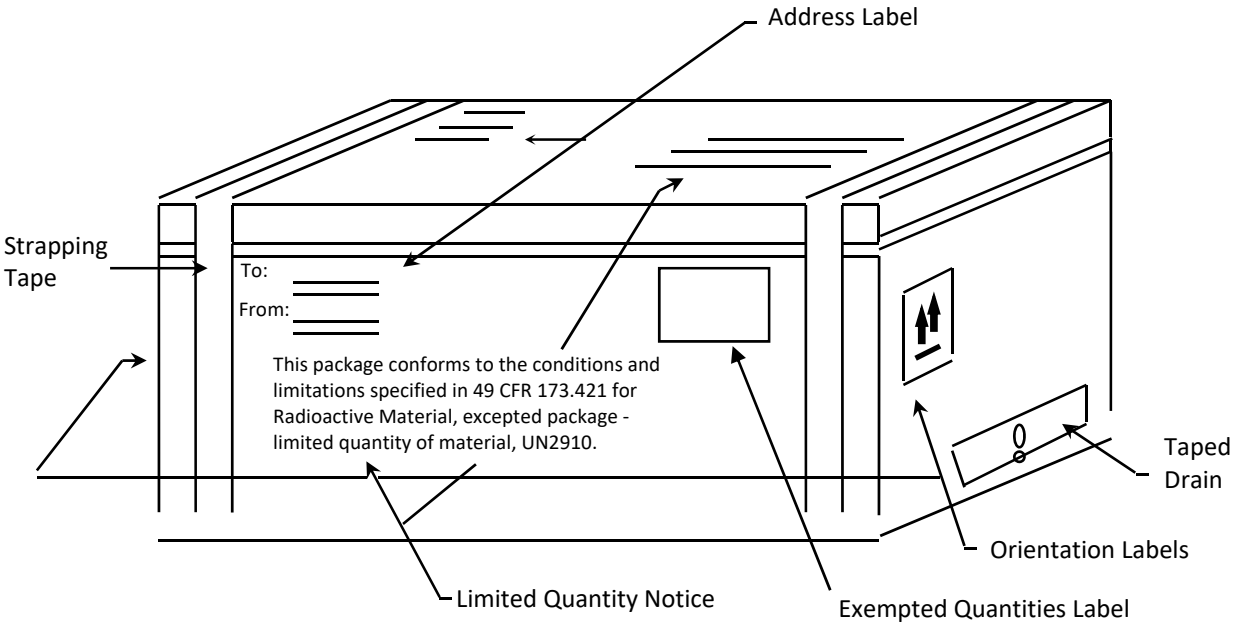
Note: Except as provided in 49 CFR 173.426, the package will not contain more than 15 grams of ^{235}U.

Note: A declaration of dangerous goods is not required.

Figure 2
Radioactive Material – Limited-Quantity Cooler Marking Example



8.0 References

U.S. Environmental Protection Agency. May 2015, or current revision. Region 4. The Field Branches Quality System and Technical Procedures, Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples SESDPROC-209-R3. February.

_____. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Sample and Evidence Management SESDPROC-005-R2. January.

_____. 2007 or current revision. *Sampler's Guide, Contract Laboratory Program, Guidance for Field Samplers*, EPA-540-R-07-06.

Title 49 Code of Federal Regulations, Department of Transportation. 2015 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

Title 49 Code of Federal Regulations, Department of Transportation. 2015 or current revision. *Shippers - General Requirements for Shipments and Packaging*, 49 CFR 173.

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Appendix A

Dangerous Goods and Hazardous Materials Inspection Checklist for Shipping Excepted or Limited-Quantity

Sample Packaging

Yes	No	N/A	
☐	☐	☐	The VOA vials are wrapped in bubble wrap and placed inside a zip-type bag.
☐	☐	☐	The VOA vials are placed inside a polyethylene bottle, filled with an absorbent, and tightly sealed.
☐	☐	☐	The drain plug is taped inside and outside to ensure control of interior contents.
☐	☐	☐	The samples have been placed inside garbage bags with sufficient bags of ice to preserve samples at 4°C.
☐	☐	☐	The cooler weighs less than the 66-pound limit for limited-quantity shipment.
☐	☐	☐	The garbage bag has been sealed with tape (or tied) to prevent movement during shipment.
☐	☐	☐	The chain-of-custody has been secured to the interior of the cooler lid.
☐	☐	☐	The cooler lid and sides have been taped to ensure a seal.
☐	☐	☐	The custody seals have been placed on both the front and back hinges of the cooler, using waterproof tape.

Air Waybill Completion

Yes	No	N/A	
☐	☐	☐	Section 1 has the shipper's name, company, and address; the account number, date, internal billing reference number; and the telephone number where the shipper can be reached.
☐	☐	☐	Section 2 has the recipient's name and company along with a telephone number where they can be reached.
☐	☐	☐	Section 3 has the **Bill Sender** box checked.
☐	☐	☐	Section 4 has the **Standard Overnight** box checked.
☐	☐	☐	Section 5 has the **Deliver Weekday** box checked.
☐	☐	☐	Section 6 has the number of packages and their weights filled out. Was the total of all packages and their weights figured up and added at the bottom of Section 6?
☐	☐	☐	Under the **Transport Details** box, the **Cargo Aircraft Only** box is obliterated, leaving only the **Passenger and Cargo Aircraft** box.
☐	☐	☐	Under the **Shipment Type**, the **Radioactive** box is obliterated, leaving only the **Non-Radioactive** box.
☐	☐	☐	Under the **Nature and Quantity of Dangerous Goods** box, the **Proper Shipping Name, Class or Division, UN or ID No., Packing Group, Subsidiary Risk, Quantity and Type of Packing, Packing Instructions, and Authorization** have been filled out for the type of chemical being sent.
☐	☐	☐	The **Name, Place and Date, Signature, and Emergency Telephone Number** appears at the bottom of the FedEx airbill.
☐	☐	☐	The statement "In accordance with IATA/ICAO" appears in the **Additional Handling Information** box.
☐	☐	☐	The **Emergency Contact Information** at the bottom of the FedEx airbill lists someone who can truly respond any time of the day or night.

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Proper Shipping Name	*Class or Division*	*UN or ID No.*	*Packing Group*	*Sub Risk*	*Quantity*	*Packing Instruction*	*Authorization*
Hydrochloric Acid Solution	8	UN1789	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Nitric Acid Solution (with less than 20%)	8	UN2031	II		1 plastic box × 0.5 L	Y807	Ltd. Qty.
Sodium Hydroxide Solution	8	UN1824	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Sulfuric Acid Solution	8	UN2796	II		1 plastic box × 0.5 L	Y809	Ltd. Qty.
Methanol	3	UN1230	II		1 plastic box × 1 L	Y305	Ltd. Qty.

Sample Cooler Labeling

Yes	**No**	**N/A**	
ϒ	ϒ	ϒ	The proper **shipping name, UN number**, and **Ltd. Qty.** appears on the shipping container (limited quantity shipments).
ϒ	ϒ	ϒ	The corresponding **hazard labels** or **dangerous goods in excepted quantity label** are affixed on the shipping container; the labels are not obscured by tape.
ϒ	ϒ	ϒ	The **name and address of the shipper and receiver** appear on the top and side of the shipping container.
ϒ	ϒ	ϒ	The **air waybill** is attached to the top of the shipping container.
ϒ	ϒ	ϒ	**Up arrows** have been attached to opposite sides of the shipping container.
ϒ	ϒ	ϒ	Packaging tape does not obscure markings or labeling.

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Appendix B

Shipment Quality Assurance Checklist

Date: _____ Shipper: _____ Destination: _____

Item(s) Description: _____

Radionuclide(s): _____

Radiological Survey Results: surface _____mrem/hr 1 meter _____

Instrument Used: Mfgr: _____ Model: _____

S/N: _____ Cal Date: _____

Limited-Quantity or Instrument and Article

Yes	No		
___	___	1.	Strong tight package (package that will not leak material during conditions normally incidental to transportation).
___	___	2.	Radiation levels at any point on the external surface of package less than or equal to 0.5 mrem/hr.
___	___	3.	Removable surface contamination less than 20 dpm/100 cm^2 (alpha) and 1,000 dpm/100 cm^2 (beta/gamma).
___	___	4.	Outside inner package bears the marking "Radioactive."
___	___	5.	Package contains less than 15 grams of ^{235}U (check yes if ^{235}U not present).
___	___	6.	Notice enclosed within or on the package that includes the consignor or consignee and the statement, **"This package conforms to the conditions and limitations specified in 49 CFR 173.421 for radioactive material, excepted package-limited quantity of material, UN2910."**
___	___	7.	Activity less than that specified in 49 CFR 173.425. Permissible package limit: Package Quantity:
___	___	8.	On all air shipments, the statement **Radioactive Material, excepted package-limited quantity of material** shall be noted on the air waybill.

Qualified Shipper: _____ Signature: _____

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Guide to Handling Investigation-Derived Waste	SOP 2-2 Revision: 9 Date: August 2020

Approved: Ernest Ashley **Technical Review:** David Sembrot

1.0 Objective

This technical standard operating procedure (SOP) presents guidance for the management of investigation-derived waste (IDW). The primary objectives for managing IDW during field activities include:

- Leaving the site in no worse condition than existed before field activities
- Removing wastes that pose an immediate threat to human health or the environment
- Proper handling of onsite wastes that do not require offsite disposal or extended aboveground containerization
- Complying with federal, state, local, and facility applicable or relevant and appropriate requirements (ARARs)
- Careful planning and coordination of IDW management options
- Minimizing the quantity of IDW

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Hazardous Waste - Discarded material that is regulated listed waste, or waste that exhibits ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261.3 or state regulations.

Investigation-Derived Wastes - Discarded materials resulting from field activities such as sampling, surveying, drilling, excavation, and decontamination processes that, in present form, possess no inherent value or additional usefulness without treatment. Wastes may be solid, sludge, liquid, gaseous, or multiphase materials that may be classified as hazardous or nonhazardous.

Mixed Waste - Any material that has been classified as both hazardous and radioactive.

PFAS – Per- and polyfluoroalkyl substances are a group of man-made chemicals that includes PFOA, PFOS, GenX, and many other chemicals.

Radioactive Wastes - Discarded materials that are contaminated with radioactive constituents with specific activities in concentrations greater than the latest regulatory criteria (i.e., 10 CFR 20).

Treatment, Storage, and Disposal Facility (TSDF) - Permitted facilities that accept hazardous waste shipments for further treatment, storage, and/or disposal. These facilities must be permitted by the U. S. Environmental Protection Agency (EPA) and appropriate state and local agencies.

Aqueous liquid – a water based polar solution with a specific gravity at or near 1. Light non-aqueous phase liquids, also known as LNAPL), such as oils (non-polar, typically float on aqueous (polar) solutions (or pure water). Dense non-aqueous phase liquids (DNAPL), such as chlorinated organic solvents or PCB containing oils, sink in aqueous based liquids.

2.2 Discussion

Field investigation activities result in the generation of waste materials that may be characterized as hazardous or radioactive. IDWs may include drilling muds, cuttings, and purge water from test pit and well installation; purge water, soil, and other materials from collection of samples; residues from testing of treatment technologies and pump and treat systems; personal protective equipment (PPE); solutions (aqueous or otherwise) used to decontaminate nondisposable protective clothing and equipment; and other wastes or supplies used in sampling and testing potentially hazardous or radiologically contaminated material.

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2.2.1 PFAS Considerations

Currently, PFAS characterization and disposal requirements are being initiated by many disposal facilities in anticipation of regulatory initiatives on the state and federal level. These evolving regulatory standards for PFAS must be considered in the implementation of many SOPs including SOP 2-2. These same concerns must be considered in the development of general project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans and laboratory statements of work used to guide the collection of samples for PFAS analysis.

- A consideration of the PFAS-specific sampling requirements is needed during the project planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, reporting limits are all impacted with the inclusion of sampling for PFAS analysis.
- Before implementing the IDW (SOP 2.2) for collection of samples for PFAS waste characterization analysis for disposal purposes, it is important to review current disposal facility requirements and state and federal regulatory standards to determine whether site- or project-specific changes to this SOP should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith Regulatory Updates can be located at the INFOCENTER. Specific State and Federal Regulatory Limit updates can be found here: https://cdmsmith.libguides.com/PFAS_InfoCenter

Note: The client's representatives may not be aware of all potential contaminants. The management of IDW must comply with applicable regulatory requirements.

3.0 General Responsibilities

Site Manager - The site manager is responsible for ensuring that all IDW procedures are conducted in accordance with this SOP. The site manager is also responsible for ensuring that handling of IDW is in accordance with site-specific requirements.

Project Manager - The project manager is responsible for identifying site-specific requirements for the disposal of IDW in accordance with federal, state, and/or facility requirements.

Field Crew Members - Field crew members are responsible for implementing this SOP and communicating any unusual or unplanned condition to the project manager's attention.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/project specific quality assurance plan.

4.0 Required Equipment

Equipment required for IDW containment will vary according to site-specific/client requirements. Management decisions concerning the necessary equipment required shall consider: containment method, sampling, labeling, maneuvering, and storage (if applicable). Equipment must be onsite and inspected before commencing work.

The selection of the container type and size for containerizing IDW must consider:

- waste/contaminant segregation (i.e. do not mix hazardous and non-hazardous wastes or incompatible materials),
- efficiency/ability to move the containerized waste (i.e. size of equipment needed vs. accessibility and bulk vs. individual containers),
- cost of storage, (i.e. rental vs. purchase)
- transportation and disposal of the material in the containers selected.
- PFAS is present many commonly used materials. Please see the PFAS material list in Section 4.5.

4.1 IDW Containment Devices

The appropriate containment device (drums, tanks, etc.) will depend on site- or client-specific requirements and the ultimate disposition of the IDW. Typical IDW containment devices can include:

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- Plastic sheeting (polyethylene) with a minimum thickness of 20 micrometers
- Department of Transportation (DOT)-approved steel containers
- Polyethylene or steel bulk storage tanks

Containment of IDW shall be segregated by waste type (i.e., solid or liquid, corrosive or flammable, etc.) and source location. Volume of the appropriate containment device will depend on site-specific requirements.

4.2 IDW Container Labeling

A "Waste Container" or "IDW Container" label or indelible marking shall be applied to each container. Labeling or marking requirements for onsite IDW not expected to be transported offsite are as detailed below.

- Labels and markings must contain the following information: project name, generation date, location of waste origin, container identification number, sample number (if applicable), and contents (drill cuttings, purge water, PPE, etc.).
- Each label or marking will be applied to the upper one-third of the container at least twice, on opposite sides.
- Containers that are 5 gallons or less may only require one label or set of markings.
- Labels or markings will be positioned on a smooth part of the container. The label must not be affixed across container bungs, seams, ridges, or dents.
- Labels must be constructed of a weather-resistive material with markings made with a permanent marker or paint pen and capable of enduring the expected weather conditions. If markings are used, the color must be easily distinguishable from the container color.
- Labels will be secured in a manner to ensure that they remain affixed to the container.

Labeling or marking requirements for IDW expected to be transported offsite must be in accordance with the requirements of 49 CFR 172.

4.3 IDW Container Movement

Staging areas for IDW containers shall be predetermined and in accordance with site-specific and/or client requirements. Arrangements shall be made before field mobilization as to the methods and personnel required to safely transport IDW containers to the staging area. Transportation of IDW containers offsite via a public roadway is prohibited unless 49 CFR 172 requirements are met.

4.4 IDW Container Storage

Containerized IDW awaiting results of pending chemical analysis or further onsite treatment shall be staged on site. Staging areas and bulk storage procedures are to be determined according to site-specific requirements. Containers are to be stored in such a fashion that the labels can be easily read. A secondary/spill container must be provided for liquid IDW storage and as appropriate for solid IDW storage (e.g., steel drums shall not be stored in direct contact with the ground).

5.0 Procedures

The three general options for managing IDW are: (1) collection and onsite disposal, (2) collection for offsite disposal, and (3) collection and interim management. Attachment 1 summarizes media-specific information on generation processes and management options. The option selected shall take into account the following factors:

- Type (soil, sludge, liquid, debris), quantity, and source of IDW
- Risk posed by managing the IDW onsite
- Compliance with regulatory requirements
- IDW minimization and consistency with the IDW remedy and the site remedy

In all cases the client shall approve the plans for IDW. Formal plans for the management of IDW must be prepared as part of a work plan or separate document.

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5.1 Collection and Onsite Disposal

5.1.1 Soil/Sludge/Sediment

The options for handling soil/sludge/sediment IDW are:

1. Return IDW to boring, pit, or source immediately after generation as long as returning the media to these areas will not increase site risks (e.g., so that "clean" areas are not contaminated, the IDW material will not be replaced at a greater depth, or in a different area than from where it was originally obtained.

2. Spread IDW around boring, pit, or source within the area of contamination (AOC) as long as returning the media to these areas will not increase site risks (e.g., direct contact with surficial contamination).

3. Consolidate IDW in a pit within the AOC as long as returning the media to these areas will not increase site risks (e.g., the contaminated soil will not be replaced at a greater depth than where it was originally so that it will not contaminate "clean" areas).

4. Send to onsite TSDF. This option may require results of laboratory analysis before treatment/disposal.

Note: These options may require client and/or regulatory approval.

5.1.2 Aqueous Liquids

The options for handling aqueous liquid IDW are:

1. Discharge to surface water, only when IDW is not contaminated, and with written client approval.

2. Discharge to ground surface close to the well from which it was extracted, only if soil contaminants will not be mobilized in the process and the action will not contaminate clean areas. If IDW from the sampling of background upgradient wells is not a community concern or associated with soil contamination, this presumably uncontaminated IDW may be released on the ground around the well with written client approval.

3. Discharge to sanitary sewer, only when IDW is not contaminated and with written client approval.

4. Send to onsite treatment/disposal facility, with facility acceptance and written client approval.

Note: These options may require results of laboratory analysis to obtain client and/or regulatory approval.

5. When small amounts (i.e., less than 5 gallons) of used decontamination fluids are generated during site characterization activities (e.g., during soil sampling using direct push technology methods), the fluids may be allowed to evaporate by spreading them on an asphalted surface, or allowing for evaporation from an open bucket.

5.1.3 Disposable PPE

The options for handling disposable PPE are:

1. Double-bag contents in nontransparent trash bags and place in onsite industrial dumpster, only if PPE is not contaminated.

2. Containerize, label, and send to onsite TSDF. This may require results of laboratory analysis before treatment/disposal.

5.2 Collection for Offsite Disposal

Before sending IDW to an offsite TSDF or to a publicly owned treatment works (POTW), laboratory analysis may be required. Manifests are required to accompany any IDW determined to be hazardous. In some instances, a bill of lading can be used for nonhazardous solid IDW (i.e., wooden pallets, large quantities of plastic sheeting). Arrangements must be made with the client

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responsible for the site to sign as generator on any waste profile and all manifests or bill of ladings; it is CDM Smith's policy not to sign any waste profile or manifest. The TSDF and transporter must be permitted for the respective wastes. Nonbulk containers (e.g., drums) must have a DOT-approved label adhered to the container and all required associated placard stickers before leaving for an offsite TSDF. These labels must include information as required in 49 CFR 172. Bulk containers (i.e., rolloffs, tanks) do not require container specific labels for transporting offsite, but must include appropriate placards as required in 49 CFR 172.

5.2.1 Soil/Sludge/Sediment

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., drummed, covered in a waste pile) or returned to its source until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.2 Aqueous Liquids

When the final site remedy requires offsite treatment and disposal, the IDW may be stored (e.g., mobile tanks or drums with appropriate secondary containment) until final disposal. The management option selected shall take into account the potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.2.3 Disposable PPE

When the final site remedy requires offsite treatment and disposal, the IDW may be containerized and stored. The management option selected shall take into account potential for increased risks, applicable regulations, and other relevant site-specific factors (e.g., weather, storage space, and public concern/perceptions).

5.3 Collection and Interim Management

All interim measures must be approved by the client and regulatory agencies.

1. Storing IDW onsite until the final action may be practical in the following situations:
   - Returning wastes (especially sludges and soils) to their onsite source area would require reexcavation for disposal as determined for the final site remedy.
   - Interim storage in containers may be necessary to provide adequate protection to human health and the environment.
   - Offsite disposal options may trigger land disposal regulations under the Resource Conservation and Recovery Act (RCRA). Storing IDW until the final disposal of all wastes from the site will eliminate the need to address this issue more than once.
   - Interim storage may be necessary to provide time for sampling and analysis.

2. Segregate and containerize all waste for future treatment and/or disposal.
   - Containment options for soil/sludge/sediment may include drums or covered waste piles in AOC.
   - Containment options for aqueous liquids may include mobile tanks or drums.
   - Containment options for PPE may include drums or roll-off boxes.

6.0 Restrictions/Limitations

Site managers shall determine the most appropriate disposal option for aqueous liquids on a site-specific basis. Parameters to consider, especially when determining the level of protection, include the volume of IDW, the contaminants present in the aqueous liquid, the nature of contaminants present in the site soil , and whether groundwater or surface water is a drinking water supply, and if obtained from contaminated groundwater, whether the plume is contained or migrating. Special disposal/handling may be needed for drilling fluids because they may contain significant solid components and therefore may need to be handled, treated, disposed as non-liquid wastes.

Disposable sampling materials, disposable PPE, decontamination fluids, etc. will always be managed on a site-specific basis. Under no circumstances shall these types of materials be stored in a site office or warehouse.

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7.0 References

Environmental Resource Center. 1997. *Hazardous Waste Management Compliance Handbook 2nd Edition*. Karnofsky (Editor).

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Title 49 Code of Federal Regulations, Department of Transportation. 2005 or current revision. *Hazardous Materials Table, Special Provisions, Hazardous, Materials Communications, Emergency Response Information, and Training Requirements*, 49 CFR 172.

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CDM Smith Regulatory Updates can be located at the INFOCENTER. Specific State and Federal Regulatory Limit updates regarding PFAS can be found here: https://cdmsmith.libguides.com/PFAS_InfoCenter

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Attachment 1
IDW Management Options

Type of IDW	*Generation Processes*	*Management Options*
Soil	▪ Well/Test pit installations ▪ Borehole drilling ▪ Soil sampling	**Onsite Disposal** ▪ Return to boring, pit, or source immediately after generation ▪ Spread around boring, pit, or source within the AOC ▪ Consolidate in a pit (within the AOC) ▪ Send to onsite TSDF **Offsite Disposal** ▪ Client to send to offsite TSDF **Interim Management** ▪ Store for future treatment and/or disposal
Sludge/Sediment	▪ Sludge pit/sediment sampling	**Onsite Disposal** ▪ Return to pit or source immediately after generation ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* **Interim Management** ▪ Store for future treatment and/or disposal
Aqueous Liquids (groundwater, surface water, drilling fluids, wastewater)	▪ Well installation/development ▪ Well purging during sampling ▪ Groundwater discharge during pump tests ▪ Surface water sampling ▪ Wastewater sampling	**Onsite Disposal** ▪ Pour onto ground close to well (nonhazardous waste) ▪ Discharge to sewer ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* ▪ Client to send to publicly owned treatment works (POTW) **Interim Management** ▪ Store for future treatment and/or disposal
Decontamination Fluids	▪ Decontamination of PPE and equipment	**Onsite Disposal** ▪ Send to onsite TSDF ▪ Evaporate (for small amounts of low contamination organic fluids) ▪ Discharge to ground surface **Offsite Disposal** ▪ Send to offsite TSDF* ▪ Discharge to sewer **Interim Management** ▪ Store for future treatment and/or disposal
Disposable PPE and Sampling Equipment	▪ Sampling procedures or other onsite activities	**Onsite Disposal** ▪ Place in onsite industrial dumpster ▪ Send to onsite TSDF **Offsite Disposal** ▪ Send to offsite TSDF* **Interim Management** ▪ Store for future treatment and/or disposal

* Client must sign waste profile, manifest, etc. for any waste sent offsite.
Adapted from U. S. Environmental Protection Agency, *Guide to Management of Investigation-Derived Wastes*, 9345-03FS, January 1992.

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Field Logbook Content and Control	SOP 4-1 Revision: 9 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Catherine Love

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to set criteria for field logbook content entry and form. Field logbooks are an essential tool to document field activities for historical and legal purposes.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Biota - The flora and fauna of a region

Magnetic Declination Corrections - Compass adjustments to correct for the angle between magnetic north and geographical meridians

2.2 Discussion

Information recorded in field logbooks includes field team names; observations; data; calculations; date and time; weather; and a description of the data collection activity, methods, instruments, and results. Additionally, the logbook may contain record of deviations from plans. It may also hold descriptions of wastes, biota, geologic material, and site features, including sketches, maps, drawings, or photographic or videographic logs, as appropriate.

3.0 General Responsibilities

Project Manager (PM) - The PM is responsible for overall project management. They may be brought into decisions regarding data management.

Field Team Leader (FTL) - The FTL is responsible for ensuring that data entry format and content are in accordance with this procedure.

Site Personnel - All CDM Smith employees who make entries in field logbooks during onsite activities are required to read this procedure before engaging in this activity. The FTL will assign field logbooks to site personnel who will be responsible for their care and maintenance. Site personnel will return field logbooks to the records file at the end of the assignment.

Data Manager (DM) - For projects using cloud base field data collection tools, the DM is responsible ensuring that a copy of the initial data collected is stored as an official record in un-editable file. The DM is responsible for regularly backing up all data generated during electronic field collection, including tabular and spatial data and photographs.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities should be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Equipment

- Site-specific plans
- Field logbook
- Indelible black or blue ink pen
- Ruler or similar scale

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5.0 Procedures

5.1 Preparation

In addition to this SOP, site personnel responsible for maintaining logbooks should be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety, sample collection, packaging, decontamination, and documentation requirements. These procedures should be available electronically or located at the field office or vehicle for easy reference.

Field logbooks shall be bound with lined, consecutively numbered pages. All pages should be numbered before initial use of the logbook. Before use in the field, each logbook will be marked with a specific document control number issued by the document control administrator, if required by the contract quality implementation plan (QIP). Not all contracts require document control numbers. The following information shall be recorded on the cover of the logbook:

- Field logbook document control number (if applicable)
- Activity (if the logbook is to be activity-specific), site name, and location
- Name of CDM Smith contact and phone number(s) (typically the project manager)
- Start date of entries
- End date of entries
- In specific cases, special logbooks may be required (e.g., waterproof paper for stormwater monitoring).

The first few (approximately three) pages of the logbook will be reserved for a table of contents (TOC). Mark the first page with the heading and enter the following:

Table of Contents

Date/Description	Pages
(Start Date)/Reserved for TOC	1-3

The table of contents remaining pages will be designated as such with “TOC” written on the top center of each page. The table of contents should be completed as activities are completed and before placing the logbook in the records file.

5.2 Operation

Follow these requirements when using a logbook:

- Record work, observations, quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are specified by an activity-specific plan, this information does not need to be duplicated in the logbook. However, the logbook should reference any forms used to record site information.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not erase or blot out any entry at any time. Indicate any deletion by a single line through the material to be deleted. Initial and date each deletion. Take care to not obliterate what was written previously.
- Do not remove any pages from the book.
- All entries should be clearly written and legible.

Specific requirements for field logbook entries include:

- Initial and date each page
- Sign and date the final page of entries for each day
- Initial and date all changes
- Multiple authors should sign out the logbook by inserting the following:
  Above notes authored by:
  - (Sign name)
  - (Print name)

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- (Date)
- A new author should sign and print his/her name before additional entries are made.
- Draw a diagonal line through the remainder of the final page at the end of the day.
- Record the following information on a daily basis:
  - Date and time
  - Name of individual making entry
  - Names of field team and other persons onsite
  - Description of activity being conducted, including station or location (i.e., well, boring, sampling location number) if appropriate
  - Weather conditions (i.e., temperature, cloud cover, precipitation, wind direction, and speed) and other pertinent data
  - Level of personal protection used
  - Serial numbers of instruments
  - Equipment calibration information
  - Serial/tracking numbers on documentation (e.g., carrier air bills)

An observation's time (written in military/24-hour units) should go before the entry. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. Record all measurements made and samples collected unless they are documented by automatic methods (e.g., data logger), a field data collection tool, or on a separate form required by an operating procedure. In these cases, the logbook should reference the automatic data record or form. Site personnel collecting data with cloud-base field data collection tools are responsible for sending data to the cloud database as data is collected or when cellular or wi-fi service becomes available. If the tool is working in off-line editing, then data should be uploaded or synced at the end of the work day.

At each station where a sample is collected or an observation or measurement made, a detailed description of the location of the station is required. Use a compass (include a reference to magnetic declination corrections), scale, or nearby survey markers, as appropriate. A sketch of station location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Maps, sketches, figures, or data that will not fit on a logbook page should be referenced and attached to the logbook to prevent separation.

Other events and observations that should be recorded include:

- Changes in weather that impact field activities
- Deviations from procedures outlined in any governing documents. Record the reason for any noted deviation.
- Problems, downtime, or delays
- Upgrade or downgrade of personal protection equipment
- Visitors to the site

5.3 Post-Operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages shall be periodically photocopied or scanned (weekly, at a minimum) and forwarded to the field or project office. Other field records shall be photocopied or scanned and submitted regularly and as promptly as possible to the office. Information from a data logger shall be exported daily onto external storage device (e.g. a flash drive or external drive) and saved to ProjectWise on a daily basis if possible (weekly, at a minimum).

At the conclusion of each activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated and that corrections were made properly (single lines drawn through incorrect information, then initialed and dated). The completed logbook shall be submitted to the records file.

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For cloud-based field data collection tools, all data (backups, raw inspection data) will be saved daily to the ProjectWise directory. Alternative timed uploads can be set to weekly data uploads at a minimum, if agreed upon by the DM and PM.
Field data collection tools should be designed so that a copy of the originally captured data is exported daily and saved on ProjectWise. This data serves as the official record and should not be altered or edited. Developers of the field tools should understand retention policies of their cloud platform and ensure that final data is stored on ProjectWise to meet project data retention requirements.

All data collection methods should follow the records control procedures specified in the site-specific plan.

6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by CDM Smith personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and objective. Field logbooks, and entries within, are not to be used for personal use.

Consider the evolving regulatory standards for per- and polyfluoroalkyl substances (PFAS) when implementing SOP 4-1 for specific sites. Also consider them when developing project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans, and laboratory statements of work used to guide the collection of samples for PFAS analysis.

A consideration of the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, reporting limits are all impacted with the inclusion of sampling for PFAS analysist.

Before implementing SOP 4-1 associated with collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether site- or project-specific changes to SOP 4-1 should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith Regulatory Updates, including specific state and federal regulatory limits, can be located on the PFAS InfoCenter: https://cdmsmith.libguides.com/PFAS_InfoCenter

When conducting PFAS sampling, field staff should avoid cross contamination which can occur with use of items in the field, such as sticky notes, waterproof field books, and aluminum foil. Staff will need to avoid use of markers, if possible, during sampling use only regular ink pens. If needed, write over regular ink with marker after the sample containers are sealed. For a current list of materials to avoid, staff should refer to appropriate sampling technical operating procedures and project requirements.

7.0 References

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Photographic Documentation of Field Activities	SOP 4-2 Revision: 10 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Catherine Love

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to provide standard guidelines and methods for photographic documentation, which include digital photography and recordings of field activities and site features (geologic formations, core sections, lithologic samples, water samples, general site layout, etc.). This SOP is intended for circumstances when formal photographic documentation is required. Based on project requirements, it may not be applicable for all photographic activities.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Photographer - The camera operator of digital photography or recordings, whose primary function in this SOP is to produce documentary or data-oriented visual media

Identifier Component - Visual components used within a photograph, such as visual slates, reference markers, and pointers

Standard Reference Marker - A reference marker used to indicate a feature size in the photograph. It should be a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Slates - Blank white index cards, marker boards, or paper used to present information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black indelible marking pens.

Arrows and Pointers - Markers/pointers used to indicate and/or draw attention to a special feature within the photograph

Camera - A standalone digital camera or a camera that is embedded in a smart phone or tablet

Contrasting Backgrounds - Backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

Date Stamp - A built-in feature that will record the date and time directly on a digital image or recording

Field Tool Application - An application that runs on a smart phone or tablet. The application's purpose is to collect a specific set of information, which may include the ability to take and document photographs. Data collection with the field tool is stored in the field tool application database. The field tool application can be designed to capture information typically stored in the project logbook, however the tool cannot completely replace the log book.

Photographic Documentation - Any information collected in a logbook or in a field tool application that is required to describe the photograph. Documentation might include the file name of the photo, photo sequence, photographer, date and time, a description of the photo, or the direction the picture was taken.

Site Photo Map - A sketch of the site that marks where photographs were taken

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*

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2.3 Discussion

Digital photographs and recordings made during field investigations are used as an aid in documenting and describing site features, sample collection activities, equipment used, and possible lithologic interpretation. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. How to use a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the images or recordings and ensure the representative nature of the photographic documentation.

3.0 General Responsibilities

Project Manager - The project manager (PM) will ensure the logbook and photographs fulfill contract requirements and is distributed appropriately. They will also provide guidance on site-specific photography requirements.

Field Team Leader - The field team leader (FTL) is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The FTL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting. The FTL is responsible for making sure the photographer understands and follows data management process for logging, documenting, and backing up the photographic files. If a field tool application is being used to take photographs, the FTL is responsible for working with the data manager to ensure the application collects the proper metadata data about the photographs and that the photographs are being stored and backed up properly.

Data Manager - The data manager (DM) is responsible for ensuring that the field application tool collects and stores the proper photographic documentation about the photographs. The DM is also in charge of verifying that photographic files and documentation have been downloaded from the field tool application and stored in the project file.

Photographer - The photographer shall seek direction from the FTL and regularly discuss the visual documentation requirements and schedule. The photographer is responsible for maintaining a logbook per Sections 5.1, 5.2.4, and 5.3.1 of this SOP. The photographer's responsibilities will be defined in the project sampling plan.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site/quality assurance project plan (QAPP). Some clients and/or client facilities have specific restrictions on photography (e.g., some do not allow photographs to show secure or sensitive buildings). Obtain permission to use a camera on these facilities in advance. The photographer may need to review the photo record with responsible site personnel.

4.0 Required Equipment

A general list of equipment that may be used:

- Smart phone or tablet with camera or digital camera and appropriate storage media
- Extra battery pack and charging cord for smart phone or tablet
- Extra batteries for camera
- Digital video camera and appropriate storage media (e.g., SD card)
- Logbook
- field tool application
- Indelible black or blue ink pen
- Standard reference markers (e.g. ruler)
- Slates or marker boards
- Arrows or pointers
- Contrasting backgrounds

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5.0 Procedures

5.1 Documentation

Documentation requirements for digital photographs and recordings should be specified in the site-specific plan. Otherwise, a commercially available, bound logbook will be used to log and document photographic activities. Alternatively, the project may develop a field tool application designed to run on a smart phone or tablet to take pictures and document the photographic activities. Review SOP 4-1, *Field Logbook Content and Control* and prepare all supplies needed for logbook entries.

Note: A separate photographic logbook is not required. A portion of the field logbook may be designated as the photographic log and documentation section.

Field Health and Safety Considerations

There are no hazards that an individual will be exposed to specific to photographic documentation. However, site-specific hazards may arise depending on the location or operation. The photographer should be aware of site-specific terrain to avoid tripping hazards. Personal protective equipment used in this operation will be site-specific and dictated through requirements set by the site safety officer, site health and safety plan, and/or prescribed by the CDM Smith Corporate Health and Safety Program. The photographer should contact the site safety officer for health and safety orientation before commencing field activities. Read, and sign the acknowledgement of, the site health and safety plan before entering the site.

The photographer should be aware of any potential physical hazards while photographing the subject (e.g., traffic, low overhead hazard, edge of excavation).

5.2 Operation

5.2.1 General Photographic Activities in the Field

The following sections provide general guidelines that should be followed to visually document field activities and site features using digital still and video cameras. General suggestions the photographer should consider when performing activities under this SOP are as follows:

- The photographer should be prepared to make a variety of shots, from close-up to wide-angle. Many shots will be repetitive in nature or format, especially close-up site feature photographs. Consider designing a system, camera settings, or technique that will provide a reliable repetition of performance. Not any special enhancement techniques in the log book or field tool application.
- Digital cameras have multiple photographic quality settings. A camera that produces higher resolution (quality) photographs has a higher number of pixels and will store a fewer number of photographs per digital storage medium. Determine project photo resolution requirements before implementing field work.
- If using a smart phone or tablet camera with a field tool application, consider that the device will need cellular service or WiFi to upload photos and documentation to the field tool application database. If using a smart phone or tablet camera that is not associated with a field tool application, the photographer shall develop a procedure for how the photographs will be extracted from the device and correlated with the photographic documentation in the log book.
- No preference of digital storage medium for a digital camera is specified. The photographer has discretion.
- Regardless of the type of camera, the photograph needs to be stored in a location that receives regular backups such as ProjectWise, OneDrive or Teams. For staff in the field without access to a regularly backed-up storage location, email can provide a temporary backup location until a more permanent location can be accessed.
- For projects dealing with confidential information or criminal investigation, the photographer should look at client-specific requirements for chain of custody procedures.

5.2.2 General Guidelines for Still Photography

Caption Information

Unless otherwise specified in a site-specific plan, all still photographs will have a full caption added after the images are downloaded on a photo log sheet. The caption should contain the following information:

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- Photograph sequence number
- Date and time
- Photographer (required for enforcement cases)
- Description of activity/item shown (e.g., name of facility/site, specific project name, project number)
- Direction (if applicable)
- Site photo map (if applicable)

When directed by the sampling plan, a standard reference marker should be used in all documentary visual media. While the standard reference marker will be predominantly used in close-up feature documentation, consider including it in all scenes.

Images taken with a digital camera, smart phone, or tablet, should be downloaded at least once each day to a personal computer; the files should be in either "JPEG" or "TIFF" format. Files should be renamed at the time of download to correspond to the logbook or as directed in the site-specific plan.

Images taken with a camera as part of a field tool application that resides on a smart phone or tablet should fill out the photographic documentation in the application. The photographer will need to upload the data each night (at a minimum) to the field tool application database. The photographer should verify with the DM that photographs are properly stored and backup via the field tool application.

Regardless of the type of camera used, the photographs should be compared to the photo documentation to ensure that the log and the photographs match.

Close-Up and Feature Photography
When directed by the sampling plan, close-up photographs should include a standard reference marker of appropriate size, to indicate feature size. The photos should contain a slate or marker board marked with the site name and any identifying label, such as a well number or core depth, that clearly tells the viewer the specific feature being photographed.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their in situ locations. This enables a more accurate record of their initial condition and color. When directed by the sampling plan, include a standard reference color strip, like a color chart such as Munsell Soil Color Chart, within the scene. This serves as a reference aid to the viewer for formal lithologic observations and interpretations.

Site Photography
Site photography, in general, will consist predominantly of medium- and wide-angle shots. If required by the sampling plan, a standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane.

While it is encouraged that a standard reference marker and slate/marker board be included in the scene, it is understood that situations will arise where they can't be included. This will be especially true of wide-angle shots. In such a case, the image number shall be entered in the photographic logbook along with other information pertinent to the scene.

5.2.3 General Photographic Documentation Using Digital Video Cameras

Documentary digital recordings of field activities may include an audio slate for all scenes. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking close-up shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

A standard reference marker and slate/marker board should be included in all scenes. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

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Original video recordings will not be edited. This will maintain the integrity of the information contained on the videotape or DVD. If editing is desired, a working copy of the original video recording can be made.

A digital recording filename should be created with the appropriate identifying information (project name, project number, date, location, etc.).

5.2.4 Photographic Documentation

If required by the site-specific plan, photographic activities should be documented in a photographic logbook or in a section of the field logbook or as part of the field tool application. The photographer will be responsible for making proper entries.

In addition to following the technical standards for logbook entry as referenced in SOP 4-1, the following information should be maintained in the appropriate logbook:

- Photographer name
- Sequential tracking number for each photograph taken (the camera-generated number may be used)
- Date and time (military time)
- Location
- A description of the activity/item photographed
- If needed, a description of the general setup, including approximate distance between the camera and the subject
- Record as much other information as possible to help identify the photographic document.

5.3 Post Operation

The photographer shall be responsible for downloading image files or recordings to the project files.

As required, the photographer(s) will ensure that the appropriate logbook is completely filled out and maintained as outlined in SOP 4-1. Images and recordings will be handled according to contract records requirements. The PM will ensure their proper distribution. Completed pages of the appropriate logbook will be copied weekly and submitted to the project files.

6.0 Restrictions/Limitations

This document is designed to provide a set of guidelines to maintain an effective and standardized program of visual documentation.

It is not within the scope of this document to provide instruction in photographic procedures, nor is it within the scope of this document to set guidelines for presentation or “show” photography.

The procedures outlined herein are general by nature. The photographer is responsible for specific operational activity or procedure described in site-specific plans. Questions concerning specific procedures or requirements should be directed to the PM or FTL.

Note: Some sites do not permit photographic documentation. Check with the site contact for any restrictions.

7.0 References

U. S. Environmental Protection Agency. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Logbooks. SESDPROC-005-R5. January.

_____. 2013. Region 4. The Field Branches Quality System and Technical Procedures, Sample and Evidence Management SESDPROC-005-R2. January.

_____. 1992. National Enforcement Investigations Center. *Multi-Media Investigation Manual*, EPA-330/9-89-003-R. p. 85. Revised March.

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Well Development and Purging	SOP 4-3 Revision: 8 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Stuart Barden

1.0 Objective

The purpose of this technical standard operating procedure (SOP) is to define the procedural requirements for well development and purging.

2.0 Background

Monitoring wells are developed to repair damage to the formation wall caused by drilling activities and to remove fines from the filter pack. Wells shall not be developed for at least 24 to 48 hours after completion when a cement bentonite grout is used to seal the annular space; however, wells may be developed before grouting if conditions warrant. Wells are purged immediately before groundwater sampling to remove stagnant water and to obtain samples representative groundwater conditions. Wells shall be sampled within 3 hours of purging (optimum) to 24 hours after purging (maximum, for low recharge conditions).

2.1 Associated Procedures

- SOP 1-6, *Water Level Measurement*
- SOP 2-2, *Guide to Handing of Investigation Derived Waste*
- SOP 4-1, *Field Logbook Content and Control*
- SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*

3.0 General Responsibilities

Project Manager - The project manager is responsible for ensuring that field personnel are trained to use this procedure and verify that development and purging are carried out in accordance with this procedure.

Field Team Leader - The field team leader is responsible for complying with this procedure.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Required Equipment

4.1 General

- Pump, pump tubing, or bailer and rope or wire line
- Surge blocks or equivalent
- Power source (e.g., generator), if required
- Electronic water-level meter
- Temperature, conductivity, pH, and turbidity meters
- Personal protective equipment as specified in the site-specific health and safety plan
- Decontamination supplies, as required, according to SOP 4-5, *Field Equipment Decontamination at Nonradioactive Sites*
- Disposal drums, if required
- Photoionization detector (PID) or equivalent as specified in site-specific health and safety plan

***If the well will be sampled for per- and polyfluoralkyl substances (PFAS)**, the potential for cross-contamination from common materials should be evaluated. Commonly used sampling materials and equipment to avoid (or verify composition) include:

- **PTFE, LDPE**, **Teflon**, sticky notes, waterproof field book, aluminum foil, plastic sheeting

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Consult materials checklists for other equipment concerns. A checklist containing common materials and sampling equipment that contain PFAS compounds is attached, and an up-to-date list will be maintained here:

https://www.yammer.com/cdmsmith.com/#/files/214861635584

5.0 Procedures

5.1 Well Development

The following steps should be followed when developing wells:

1. Review site-specific health and safety plan and project plans before initiating sampling activity.
2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.
3. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any.
4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.
5. Determine the depth to static water level and depth to bottom of the well (SOP 1-6).
6. Prepare the necessary equipment to develop the well. There are a number of techniques that can be used to develop a well. Some of the more common methods are bailing, overpumping, backwashing, mechanical surging, surge and pump, wire brush, swabbing, and high-velocity jetting. All of these procedures may be acceptable; however, final approval of the development method rests with the appropriateness of a specific method to the geologic conditions, well type and approved work plans, field sampling plans, etc. In general, well development should involve pumping and surging the well screen to mobilize fines in the adjacent formation wall and filter pack. Surging activities are typically followed by pumping until the water produced runs clear. This may require repeated cycles of surging and pumping followed by efforts to remove all sediment from the bottom of the well.
7. For screened intervals longer than 10 feet (3 meters [m]), develop the well in 2- or 3-foot (0.75- or 1-m) intervals from bottom to top. This will ensure proper settling of the filter pack.
8. Continue well development until produced water is clear and free of suspended solids, as determined by a turbidity meter or when pH, conductivity, and temperature have stabilized. Record pertinent data in the field logbook and on appropriate well development forms. Remove the pump assembly or bailers from the well, decontaminate (if required), and clean up the area. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plans.

5.2 Volumetric Method of Well Purging

The following steps shall be followed when purging a well by the volumetric method:

1. Review site-specific health and safety plan and project plans before initiating well purging activity.
2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.
3. Open the well cover and check condition of the wellhead, including the condition of the surveyed reference mark, if any.
4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.
5. Determine the depth to static water level and depth to bottom of well casing according to SOP 1-6, *Water Level Measurement*. Calculate the volume of water within the well bore using the following formula (or equivalent):

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$$7.4805 \left[\frac{D^2\pi}{(4)} \right] dH = \text{volume (in gallons)}$$

where
D = casing diameter in feet. (***Note***: This equation is used for grouted wells with short screens. For wells with long screens and/or ungrouted wells, the D = borehole diameter in feet).
dH = the distance from well bottom to static water level in feet.
π = 3.1416.

Alternatively, the volume may be determined using a casing volume per foot factor for the appropriate diameter well, similar to that in Table 1. The water level is subtracted from the total depth, providing the length of the water column. This length is multiplied by the factor in Table 1 corresponding to the appropriate well diameter, providing the amount of water, in gallons, contained in the well.

TABLE 1
WELL CASING DIAMETER vs. VOLUME

WELL CASING DIAMETER (inches) vs. VOLUME (gals.)/FEET of WATER	
CASING	GALLONS/FT
1	0.041
2	0.163
3	0.367
4	0.653
5	1.02
6	1.469
7	1.999
8	2.611
9	3.305
10	4.08
11	4.934
12	5.875

Note: Record all data and calculations in the field logbook.

6. Prepare the pump and tubing, or bailer, and lower it into the casing.

7. Remove the number of well volumes specified in the site-specific plans. Generally, three to five well volumes will be required. Conductivity, pH, temperature, and turbidity shall be measured and recorded. Purging shall continue until the field parameters have stabilized. Groundwater quality parameters are considered stable when three consecutive readings are within ± 0.1 for pH, ±3 percent for conductivity, ±10 mv for redox potential (ORP), and ±10 percent for turbidity if greater than 10 NTU, in accordance with site-specific plans. Efforts shall be made to get turbidity below 10 NTU, especially if groundwater samples are to be collected for metals or PCB analyses.

8. In low recharge aquifers, the following steps shall be followed: (1) If the initial water level is less than 10 feet above the top of the screen, then purge to dryness and allow sufficient recharge to collect samples. (2) If initial water level in the well is more than 10 feet above the top of the screen, then care shall be taken to prevent the dewatering of the screened interval. (3) Continue purging until the water level is between 1 and 5 feet above the top of the screen. (4) Allow well to recharge then continue purging until at least 1 full initial well volume has been purged. (5) Record pertinent data in the field logbook.

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9. Groundwater sampling shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment that was used for purging. Unfiltered samples shall be collected first, beginning with volatile organic compounds (VOCs). After all unfiltered samples have been collected, a 0.45 micron in-line filter shall be installed in the discharge line for collection of filtered samples, if required.

10. After sampling activities have been completed, remove the pump assembly or bailer from the well, decontaminate it (if required), and clean up the site. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plan.

5.3 Indicator Parameter Method of Well Purging

1. Review site-specific health and safety plan and project plans before initiating well purging activity.

2. Don personal protective clothing and equipment as specified in the site-specific health and safety plan.

3. Open the well cover and check the condition of the wellhead, including the condition of the surveyed reference mark, if any.

4. Monitor the air space at the wellhead, using a PID or equivalent, as soon as well cover is removed according to health and safety requirements.

5. Determine the depth to static water level and depth to bottom. Set up surface probe(s), (e.g., pH, conductivity) at the discharge orifice or dedicated probe port of the pump assembly or within the flow-through chamber. Allow probe(s) to equilibrate according to manufacturer's specifications. Record the equilibrated readings in the field logbook.

6. Assemble the pump and tubing and lower into the casing.

7. Begin pumping or bailing the well. Record indicator parameter readings for every 5 minutes or purge volume, whichever is sooner. Maintain a record of the approximate volumes of water produced. Care shall be taken to minimize drawdown (0 to 0.2 feet).

8. Continue pumping or bailing until indicator parameter readings remain stable within ± 0.1 for pH, ±3 percent for conductivity, ±10 mv for redox potential (ORP), and ±10 percent for turbidity if greater than 10 NTU for three consecutive recording intervals and a minimum of 1 well volume is removed, or in accordance with site-specific plans. Purging shall continue until the discharge stream is clear or turbidity becomes asymptotic-low or meets project requirements.

9. For a low recharge aquifer, follow the guidelines of Section 5.2, Paragraph 7.

10. Groundwater sampling shall be performed immediately upon completion of purging (unless time for recharge is required for low-recharge wells) using the same equipment that was used for purging. Unfiltered samples shall be collected first, beginning with VOCs. After all unfiltered samples have been collected, a 0.45 micron in-line filter shall be installed in the discharge line for collection of filtered samples, if required.

11. Remove the pump assembly or bailer from the well, decontaminate (if required), and clean up the site. Lock the well cover before leaving. Containerize and/or dispose of development water as required by the site-specific plans.

5.4 Procedural Considerations for PFAS

Development of PFAS awareness in guidance and incorporation into specific SOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. For example, Federal drinking water MCL for Arsenic is 10,000 ppt where the EPA health advisory level is a combined 70 ppt for PFOA and PFOS.

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Procedural modifications to field activities when sampling for PFAS analysis involve the concerns listed below. Practice good housekeeping, document these extra steps in detail, and maintain vigilance to adhere to clearly defined exclusion zones.

Good practices

- Use good safety planning and institute exclusion zones, contamination reduction zones, and support zones (no visitors within 30 feet of sampling).
- Do not eat in or near sampling areas (food packaging may contain PFAS).
- Be vigilant about PPE donning and doffing practices.
- Wash hands before and after eating.
- Wear powderless nitrile gloves and change them frequently.
- Open sample container only during sample collection and never set the sample container lid down.
- Keep hands away from the container opening when sampling and keep lid protected.
- Develop a project-specific checklist for review with field personnel.
    - It should be project-specific
    - Completed prior to beginning PFAS sampling event, or when there are staffing changes, or when sample media change and associated sampling/field equipment changes
    - Consider appropriate sampling/field work sequences for each matrix
    - Sampling/field work sequences
    - PFAS sampling/field work should occur first on sites where other contaminants will also be sampled.
    - CDM Smith PFAS Sampling Guidance Team can assist with development of your project-specific checklist.

Avoid

- **PTFE, LDPE**, sticky notes, waterproof field book, aluminum foil
- Field filtering of water samples because of glass fiber filters
    - Avoid using markers, if possible. Use only regular ink pens during sampling. If needed, write over regular ink with marker after the sample containers are sealed.

Consult materials checklists for equipment concerns

6.0 Restrictions/Limitations

Where flammable, free, or emulsified product is expected, or known to exist on or in groundwater, use intrinsically safe electrical devices only and place portable power sources (e.g., generators) 50 feet (15 m) or further from the wellhead and disposal drums. Where sampling for PFAS is anticipated, ensure that the development, purging and sampling equipment and supplies are free from Teflon®, Viton®, and LDPE-containing materials and the equipment materials and clothing that may contain PFAS does not come in contact with down hole equipment.

7.0 References

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PFAS Sampling Checklist

Development of PFAS sampling guidance SOPs is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the parts per trillion (ppt) range. The levels of awareness for PFAS cross contamination vary by the opportunities to introduce PFAS into the environment media under investigation.

1. Critical: Items in direct contact of environmental media under investigation. These can include, but are not limited to, sample containers, sampling parts and equipment, drilling equipment, well construction items and materials, parts and equipment for hydrogeological testing, in-situ treatment parts and equipment.
2. Very Important: PPE, personal hygiene that are used by sampling personnel.
3. Important: Items used in coolers for shipping and transporting PFAS samples.
4. Less important/Awareness level concern: Activities in the staging area away from immediate PFAS investigation area.

Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Field Clothing or PPE				
Clothing or boots containing "water resistance" or "stain-treated" fabrics			X	
Cloths washed with fabric softeners			X	Fabric softeners may contain PFAS
New and unwashed clothing			X	Fabric treatment may contain PFAS
Uncoated Tyvek		X		USEPA PFAS sampling guidance from Region 2 prohibits use of Tyvek
Coated Tyvek			X	
PVC or wax-coated fabrics	X			
Neoprene	X			
Synthetic and natural fibers (preferably cotton)	X			
Steel-toed boots made with polyurethane and PVC	X			If it is not possible to find PFAS-free steel-toed footwear, PFAS-free over boots may be worn. The over boots should be put on and the hands washed after putting on the over boots prior to the sampling activities beginning. Over boots may only be removed in the staging area and after the sampling activities have been completed.
Well laundered clothes	X			several times from time of purchase
Well washed cotton coveralls	X			washed several times

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Personnel Hygiene and Protective Skin Products				
Sunscreens		X		Good to use: Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss My Face, and baby sunscreens that are "free" or "natural"
Insect repellents		X		Good to use: Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics
International brands of sunscreens and insect repellents		X		Should be evaluated on a case-by-case basis
Field Sampling Items				
Water proof field paper or books			X	Use loose plain paper
Post-it notes			X	
Aluminum foil			X	
Brand-name markers			X	Sharpie may be used to label sample bottles in the staging area, but markers should not be used in the immediate sampling environment
Off-brand markers		X		
Ball point pens	X			
Plastic clipboards			X	NJ DEP sampling guidance, use metal clipboard
Plastic table cover			X	
Sampling Equipment				
Item containing high-density polyethylene (HDPE)	X			
Item containing polypropylene	X			PP sample bottles should be used for drinking-water samples in accordance with USEPA method 537.1.1
Item containing polyurethane	X			
Item containing Polyvinyl chloride (PVC)	X			
Item containing silicon	X			

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Item made of stainless steel	X			
Alconox®	X			
Citronex®	X			
Liquinox®	X			
Powderless nitrile gloves	X			
HDPE Hydrasleeves or sonic core bags	X			
Neoprene	X			
Crisco® or other vegetable-based greases for lubricating parts	X			
Item containing PTFE			X	Items or equipment that contains PTFE parts that will be in direct contact with sampling media
Item containing Teflon®			X	Field sampling items or equipment that contains Teflon® and that will be in direct contact with the sampling media
Item containing fluoropolymer			X	
Low-density polyethylene (LDPE)			X	Items or equipment that contains LDPE parts and that will be in direct contact with the sampling media
Viton® O-rings			X	Viton® O-rings used in pressure washers used for sampling equipment decontamination
Glass sample containers			X	
Field filter			X	Field filtration should be avoided regardless of filter types.
Decon 90			X	
Items containing fluorosurfactants			X	
Teflon-bearing plumber's tape			X	
Blue (or chemical) ice			X	Later data (unpublished) suggest no cross contamination from blue ice. The category may be changed after data are published.
Water ice	X			Double bag in polyethylene bags
Internal valves and equipment parts for sampling or decon		X		

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Methanol or other solvents		X		
LDPE plastic bags (e.g., Ziploc® bags)		X		For larger biota sampling, Ziploc bags may be used, but collecting an equipment blank is recommended because these bags may be made of LDPE
Drilling fluids		X		
LDPE sonic core sample bags		X		Manufactured by Boart Longyear and Hole Products
Equipment with moving parts that may be lubricated with PFAS containing lubricants or greases		X		
Rental equipment		X		Should be verified to have no PFAS-bearing parts prior to use
Others				
Food wrappers			X	Field personnel should wash hands after having food wrapped with grease repelling paper

(1) If an item which may contain PFAS but alternative is not available, the item should be tested for PFAS before use
(2) This mostly refers to the immediate sampling environment, particularly, the item is in contact with environmental media to be sampled

There are no standard operation procedures on how an item can be verified. Please contact PFAS experts for advice on the best practice to test a potential PFAS containing item.

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Field Equipment Decontamination at Nonradioactive Sites	SOP 4-5 Revision: 11 Date: March 2020

Approved: Ernest Ashley **Technical Review:** David Sembrot

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to describe the general procedures required for decontamination of field equipment at nonradioactive sites. This SOP serves as a general guide and is applicable at most sites; however, it shall be noted that site-specific conditions (i.e., type of contamination, type of media sampled), the governing agency (e.g., EPA, DOE, USACE), and site-specific work plans, sampling and analysis plans and/or quality assurance (QA) project plans may require modifications to the decontamination procedures provided in this SOP. Decontamination of field equipment is necessary to ensure acceptable quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants offsite.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Acid Rinse - A solution of 10 percent nitric or hydrochloric acid made from reagent grade acid and analyte-free water

Analyte-Free Water - Tap water that has been treated so that the water contains no detectable heavy metals or other inorganic compounds. Analyte-free water shall be stored only in clean glass, stainless steel, or plastic containers that can be closed when not in use.

Clean - Free of contamination and when decontamination has been completed in accordance with this SOP

Cross Contamination - The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or noncontaminated samples or areas.

Decontamination - The process of rinsing or otherwise cleaning the surfaces of sampling or other equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure to personnel

Safety Data Sheets (SDS) - These documents discuss the proper storage and physical and toxicological characteristics of a particular substance used during decontamination. These documents, generally included in site health and safety plans, shall be kept on site at all times during field operations.

Organic-Free/Analyte-Free Water - Tap water that has been treated so that the water meets the analyte-free water criteria and contains no detectable organic compounds. Organic-free/analyte-free water shall be stored only in clean glass, Teflon™, or stainless steel containers that can be closed when not in use.

PFAS – Per- and polyfluoroalkyl substances are a group of man-made chemicals that includes PFOA, PFOS, GenX, and many other chemicals.

Potable Water - Tap water may be obtained from any municipal system. Chemical analysis of the water source may be required before it is used for decontamination purposes.

Sampling Equipment - Equipment that comes into direct contact with the sample media. Such equipment includes split spoon samplers, well casing and screens, and spatulas or bowls used to homogenize samples.

Soap - Low-sudsing, nonphosphate detergent such as Liquinox™.

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Solvent Rinse - Pesticide grade, or better, isopropanol, acetone, or methanol.

2.2 Associated Procedures

- SOP 1-1 – *Surface Water Sampling*
- SOP 1-3 – *Surface Soil Sampling*
- SOP 1-4 – *Subsurface Soil Sampling*
- SOP 1-5 – *Groundwater Sampling Using Bailers*
- SOP 1-7 – *Wipe Sampling*
- SOP 1-9 – *Tap Water Sampling*
- SOP 1-11 – *Sediment/Sludge Sampling*
- SOP 1-12 – *Low Flow (Low-Stress) Groundwater Sampling*
- SOP 1-13 – *Drum Sampling*
- SOP 1-14 – *Lagoon Sampling*
- SOP 1-15 – Procedures for Determination of Screening-Level Elemental Concentrations in Soil and Sediment using Field Portable X-Ray Fluorescence Spectrometry
- SOP 2-2 – *Guide to Handling Investigation-Derived Waste*
- SOP 3-1 – *Geoprobe® Sampling*

3.0 Responsibilities

The project manager or designee, generally the field team leader (FTL), ensures that field personnel are trained in the performance of this procedure and that decontamination is conducted in accordance with this SOP and site-specific work plans. The FTL may also be required to collect and document rinsate samples (also known as equipment blanks) to provide quantitative verification that these procedures have been correctly implemented.

Note: Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance project plan (QAPP).

4.0 Required Equipment

- Stiff-bristle scrub brushes
- Plastic buckets and troughs
- Soap
- Nalgene or Teflon sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayer (pump sprayer material must be compatible with the solution used)
- Plastic sheeting, plastic bags, and/or aluminum foil to keep decontaminated equipment clean between uses
- Disposable wipes, rags, or paper towels
- Potable water*
- Analyte-free water
- PFAS-free water, as determined by regulatory needs of the project and obtained from your laboratory
- Organic-free/analyte-free water
- Gloves, safety glasses, and other protective clothing as specified in the site-specific health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source (e.g., 10 percent and/or 1 percent nitric acid [HNO_3], acetone, methanol, isopropanol, hexane)
- Tools for equipment assembly and disassembly (as required)
- 55-gallon drums or tanks for temporary storage of decontamination water (as required)
- Pallets for drums or tanks holding decontamination water (as required)

* Potable water may be required to be tested for contaminants before use. Check field plan for requirements.

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4.1 PFAS Materials Considerations

Consider the PFAS-specific sampling requirements is needed during the planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels and reporting limits are impacted with the inclusion of sampling for PFAS analysis.

Before implementing TSOP 4-5 for field equipment decontamination where samples for PFAS analysis may be collected, it is important to review current regulatory standards to determine whether site- or project-specific changes to TSOP 1-5 should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith Regulatory Updates can be located in the InfoCenter. Specific state and federal regulatory limits updates can be found at:
https://cdmsmith.libguides.com/PFAS_InfoCenter.

Because of the evolving nature of PFAS regulation and guidance, the information provided here is based on our current knowledge. Some of the recommendations made here may reflect an overly cautious approach to avoid cross-contamination by removing items from the immediate sampling environment. As additional information about PFAS is confirmed the checklist included at the end of this document will be revised to reflect the recommended practices for PFAS sampling. Procedures and Regulatory Updates will be provided separately. Awareness of these concerns is essential to ensure our field sampling teams accurately document field activities they are performing or overseeing.

5.0 Procedures

All reusable equipment (nondedicated) used to collect, handle, or measure samples shall be decontaminated before coming into contact with any sampled media or personnel using the equipment. Decontamination of equipment shall occur either at a central decontamination station or at portable decontamination stations set up at the sampling location, drill site, or monitoring well location. The centrally located decontamination station shall include an appropriately sized bermed and lined area on which equipment decontamination shall occur and shall be equipped with a collection system and storage vessels. In certain circumstances, berming is not required when small quantities of water are being generated and for some short duration field activities (i.e., pre-remedial sampling). Equipment shall be transported to and from the decontamination station in a manner to prevent cross contamination of equipment and/or area. For example, precautions taken may include enclosing augers in plastic wrap while being transported on a flatbed truck.

The decontamination area shall be constructed so that contaminated water is either collected directly into appropriate containers (5-gallon buckets or steel wash tubs) or within the berms of the decontamination area that then drains into a collection system. Water from the collection system shall be transferred into 55-gallon drums or portable tanks for temporary storage. Typically, decontamination water shall be staged until sampling results or waste characterization results are obtained and evaluated and the proper disposition of the waste is determined (SOP 2-2, *Guide to Handling Investigation-Derived Waste*). The exact procedure for decontamination waste disposal shall be discussed in the work plan. Also, solvent and acid rinse fluids may need to be segregated from other investigation-derived wastes.

All items that shall come into contact with potentially contaminated media shall be decontaminated before use and between sampling and/or drilling locations. If decontaminated items are not immediately used, they shall be covered either with clean plastic or aluminum foil depending on the size of the item. All decontamination procedures for the equipment being used are as follows:

General Guidelines

- Potable, analyte-free water shall be free of all contaminants of concern. Following the field QA sampling procedure described in the sampling plan, analytical data from the water source may be required.
- If sampling for PFAS compounds, PFAS-free water will need to be obtained from your project selected laboratory. (Note: PFAS free water, if needed, must be obtained from your selected project laboratory and the analyte list and detection limits of PFAS will be site-specific and dependent on latest regulatory updates from applicable state and federal regulators.)
- PFAS containing materials should be avoided. (See PFAS Materials Table at the end of this document)

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- Sampling equipment that has come into contact with oil and grease shall be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or client requirements regarding solvent use shall be stated in the sampling plan.
- All solvents and acids shall be pesticide grade or better and traceable to a source. If provided, certificates of analyses should be placed in the project files. The corresponding lot numbers shall be recorded in the appropriate logbook.

Note: Solvents and acids are potentially hazardous materials and must be handled, stored, and transported accordingly. Solvents shall never be used in a closed building. See the site-specific health and safety plan and/or the chemical's MSDS for specific information regarding the safe use of the chemical.

- Decontaminated equipment shall be allowed to air dry before being used.
- Documentation of all equipment type, date, time, and method of decontamination along with associated field QA sampling shall be recorded in the appropriate logbook.
- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment shall be used as specified in the site-specific health and safety plan.

5.1 Heavy Equipment Decontamination

Heavy equipment includes drilling rigs, well development rigs, and backhoes. Follow these steps when decontaminating this equipment:

1. Establish a bermed decontamination area that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be used; otherwise, use one or more layers of heavy plastic sheeting to cover the ground surface and berms. All decontamination pads shall be upwind of the area under investigation.

2. With the rig in place, spray areas (rear of rig or backhoe) exposed to contaminated media using a hot water high-pressure sprayer. Be sure to spray down all surfaces, including the undercarriage.

3. Use brushes, soap, and potable water to remove dirt whenever necessary.

4. Remove equipment from the decontamination pad and allow it to air dry before returning it to the work site.

5. After decontamination activities are completed, collect all contaminated wastewater, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the field plan. Liquids and solids must be drummed separately.

5.2 Downhole Equipment Decontamination

Downhole equipment includes hollow-stem augers, drill pipes, rods, stems, etc. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area shall be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.

2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air-drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination pads shall be upwind of any areas under investigation.

3. Place the object to be cleaned on aluminum foil or plastic-covered wooden sawhorses or other supports. The objects to be cleaned shall be at least 2 feet above the ground to avoid splashback when decontaminating.

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4. Using soap and potable water in the hot water high-pressure sprayer (or steam unit), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.

5. If using soapy water, rinse the equipment using clean, potable water. If using hot water, the rinse step is not necessary if the hot water does not contain a detergent. If the hot water contains a detergent, this final clean water rinse is required.

6. Using a suitable sprayer, rinse the equipment thoroughly with analyte-free water.

7. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.

8. After decontamination activities are completed, collect all contaminated wastewaters, plastic sheeting, and disposable gloves, boots, and clothing in separate containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal. Liquids and solids must be drummed separately.

5.3 Sampling Equipment Decontamination

Follow these steps when decontaminating sampling equipment:

1. Set up a decontamination line on plastic sheeting. The decontamination line shall progress from "dirty" to "clean." A clean area shall be established upwind of the decontamination wash/rinse activities to dry the equipment. At a minimum, clean plastic sheeting must be used to cover the ground, table, or other surfaces that the decontaminated equipment is placed for drying.

2. Disassemble any items that may trap contaminants internally. Do not reassemble the items until decontamination and air drying are complete.

3. Wash the items with potable water and soap using a stiff brush as necessary to remove particulate matter and surface films. The items may be steam cleaned using soap and hot water as an alternative to brushing. Note: Polyvinyl chloride or plastic items shall not be steam cleaned. Items that have come into contact with concentrated and/or oily contaminants may need to be rinsed with a solvent such as hexane and allowed to air dry prior to this washing step.

4. Thoroughly rinse the items with potable water.

5. The specific chemicals and/or fluids to be used in the decontamination process will be defined in the sampling plan. If sampling for metals, typically the potable water and soap wash is followed by a potable water rinse then an analyte-free water rinse; alternatively, an acid solution (e.g., 10 percent nitric acid) rinse followed by a rinse using analyte-free water may be specified in some instances. If sampling for organic compounds, thoroughly rinse the items with solvent (e.g., isopropanol) followed by a rinse using organic-free/analyte-free water. Again, the specific chemicals used in any acid rinse or solvent rinse phases shall be specified in the sampling plan. Any acid rinsate and solvent rinsate must each be containerized separately. Acids and solvents are potentially hazardous materials and care must be exercised when using these chemicals to prevent adverse health affects (e.g., skin burns, irritation to the eyes and respiratory system). Appropriate personal protective equipment must be worn when using these chemicals. The use of acids and solvents for decontamination should be carefully considered. These chemicals (including spent rinsate) must be managed and stored appropriately. Special measures such as proper labels, paperwork, notification, etc. may be required when transporting or shipping these chemicals.

6. Allow the items to air dry completely.

7. After drying, reassemble the parts as necessary and wrap the items in clean plastic wrap or in aluminum foil.

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8. After decontamination activities are completed, collect all contaminated waters, used solvents and acids, plastic sheeting, and disposable personal protective equipment. Place the contaminated items in properly labeled drums for disposal. Liquids and solids must be drummed separately. Refer to site-specific plans for labeling and waste management requirements.

5.4 Pump Decontamination

Follow the manufacturer's recommendation for specified pump decontamination procedures. At a minimum, follow these steps when decontaminating pumps:

1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other surfaces. Set up four containers: the first container shall contain dilute (nonfoaming) soapy water, the second container shall contain potable water, the third container shall be empty to receive wastewater, and the fourth container shall contain analyte-free water.

2. The pump shall be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first container. Place the discharge outlet in the wastewater container above the level of the wastewater. Pump soapy water through the pump assembly until it discharges to the waste container. Scrub the outside of the pump and other wetted parts with a metal brush.

3. Move the pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.

4. Move the pump intake to the analyte-free water container. Pump the water through the pump assembly. Pump the volume of water through the pump specified in the field plan. Usually, three pump-and-line-assembly volumes shall be required.

5. Decontaminate the discharge outlet by hand, following the steps outlined in Section 5.3.

6. Remove the decontaminated pump assembly to the clean area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices shall be covered with aluminum foil to prevent the entry of airborne contaminants and particles.

5.5 Low Stress (Low Flow) Sampling Pump Decontamination

Sampling equipment used for Low Stress (Low Flow) Groundwater Sampling (SOP 1-12) must be decontaminated thoroughly each day before use (daily decontamination) and after each well is sampled (between-well decontamination). All non-disposable equipment, including the pump (support cable and electrical wires which are in contact with the sample) will be decontaminated as described below. Dedicated, in-place pumps and tubing must be thoroughly decontaminated using "daily decontamination" procedures prior to their initial use or installation.

5.5.1 Prior to Sampling Event Decontamination

Please Note: Steps 5 through 13 should only be performed once (for each pump that is to be used) before the commencement of a particular sampling event by a person qualified to disassemble pumps.

1. Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and thoroughly flush other equipment with potable water.

2. Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Liquinox™, for 5 minutes and thoroughly flush other equipment with fresh detergent solution. Use the detergent sparingly.

3. Rinse: Operate pump in a deep basin of potable water for 5 minutes and thoroughly flush other equipment with potable water for 5 minutes.

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4. Analyte-Free Rinse: Operate pump in a deep basin of analyte-free water to pump out 1 to 2 gallons of this final rinse water. (Note: PFAS free water, if needed, must be obtained from your selected project laboratory and the analyte list and detection limits of PFAS will be site-specific and dependent on latest regulatory updates from applicable state and federal regulators.)

5. Disassemble pump.

6. Wash pump parts (inlet screen, shaft suction interconnector, motor lead assembly, stator house): Place the disassembled parts of the pump into a deep basin containing 8 to 10 gallons of non-phosphate detergent solution. Scrub all pump parts with a test tube brush.

7. Rinse pump parts with potable water for five minutes.

8. Rinse the pump parts with demonstrated analyte-free water.

9. If sampling for metals, an acid rinse may be specified in the sampling plan; if so, place impeller assembly in a large glass beaker and rinse with 1% nitric acid (HNO3).

10. Rinse impeller assembly with potable water for five minutes.

11. If sampling for organics, a solvent rinse may be specified; if so, place impeller assembly in a large glass bleaker and rinse with isopropanol or appropriate organic solvent specified in the site-specific plan.

12. Thoroughly rinse impeller assembly with demonstrated analyte-free water.

13. Reassemble pump.

5.5.2 Daily and Between-Well Decontamination

1. Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and thoroughly flush other equipment with potable water for five minutes.

2. Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Liquinox™, for 5 minutes and thoroughly flush other equipment with fresh detergent solution. Use the detergent sparingly.
3. Rinse: Operate pump in a deep basin of potable water for 5 minutes and thoroughly flush other equipment with potable water for five minutes.
4. Final Rinse: Operate pump in a deep basin of analyte-free water to pump out 1 to 2 gallons of this final rinse water.

5.6 Instrument Probe Decontamination

Instrument probes used for field measurements such as pH meters, conductivity meters, etc. shall be decontaminated between samples and after use with analyte-free, or better, water.

5.7 Waste Disposal

Refer to site-specific plans and SOP 2-2 for waste disposal requirements. The following are guidelines for disposing of wastes:

- All wash water, rinse water, and decontamination solutions that have come in contact with contaminated equipment are to be handled, packaged, labeled, marked, stored, and disposed of as investigation-derived waste.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions shall be generated, each type of waste shall be contained in separate containers.
- Unless otherwise required, plastic sheeting and disposable protective clothing may be treated as solid, nonhazardous waste.

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- Waste liquids shall be sampled, analyzed for contaminants of concern in accordance with disposal regulations, and disposed of accordingly.
- PFAS disposal requirements are being initiated by many disposal facilities in anticipation of regulatory initiatives on the state and federal level.

5.7.1 PFAS Considerations for IDW disposal

Currently, PFAS characterization and disposal requirements are being initiated by many disposal facilities in anticipation of regulatory initiatives on the state and federal level. These evolving regulatory standards for PFAS must be considered in the implementation of many SOPs including SOP 2-2 and SOP 4-5. These same concerns must be considered in the development of general project planning documents such as: work plans, sampling and analysis plans, quality assurance project plans, data management plans, accident prevention plans, health and safety plans and laboratory statements of work used to guide the collection of samples for PFAS analysis.

- A consideration of the PFAS-specific sampling requirements is needed during the project planning phase because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. This means that the development of project data quality objectives (DQOs), and selection of action levels, reporting limits are all impacted with the inclusion of sampling for PFAS analysis.

- Before implementing the IDW (SOP 2.2) for collection of samples for PFAS waste characterization analysis for disposal purposes, it is important to review current disposal facility requirements and state and federal regulatory standards to determine whether site- or project-specific changes to this SOP should be included in project planning documents to align with the latest regulatory standards and sampling guidance. CDM Smith Regulatory Updates can be located at the INFOCENTER. Specific State and Federal Regulatory Limit updates can be found here: https://cdmsmith.libguides.com/PFAS_InfoCenter

6.0 Restrictions/Limitations

Nitric acid and polar solvent rinses are necessary only when sampling for metals or organics, respectively. These steps shall not be used, unless required, because of the potential for acid burns and ignitability hazards.

If the field equipment is not thoroughly rinsed and allowed to completely air dry before use, volatile organic residue, which interferes with the analysis, may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted. In the summer, volatilization is rapid, and in the winter, volatilization is slow. Check with your EPA region, state, and client for approved decontamination solvents.

7.0 References

American Society for Testing and Materials. 2015. *Standard Practice for Decontamination of Field Equipment at Waste Sites*, ASTM D5088-15. January 15.

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PFAS Sampling Checklist

Development of PFAS sampling guidance is necessary because cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the parts per trillion (ppt) range. The levels of awareness for PFAS cross contamination vary by the opportunities of introducing PFAS into the environment media under investigation.

1. Critical: Items in direct contact of environmental media under investigation. These can include, but not limited to, sample containers, sampling parts and equipment, drilling equipment, well construction items and materials, parts and equipment for hydrogeological testing, in-situ treatment parts and equipment.

2. Very Important: PPE, personal hygiene that are used by sampling personnel.

3. Important: Items used in coolers for shipping and transporting PFAS samples.

4. Less important/Awareness level concern: Activities in the staging area away from immediate PFAS investigation area.

Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Field Clothing or PPE				
Clothing or boots containing "water resistance" or "stain-treated" fabrics				
Cloths washed with fabric softeners				Fabric softeners may contain PFAS
New and unwashed clothing				fabric treatment may contain PFAS
Uncoated Tyvek				USEPA PFAS sampling guidance from Region 2 prohibits use of Tyvek
Coated Tyvek				
PVC or wax-coated fabrics				
Neoprene				
Synthetic and natural fibers (preferably cotton)				
Steel-toed boots made with polyurethane and PVC				If it is not possible to find PFAS free steel-toed footwear, PFAS-free over boots may be worn. The over boots must be put on and the hands washed after putting the over boots on prior to the beginning of the sampling activities. Over boots may only be removed in the staging area and after the sampling activities have been completed
Well laundered clothes				several times from time of purchase
Well washed cotton coveralls				washed several times

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Personnel Hygiene and Protective Skin Products				
Sunscreens				Good to use: Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss my face, and baby sunscreens that are "free" or "natural
Insect Repellents				Good to use: Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellant, Herbal Armor, California Baby Natural Bug Spray, BabyGanics
International Brands of sunscreens and insect repellents				Must be evaluated on a case-by-case basis
Field Sampling Items				
Water proof field paper or books				Use loose plain paper
Post-it notes				
Aluminum foil				
Brand-name markers				Sharpie may be used to label sample bottles in the staging area, but markers should not be used in the immediate sampling environment
Off-brand markers				
Ball point pens				
plastic clipboards				NJ DEP sampling guidance, use metal clipboard
Plastic table cover				
Sampling Equipment				
Item containing high-density polyethylene (HDPE)				
Item containing polypropylene				PP sample bottles must be used for drinking-water samples in accordance with USEPA method 537.1.1
Item containing polyurethane				
Item containing Polyvinyl chloride (PVC)				
Item containing silicon				

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Item made of stainless steel				
Alconox®				
Citronex®				
Liquinox®				
Powderless nitrile gloves				
HDPE Hydrasleeves or sonic core bags				
Neoprene				
Crisco® or other vegetable-based greases for lubricating parts				
Item containing PTFE				Items or equipment that contains PTFE parts that will be in direct contact with sampling media
Item containing Teflon®				Field sampling items or equipment that contains Teflon® and that will be in direct contact with the sampling media
Item containing fluoropolymer				
Low-density polyethylene (LDPE)				Items or equipment that contains LDPE parts and that will be in direct contact with the sampling media
Viton® O-rings				Viton® O-rings used in pressure washers used for sampling equipment decontamination
Glass sample containers				
Field filter				Field filtration should be avoided regardless of filter types
Decon 90				
Items containing fluorosurfactants				
Teflon-bearing plumber's tape				
Blue (or chemical) ice				Later data (unpublished) suggest no cross contamination from blue ice. The category may be changed after data are published
Water ice				Double bag in polyethylene bags
Internal valves and equipment parts for sampling or decon				

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Methanol or other solvents				
LDPE plastic bags (e.g., Ziploc® bags)				For larger biota sampling, Ziploc bags may be used, but collecting an equipment blank is recommended because these bags may be made of LDPE
Drilling fluids				
LDPE sonic core sample bags				Manufactured by Boart Longyear and Hole Products
Equipment with moving parts that may be lubricated with PFAS containing lubricants or greases				
Rental equipment				Must be verified to have no PFAS-bearing parts prior to use
Others				
Food wrappers				Field personnel must wash hands after having food wrapped with grease repelling paper

(1) If an item which may contain PFAS but alternative is not available, the item should be tested for PFAS before use

(2) This mostly refers to the immediate sampling environment, particularly, the item is in contact with environmental media to be sampled

(3) There are no standard operation procedures on how an item can be verified, please contact PFAS experts for advices on the best practice of testing a potential PFAS containing item.

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Control of Measurement and Test Equipment	SOP 5-1 Revision: 11 Date: August 2020

Approved: Ernest Ashley **Technical Review:** Jeniffer Oxford

1.0 Objective

The objective of this technical standard operating procedure (SOP) is to establish the baseline requirements, procedures, and responsibilities inherent to the control and use of all measurement and test equipment (M&TE). Contractual obligations may require more specific or stringent requirements be implemented.

2.0 Background

2.1 Definitions

Note: Definitions are often promulgated or codified in state or local statutes, regulations, or ordinances and can vary between regulatory agencies. Definitions should be verified against the definitions provided by agencies regulating the work when applicable.

Traceability - The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification

2.2 Associated Procedures

- SOP 4-1, *Field Logbook Content and Control*
- CDM Smith Quality Requirements – As Required Activity, *Procuring M&TE* https://cdmsmithonline.sharepoint.com/sites/PM/Projects/PDS/Pages/PDS.aspx#
- Manufacturer's operating and maintenance and calibration procedures

2.3 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that accountable and high-integrity measurements result from using this equipment. To facilitate that, the equipment shall be used with full understanding of and in compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures, and in accordance with any other related project-specific requirements.

3.0 Responsibilities

All staff responsible for the direct control and/or use of M&TE need to know, understand, and implement the following requirements, as well as any other related project-specific requirements.

Project Manager - The project manager (PM) or designee (equipment coordinator, quality assurance specialist, field team leader, etc.) is responsible for initiating and tracking the following requirements.

Field Team Leader (FTL) - Read/review field plans and coordinate assigned tasks with the field team leader before conducting the field work

The M&TE requisitioner is responsible for:

- Determining project-specific technical and quality requirements for M&TE
- Determining if acceptance testing will be performed
- Determining the accuracy, precision, range, and units required for M&TE
- Determining if the item requires calibration. ***Note***: If calibration is required, then one must also document a determination whether calibration traceability to national standards is required.
- Determining the responsible party for calibrating the M&TE, such as the supplier or CDM Smith
- Determining if M&TE may contain material with potential for per- and polyfluoroalkyl substances (PFAS) contamination
- Submitting the purchase requisition via iProcurement

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- Determining if purchased items are acceptable
- Arranging and documenting acceptance testing, if required

Personnel identifying nonconforming items affecting quality are responsible for segregating the items and removing them from use. Additional project-specific responsibilities may include labeling or tagging nonconforming items, documenting the nonconformance, and communicating the deficiency to the PM, field team leader, or Project Technical Leader (PTL).

***Note*:** Responsibilities may vary from site to site. Therefore, all field team member responsibilities shall be defined in the field plan or site-/project-specific quality assurance plan.

4.0 Requirements for M&TE

- Review and implement M&TE-related project-specific requirements
- Obtain maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment log or a field log as appropriate (Figure 1)
- Maintain M&TE records
- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using appropriate procedures and calibration standards
- Identify and act on nonconforming M&TE

5.0 Procedures

5.1 Determine if Other Project-Specific Requirements Apply (All M&TE)

The PM or designee shall determine if M&TE related project-specific requirements apply. If M&TE related project-specific requirements apply, obtain a copy of them and review and implement as appropriate. Refer to Figure 2 for a list of per- and polyfluoroalkyl substances (PFAS)-related considerations.

5.2 Obtain Operating and Maintenance and Calibration Documents

A. GF M&TE to be procured:
Requisitioner - Specify that the maintenance and calibration procedures are included with equipment

B. GF M&TE acquired from a property transfer:
Receiver - Inspect the M&TE to determine if maintenance and calibration procedures are included with the item. If missing or incomplete, order the appropriate documentation from the manufacturer.

C. M&TE rented or leased from an outside vendor:
Requisitioner - Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, ask the procurement division to request it from the vendor.

D. All M&TE
Receiver - Inspect the M&TE to determine that there are no obvious defects, and the required tolerances for accuracy, precision, range and units meet those requested

Requisitioner - Specify that the M&TE material list be included with equipment

Receiver - When sampling for PFAS analysis, inspect M&TE material list prior to use to verify that no PFAS-bearing parts are included

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5.3 Prepare and Record Maintenance and Calibration Records

All M&TE:

PM or Designee - Record all maintenance and calibration events in a field log unless other project-specific requirements apply.

GF M&TE only (does not apply to rented or leased M&TE):

If an equipment log is a project-specific requirement, perform the following:

Receiver - Notify the PM or designee for the overall property control of the equipment upon receipt of an item of M&TE.

PM or Designee and User:

- Prepare a sequentially page numbered equipment log for the item using the maintenance and calibration form (or equivalent) (Figure 1)
- Record all maintenance and calibration events in an equipment log
- Refer to Figure 2, PFAS Sampling Materials Checklist, if applicable

5.4 Label M&TE Requiring Calibration (GF M&TE only - does not apply to rented or leased M&TE)

If calibration labeling is a project-specific requirement, perform the following:

FTL or Designee and User:

- Read the maintenance and calibration procedures to determine the calibration frequency required.
- If an M&TE item requires calibration before use, affix a label to the item stating, "Calibrate Before Use."
- If an M&TE item requires calibration at other scheduled intervals, e.g., monthly, annually, etc., affix a label listing the date of the last calibration, the date the item is next due for a calibration, the initials of the person who performed the calibration, and a space for the initials of the person who shall perform the next calibration.

5.5 Operating, Maintaining or Calibrating an M&TE Item (All M&TE)

FTL or Designee and User - Operate, maintain, and calibrate M&TE in accordance with the manufacturer's maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log. Handle and store M&TE using good judgement and the manufacturer's or vendor's recommendation.

5.6 Shipment

A. GF M&TE:

Supplier - Inspect the item to ensure that maintenance and calibration procedures are attached or included with the shipping case, and that a copy of the most recent equipment log entry page (if required) is included with the shipment. If the maintenance and calibration procedures and/or the current equipment log page (if required) is missing or incomplete, do not ship the item.

B. M&TE that is rented or leased from an outside vendor:

Supplier - Inspect the item to ensure that the maintenance and calibration procedures and latest calibration and standards certification records are included prior to shipment. If any documentation is missing or incomplete, do not ship the item. Immediately contact the procurement division and request that they obtain the documentation from the vendor.

Inspect the M&TE to verify that no PFAS containing materials are used for the item.

Some M&TE equipment must remain upright to maintain calibration. Such M&TE should be shipped only in containers labeled appropriately with "This End Up" labels.

Receiver - Some M&TE equipment must remain upright to maintain calibration. Upon receipt, inspect the container to verify that it is upright per "This End Up" labels. If it is not upright, notify the PM and vendor. Immediately contact the vendor and request a replacement for any missing items.

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When M&TE is to be used to support sample collection for PFAS analysis, inspect the M&TE to verify that no PFAS containing materials are used for the item. Immediately contact the vendor and request a replacement item.

5.7 Records Maintenance

GF M&TE:

FTL or Designee - Create a file on the initial receipt of an M&TE item or calibration standard. Organize the files by contract origin and by M&TE item and calibration standard. Store all files in a cabinet, file drawer, or other appropriate storage media at the pertinent warehouse/onsite field trailer or office location. Scan copies and store on ProjectWise.

Receiver - Forward the original packing slip to the procurement division and a photocopy to the FTL or designee for storage on ProjectWise.

FTL or Designee and User:

- Maintain all original documents in the equipment file except for the packing slip and field log.
- File the photocopy of the packing slip in the M&TE file.
- Record all maintenance and calibration in an equipment log or field log (as appropriate). File the completed equipment logs in the M&TE records. Forward completed field logs to the PM for inclusion in the project files on ProjectWise.

M&TE rented or leased from an outside vendor:

Receiver - Forward the packing slip to the procurement division.

User:

- Forward the completed field log to the PM for inclusion in the project files on ProjectWise.
- Retain the most current maintenance and calibration record and calibration standards certifications with the M&TE item and forward previous versions to the PM for inclusion in the project files on ProjectWise.

5.8 Traceability of Calibration Standards (All M&TE)

FTL or Designee and User:

- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Safety Data Sheets be provided with standards.
- Monitor standards that are perishable and consume or dispose of them on or before the expiration date.

5.9 M&TE That Fails Calibration (Any M&TE item that cannot be calibrated or adjusted to perform accurately)

FTL or Designee

- Immediately discontinue use and tag and segregate the item from other equipment. Notify the appropriate PM and take action to replace the nonconforming items.
- Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected. Share the review results with the appropriate PM(s).

6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this SOP may be granted by Headquarters health and safety manager and/or Headquarters quality assurance manager. All exemptions shall be documented and included in the equipment records.

7.0 Regulatory Considerations

Consider the evolving regulatory standards for per- and polyfluoroalkyl substances (PFAS) when implementing this SOP. Also consider them and during the development of project-specific sampling and analysis plans, and quality assurance project plans used to collect PFAS analysis samples.

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Before implementing this SOP for control of M&TE related to the collection of samples for PFAS analysis, it is important to review current regulatory standards to determine whether any site- or project-specific changes related to control of M&TE should be included in project planning documents. CDM Smith Regulatory, as well as specific state and federal regulatory limits, can be found on the PFAS InfoCenter: https://cdmsmith.libguides.com/PFAS_InfoCenter

8.0 Other Procedural Cautions

Cross-contamination is a prominent concern for data quality, particularly with PFAS action levels in the low parts per trillion (ppt) range. Thus, special care is required to select appropriate M&TE when sampling for PFAS analysis. Sites with PFAS sampling need to include the following considerations and awareness.

- Wear powderless nitrile gloves and change them frequently.
- Avoid polytetrafluoroethylene (PTFE), low-density polyethylene (LDPE), sticky notes, waterproof field book, and aluminum foil
- Avoid using markers, if possible, during recording of M&TE results. Use only regular ink pens. If needed, write over regular ink with marker after the sample containers are sealed.
- Consult the materials checklists for equipment concerns.
- Evaluate the parts and equipment used for hydrogeological testing, and in-situ testing against the list in Figure 2.

Refer to Figure 2, *PFAS Sampling Materials Checklist,* for acceptable materials and those that will need verification prior to sampling for PFAS.

9.0 References

American Society for Quality/ American National Standard Institute. 2014. *Quality Management Systems for Environmental Information and Technology Programs – Requirements with Guidance for Use*. ASQ/ANSI E4:2014.

CDM Smith. 2018. Firmwide *Quality Manual.* Revision 5. February.

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Figure 1

CDM Smith A subsidiary of Camp Dresser & McKee Inc.

Maintenance and Calibration

Date: Time: (a.m./p.m.)

Employee Name: Equipment Description:

Contract/Project: Equipment ID No.:

Activity: _____ Equipment Serial No.: _____

Maintenance

Maintenance Performed:

Comments:

Signature: _____ Date: _____

Calibration/Field Check

Calibration Standard: Concentration of Standard:

Lot No. of Calibration Standard: Expiration Date of Calibration Standard:

Pre-Calibration Reading: Post-Calibration Reading:

Additional Readings: Additional Readings:

Additional Readings: Additional Readings:

Pre-Field Check Reading: Post-Field Check Reading:

Adjustment(s):

Calibration: ☐ Passed ☐ Failed

Comments:

Signature: _____ Date: _____

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Figure 2
PFAS Materials Checklist

Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Sampling Equipment				
Item containing high-density polyethylene (HDPE)				
Item containing polypropylene				
Item containing polyurethane				
Item containing Polyvinyl chloride (PVC)				Polypropylene sample bottles must be used for drinking-water samples in accordance with EPA method 537.1.1
Item containing silicon				
Item made of stainless steel				
Alconox®				
Citronex®				
Liquinox®				
Powderless nitrile gloves				
HDPE Hydrasleeves or sonic core bags				
Neoprene				
Crisco® or other vegetable-based greases for lubricating parts				
Item containing PTFE				Items or equipment that contains PTFE parts that will be in direct contact with sampling media
Item containing Teflon®				Field sampling items or equipment that contains Teflon® and that will be in direct contact with the sampling media
Item containing fluoropolymer				
Low-density polyethylene (LDPE)				Items or equipment that contains LDPE parts and that will be in direct contact with the sampling media
Viton® O-rings				Viton® O-rings used in pressure washers used for sampling equipment decontamination
Glass sample containers				
Field filter				Field filtration should be avoided regardless of filter types
Decon 90				
Items containing fluorosurfactants				
Teflon-bearing plumber's tape				
Blue (or chemical) ice				Later data (unpublished) suggest no cross contamination from blue ice. The category may be changed after data are published.

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Item	Good to Use	Need Verification (2)(3)	Should Avoid (1)(2)	Comments
Water ice				Double bag in polyethylene bags
Item	**Good to Use**	**Need Verification (2)(3)**	**Should Avoid (1)(2)**	**Comments**
Internal valves and equipment parts for sampling or decontamination				
Methanol or other solvents				
LDPE plastic bags (e.g., Ziploc® bags)				For larger biota sampling, Ziploc bags may be used, but collecting an equipment blank is recommended because these bags may be made of LDPE
Blue (or chemical) ice				
Drilling fluids				
LDPE sonic core sample bags				Manufactured by Boart Longyear and Hole Products
Equipment with moving parts that may be lubricated with PFAS containing lubricants or greases				
Rental equipment				Must be verified to have no PFAS-bearing parts prior to use

(1) If an item may contain PFAS but an alternative is not available, the item should be tested for PFAS before use.
(2) This mostly refers to the immediate sampling environment, particularly, the item is in contact with environmental media to be sampled.
(3) There are no standard operation procedures on how an item can be verified, please contact PFAS experts for advice on best practices for testing a potential PFAS containing item.

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Appendix B

Analytical Services Branch Laboratory Information

QAPP Worksheets

QAPP Worksheet #12a: Measurement Performance Criteria for ASB Laboratory

Matrix	Water				
Analytical Group	ASB Inorganics				
Concentration Level	(Low/medium) ug/L				
Sampling Procedure	**Analytical Method/SOP**	**Data Quality Indicators (DQIs)**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S & A)**
See Worksheets #18 and #21	SOP Metals003A ICP Metals – Thermo (EPA 200.7/SW-848: 6010D) Version 8 QAPP Worksheet #23 lists all applicable SOPs.	Precision	≤30% RPD	Field Duplicate	S & A
		Accuracy/bias	No Analyte > RL; No analyte > MDL	Equipment Blank; Laboratory Control Blank,	S & A A
		Precision	RPD ≤ 20%	Laboratory Duplicate	S & A
		Accuracy/bias	±20% of true value; %R within 85-115% %R within 75-125%	Interference Check sample; BS/LCS; MS	A
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare to historical data calculate RPD at sampling point from previous event,	S
		Sensitivity	See QAPP Worksheet #15b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assess valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Note

% R – Percent Recovery
≤ – Less than or equal to
MDL – Method Detection Limit
ICP – Inductively Coupled Plasma
BS/LCS – Blank Spike/ Laboratory Control Sample
RL – Reporting Limit
RPD – Relative Percent Difference
ASB – Analytical Services Branch
MS – Matrix Spike
SOP – Standard Operating Procedure

QAPP Worksheet #12b: Measurement Performance Criteria Table for ASB Laboratory

Matrix	Water				
Analytical Group	ASB VOCs				
Concentration Level	Low /medium (µg/l)				
Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
See Worksheet #18 and #21	ASB SOP MS023 VOA in Water (SW-846 :8260C, Modified) Version 7 QAPP Worksheet #23 lists the applicable SOPs	Precision/Bias	≤30% RPD	Field Duplicate	S & A
		Accuracy	No analyte > RL	Method Blank Trip Blank	A S & A
		Precision/Bias	Per SOP Attachment 6. QC Criteria Summary	MS/MSD	S & A
		Accuracy	Per SOP Attachment 6. QC Criteria Summary	MS/MSD Surrogate Spike	A
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare data to historical data, calculate RPD at sampling point from previous event	S
		Sensitivity	See QAPP Worksheet #15a and b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assesses valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Notes:

% – Percent MS/MSD – Matrix Spike/ Matrix Spike Duplicate ≤ – Less than or equal to VOA – Volatile Organic Analysis

RL – Reporting Limit

MDL – Method Detection Limit
ASB – Analytical Services Branch

RPD – Relative Percent difference
RL – Reporting Limit

SOP – Standard Operating Procedure
QC – Quality Control

QAPP Worksheet #12c: Measurement Performance Criteria Table

Matrix	Water				
Analytical Group	1,4-Dioxane				
Concentration Level	Low (µg/l)				
Sampling Procedure	Analytical Method/SOP	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
See Worksheet #18 and #21	Region 5 ASB Laboratory Method SOP MS035, Version 2 QAPP Worksheet #23 lists the applicable SOPs	Precision	≤30% RPD	Field Duplicate	S & A
		Accuracy/Bias	No analyte > RL	Method Blank Trip Blank	A S & A
		Precision	RPD and R per Section 10; Table 2 and Table 4 SOP MS035	MS/MSD; LCS	S & A A
		Accuracy/Bias	Per Section 10, Table 2 and Table 4 SOP MS035	Surrogate; MS/MSD	A
		Representativeness Comparability	Variability at a sampling point ≤ 30% RPD	Adherence to sampling SOP for collection procedures and quantity of samples to collect Compare data to historical data, calculate RPD at sampling point from previous event	S
		Sensitivity	See QAPP Worksheet #15a and b	MDL study	A
		Completeness	90% of samples collected and valid analytical data received versus planned	Project manager assesses valid data collected versus planned; laboratory project manager assesses completeness of analytical requirements per the QAPP	S & A

Notes: For samples sent to the Analytical Services Branch laboratory see SOP in Appendix B.

% – Percent
MS/MSD – Matrix Spike/ Matrix Spike Duplicate
≤ – Less than or equal to
R - % Recovery
RL – Reporting Limit
MDL – Method Detection Limit
RPD – Relative Percent difference
SOP -standard operating procedure

QAPP Worksheet #28a: Analytical Quality Control and Corrective Action for ASB Laboratory

Analytical Group:	VOC	**Concentration Level:**	Low
Analytical Method/ SOP Reference:	SOP MS023 VOA in Water (SW-846 :8260C) Version 7[1]	**Sampler's Name:**	TBD
Matrix:	Water (influent, effluent and groundwater)	**Field Sampling Organization:**	CDM Smith
Sampling SOP:	See Worksheet #18, #21, and Appendix A	**Analytical Organization:**	USEPA Regions 5 ASB Laboratory[1]
		No. of Sample Locations:	26 wells sampled quarterly 1 effluent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples [2]	NA	Notify Site PM, flag duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Method blank	1 per batch analyzed every 12 hours	≤ RL	Find source/rectify	Laboratory analyst/Data validator	Laboratory contamination	≤ RL
Surrogates	Added to every sample, QC sample and standard	SOP MS023 Attachment 6. QC Criteria Summary	Check calculation /instruments -reanalyze samples. Failure allowances as per SOP.	Laboratory analyst/Data validator	Laboratory accuracy/bias	% recovery varies see SOP MS023 Attachment 6. QC Criteria Summary
MS/MSD	1 per 20 samples	≤ 60% RPD 60-110% Recovery	Flag outliers	Laboratory analyst/Data validator	Precision Matrix interference/ laboratory accuracy	≤ 60% RPD 60-110% Recovery
Internal standards	Added to every sample and standard	50-200% Recovery see SOP MS023 Attachment 6. QC Criteria Summary	Check calculation / instruments -reanalyze samples	Laboratory analyst/Data validator	Accuracy/bias	50-200% Recovery see SOP MS023 Attachment 6. QC Criteria Summary

Notes:

[1]See ASB SOP MS023 Version 7 for analytical quality control and corrective actions

[2] One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed, and field duplicates mal be collected for subsequent sampling events until sample and duplicate agreement are within the defined RPD.

DQI – data quality indicator
MS/MSD – matrix spike/matrix spike duplicate
SOP – standard operation procedure
VOC – volatile organic compound
RL – Reporting Limit
PM – Project manager
QC – quality control
RPD – relative percent difference
ASB – Analytical Services Branch
TBD – To be determined
VOA – Volatile organic analysis

QAPP Worksheet #28b: Analytical Quality Control and Corrective Action for ASB LAboratory

Analytical Group:	ASB Metals	**Concentration Level:**	Low/medium
Analytical Method/ SOP Reference:	SOP Metals003A ICP Metals – Thermo (EPA 200.7/SW-846: 6010D) [1]	**Sampler's Name:**	TBD
Matrix:	Water (influent, effluent and groundwater)	**Field Sampling Organization:**	CDM Smith
Sampling SOP:	See Worksheet #18, #21, and Appendix A	**Analytical Organization:**	ASB Laboratory[1]
		No. of Sample Locations:	14 wells sampled quarterly 1 effluent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action [1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples[2]	NA	Notify Site PM, flag duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Laboratory control blank	1 per 20 samples or less	≤ MDL	Suspend analysis/rectify problem; redigest and reanalyze affected samples	Laboratory analyst /Data validator	Laboratory contamination	≤ MRL
ICV	Start of each run	±1 times MRL	Check calculations and instruments; reanalyze affected samples		Laboratory accuracy/bias	See Method/ SOP QC Acceptance Limits Section 10.3.12
BS/LCS	1 per 20 samples or less	85-115 %R	Suspend analysis/rectify problem; reanalyze affected samples		Laboratory accuracy	
MS	1 per 10 samples or less	75-125% R	Flag outliers		Matrix interference	
Laboratory duplicate	1 per 10 samples or less	RPD ≤ 20%	Flag outliers		Laboratory precision	

Notes:

[1]See ASB SOP Metals003A ICP Metals Version 8 for analytical quality control and corrective actions

[2] One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed, and field duplicates mal be collected for subsequent sampling events until sample and duplicate agreement are within the defined RPD.

DQI – data quality indicator
QC – quality control
ICV – initial calibration verification
%R – Percent recovery
BS/LCS – Blank spike/laboratory control sample
MS – matrix spike
SOP – standard operation procedure
RPD – relative percent difference
PM – Project Manager

QAPP Worksheet #28b: Analytical Quality Control and Corrective Action

Analytical Group:	1,4-Dioxane	**Concentration Level:**	Low
Analytical Method/SOP Reference:	SOP MS035[1]	**Sampler's Name:**	TBD
Matrix:	Water (influent, effluent and groundwater)	**Field Sampling Organization:**	CDM Smith
Sampling SOP:	See Worksheet #18, #21, and Appendix A	**Analytical Organization:**	ASB Laboratory[1]
		No. of Sample Locations:	40 wells sampled quarterly 1 effluent and 1 influent point sampled monthly/quarterly

QC Sample[1]	Frequency / Number[1]	Method/SOP QC Acceptance Limits[1]	Corrective Action[1]	Person(s) Responsible for Corrective Action[1]	DQI	Measurement Performance Criteria[1]
Field duplicate	1 per 10 samples [2]	NA	Notify Site PM, flag duplicate results	CDM Smith PM	Precision	RPD ≤ 30%
Method blank	1 per batch of 20 or fewer samples extracted/analyzed	Sample concentration must be >5x blank concentration	Find source/rectify	Laboratory analyst/Data validator	Laboratory contamination	Sample concentration must be >5x blank concentration
LCS	1 per batch of 20 or fewer samples extracted/analyzed	% recovery 70-130%	Re-prepare and re-analyze samples; Qualify data for out of control analyte	Laboratory analyst/Data validator	Laboratory accuracy/bias	Within initial recovery limits, See SOP Table 4
Surrogate	Added to every field and QC sample	% recovery 70-130%	Check calculation / instruments - reanalyze samples. Failure allowances as per SOP.	Laboratory analyst/Data validator	Laboratory accuracy/bias	Within initial recovery limits, See SOP, Table 2 and Table 4
MS/MSD	1 per batch of 20 or fewer samples extracted/analyzed	% recovery 70-130% RPD <30%	Qualify outliers	Laboratory analyst/Data validator	Precision Matrix interference/ laboratory accuracy	See SOP, Table 2 and Table 4
Internal standards	Added to every field sample, QC sample and CCV	50-200% of CCV	Check calculation / instruments - reanalyze samples	Laboratory analyst/Data validator	Accuracy/bias	See SOP Table 4

Notes:

[1]See ASB SOP MS035, Version 2 for analytical quality control and corrective actions

[2]One field duplicate will be collected for the first effluent sampling event each year and evaluated for acceptable agreement. If agreement is outside the relative percent difference (RPD defined in the QAPP, field procedures will be reviewed, and field duplicates may be collected for subsequent sampling events until sample and duplicate agreement are within the defined RPD.

DQI – data quality indicator

QC – quality control

MS/MSD – matrix spike/matrix spike duplicate

RPD – relative percent difference

SOP – standard operation procedure

ASB – Analytical Services Branch

RL – reporting limit

Analytical Methods Standard Operational Procedures

Standard Operating Procedure for the
Measurement of Volatile Organic Analytes in
Water by Purge & Trap Gas Chromatography/Mass Spectrometry

ASB SOP MS023: VOA in Water
(Based on EPA Method SW-846:8260C, Modified)

United States Environmental Protection Agency
Region 5 Analytical Services Branch
536 S. Clark Street, 10th Floor (LAB-10C)
Chicago, Illinois 60605

Section #	Section Title	Starting Page #
1.	**SCOPE AND APPLICATION**	3
2.	**SUMMARY OF METHOD**	3
3.	**ABBREVIATIONS AND DEFINITIONS**	4
4.	**HEALTH, SAFETY AND WASTE HANDLING**	7
5.	**CAUTIONS AND INTERFERENCES**	8
6.	**EQUIPMENT AND SUPPLIES**	9
6.1	Purge-and-trap system	9
6.2	GC/MS system	9
6.3	Glassware	10
6.4	Other equipment and supplies	10
6.5	Certified supporting equipment	10
7.	**REAGENTS AND STANDARDS**	10
7.1	Reagents	11
7.2	Standards	11
7.3	Preparation and handling of stocks and standards	12
7.4	Spike preparation for calibration-level and batch-level quality controls	14
7.5	Calibration preparation	17
8.	**SAMPLE HANDLING AND PRESERVATION**	18
9.	**SAMPLE PREPARATION AND ANALYSIS**	19
9.1	Sample preparation	19
9.2	Purge-and-trap	20
9.3	Gas chromatography/mass spectrometry	22
9.4	System calibration and sample data collection	24
9.5	Qualitative and quantitative analysis	27
10.	**QUALITY CONTROL**	29
10.1	Demonstration of capability	29
10.2	Procedural quality controls	29
11.	**DATA AND RECORDS MANAGEMENT**	32
12.	**TROUBLESHOOTING**	35
13.	**PREVENTATIVE MAINTENANCE**	37
14.	**REFERENCES**	39
15.	**REVISION HISTORY**	40
	APPENDICES	43
	Table 1. Method Analyte CAS Numbers, MS Ions, and GC Retention Times	43
	Table 2. MDLs, RLs, and Estimated Measurement Uncertainties	46
	Table 3. BFB Abundance Criteria	49
	Attachment 1. Sample Calculations and Equations	50
	Attachment 2. Deviations from SW-846 Method 8260C	54
	Attachment 3. Field Sample Screening Procedure	55
	Attachment 4. Preparation Procedure of Pre-Acidified Vials for Field Sampling	56
	Attachment 5. VOA Data Package Components	57
	Attachment 6. QC Criteria Summary	59

1. SCOPE AND APPLICATION

1.1. Purpose and scope

This procedure applies to the analysis of water samples for purgeable volatile organic compounds by the US EPA Region 5 Analytical Services Branch Laboratory. This is the ASB implementation of EPA method 8260C: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). There are minor modifications from EPA method 8260C in this SOP, which are summarized in Attachment 2 of the Appendix.

1.2. Target compounds

The compounds measured by this method, as well as chromatographic and mass spectral parameters used for their qualitative and quantitative determination, are listed in Table 1 of the Appendix. Five of the listed compounds - methyl tert-butyl ether, 1,4-dioxane, acrolein, acrylonitrile, and 2-chloroethylvinyl ether – are not routinely analyzed, but may be determined by this method upon request.

1.3. Sample matrices

This SOP applies to all water samples except those subject to regulation under the Clean Water Act (like NPDES permit enforcement and compliance monitoring), which is covered in ASB SOP MS024, and those regulated under the Safe Drinking Water Act, which are not analyzed for VOA at ASB.

1.4. Calibration range

The calibration range described in this SOP is 2 – 100 ppb for most analytes, but may be modified at the discretion of the analyst to meet the needs of the project.

1.5. Method Detection Limits (MDL) and Method Reporting Limits (MRL)

The MDL and MRL for each analyte are listed in Table 2 of the Appendix. The RL for a specific water sample may differ from that listed, depending on the nature of the interferences in the sample matrix and the extent of dilution necessary. Data generated by this SOP are reported to the RL.

1.6. Measurement uncertainty. Variability in historical LCS spike recovery provided in the associated control charts may be used to estimate the minimum laboratory contribution to uncertainty in the measured concentrations of the target analytes generated with this SOP. Uncertainty estimates based on variability in LCS recovery are conservative because some sources of variability are not included, such as matrix effects on analyte recovery, as well as uncertainty related to field operations and sampling.

2. SUMMARY OF METHOD

2.1. Purge-and-trap analysis of aqueous samples

Volatile organic compounds are purged from the sample matrix by sparging a 5 mL sub-sample with nitrogen gas. The purged analytes are then trapped in a sorbent tube until purging is

complete, at which time the sorbent tube is heated and back-flushed with helium, desorbing the trapped analytes to a GC/MS, where they are then separated and detected.

2.2. Identification and quantification of target compounds

Qualitative identification of the parameters in the sample is performed using the retention time (RT) and relative abundance of two or more characteristic masses (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

2.3. Identification and quantification of tentatively identified compounds (TICs)

TICs are identified by comparing the resultant mass spectra to mass spectra contained in an electronic mass spectral library (e.g. Wiley, NIST). TICs are quantified by comparing the mass spectral response from the reconstructed total ion chromatogram for the non-target compound peaks to that of the internal standard with the nearest RT. A relative response factor (RRF) of 1.0 is assumed for quantitation.

3. ABBREVIATIONS AND DEFINITIONS

3.1. Abbreviations

ADOC – Analyst demonstration of capability
amu – Atomic mass unit
ASB – EPA Region 5's Analytical Services Branch (this laboratory, formerly known as CRL)
BFB – 4-Bromofluorobenzene
BS – Blank spike, also known as LCS or midlevel CCV
BSD – Blank spike duplicate, also known as LCSD or midlevel CCV
CAS – Chemical Abstracts Service
CCV – For continuing calibration verifications performed at the mid-level of the calibration, see BS/BSD and LCS/LCSD
"-CCV" – In this SOP, the notation dash-CCV indicates a specific code in the LIMS to denote a distinct procedural calibration verification performed at the lowest calibration level.
CID – Criminal Investigation Division
CRL – Chicago Regional Laboratory (see ASB)
DI – Deionized
EI – Electron ionization
FEP – Fluorinated ethylene propylene
GC/MS – Gas chromatography/mass spectrometry
H_2SO_4 – Sulfuric acid
HCl – Hydrochloric acid
ICAL – Initial calibration
ICV – Initial calibration verification
ID – Identification number
i.d. – Internal diameter
IDOC – Initial demonstration of capability
IS/SS – Internal standards/surrogate standards
L.A.B. – Laboratory Accreditation Bureau
LCL – Lower control limit
LCS – Laboratory control sample, also known as BS or midlevel CCV
LCSD – Laboratory control sample duplicate, also known as BSD or midlevel CCV

LIMS – Laboratory Information Management System
MB – Method blank
MDL – Method detection limit
MRL – Method reporting limit
MS – Matrix spike
MSD – Matrix spike duplicate
m/z – Mass-to-charge ratio
n/a – Not applicable
$NaHSO_4$ – Sodium bisulfate
NIST – National Institute for Standards and Technology
NPDES – National Pollutant Discharge Elimination System
P&A – Precision and accuracy
%D – Percent difference
%RSD – Percent relative standard deviation
PFA – Perfluoroalkoxy
PFTBA – Perfluorotributylamine
p/n – Part number
ppb – Parts per billion (ug/L)
PPE – Personal protective equipment
psi – Pound per square inch
PTFE – Polytetrafluoroethylene
QA – Quality assurance
QC – Quality control
QMP – Quality Management Plan
r^2 – Coefficient of determination
RL – Reporting limit
RPD – Relative percent difference
RRF – Relative response factor
RRF_{avg} – Average relative response factor
RT – Retention time
SDS – Safety data sheet
SOP – Standard operating procedure
SP – Service pack
TC – Target compound
TIC – Tentatively identified compound
UCL – Upper control limit
UI – Ultra inert
US EPA – United States Environmental Protection Agency
VOA – Volatile organic analysis
VOC – Volatile organic compound

3.2. Definitions

Batch – A group of ≤ 20 samples prepared using the same reagents, standards, equipment, and method (i.e. processed as a unit), and analyzed during the same analytical period (i.e. the 12-hour analysis window established by the GC/MS instrument performance check of BFB). Each group of samples contains the procedural quality controls outlined in this SOP and is entered as a unique batch number with an associated bench sheet in the LIMS.

Blank spike – Name used interchangeably with laboratory control sample.

Continuing calibration verification – In the reference method, a continuing calibration verification is a laboratory control sample analyzed in the course of the analysis, which is run to demonstrate that the initial calibration determined at the beginning of the analysis or in a prior analytical run remains valid through the course of the current analytical run. It is usually spiked at the mid-level of the calibration range. Because calibration standards are carried through the entire purge and trap procedure in the same manner as samples, the blank spike/blank spike duplicate (-BS/BSD) serves to verify the calibration, so separate continuing calibration verification standards in addition to the BS/BSD are not required. Throughout this SOP, the abbreviation BS/BSD or LCS/LCSD will be used to refer to midlevel continuing calibration verifications. The CCV abbreviation preceded by a dash ("-CCV") used within this SOP indicates a LIMS entry for one of the reporting limit checks.

Electron ionization – A type of mass spectrometer source where sample molecules are introduced into the source, ionized, repelled out of the chamber, focused, and admitted into the mass analyzer as a monoenergetic ion beam.

Initial calibration – A set of prepared standards diluted from a stock standard solution in a range of concentrations are used to calibrate the instrument response relative to the selected internal standards with respect to known analyte concentrations.

Initial calibration verification – A second-source control standard, analyzed after the initial calibration standards to demonstrate that the instrument is capable of acceptable performance and that the initial calibration is valid.

Laboratory control sample – An aliquot of reagent water to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a sample with all preparation steps. The purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Also called a blank spike. In the LIMS, LCSs are added to a bench sheet by selecting "Lab control sample" or "lab control sample duplicate" from a pull-down menu, but the bench sheet label itself will show "-BS" or "-BSD")

Matrix spike – An aliquot of an environmental sample to which known quantities of method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample with all preparation steps that the parent sample received. The purpose is to determine whether the sample matrix contributes bias to the analytical results. Background concentrations of the analytes in the sample matrix must be determined in the parent sample and used to correct the measured values in the matrix spike.

Method blank – An aliquot of reagent water treated exactly like a sample, including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

Method detection limit – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. MDLs are specified in Appendix Table 2. Data are not reported to the MDL except by special request.

Method reporting limit – The lowest concentration of an analyte routinely reported by the ASB. MRLs are specified in Appendix Table 2.

Reporting limit –The reporting limit usually equals the MRL. For specific projects and analytes, actual RLs may be raised above the MRL depending on interferences in the sample matrix and the extent of dilution necessary

Reporting limit check – An initial calibration standard(s) used to demonstrate analyte recovery at the listed MRLs.

Work order – A group of samples arriving at the ASB on the same day from the same sampling project.

4. HEALTH, SAFETY AND WASTE HANDLING

4.1. Safety data sheets

SDSs are available online. See the ASB Chemical Hygiene Plan for more information. Review SDSs for specific physical and health and hazards including appropriate PPE to be used. SDSs may be accessed at: www.sigmaaldrich.com. Alternatively, other vendor websites may be used to locate relevant information.

4.2. Health hazards

The toxicity and carcinogenicity of each reagent used in this SOP have not been precisely defined. However, each chemical compound is treated as a health hazard. Exposure to these chemicals should be reduced to the lowest possible level and the appropriate PPE must be utilized.

WARNING: Many of the chemicals used in this SOP are toxic, carcinogenic, and/or reproductive hazards. Safety must be a primary consideration when handling and preparing chemical standards associated with this SOP.

For a comprehensive guide to health and safety in the ASB, please see the ASB Chemical Hygiene Plan.

4.3. Waste handling

A green tag identifying the contents as “Hazardous Waste” is placed on any container designated for temporary storage of aqueous, solvent-containing wastes generated in this SOP. A carboy with a green “Hazardous Waste” label is kept in the VOA laboratory and is used exclusively for temporary waste storage. It is important to always attach the appropriate chemical waste label (in this case, the green “Hazardous Waste” label) and write the date on which waste collection began in it.

Used VOA vials are emptied of their aqueous content and disposed of in an approved broken glass box along with all other glass waste. The vial caps may be discarded in a regular trash bin. However, used VOA vials that contain residual client samples should be returned to the refrigerator immediately after analysis, then returned to the Sample Custodian. All aqueous organic solvent waste created in this SOP is appropriate for disposal in a green-labeled waste carboy.

Additional information on waste reduction can be found in the ASB Environmental Compliance Plan and additional information on hazardous waste spills can be found in the ASB Hazardous

Materials/Hazardous Waste Contingency Plan. Report all major spills to the ASB Chemical Hygiene Officer.

5. CAUTIONS AND INTERFERENCES

5.1. Causes

Method interferences may be caused by contaminants in the solvent, reagent water, glassware, laboratory conditions, and/or the purge-and-trap system, which can lead to discrete artifacts or an elevated baseline in total ion current profiles. Matrix interferences may be caused by contaminants that are co-extracted from the samples. The extent of matrix interferences will vary considerably from sample source to sample source depending upon variations in the sample matrix. Whenever an interference is encountered, it must be narrowed down to the source so that a corrective action can be taken and the contamination eliminated. For matrix interferences, no corrective action is possible since the interference is an integral part of the sample.

5.2. Minimization

5.2.1. Glassware cleaning

All glassware is cleaned according to ASB SOP GEN008.

5.2.2. High purity reagents

The use of purge-and-trap grade methanol and deionized reagent water that has been filtered/purified (ultrapure water) helps to minimize reagent-based interference problems.

5.2.3. Preventing carryover contamination

Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. After a high concentration sample is analyzed, the following sample(s) must be carefully examined for evidence of cross-contamination. Samples that are suspected to contain target analytes above the standard calibration range may also be screened prior to analysis, providing that an adequate amount of vials were received for each sample. For sample screening, see Attachment 3 of the Appendix.

5.2.4. Evaluation of method blanks

All materials used during sample analysis, including reagent water, solvents, glassware, and the purge-and-trap system, are routinely demonstrated to be free from interferences by the analysis of method blanks. The formation of the thermal decomposition products bromomethane and chloromethane are also monitored by the routine analysis of method blanks.

5.2.5. **CAUTION**: High purity non-volatile tubing

The use of plastic tubing containing VOCs in the purge-and-trap system or in relation to any preparation steps should be avoided as such materials outgas VOCs, contaminating the system. Metal tubing (stainless steel or copper) should be used in the system whenever

possible to avoid contamination. If plastic tubing is necessary for preparation, ensure that volatile-free tubing is utilized (e.g. PTFE, PFA, or FEP tubing).

5.2.6. **CAUTION**: Common laboratory contaminants

Precaution must be taken to avoid methylene chloride and acetone contamination in an organic laboratory as they are commonly used extraction solvents. The analytical and sample storage area should be isolated from all atmospheric sources of these contaminants and the purge-and-trap system must be properly baked prior to use to ensure it is contaminant-free (see Section 9.2.2.1).

5.2.7. Evaluation of trip blanks

Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. If provided, a trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

6. EQUIPMENT AND SUPPLIES

All of the vendors and part numbers for the equipment and supplies listed below are what the ASB currently uses as of the date of this SOP and are listed specifically to facilitate the ordering process. The mention of trade names or commercial products in this SOP is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. Analysts are encouraged to seek out equivalent alternatives to the items listed below. These specific vendors and part numbers are provided as examples, and other materials of equivalent quality may be substituted without altering this SOP. Refer to ASB SOP GEN026 for instructions and analyst responsibilities when purchasing equipment and supplies.

6.1. Purge-and-trap system

6.1.1. Teledyne Tekmar Atomx Automated Sample Prep System

6.1.1.1. Sparge vessel – Teledyne Tekmar 5 mL frit sparger, ½” neck (p/n 14-2337-024) or 25 mL frit sparger, ½” neck (p/n 14-2334-024)

6.1.1.2. Sorbent trap – Agilent p/n 5188-8820, Teledyne Tekmar VOCARB 3000 (#K) trap p/n 14-5864-403, or #9 trap p/n 14-9908-403

6.1.1.3. Computer system with the Teledyne Tekmar Atomx TekLink software

6.2. GC/MS system

6.2.1. Gas chromatograph – Agilent model # 7890A or 7890B with traditional split/splitless inlet

6.2.1.1. GC column – J&W p/n 121-1324UI, DB-624 UI, 20 m x 0.18 mm x 1.0 um

6.2.1.2. Inlet liner – Agilent p/n 5190-4047, straight-through 1.0 mm UI liner

6.2.1.3. Inlet septum – Supelco p/n 23163, Thermogreen LB-2, 11 mm, conditioned

6.2.1.4. Inlet liner o-ring – Agilent p/n 5188-5366, non-stick flip-top

6.2.1.5. GC column ferrules – Agilent p/n 5181-3323 (inlet side) p/n 5062-3508 (mass spectrometer side), 0.4 mm i.d., 85%:15% vespel-graphite

6.2.2. Mass spectrometer – Agilent model # 5975C or 5977A with 6 mm draw-out plate

6.2.3. Computer system with the Agilent Chemstation™ software (version E.02.02 SP1 or newer), a mass spectral library, the LIMS database, and a printer

6.3. Glassware

6.3.1. VOA vials – 40 mL with PTFE-lined caps

6.3.2. Mininert vials – 1.0 mL with screw cap valves

6.3.3. Pasteur pipettes – disposable borosilicate glass, 5.75” with bulbs

6.3.4. Volumetric flasks, class A – 10, 50, and 100 mL with ground-glass stoppers

6.3.5. Beakers – various sizes as needed

6.3.6. Autosampler vials – 1.0 mL with caps

6.4. Other equipment and supplies

6.4.1. Refrigerator capable of maintaining a temperature of < 6°C

6.4.2. pH paper – EMD Chemicals Inc. (Merck) p/n 9590, colorpHast pH-indicator strips

6.4.3. Gases – nitrogen and helium, ultra-high purity

6.5. Certified supporting equipment

6.5.1. Gas-tight graduated syringes – 5, 10, 25, 50, 100, 250, 500, and 1000 uL

NOTE: See ASB SOP GEN026 for re-certification frequency when applicable.

7. REAGENTS AND STANDARDS

All of the vendors and part numbers for the reagents and standards listed below are what the ASB currently uses as of the date of this SOP and are listed specifically to facilitate the ordering process. The mention of trade names or commercial products in this SOP is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. Analysts are encouraged to seek out equivalent alternatives to the items listed below. These specific vendors and part numbers are provided as examples, and other materials of equivalent quality may be substituted without altering this SOP. Refer to ASB SOP GEN026 for instructions and analyst responsibilities when purchasing reagents and standards.

The described preparations of the spiking standards, calibration standards, and QC samples below are not absolute requirements. Rather, the prepared concentrations, spike volumes, and overall calibration range may be tailored to the needs of the project, with consideration given to any relevant action limits or regulatory thresholds, as well as to the nature of the samples and any interferences present therein.

NOTE: Solvents used for sample preparation must be tracked by a LIMS ID.

7.1. Reagents

7.1.1. Reagent water – deionized water supplied throughout the ASB by an in-house reverse osmosis water filtration system. This DI water is then filtered/purified by a Millipore™ system with a VOC-polishing filter (ultrapure water).

7.1.2. Methanol – B&J Brand p/n 232-235 (purge-and-trap grade)

7.2. Standards

7.2.1. Internal standards/surrogates stock solution (IS/SS stock) – AccuStandard p/n M-8260A/B-IS/SS-10X, Internal/Surrogate Standard Mix, 2000 ug/mL in methanol

7.2.2. Instrument performance check stock solution (BFB stock) – AccuStandard p/n M-624-SS-03-10X, p-Bromofluorobenzene, 2000 ug/mL in methanol

7.2.3. AB mix target compounds stock solution – Supelco p/n 47933, EPA 524 VOC Mix A, 200 ug/mL each component in methanol

7.2.4. D mix (ketones(D-K) target compounds stock solution – p/n 572028 Restek EPA VOA 8260D Standard #1 custom mix, varied concentrations 200-1000 ug/mL in 10%:90% DI water-methanol. See Table in 7.3.3.1 below for details.

7.2.5. D mix Acrolein/Acrylonitrile (D-A/A) target compounds in stock solution –AccuStandard p/n M-603-M-5X, 5000 ug/mL each component in 10%:90% DI water-methanol

7.2.6. C mix (gases) target compounds stock solution – AccuStandard p/n M-502B, Volatile Organic Compounds - Gases, 200 ug/mL each component in methanol

7.2.7. Methyl tert-butyl ether stock solution – Restek p/n 30402, 2000 ug/mL in methanol

7.2.8. 2-Chloroethylvinyl ether stock solution – AccuStandard p/n M-601C-10X, 2000 ug/mL in methanol

7.2.9. 1,4-Dioxane stock solution – Restek p/n 30287, 2000 ug/mL in methanol

7.2.10. Second source AB mix ICV stock solution – Restek p/n 30432, 502.2 Cal200 MegaMix, 200 ug/mL each component in methanol

7.2.11. Second source C mix (gases) ICV stock solution – Restek p/n 30042, 502.2 CalMix #1, 2000 ug/mL in methanol

NOTE: The second source ICV stock solutions should be obtained from a different vendor than the target compounds stock solutions used to make the ICAL. However, if none are available, a second lot number from the primary source is sufficient.

NOTE: The D mix (ketones), 1,4-dioxane, methyl tert-butyl ether, and 2-chloroethylvinyl ether are not included amongst the second source ICV standards.

7.3. Preparation and handling of stocks and standards

Stock solutions are received as sealed ampoules from the manufacturer. Stock ampoules must be entered in LIMS as soon as possible after receipt of order, labelled with LIMS id's, and stored in "VOA standard storage freezer" -28ºC, or "ambient standard storage" drawer (BFB and IS/SS only). Open ampules are stored in trays separate from unopened ampules. Certificates of analysis provided by the manufacturer must be filed in the VOA Certificates of Analysis binders located in VOA lab 1010, and preferably also scanned and uploaded into LIMS. Once an ampoule is opened, any unused stock must be transferred to a GC vial with minimal headspace, labelled with the LIMS id of the stock, date opened, analyst's initials, and date the excess stock vial expires. GC vials of excess stock must be stored in the VOA standard freezer. Excess stock vials have the following shelf lives: IS/SS stock and BFB stock: 6 months; C and ICV-C stocks: 1 week; all other stocks: 1 month. Similarly, prepared spiking solutions are also stored in athe VOA standard storage freezer, with the same shelf lives noted above.

NOTE: The volumes specified in the standard preparation instructions below can be measured using gas-tight graduated syringes. The stock solutions must be introduced directly into the dilution solvent (below the meniscus in the mininert or small autosampler vials) to avoid potential loss of analytes.

NOTE: Stocks and standards must be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

7.3.1 Record-keeping and labeling

7.3.1.1 All standards purchased or prepared by mixing and/or diluting stock solutions must be entered into LIMS for identification and labeling. Details of the preparations are recorded in the VOA Standards Preparation Log at minimum, and preferably also as a narrative description in the LIMS entry for the standard. The LIMS and log book entries must minimally contain the manufacturer and lot number or LIMS ID of any dilution solvents or stock solutions used, the volumes of each, the nominal concentration and preparation date of the standard, preparers initials and the new standard's assigned LIMS ID.

7.3.1.2 The prepared spiking solutions are labeled with the standard concentration, LIMS ID, date of preparation and expiration, and the name of the preparing chemist. The preparation of each spiking solution can be traced back to the preparation logbook by the date.

7.3.1.3 The spiking solutions used for a batch of samples are recorded in the data package using the assigned LIMS IDs on the bench sheet. The ICAL data files from the instrument must contain the LIMS IDs of the spiking solutions used to prepare the individual calibration and verification standards.

7.3.2 Internal standards/surrogates spiking solution

The compounds used as internal standards to calculate analyte concentrations in this SOP are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. The compounds used as surrogates to monitor method performance in this SOP are dibromofluoromethane, 1,2-dichloroethane-d_4, toluene-d_8, and 4-bromofluorobenzene. The Atomx purge-and-trap system can be programmed to automatically add a set volume of IS/SS spiking solution to each sample aliquot prior to analysis.

7.3.2.1 An IS/SS spiking solution at 10 ug/mL concentration may be prepared by adding 50 uL of the 2000 ug/mL IS/SS stock solution to a 10 mL volumetric flask and diluting to volume with methanol. The prepared IS/SS spiking solution may then be transferred to a 15 mL standard vessel on the Atomx standard injection system (be sure to prime the injection lines by selecting 'Prime/Change Standard' from the 'Tools' menu in the Atomx TekLink software). The prepared IS/SS spiking solution remain in the standard vessel on the Atomx instrument, under ambient conditions, and expires after 6 months.

7.3.3 ABD mix target compounds spiking solution

The AB and D mix target compounds for this SOP are listed in Table 1 of the Appendix.

7.3.3.1 An ABD mix target compounds spiking solution at 50 ug/mL concentration may be prepared by mixing 250 uL of the AB mix target compounds stock solution at 200 ug/mL with 250 uL of the D mix (ketones) target compounds stock solution at 200 - 1000 ug/mL, 10 uL of the D mix (D-A/A) (acrolein/acrylonitrile, upon request) target compounds stock solution at 5000 ug/mL, and 490 uL of methanol in a mininert or autosampler vial. If acrolein/acrylonitrile is not requested, the D-A/A stock is omitted and 500 uL of methanol is used.

NOTE: The D mix (ketones) target compounds stock solution is a custom mix with varied concentrations of analytes. If the target compounds spiking solution is prepared as above (Section 7.3.3.1), the concentrations of the D mix ketones, as well as acrolein and acetonitrile (if requested) will be as follows:

D mix analytes	Concentrations in D mix ketones or D mix AA stock	Concentrations in 50 ug/mL ABD mix spiking solution
Acrolein	5000 ug/mL	50 ug/mL
Acrylonitrile	5000 ug/mL	50 ug/mL
Acetone	1000 ug/mL	250 ug/mL
2-Butanone	1000 ug/mL	250 ug/mL
4-Methyl-2-pentanone	400 ug/mL	100 ug/mL
2-Hexanone	400 ug/mL	100 ug/mL
Carbon disulfide	200 ug/mL	50 ug/mL

NOTE: Because m- and p-xylene co-elute and have identical mass spectra, these analytes are reported as m+p-xylene and their concentration will be 100 ug/mL in the ABD mix spiking solution when prepared as described above (Section 7.3.3.1).

7.3.4 C mix target compounds spiking solution

The C mix target compounds for this SOP are listed in Table 1 of the Appendix.

7.3.4.1 A C mix (gases) target compounds spiking solution at 50 ug/mL concentration may be prepared by mixing 250 uL of the C mix target compounds stock solution at 200 ug/mL with 750 uL of methanol in a mininert or autosampler vial.

7.3.5 Methyl tert-butyl ether (MTBE) spiking solution (by request)

7.3.5.1 A methyl tert-butyl ether spiking solution at 50 ug/mL concentration may be prepared by mixing 25 uL of the methyl tert-butyl ether stock solution at 2000 ug/mL with 975 uL of methanol in a mininert or autosampler vial. Alternatively, this may be prepared as part of the ABD mix described in 7.3.3.1 by adding the 25 uL MTBE stock into the ABD mix, and decreasing the methanol by 25 uL.

7.3.6 2-Chloroethylvinyl ether (2-CEVE) spiking solution (by request)

7.3.6.1 A 2-chloroethylvinyl ether spiking solution at 50 ug/mL concentration may be prepared by mixing 25 uL of the 2-chloroethylvinyl ether stock solution at 2000 ug/mL with 975 uL of methanol in a mininert or autosampler vial. Alternatively, this may be prepared as part of the ABD mix described in 7.3.3.1 by adding the 25 uL of 2-CEVE stock into the ABD mix, and decreasing the methanol by 25 uL.

7.3.7 1,4-Dioxane spiking solution (1,4-D) (by request)

7.3.7.1 A 1,4-dioxane spiking solution at 100 ug/mL may be prepared by diluting 50 uL of a 2000 ug/mL stock standard with 950 uL of methanol in a mininert or autosampler vial. If 1,4-dioxane is requested, its spiking solution must be prepared separately, and not as part of the ABD mix described in 7.3.3.1.

7.3.8 Second source AB mix ICV spiking solution

7.3.8.1 An AB mix ICV spiking solution at 50 ug/mL concentration may be prepared by mixing 25 uL of the second source AB mix ICV stock solution at 2000 ug/mL with 975 uL of methanol in a mininert or autosampler vial.

NOTE: Because m- and p-xylene co-elute and have identical mass spectra, these analytes are reported as m+p-xylene and their concentration will be 100 ug/mL in the AB mix ICV spiking solution when prepared as described above (Section 7.3.8.1).

7.3.9 Second source C mix ICV spiking solution

A C mix (gases) ICV spiking solution at 50 ug/mL concentration may be prepared by mixing 25 uL of the second source C mix ICV stock solution at 2000 ug/mL with 975 uL of methanol in a mininert or autosampler vial.

7.4. Spike preparation for calibration-level and batch-level quality controls

7.4.1.IS/SS spikes: All samples, including ICAL standards, field samples, and associated QC samples, will have internal standards and surrogates added to them in order to calculate analyte concentrations and monitor method performance in each sample. The Atomx purge-

and-trap system can be programmed to automatically spike the IS/SS spiking solution into each sample aliquot prior to analysis. The addition of 5 uL of the 10 ug/mL IS/SS spiking solution (Section 7.3.2) to a 5 mL sample aliquot results in a final concentration of 10 ppb, or 50 ng to the GC assuming 100% recovery.

7.4.2. Target compound spikes: The RL checks, blank spike, and matrix spike QC samples will have target compounds added to them to assess method performance at the RL (-MRL, -CCV) and at the mid-point of the calibration (BS/BSD and MS/MSD). The RL checks are prepared in reagent water, while the mid-range spikes are performed in both reagent water (BS/BSD) and a field sample matrix (MS/MSD). The target compounds must be manually spiked into these QC samples prior to analysis, except that, as noted in 7.5.1, and consistent with the guiding method (8260C:11.4.5.6), the 10 ng (2 ppb) ICAL standard is reprocessed as the -MRL check QC sample.

7.4.2.1. For the RL check (i.e. -CCV1), the addition of 4 uL of the target compounds spiking solutions (Sections 7.3.3 – 7.3.6) to a 100 mL volumetric flask results in a final concentration of 2 ppb, or 10 ng to the GC, assuming 100% recovery of a 5-mL sample aliquot.

7.4.2.2. For BS/BSDs, the addition of 50 uL of the target compounds spiking solutions (Sections 7.3.3 – 7.3.6) to a 100 mL volumetric flask results in a final concentration of 25 ppb, or 125 ng to the GC, assuming 100% recovery of a 5-mL sample aliquot.

7.4.2.3. For MS/MSDs, the addition of 20 uL of the target compounds spiking solutions (Sections 7.3.3 – 7.3.7) to each of two replicate field sample vials (40 mL VOA vials) results in a a final concentration of 25 ppb added, or 125 ng to the GC, assuming 100% recovery of a 5-mL sample aliquot. For best results, slightly loosen the VOA vial cap, inject the through the VOA vial septum, re-tighten the cap and mix well by inverting the vial multiple times.

7.4.3. Method blanks: For each batch of ≤ 20 field samples, at least one method blank QC sample, consisting of reagent water processed through laboratory glassware and a VOA vial, is analyzed to monitor for systemic contamination with any of the target compounds. The method blank sample is only spiked with the IS/SS spiking solution. The method blank is distinct from an instrument blank, which consists of only the AtomX instrument's reagent water and IS/SS, with no VOA vial.

NOTE: Instrument blanks are not considered to be batch QC, and their use in the analytical sequence is described in section 9.2.2.3. As an alternative to an instrument blank, a vial blank may be run. A vial blank is identical to a method blank, but is not routinely added to the bench sheet. A vial blank may be preferable to an instrument blank for clearing carryover contamination after analysis of highly contaminated samples.

7.4.4. RL checks: For each calibration, two RL check QC samples (reagent water) are analyzed to verify method performance at or near the RL. The RL check QC samples are spiked with the IS/SS and target compounds spiking solutions as described in 7.4.2.1. One is the 10 ng (2 ppb) ICAL standard reprocessed through the calibration (entered in LIMS as -MRL). The other is a separate injection from a separate vial (entered in LIMS as -CCV). See section 10.2.5 for additional detail.

7.4.5. Blank spike/blank spike duplicate: For each batch of ≤ 20 field samples and every 12 hours of analysis, at least one BS/BSD pair is analyzed to compile method performance data for a clean sample matrix. The blank spike QC samples are prepared by spiking reagent water with the IS/SS and target compounds spiking solutions as described in 7.4.2.2. For analytical runs opening with an initial calibration, the 125 ng (25 ppb) ICAL standard is reprocessed to generate the opening BS. The closing BS, and any mid-sequence BSs needed for longer sequences, also serve to periodically verify the calibration. In LIMS, BSs are added to the LIMS bench sheet by selecting "add laboratory control sample" but they show on the bench sheet as -BS/BSD.

7.4.6. Matrix spike/matrix spike duplicate: At least one MS/MSD pair performed on replicate field samples is analyzed per batch of ≤20 field samples from a given project. For projects consisting of multiple small work orders (each a few samples) arriving in rapid succession and batched together, each project must have an MS/MSD. To the extent that sample vials permit, an MS/MSD result at an appropriate dilution must be present for each analyte. Requirements for performing MS/MSDs are contingent upon sufficient sample being provided. A minimum of 5 vials is required for an MS/MSD; more are recommended for samples with widely variable analyte concentrations.

The MS/MSD verifies method performance in the field sample matrix. The QC samples are spiked with the IS/SS and target compounds spiking solutions at similar concentrations as the blank spike QC samples. As described in 7.4.2.3, for a 40 mL sample, 20 µL of the normal spiking solutions are added to each vial to reach a 25 µg/L spike concentration. For samples with high or highly variable analyte concentrations, it may be necessary to prepare MS/MSDs from dilutions to obtain valid MS/MSD results for some analytes. The following procedures provide examples of how to perform MS/MSDs on samples requiring dilution. Regardless which dilution approach is used, the source sample must be treated the same way as the MS/MSD vials.

7.4.6.2. Spike after volumetric bench dilutions. An appropriate aliquot from each of three replicate sample vials (source vial, MS vial, and MSD vial) is transferred into reagent water in three volumetric flasks (E.g., for 50x dilutions, use 2 mL of sample per 100 mL flask.) Ensure the flask is mostly filled with reagent water before adding sample aliquots or spikes. After addition of sample, bring to volume without target compound spikes to prepare the bench dilution of the source sample. For the MS/MSD, spike with 50 uL of the target compound spiking solutions (Sections 7.3.3 – 7.3.6) before bringing to 100 mL, resulting in a spiked concentration of 25 ppb added, or 125 ng to the GC, assuming 100% recovery of a 5-mL sample aliquot. (The internal standards and surrogates are added at the Atomx purge & trap instrument as described in 7.4.1) To ensure accurate MS/MSD recovery and RPD calculations in LIMS, ensure the dilution factor is applied to the source and spiked samples in the data entry/review screen. On the bench sheet, the spike volume equals the ordinary spike volume (per 5 mL aliquot) times the dilution factor.

7.4.6.3. High spike before Atomx dilution. Alternatively, higher concentrations of target compounds can be spiked directly into the MS/MSD VOA vials in order to spike at concentrations comparable to or above the concentrations in the source sample. In this case, the source and MS/MSD dilutions would all be performed on the Atomx. In order to keep spike volumes low, this approach might require spiking with stock solutions, rather than with the target compound spiking solutions in sections 7.3.3-7.3.6.

7.5 Calibration preparation

NOTE: Calibration standards must be prepared fresh daily.

7.5.1 Initial calibration standards, the ICV standard, and the mid-level CCV standard(s) may be prepared in 100 mL class A volumetric flasks with reagent water (Section 7.1.1), 50 ug/mL spiking solutions (Sections 7.3.3 - 7.3.8), and gas-tight graduated syringes in the following scheme:

Calibration Level	**Level 1†**	**Level 2**	**Level 3**	**Level 4***	**Level 5**	**Level 6**
Volume of 50 ug/mL spiking solution	4 uL	10 uL	20 uL	50 uL	100 uL	200 uL
ppb of standard	2 ppb	5 ppb	10 ppb	25 ppb	50 ppb	100 ppb
ng to GC#	10 ng	25 ng	50 ng	125 ng	250 ng	500 ng
Volume of 100 ug/mL 1,4-D spiking solution	25 uL	50 uL	100 uL	150 uL	200 uL	
ppb of standard	25 ppb	50 ppb	100 ppb	150 ppb	200 ppb	
ng to GC#	125 ng	250 ng	500 ng	750 ng	1000 ng	

† Level 1 serves as the RL check spiking concentration (-MRL and -CCV).

* Level 4 ordinarily serves as the ICV and BS/BSD spiking concentration. Level 3 serves for 1,4-dioxane.

Assuming a 5-mL sample aliquot and 100% purge & trap efficiency from AtomX. Stated ng do not account for constant, 150:1 split at the GC injector (ie, ng are before the split)

NOTE: In sequences opening with an initial calibration, the 125 ng (25 ppb) ICAL standard is reprocessed as the BS QC sample (-BS1). The 10 ng (2 ppb) ICAL standard is reprocessed as the RL check QC sample (-MRL).

7.5.2 The spiking solutions must be introduced directly into the reagent water (below the meniscus in the volumetric flask) to avoid potential loss of analytes. Once a volumetric flask has been spiked, it can be brought up to volume with reagent water, fitted with a ground-glass stopper, and inverted no more than 3 times to mix the solution before filling up the corresponding 40 mL VOA vial(s) with no headspace.

The following tables outline the corresponding concentrations in the calibration standards and the ng transferred to the GC (assuming 5 mL aliquot and 100% recovery) for the analytes that have variable concentrations in the nominally 50 ug/mL spiking solutions. See the notes under Section 7.3.3 for further details.

250 ug/mL concentration in 50 ug/mL spiking solution	**Level 1**	**Level 2**	**Level 3**	**Level 4***	**Level 5**	**Level 6**
Acetone **2-Butanone**	10 ppb 50 ng	25 ppb 125 ng	50 ppb 250 ng	125 ppb 625 ng	250 ppb 1250 ng	500 ppb 2500 ng
100 ug/mL concentration in 50 ug/mL spiking solution	**Level 1**	**Level 2**	**Level 3**	**Level 4***	**Level 5***	**Level 6**
4-Methyl-2-pentanone **2-Hexanone** **m+p-Xylene**	4 ppb 20 ng	10 ppb 50 ng	20 ppb 100 ng	50 ppb 250 ng	100 ppb 500 ng	200 ppb 1000 ng

7.5.3 A method blank is prepared by collecting the same reagent water used to prepare the calibration standards into a 100 mL volumetric flask, then transferring to a 40 mL VOA vial with no headspace. Instrument or vial blanks may also be run to periodically check for carryover contamination in the system after highly concentrated standards (i.e. at the end of a calibration) or field samples. The Atomx purge-and-trap system can be programmed to automatically introduce instrument blanks from the attached reagent water reservoir.

8. SAMPLE HANDLING AND PRESERVATION

Samples are collected in 40 mL VOA vials with PTFE-lined caps and no headspace. A minimum of three vials per distinct sampling location are requested by ASB for analysis; five vials of at least some sampling locations are preferred in order to permit matrix spike and field duplicates to be performed. Aqueous samples with no residual chlorine should be preserved by adjusting the pH of the vials in the field to < 2 with acid, e.g. H_2SO_4, HCl, or solid $NaHSO_4$ (SW-846 Ch4, Table 4.1). If the vials need to be pre-acidified at the ASB prior to field sampling, see Attachment 4 of the Appendix.

NOTE: If 2-chloroethylvinyl ether is requested, un-acidified samples are recommended (SW-846 Ch 4, Table 4.1).

NOTE: If acrolein or acrylonitrile is requested, pH 4-5 or unpreserved samples are recommended (SW-846 Ch 4, Table 4.1; 40 CFR 136, Table II, Footnote 9).

All samples are iced or refrigerated at ≤ 6°C from the time of collection until analysis (SW-846 Ch 4, Table 4.1).

VOA samples are stored separately from all other non-VOA samples as well as from VOA stocks and spiking solutions. When non-CID VOA samples arrive at the ASB, they are immediately checked out from the sample custodian and stored in the VOA refrigerator along with a storage blank comprised of reagent water in a 40 mL VOA vial (usually three). For CID VOA sample storage, see ASB SOP GEN007. Record the prep date of the storage blank on the bench sheet in LIMS. Storage blanks are full vials of reagent water, prepared three at a time on the day samples are received in the VOA refrigerator.

Non-preserved aqueous samples must be analyzed within 7 days of collection. Non-preserved samples for acrolein and acrylonitrile are recommended for analysis within 3 days. Preserved aqueous samples must be analyzed within 14 days of collection. If non-preserved samples exceed the 7 day holding time, the aromatic compounds as well as 2-chloroethyl vinyl ether, acrolein and acrylonitrile must be qualified

(40 CFR 136, SW-846:4). Samples that exceed the 14 day holding time must have all data qualified (see ASB SOP MS037). After analysis, the pH of preserved samples is verified with pH paper.

9. SAMPLE PREPARATION AND ANALYSIS

The described preparations of the field samples as well as the specific operating conditions for both the purge-and-trap pre-concentrator and GC/MS are not absolute requirements. Rather, the prepared concentrations, spike volumes, and instrument settings may be tailored to the needs of the project with consideration given to any action limits or regulatory thresholds the samples are being analyzed against and the nature of the samples and any interferences present therein.

9.1. Sample preparation

9.1.1. Field samples collected in 40 mL VOA vials do not require any bench-top preparation and can be directly analyzed. Samples that are collected in alternative containers will need to be sub-sampled into 40 mL VOA vials and the resultant data considered for qualification as some of the analytes may volatilize during the transfer.

NOTE: Samples that have an air bubble larger than pea-sized (6 mm, ¼ inch) must be considered for data qualification due to potential loss of volatile analytes (SW-846:5021a).

9.1.2. Samples that are suspected to contain target analytes above the calibration range may be screened prior to analysis, providing that an adequate amount of vials were received for each sample. Screening of the sample prior to analysis may provide guidance on whether sample dilution is necessary. For sample screening, see Attachment 3 of the Appendix.

NOTE: Only one aliquot may be removed from each field sample vial for analysis using the autosampler because once the vial has headspace, target compounds will be lost. When preparing multiple dilutions at the bench using a gas-tight syringe, multiple aliquots may be removed (e.g. 100 mL, 50 µL) during a single preparation session for that sample.

9.1.3. If the concentration of any target analytes in a field sample exceeds the calibration range, the purge-and-trap instrument can be programmed to automatically dilute a sample during analysis.

NOTE: If sample dilutions are to be performed by the purge-and-trap instrument, a reagent water reservoir blank will need to be analyzed as part of the instrument QC.

9.1.4. For MS/MSD QC samples, the 40 mL VOA vials are spiked at similar concentrations as the BS/BSD QC samples. If the desired amount of target compounds is 125 ng (25 ppb), then this may be accomplished by opening the VOA vial, quickly adding 20 uL of the 50 ug/mL target compounds spiking solutions, resealing the vial, and inverting it several times to evenly distribute the spiked analytes. If sub-sampling is required, the MS/MSD QC samples may be prepared using a volumetric flask, just like the BS/BSD QC samples.

9.1.5. After analysis, the pH of acid preserved samples is verified with wide-range pH paper. pH as well as the LIMS id of the pH paper used are recorded on the bench sheet(s).

9.2. Purge-and-trap

9.2.1. Purge-and-trap operating conditions (run method)

Purge		**Desorb**	
Valve Oven Temp	125°C	Methanol Needle Rinse	Off
Transfer Line Temp	125°C	Methanol Needle Rinse Volume	0.0 mL
Sample Mount Temp	40°C	Water Needle Rinse Volume	7.0 mL
Water Heater Temp	80°C	Sweep Needle Time	0.25 min
Sample Vial Temp	20°C	Desorb Preheat Temp	245°C
Sample Equilibrate Time	0.00 min	GC Start Signal	Start of Desorb
Soil Valve Temp	110°C	Desorb Time	0.50 min
Standby Flow	10 mL/min	Drain Flow	100 mL/min
Purge Ready Temp	40°C	Desorb Temp	250°C
Condensate Ready Temp	45°C		
Presweep Time	0.25 min	**Bake** (in-run)	
Prime Sample Fill Volume	3.0 mL	Methanol Glass Rinse	Off
Sample Volume	5.0 mL	Number of Methanol Glass Rinses	0
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	0.0 mL
Sweep Sample Flow	100 mL/min	Number of Water Bake Rinses	3
Sparge Vessel Heater	Off	Water Bake Rinse Volume	7.0 mL
Sparge Vessel Temp	20°C	Bake Rinse Sweep Time	0.40 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100 mL/min
Prepurge Flow	0 mL/min	Bake Rinse Drain Time	0.60 min
Purge Time	11.00 min	Bake Time	6.00 min
Purge Flow	40 mL/min	Bake Flow	250 mL/min
Purge Temp	20°C	Bake Temp	260°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	2.00 min		
Dry Purge Flow	100 mL/min	**Cryo**	
Dry Purge Temp	20°C	n/a	

9.2.2. Purge-and-trap instrument preparation

9.2.2.1. Before each sequence, the sorbent trap must be baked at 260°C with 200 mL/min of flow for approximately 10 minutes. Instrument blanks may be analyzed as an alternative. Verify that there is enough nitrogen in the attached tank to perform the analysis. Tanks are typically replaced at 200-300 psi.

9.2.2.2. The green label waste carboy dedicated to the purge-and-trap instrument must be regularly maintained along with the reagent water reservoir used to dilute samples, rinse the sparge vessel, and provide instrument blanks, when necessary.

9.2.2.3. Once the system is prepared, the VOA vials can be loaded onto the autosampler carousel and the purge-and-trap schedule (sequence) can be created. The dilution factor (if necessary) of the sample and the volume of IS/SS spiking solution to be

automatically added to the 5 mL sample aliquot (see Section 7.4.1) are entered into the purge-and-trap schedule. An example of a suitable analysis sequence beginning with an initial calibration is as follows:

Analysis system/reagent H_2O blank check
Instrument performance check (BFB)
Calibration sequence:
- CAL blank (reagent water from a volumetric flask)
- ICAL level 1: 10 ng (2 ppb) [reprocess as -MRL1]
- ICAL level 2: 25 ng (5 ppb)
- ICAL level 3: 50 ng (10 ppb)
- ICAL level 4: 125 ng (25 ppb) [reprocess as -BS1]
- ICAL level 5: 250 ng (50 ppb)
- ICAL level 6: 500 ng (100 ppb)
- Instrument or vial blank(s) carryover check
- RL check: 10 ng (2 ppb). Separate vial. [enter in LIMS as -CCV1]
- ICV: 125 ng (25 ppb)

Method blank QC samples(s) (including storage blank per project)
Field sample(s) [including MS/MSD
Closing BSD: 125 ng (25 ppb) [-BSD1]

For existing calibrations, follow a similar sequence, replacing the ICAL sequence (CAL blank through ICV) with a midlevel continuing calibration verification (-BS1) run between the instrument performance check and the method blank.

With the exception of direct-injection BFB, each injection requires ~30 minutes to complete. Add blanks if needed to address carryover, ensure at least one BFB check per 12-hours of analysis, and one analytical blank per ≤20 field samples. Add BS as needed to ensure on BS per ≤20 field sample and one continuing calibration verification every 12 hours.

At the conclusion of a sequence, save (pdf print) the sequence logs from the GC/MS and from the associated Atomx instrument for inclusion in the electronic data package.

9.3. Gas chromatography/mass spectrometry

9.3.1. GC/MS operating conditions

GC Inlet		**GC Auxiliary Heater**	
Heater	200°C	Heater	On
Pressure	14.125 psi	Temperature	250°C
Total Flow	110.7 mL/min	**GC Inlet/Injection Type (Parameters)**	
Septum Purge Flow	5 mL/min		
Mode	Split	Sample Inlet	GC
Split Ratio	150:1	Injection Source	External Device
Split Flow	105 mL/min	Mass Spectrometer	Enabled
Gas Saver	Off	**Mass Spectrometer Scan Parameters**	
GC Column		Solvent Delay	1.05 min
Constant Flow	0.7 mL/min	EMV Mode	Gain Factor
Average Velocity	37.062 cm/sec	Gain Factor	5.00
Holdup Time	0.8994 min	Acq. Mode	Scan
		Tune File	BFB_Atune.u
GC Oven		Low Mass	35.00 amu
Oven Temp	On	High Mass	300.00 amu
Equilibration Time	0 min	Threshold	100
Maximum Oven Temp	260°C	Samples	2
Initial	35°C hold for 4 min	A/D Samples	4
Ramp 1	15°C/min to 240°C hold for 0.3333 min	Trace Ion Detection	On
Run Time	18 min		
GC Configuration		**Mass Spectrometer Temperatures**	
		Source	250°C
Slow Fan	Disabled	Quad	200°C
Column	See Section 6.2.1.1		
Column Inlet	Front Inlet		
Column Outlet	Vacuum		
Front SS Inlet	He		

9.3.2. GC/MS instrument preparation

9.3.2.1. Verify that there is enough helium in the attached tank to perform the analysis. Tanks are typically replaced at 200-300 psi.

9.3.2.2. Create the GC/MS sequence. Be sure to match the GC/MS sequence to the corresponding purge-and-trap schedule.

9.3.3. GC/MS instrument performance check

9.3.3.1. Before analyzing any calibration standards or samples, the spectral fidelity of 4-bromofluorobenzene must be evaluated against the ion abundance criteria listed in Table 3 of the Appendix. This may be done one of two ways: 1) An instrument blank or vial blank can be purged and analyzed using the same system parameters

as the rest of the sequence or 2) 1 uL of the instrument performance check stock solution containing 4-bromofluorobenzene (Section 7.2.2) can be directly injected onto the GC column and analyzed using the following GC/MS operating conditions:

GC Inlet		**GC Auxiliary Heater**	
Heater	200°C	Heater	On
Pressure	14.517 psi	Temperature	250°C
Total Flow	281.2 mL/min	**GC Inlet/Injection Type (Parameters)**	
Septum Purge Flow	0.5 mL/min		
Mode	Split	Sample Inlet	GC
Split Ratio	400:1	Injection Source	Manual
Split Flow	280 mL/min	Mass Spectrometer	Enabled
Gas Saver	Off	**Mass Spectrometer Scan Parameters**	
GC Column		Solvent Delay	3.00 min
Constant Flow	0.7 mL/min	EMV Mode	Gain Factor
Average Velocity	37.157 cm/sec	Gain Factor	5.00
Holdup Time	0.89709 min	Acq. Mode	Scan
		Tune File	BFB_Atune.u
GC Oven		Low Mass	35.00 amu
Oven Temp	On	High Mass	300.00 amu
Equilibration Time	0 min	Threshold	100
Maximum Oven Temp	260°C	Samples	2
Initial	40°C hold for 1 min	A/D Samples	4
Ramp 1	25°C/min to 200°C hold for 0.6 min	Trace Ion Detection	On
Run Time	8 min		
GC Configuration		**Mass Spectrometer Temperatures**	
		Source	250°C
Slow Fan	Disabled	Quad	200°C
Column	See Section 6.2.1.1		
Column Inlet	Front Inlet		
Column Outlet	Vacuum		
Front SS Inlet	He		

9.3.3.2. A background corrected mass spectrum of 4-bromofluorobenzene is obtained to confirm that the m/z criteria in Table 3 of the Appendix are achieved. If the criteria cannot be achieved, the mass spectrometer is re-tuned according to Section 9.3.4 and the instrument performance check is repeated. If the criteria still cannot be met, some preventative maintenance may be necessary (Section 13). This check must be repeated once every 12 hours and the analysis of this check starts the 12-hour clock during which calibration standards and samples may be analyzed.

9.3.3.3. The Chemstation™ software has a built-in macro that automatically finds and performs a background subtraction for BFB ('Tuner → Evaluate BFB… → Autofind BFB to Screen'). The autofind macro also gives the option of either evaluating BFB as the average of three scans across the chromatographic peak minus a background scan or evaluating BFB as the average across the entire peak minus a background scan. Alternatively, a single scan may be selected manually as long

as the total ion chromatogram response of the selected scan is ≥ 50% of the peak height. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of BFB.

9.3.3.4. The BFB performance check is required to be repeated every 12 hours, as described in 10.2.8. Consistent with 8260C § 11.4.1, the initial BFB performance check must precede analysis of calibration standards or samples, so must be performed by direct injection or on an instrument or vial blank as described in 9.3.3.1. However, if subsequent BFB performance checks are required during long sequences, they may be performed on any blank or QC sample (except for matrix spikes), such as instrument blank, vial blank, analytical blank, storage blank or even a mid-sequence calibration verification sample (-BS), as permitted by 8260C § 11.4.3.

9.3.4. PFTBA mass spectrometer tuning

9.3.4.1. When a BFB tune check cannot be passed or when changes to the mass spectrometer warrant it, a PFTBA system tune must be performed. More frequent tunes such as before each calibration are recommended. The tuning algorithm (e.g. BFB Autotune or BFB.U) is run via the mass spectrometer instrument software and the resultant optimization report is evaluated and then filed in the tuning binder for the instrument. The BFB Autotune algorithm adjusts the PFTBA tune to more closely match source conditions that will provide a satisfactory BFB spectrum. It may be helpful to conduct an ordinary PFTBA autotune algorithm first, followed by the BFB autotune algorithm. Typically, following a BFB autotune on PFTBA, the ratio of the 219 m/z : 69 m/z PFTBA fragments will be in the range of 45% – 75% while the repeller settings will range from 20 V – 32 V. It is also normal for the emission settings to deviate from 35 uA with the BFB Autotune algorithm. Air and water backgrounds can be checked in the BFB Autotune results, or through an independent air and water check. Backgrounds of 1-3% are typical for both m/z 18 and 28.

9.4. System calibration and sample data collection

NOTE: For sample calculations and equations, see Attachment 1 of the Appendix. For an example sequence, see 9.2.2.3.

9.4.1. Initial calibration

9.4.1.1. A series of prepared calibration standards are analyzed to generate calibration curves for individual analytes. The reviewed data files for each standard are uploaded into the calibration method and reports are generated showing, depending on the calibration model, the RRF_{avg} and %RSD and/or the linear or quadratic regression coefficients and r^2 fit for each target compound and surrogate.

9.4.1.2. The initial calibration acceptance criteria for the RRF_{avg} calibration model is %RSD ≤ 20 for all target compounds and surrogates. If the %RSD does not meet the 20% limit, or based on professional judgment of the analyst, a linear or quadratic calibration model using least-squares fit regression may be used. The minimum number of calibration points for the RRF_{avg} and linear regression models is 5, while 6 points is the minimum for quadratic regression. The acceptance criterion for either regression model is $r^2 \geq 0.99$. The regression function should be weighted

(1/concentration or 1/concentration^2) to improve the accuracy of the calibration at the low end of the curve, although weighting is not required by either 8260C or 8260D. While the simplest curve that meets the criteria is preferred, any of the calibrations listed above may be used provided their acceptance criteria are met.

9.4.1.3. Regardless of the calibration model, the low and/or high point(s) can be removed from the calibration curve to meet the acceptance criteria as long as the curve contains the minimum number of points. Consideration must be given to the effect of the change in the calibration range on the reporting limits of the samples.

9.4.1.4. If > 10% of the target compounds do not meet the initial calibration criteria, create fresh spiking solutions and evaluate fresh calibration standards.

9.4.1.5. Due to the large number of compounds that may be analyzed by this method, even when >90% of target compounds meet the initial calibration criteria, some compounds may fail to meet the criteria. If a compound fails to meet initial calibration criteria, its concentration may still be determined, but must be reported as estimated.

9.4.1.6. Update the retention times, primary/secondary ion ratios and reference mass spectra with each initial calibration using a midlevel calibration standard (typically cal level 4, 125 ng). (Procedural detail is lacking in 8260C §11.6.1. Per-calibration updates on a midlevel standard are required by 8260D §11.3.3).

A recommended practice is to retain the reference spectra from the prior calibration while initially processing the ICAL files, then update the retention times, ion ratios and reference spectra after completion of the calibration but before its use in processing subsequent samples.

- Retention times and ion ratios can be updated in ChemStation/Initial Calibration/Update levels/update level.
- Reference spectra can be updated in ChemStation/Continuing Calibration/Update Reference Spectra, performed with the appropriate calibration file loaded.

9.4.1.7. After completing the initial calibration and associated processing method, copy the level 1 and level 4 calibration data files into a new folder for re-processing with the new calibration. In the ChemStation software, edit the file information of the copied files to match the correct bench sheet QC samples (Bxxxxxx-MRL1 and -BS1).). Reprocess the copied files using the new calibration to capture -MRL1 and -BS1 calibration data for upload into LIMS. If the re-processed -MRL1 fails the 50-150% RL check acceptance limits described in 10.2.5, repeat the re-processing for successively higher calibration levels as needed, and either raise the reporting limit to the first passing level, or qualify data between the method reporting limit and the first passing level, as described in 10.2.5.

9.4.1.8. Process the CAL blank, the independent RL check standard and the ICV using the new calibration. The independent RL check standard is loaded into LIMS as -CCV1 for long-term tracking of method performoence. The ICV standard is evaluated in ChemStation ("Continuing calibration/evaluate data file as continuing calibration"). Measured concentrations are evaluated against limits of 70-130% of the expected

concentrations for all target compounds present in the ICV standard. If the measured concentration of any of these compounds varies from the expected concentration by > ± 30%, the results for the compounds exceeding these criteria are qualified (see ASB SOP MS037). If more than 10% of analytes reported for the project fail, then immediate corrective action must be taken and documented, such as re-injecting or re-preparing and re-injecting the ICV standard.

9.4.1.9. The initial calibration section of a data package must consist of the following.

(1) A quantitation report for the calibration blank and for each calibration standard, with detailed reports recommended for the cal blank, CAL 1 and CAL 4. Ensure that reports include results for internal standard and surrogate as well as all analytes, including analytes not detected. A detailed report must be generated for the data file used to update the reference spectra in the data processing method.
(2) Summary calibration reports generated by the Chemstation™ software, including (i) response factor report (Initial calibration/response factors to printer), and (ii) calibration report (enhanced 2/execute; then calibrate/list). A calibration status report (initial calibration/list calibration history) is optional but serves the function of documenting target and internal standard concentrations (or ng) at each calibration level. If it is not included, ensure that concentrations or ng are documented in the package in another manner, such as ChemStation quantitation report sample names.
(3) Quantitation reports for the re-processed -MRL1 and -BS1, as well as for the independent RL check (-CCV1) and the ICV, as well as the ChemStation continuing calibration report for the ICV.

NOTE: Ensure that the “last update” time stamp that appears on the above reports, and which is associated with a given calibration method, matches that of the sample and QC quantitation reports processed using that same method. (To ensure accurate timestamps, in ChemStation, the calibration must be saved both in Initial Calibration/Edit Compounds menu, and in Method/Save Method).

9.4.2. Continuing calibration verification

9.4.2.1. Each target compound and surrogate calibration function in the initial calibration is verified by the measurement of a mid-level calibration standard at the beginning of a sequence (for sequences opening with a calibration, use ICAL 4), every 12 hours after the initial calibration during which samples are analyzed, and again at the end of a sequence. The 70-130% acceptance criteria apply to all the target compounds and surrogates listed in Table 1 of the Appendix. If the measured concentration of any of these compounds falls outside the 70-130% limits for either bracketing -BS, the data in bracketed samples are qualified (see ASB SOP MS037).

NOTE: An separately-injected opening BS is not needed for any 12-hour analysis window that includes the instrument performance check and the initial calibration standards. A closing BS is required to be analyzed at the end of the 12-hour time period or at the end of the sequence, whichever is first. The closing BS does not have to be analyzed within 12 hours of the instrument performance check standard, but it must be analyzed immediately after the last sample in a sequence. If a sequence error or other gross error occurs overnight impacting the closing BSD, it may be re-analyzed after a BFB tune check immediately upon return of the analyst to the laboratory.

9.4.2.2. If the BS at the beginning of a sequence does not meet the 70-130% acceptance criteria for > 20% of the target compounds, fresh calibration spiking solutions must be prepared and the instrument recalibrated. Depending on specific project needs, the compounds that exceed the %D criteria may be deemed important enough to necessitate fresh standards and recalibration even if < 20% of the compounds fail the BS.

9.4.2.3. The continuing calibration verification section of a data package must consist of (1) quantitation reports for the continuing calibration check standards (BS/BSDs) and (2) their continuing calibration evaluation reports.

9.4.3. Sample data collection

9.4.3.1. Once the instrument performance check (Section 9.3.3) and initial calibration (Section 9.4.1) and/or continuing calibration (Section 9.4.2) analyses have met their respective acceptance criteria, field and QC samples may be analyzed. Sample results which exceed the upper calibration limit must be diluted and re-analyzed so the concentration falls within the given calibration range (see Section 9.1.3).

9.4.3.2. The field sample section of a data package must consist of a detailed quantitation report for each field sample, as well as for MS/MSDs, showing results for internal standards, surrogates and all analytes, including non-detected analytes.

9.5. Qualitative and quantitative analysis

9.5.1. Mass spectra are obtained for the primary m/z and at least one or two other masses for each compound. These ions should include all those listed in Table 1 of the Appendix. The following criteria should be met to make a qualitative identification:

9.5.1.1. The characteristic masses of each parameter of interest maximize in the same scan or within one scan of each other.

9.5.1.2. The retention time falls within ± 10 seconds of the retention time of the reference compound.

9.5.1.3. The relative peak intensities of the characteristic masses fall within ± 30% of the relative intensities of these masses in a reference mass spectrum. (Example: For an ion with an abundance of 50% in the reference mass spectrum, the corresponding abundance in a sample spectrum can range from 20% to 80%). This reference mass spectrum must be obtained from a mid-level standard used for initial calibration.

9.5.1.4. Structural isomers that have the same mass spectra may be explicitly identified only if the baseline to valley height between the isomers is less than 50% of the average of the two peak heights. Otherwise, isomers must be reported as isomeric pairs.

9.5.1.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a

valley between two or more maxima), appropriate selection and evaluation of the analyte spectra and background spectra is important for qualitative identification.

9.5.1.6. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

9.5.2. When an analyte has been identified, quantitation is based on the peak area of the primary characteristic m/z relative to the peak area of an associated internal standard. The internal standard used shall be the one nearest the retention time of the given analyte. If the sample matrix interferes with the primary m/z, a secondary characteristic m/z can be used to quantify the analyte. Usually, the base peak m/z is used for internal standards and surrogates. See the example calculations in Attachment 1 of the Appendix for guidance on quantitation.

9.5.3. Chromatographic peaks must be reviewed for proper integration. When the analyst believes the integration performed by the instrument is unacceptable, (s)he should consult the ASB SOP GEN029 for guidance on appropriate manual integration and documentation thereof. This applies to all standards, field samples, and QC samples. For required documentation for manual integrations below the RL, see ASB SOP GEN023.

9.5.4. Tentatively identified compounds – Upon request, a library search shall be executed for non-target compounds in the sample for the purpose of tentative identification. Guidelines for qualitative verification of TICs by comparison of mass spectra are as follows:

9.5.4.1. Major ions (> 10% relative intensity) in the reference spectrum are present in the sample mass spectrum.

9.5.4.2. The relative intensities of the major ions in the sample mass spectrum are within ± 20% of those in the reference spectrum.

9.5.4.3. Molecular ions present in the reference spectrum are present in the sample spectrum.

9.5.4.4. Ions present in the sample spectrum, but not in the reference spectrum are reviewed for possible background contamination or interference.

9.5.4.5. If no library match for a TIC is acceptable, the TIC is identified as “unknown.”

9.5.4.6. Estimated concentration, if given, use a response factor of 1.0 for both identified and “unknown” TICs.

9.5.4.7. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

10. QUALITY CONTROL

Analysts are responsible for performing data evaluation and verification, including manual verification of spreadsheet calculations and data qualification when QC parameters are not met. See ASB SOP MS037 for more information on data review and qualification parameters. Method performance data and QC limits were reviewed for the current version of the SOP, in March 2019. Updated RLs, control limits, and uncertainty values are in Table 2 and the associated control charts, and Attachment 6. Historical limits were calculated. The method allows fixed limits, which ASB uses. No other changes were required.

All data packages which are reported out to clients must be reviewed by a secondary reviewer who has demonstrated (i.e. completed ADOC or IDOC) in the use of a mass spectrometry analytical SOP and who is trained to use GC/MS instrumentation. See the ASB QMP for guidance on the implementation and tracking of formal corrective actions.

10.1. Demonstration of capability

10.1.1. For each analysis system utilized under this method, an IDOC study must be performed. See the ASB QMP and QA-WI002 for guidance and requirements regarding IDOCs.

10.1.2. For an analyst to be qualified to use this SOP to report data to clients, an initial ADOC study must be performed. See the ASB QMP and QA-WI002 for guidance and requirements regarding ADOCs. Initial ADOCs include four mid-level spikes, a method blank, reporting limit checks, and a Mass Spectrometry training checklist (with applicable sections, MS-CHKLST003) completed successfully. A continuing ADOC has fewer requirements and may be assembled from historical data or a performance testing sample data package. See the ASB QMP and QA-WI002 for guidance and requirements regarding ADOCs.

10.1.3. The acceptance criteria for P&A samples for an IDOC or an ADOC is an average recovery of 70-130% of the expected value, and a relative standard deviation of ≤20%. (The 20 %RSD limit ensures that any analyte passing an MS023 ADOC also passes the tightest %RSD limit imposed by MS024 – Volatiles by EPA 624).

10.2. Procedural quality controls

Following are the general types of quality controls that are employed with the analysis procedure described in this SOP. More information on evaluation and corrective action for each type of QC can be found in ASB SOP MS037. For a summary of this QC criteria, see Attachment 6 of the Appendix.

NOTE: For sample calculations and equations, see Attachment 1 of the Appendix.

10.2.1. Surrogates: All samples, including ICAL standards, field samples, and associated QC samples, have surrogates added to them to monitor method performance in each sample. Percent recoveries are calculated and compared to the surrogate recovery limits in Attachment 6. Two out of the four surrogates listed in this SOP must be recovered within the specified limits.

10.2.2. Internal standards: All samples, including ICAL standards, field samples, and associated QC samples, have internal standards added to them to calculate analyte concentrations in each sample. The IS peak areas in each sample must be within 50% - 200% of the most recently run mid-level calibration standard and the IS peak areas in each CCV must be

within 50% - 200% of the mid-level ICAL standard. Alternatively, the mean response of an internal standard across all ICAL standards may be used for this comparison if the response in the mid-point ICAL standard is not similar to the other standards. The IS RTs in each sample must be within ± 10 seconds of the most recently run mid-level calibration standard and the IS RTs in each CCV must be within ± 10 seconds of the mid-level ICAL standard.

10.2.3. Method blanks: For each batch, at least one method blank QC sample (reagent water) is analyzed to monitor for systemic contamination with any of the target compounds. For any analyte, a sample concentration greater than five times the concentration in the corresponding method blank(s) is considered free of interference from contamination. For convenience, the "VOA – 8260" analysis is set up in LIMS to automatically place a "Q" indicator on detections greater than the reporting limit, to assist in the evaluation.

10.2.4. Storage blanks: Analysis of the storage blank ensures that cross-contamination is not occurring in the VOA refrigerator between high-level and low-level volatile samples. One storage blank (the oldest) per project must be analyzed, at minimum. The same considerations apply as in 10.2.3 above in assessing whether a sample is considered free from contamination by a storage blank.

10.2.5. RL checks: For each calibration, two RL check QC samples in reagent water are analyzed to monitor and verify method performance at or near the RL. One RL check QC sample, the -MRL, consists of the 10 ng (2 ppb) ICAL standard. The raw instrument data for this standard is re-processed through the new calibration as if it were an unknown sample, as described in 9.4.1.7. This procedure is in accordance with reference method 8260C:11.4.5.6, which specifically requires a calibration-level verification for linear calibration models. This SOP applies RL verifications to all calibration models, substantially exceeding reference method requirements. Sample data are qualified, or reporting limits are raised for that calibration, based on the evaluation of the reprocessed procedural calibration (-MRL).

The other RL check QC sample, the -CCV, is an independent injection of a standard spiked at the reporting limit into reagent water. Unlike the -MRL check, which can be reprocessed at higher calibration levels if needed, the -CCV is always at calibration level 1, and is uploaded into LIMS for long term performance tracking at the original reporting limit. The -CCV is not used to set reporting limits for analytes using the calibration. Reporting limits for the method are verified annually and are adjusted globally based on the -CCV reporting check only during the annual RL verification described below.

Percent recoveries for the RL checks are calculated and compared to the RL recovery limits of 50% – 150%. At least ≥ 80% of the target compounds in the MRL at the first calibration level (-MRL1) must meet the criteria outlined above. If this condition is not met, repeat the calibration. Analytes in the -MRL1 which do not meet the minimum RL criteria must either have their reporting limit raised to the next acceptable level or be flagged as estimated when the concentration in the samples is at or near the original MRL. The next acceptable level is defined as the next calibration level demonstrated quantitatively to meet criteria by re-processing of the calibration standard through the calibration equation.

Neither reference method 8260C nor 8000D require batch-level RL verification. Continued sensitivity at the batch level is demonstrated by comparing internal standard responses of batch samples to the ICAL to monitor for diminished response over time, and by monitoring performance of the BS/BSDs (Refer to 10.2.2, 10.2.10.)

Consistent with guidance in EPA Method 8000D:9.7.2, at least once per year the RL is verified with QC samples (spiked reagent water at calibration level 1, -CCVs) prepared at the reporting limits and separate from the calibration standards. The annual RL verification is based on control charting of the -CCVs, and must include at least seven replicates, which should originate from separate calibrations but may be analyzed in one or more analytical batches if needed to generate sufficient data for charting**.** The verification is evaluated for performance against the 50-150% recovery limits and documented in the lab's quality system tracking software.

10.2.6. Blank spike/blank spike duplicate: For each batch, at least one LCS/LCSD pair (reagent water) is analyzed to compile method performance data for each analyte that is reported in a clean sample matrix. The blank spike QC samples can double as the 125 ng (25 ppb) ICAL standard and also serves as a mid-level calibration verification at the opening, closing and if needed, midpoints of a sequence. The BS/BSD is called LCS/LCSD in method 8260. The recovery of the target analytes in the LCS/LCSD QC samples is compiled for the purpose of establishing estimated measurement uncertainty (see current control charts). Blank spike data are reported with the midlevel continuing calibration verification limits of 70% – 130%, see also section 10.2.10. The RPD between duplicate spikes is a measure of method precision, but it is not used on its own for data qualification. The RPD limit for all TCs is 60%.

10.2.7. Matrix spike/matrix spike duplicate: For each batch, if enough sample is provided, at least one MS/MSD pair is analyzed to monitor and verify method performance for each analyte that is reported in the field sample matrix. If a batch contains samples from multiple projects, an MS/MSD is required per project. The MS/MSD QC samples are spiked at similar concentrations as the blank spike QC samples. Percent recoveries are calculated and compared to the matrix spike recovery limits of 60% – 110%. If either the MS or the MSD fall outside of recovery limits, the source sample is qualified for that analyte. (See also MS037 Data evaluation SOP.) Client-specified performance criteria may also be used. The RPD between duplicate spikes is a measure of method precision, but it is not used on its own for data evaluation. The RPD limit for all TCs is 60%.

10.2.8. Instrument performance check: A GC/MS instrument performance check is analyzed at the beginning of the analysis sequence and again every 12 hours or less while field samples and associated QC samples are being analyzed. The instrument performance check contains ≤ 50 ng on-column of BFB (after split), ensuring ≤50 ng BFB into the mass spectrometer, consistent with 8260c §11.4.1 and 8260d § 11.3.1.1. The spectral fidelity of BFB must be evaluated against and meet the ion abundance criteria listed in Table 3 of the Appendix in order to ensure proper mass spectrometer performance.

10.2.9. Initial calibration: Calibration curves are verified to be accurate through analysis of a second source ICV. The ICAL acceptance criteria specified in this SOP are: %RSD for RRF_{avg} curves ≤ 20%, $r^2 \geq 0.99$ for linear or quadratic regression functions, and ≤ 30% difference from the expected concentrations for the target analytes in the ICV. Calibration points may be removed from either the low or high end of the curve, but at least 5 calibration points are required for the RRF_{avg} and linear regression models while at least 6 point are required for quadratic regression. Linear or quadratic regression functions should be weighted (1/concentration or 1/concentration^2). ≥ 90% of the target compounds must meet the initial calibration criteria outlined above. An initial calibration remains valid until a CCV no longer meets the acceptance criteria described in Section 10.2.10, which is typically less than one week for the full VOC list.

10.2.10. Continuing calibration verification: The instrument calibration and sensitivity, along with the method performance for each analyte is monitored and verified at the mid-level in a clean matrix every 12 hours after the instrument performance check and at the end of every sequence by the analysis of a mid-level continuing calibration verification standard spiked into reagent water at calibration level 4. The acceptance criterion specified for this SOP is for calculated concentrations to be within 30% of the expected values for each compound. At least ≥ 80% of the target compounds in a CCV analyzed at the beginning of a sequence must meet the %D criteria outlined above. This QC sample is entered into LIMS on the bench sheet as a laboratory control sample (-BS/BSD).

11. DATA AND RECORDS MANAGEMENT

11.1. Data reduction

NOTE: For sample calculations and equations, see Attachment 1 of the Appendix.

11.1.1. Review the quantitation of all field and QC samples making sure that all compounds have been properly identified and integrated. Evaluate the mass spectra of all chromatographic peaks compared to their reference mass spectra taking into consideration co-eluting peaks and other interferences and delete any compound results that are not supported by the comparison. Generate quantitation reports for all field and QC samples.

11.1.2. Generate an injection log and internal standard peak area and retention time summary using the 'Custom Reports' feature in the Chemstation™ software. The injection log must include the acquisition times, and IS peak areas and RTs of all relevant analyses in a given sequence including the instrument performance check(s), all calibration standards, and field sample and QC samples. Calibration time stamps are optional, as this information is captured on individual quantitation report headers. The IS peak areas in each sample must be within 50% - 200% of the most recently run mid-level calibration standard and the IS peak areas in each CCV must be within 50% - 200% of the mid-level ICAL standard. Alternatively, the mean response of an internal standard across all ICAL standards may be used for this comparison if the response in the mid-point ICAL standard is not similar to the other standards. The IS RTs in each sample must be within ± 10 seconds of the most recently run mid-level calibration standard and the IS RTs in each CCV must be within ± 10 seconds of the mid-level ICAL standard.

Note: Calibration time stamps of the calibration standards, which are necessarily processed with a prior calibration method to obtain peak areas for developing the new calibration equations, will not match those of files processed with the new calibration. Files processed with the new calibration including -BS1, -MRL1, and the ICV as well as subsequent QC and field samples, must bear the timestamp of the calibration, which is shown on the summary calibration reports. If timestamps are included in the injection log, and discrepancies are observed between timestamps in the injection log and the ChemStation quantitation report timestamps for any file, the ChemStation quantitation file takes precedence over that in the injection log.

11.1.2.1. The injection log is produced in Excel and includes all runs associated with the project on the Raw Data tab(s) (obtained from Chemstation/instrument software export). The Report tab includes the sample date, data file name, sample name, miscellaneous

info, last calibration update (optional), retention time, and peak area response for all internal standards. Additionally, for each injection (except BS/BSDs), the percent area and the difference in retention time are calculated relative to the most recent BS/BSD. The percent area and retention time difference for each BS/BSD is calculated relative to either the ICAL midlevel standard or the ICAL average. The injection log also includes a header with the reported work order, batches, instrument, analyst, method, and SOP. It must include the ICAL sequence, even if the ICAL was previously reported or is associated with another work order. The injection log is generated using the custom reports function in Chemstation. The report tab can be customized to focus on the project (e.g. omit batches full of other project samples on an intervening day) but the Raw Data tab must include all injections from all days of analysis for the project in chronological order.

11.1.3. Generate reviewed reports of all the relevant instrument data. See ASB SOP MS037 for data review guidelines. Detailed reports must be generated for every field sample. Summary reports can be generated for ICAL and CCV standard(s) and QC samples. Ensure that the 'Sample Name' field in the data files exactly matches the LIMS IDs so that Data Tool will recognize the files when they are merged and uploaded into LIMS. Generate the final reviewed quantitation reports to the file name 'epatemp.txt' before merging the raw instrument data into LIMS.

11.1.4. Complete the bench sheet(s), upload the data to LIMS, and review the sample and QC results. The completed bench sheet(s) must make clear which quality control samples were analyzed with each batch.

11.2. Data package assembly

11.2.1. Create a tuning and calibration deliverables section with copies of sequence files, an injection log, instrument performance check parameters (BFB tune check report), initial and continuing calibration reports, and quantitation reports for each calibration standard.

11.2.2. Create a QC deliverables section with QC sample quantitation reports.

11.2.3. Create a field sample deliverables section with field sample quantitation reports and, if requested, associated TIC reports.

NOTE: For a comprehensive checklist of VOA data package components, see Attachment 5 of the Appendix

NOTE: For a general description of data package assembly and handling, see ASB SOP GEN032.

11.3. Record keeping

11.3.1. The BFB Autotune reports must be stored in the appropriate folders electronically on the instrument computer and in the instrument binder. The instrument sequence is also stored electronically on the instrument computer. The calibration reports are stored electronically with the sample data.

11.3.2. Record any instrument maintenance including consumables (carrier gas, gas line traps, septa, *etc.*) in the instrument maintenance logbook, located adjacent to or in a drawer nearby each instrument.

11.3.3. During an analytical run, back up raw instrument data and sequences daily. This is most easily done via the GCMS computer desktop links to R5CRL\Vol1\Old Folders (structure and data)\MS\VOA\backups raw data, or directly to the MS data review location R5CRL\Vol5\Organic Data Review\work order.

11.3.4. Archive all final processed data files along with any electronic copies of supporting data to the R5CRL server as detailed in GEN032.

11.4. LIMS data entry and reporting

11.4.1. All LIMS data entry is based on first creating a batch that contains the correct preparation method, batch matrix, surrogate spiking standard LIMS ID, and analysis. After these are selected, the bench sheet can be created which contains the field sample and QC sample LIMS IDs, sample volumes, surrogate and target compound spike volumes, and preparation dates. The analyst must make certain that the preparation date in LIMS matches the actual preparation date and that the selected analysis is correct before uploading instrument data. The name of the analysis in LIMS is 'VOA – 8260'.

11.4.2. When the data is ready to be entered into the LIMS, the batch or work order number is selected under 'Laboratory → Data Entry/Review…', the relevant analyses are selected, and the 'Create' button under the 'Data Entry' tab is pressed. If data is to be entered manually, click the 'Save' button. If data is to be merged automatically using the Data Tool program, click the Data Tool icon and 'Browse…' for the folder with the desired instrument data files. Selecting the appropriate file folder should populate the upper left window with the file and sample names of the data if the 'File Type' is set to epatemp.txt. Press the 'Merge Files' button to merge the instrument data with the data entry table in LIMS. Investigate for any discrepancies with LIMS IDs, target compound names, or units, and 'Save' the merged template. If either 'Unmatched Analytes' or 'Unmatched Units' appear in red, repair the cross table prior to saving the merged data entry table or some analytes or units may not populate the table correctly. Once saved, the data entry table will be populated with the instrument data.

11.4.3. Proceed to the 'Data Review' tab and press 'Query'. Fill in the instrument used in the Instrument field. Immediately lock the instrument data and verify that the results correlate to the LIMS IDs in the table. Verify that all conversions to reporting units and dilutions have been calculated correctly. Verify that reporting limits have been correctly applied. Flags may be added at this stage following the guidance given in ASB SOP MS037. Before generating a draft LIMS report for secondary data review, the status of the data must be updated to analyzed.

11.4.4. When performing manual data entry, enter the results in the 'IResult' column. For each result, enter the response in the 'Response' column, the retention time in the 'RT' column, the date of analysis in the 'Analyzed' column, and the data file name in the 'FileID' column. If dilutions were necessary for the analysis, enter the dilution factor in the 'Diln' column. The initial sample result must be the one measured and not corrected for dilution. Verify that the 'Analyst' field and 'Instrument' field are correct. Once all the data appears properly, click the 'Save' button.

11.4.5. The results are reported in ug/L for aqueous samples without correction for recovery data and to three significant figures.

11.5. LIMS report generation

11.5.1. To prepare a draft report, select 'Project Management → Reports…', select the work order and analysis, and select the 'Report Format' under the 'Format' tab. If QC is to be omitted from the report or if the client has requested that the field samples be reported down to the MDL, 'Modified Draft' can be selected. Standard procedure is to report to the RL.

11.5.2. After the data reviewer has updated the status of the analysis to 'Reviewed' and the corresponding case narrative has obtained supervisory approval, the final report may be generated. Upload the case narrative into LIMS by selecting the 'Case Narratives' tab and pressing the page icon next to the 'Work Order Case Narrative' field. Click 'Add' to cut-and-paste the narrative into the text field. Select the appropriate analysis from the column on the left and type that analysis into the 'Description' field and click 'Save'. Select the case narrative from the 'Work Order Case Narrative' pull-down menu. If the client requests an electronic copy of the data, select 'STANDARD EXCEL' from the 'Standard EDD Library' pull-down menu under the 'Format' tab to generate an Excel spreadsheet of the data. (Other formats may be requested.) The mode of generation of the report is the same as above (Section 11.5.1) and either the 'Final' or 'Modified Final' report may be selected. Use "modified draft" and "modified final" to remove the "-CCV" reporting limit check from LIMS reports to clients, as this RL check is for internal long-term RL verification only. Use only the -MRL reporting limit check on externally reported data. The final report must be electronically signed by the analyst per GEN 032.

12. TROUBLESHOOTING

NOTE: For extended troubleshooting, consult the instrument manuals located near the respective instrument.

12.1. Possible causes of inadequate abundance at high masses

12.1.1. Dirty or contaminated ion source, electron multiplier, or quadrupole rod surfaces. If a BFB Autotune shows the repeller to be at or near 35 V, the source is probably clean.

12.1.2. Potentials of ion source elements at wrong values due to open or short circuits.

12.1.3. Faulty ion source electronics, detector electronics, or power supply.

12.1.4. Abnormally high pressure (greater than 5.0×10^{-5} torr) causing ionization difficulties. Check for presence of air at m/z 28 and 32 > 10% of the m/z 69 peak of PFTBA, indicating vacuum system leak.

12.2. Possible causes of improper isotope ratios

12.2.1. High background levels of undesired substances (contamination from earlier sample analysis) which contributed additional abundance at the isotope mass. Bake out the sorbent trap and sufficiently rinse the sparge vessel with water and possibly even methanol. If necessary, bake out the ion source assembly and condition the column.

12.2.2. Resolution of adjacent masses set improperly: higher than normal ratios due to low resolution (wide peaks) or lower than normal ratios due to high resolution (narrow peaks).

12.3. Probable causes of low overall sensitivity

12.3.1. Dirty or contaminated ion source, electron multiplier, or quadrupole rod surfaces. If a BFB Autotune shows the repeller to be at or near 35 V, the source is probably clean.

12.3.2. Electron multiplier with low gain.

12.3.3. Abnormally high pressure (greater than 1.0×10^{-4} torr) causing ionization difficulties. Check for presence of air at m/z 28 and 32 (CO_2 at m/z 44) indicating vacuum system leak.

12.4. Possible causes of poor reproducibility

12.4.1. Loose or intermittent connection either to a printed circuit or to one or more ion source or quadrupole elements inside the analyzer.

12.4.2. Pressure or flow fluctuations – check using ion gauge. Check the PFTBA vial. Refill the vial, if necessary. Pressure with the PFTBA valve open should be between 1.0×10^{-6} torr and 4.0×10^{-6} torr. Ensure that the mass flow controller on the purge-and-trap instrument is working properly.

12.5. Possible causes of high background

12.5.1. Dirty or contaminated sorbent trap, sparge vessel, ion source, electron multiplier, or quadrupole rod surfaces. Bake out the trap and sufficiently rinse the sparge vessel with water or possibly even methanol. If necessary, bake out the source. If a BFB Autotune shows the repeller to be at or near 35 V, the source is probably clean.

12.5.2. Column or septum bleed, as evidenced by intense peaks at m/z 73, 147, 207, or 221. Should not have an abundance of greater than 500 counts under BFB Autotune conditions. Replace the faulty septum and condition the column.

12.5.3. Pump oils – in any oil diffusion pumped vacuum system, there is the potential for diffusion pump oil and/or mechanical pump oil to be present in the system. This is highly unlikely under normal conditions.

12.5.4. There is the possibility that some previously analyzed sample can still be present in the vacuum system long after it was thought to be evacuated. This phenomenon depends on sample volatility, temperature, etc.

12.5.5. Contamination in a recently cleaned vacuum system – after any venting of a vacuum system for maintenance, there is the potential for introducing new substances into the vacuum system. Some substances are normal and can be pumped out while others require more cleaning or baking.

12.5.5.1. Solvents used in the cleaning process – these will be present for awhile but should be pumped out as heat is applied to the vacuum system.

12.5.5.2. Water absorbed on the metal surfaces while vented. This will pump out with heat.

12.5.5.3. Fingerprints– heavy organic substances from inadequate clean room procedures may not be pumped out and may require source cleaning.

12.6. Possible causes of an unresponsive mass spectrometer

12.6.1. Mass spectrometer electronics are not on – check that the power switch is on.

12.6.2. Secondary fuse blown – check the secondary fuses on the rear of the mass spectrometer and replace the faulty fuse(s).

12.7. Possible causes of BFB tune check failures

12.7.1. Persistent failures (consecutive evaluations), typically involving the 50 m/z : 95 m/z or 174 m/z : 95 m/z abundance ratios, are more than likely due to an out of tune or dirty ion source. Either retune the ion source or, if necessary, clean the ion source and then retune.

12.8. Faulty sorbent trap indicators

12.8.1. The dichlorodifluorobenzene peak in a chromatogram will begin to tail. Consider replacing the trap.

12.8.2. The chloroethane peak in a chromatogram will begin to broaden. Consider replacing the trap.

13. PREVENTATIVE MAINTENANCE

The sequence folders contain records of analyses and an instrument logbook is used to record any preventative maintenance performed, hardware and software problems, modifications, etc. It is the responsibility of the analyst to document all maintenance items in the relevant instrument maintenance logbooks (date and initial). The logbook for a particular instrument may be found near that instrument. Sequence folders are on the instrument computer. Instrument manuals are on the instrument computer and/or in a drawer near the instrument.

13.1. Gas chromatograph and inlet

Analysts are responsible for performing routine maintenance of the instruments and much of the equipment used in this SOP. Many GC/MS maintenance tasks are performed when some quality control parameter fails (such as an instrument performance check) and are not part of a regular

maintenance schedule. The injector septum and injection port liner are typically replaced when the GC column is replaced. The GC column is changed when chromatographic peak shape becomes poor for select analytes that are more sensitive to the development of active sites in the column that develop with column age, usage, and the degree of interferences in the sample matrix. If replacing the column, liner, and injector septum does not fix the peak shapes of these analytes, replacing the gold seal in the bottom of the inlet may fix the problem.

13.2. Mass spectrometer

The ion source is cleaned if the mass spectrum of the system performance check compound (BFB) fails the prescribed tolerance limits upon consecutive evaluations or if overall sensitivity decreases sharply. Some routine maintenance tasks (e.g. changing the vacuum pump oil) may be covered under the service contract maintained for the instrument and are performed on a routine basis according to the instrument manufacturer's recommended schedule.

13.3. Purge-and-trap instrument

The sorbent trap is replaced when a decline in trapping efficiency becomes apparent. Typical indicators are when the dichlorodifluorobenzene peak in a chromatogram begins to tail and/or when the chloroethane peak begins to broaden. The autosampler carousel will also periodically need to be re-aligned, depending on the frequency with which it is used. See the instrument manual for instructions. Some routine maintenance tasks may be covered under the service contract maintained for the instrument and are performed on a routine basis according to the instrument manufacturer's recommended schedule.

14. REFERENCES

40 CFR Part 136, Table 2, Footnote 9, https://www.ecfr.gov/cgi-bin/text-idx?SID=b9f7e42b56feeb84c90d7bb6b522fbd0&mc=true&node=pt40.25.136&rgn=div5 [accessed 3/30/17]

SW-846 Chapter 4 Revision 4 February 2007 Organic Analytes https://www.epa.gov/hw-sw846/chapter-four-sw-846-compendium-organic-analytes [accessed 3/30/17]

SW-846 Method 5021A Revision 2 July 2014 Volatile Organic Compounds in Various Sample Matrices Using Equilibrium Headspace Analysis https://www.epa.gov/hw-sw846/sw-846-test-method-5021a-volatile-organic-compounds-vocs-various-sample-matrices-using [accessed 3/30/17]

SW-846 Method 5030C Revision 3 May 2003 Purge-and-Trap for Aqueous Samples https://www.epa.gov/hw-sw846/sw-846-test-method-5030b-purge-and-trap-aqueous-samples [accessed 3/30/17]

SW-846 Method 8260C Revision 3 August 2006 Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) https://www.epa.gov/hw-sw846/sw-846-test-method-8260b-volatile-organic-compounds-gas-chromatographymass-spectrometry [accessed 3/30/17]

SW-846 Method 8260D Revision 4, June 2018, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) https://www.epa.gov/sites/production/files/2018-06/documents/method_8260d_update_vi_final_06-11-2018.pdf [accessed 3/27/20]

J.S. Hollis and H. Prest, *Volatile Organic Compound Analysis Using Purge and Trap: Success with VOC analysis using the Agilent 5975C Mass Selective Detector*, Agilent Technologies, Inc. Application Note 5991-0029EN (2012).

AGILENT Gas Chromatograph 7890 Operator's Manual

AGILENT Mass Spectrometer 5975 or 5977 Operator's Manual

TELEDYNE TEKMAR Atomx Operator's Manual

15. REVISION HISTORY

Version	Status	Location of Change History
Qualtrax Version 7		Cover. Updated ANAB logo. Global update to replace CRL with ASB Global update to further explain reprocessed (-MRL) and independent (-CCV) reporting limit checks, and to minimize “CCV” language Global update documenting LIMS and ChemStation mechanics of implementing method Sections 7.4 and 7.5. Replace on-column with to-GC language. Add extended detail regarding instrument, vial and method blanks, MS/MSD requirements (in preparation for upgrade to 8260D) and procedures, and re-processing procedure for ICAL -BS1 and -MRL1. Incorporate P&I 18916. Section 8. Add detailed procedure for storage blanks. Section 9.2, 9.3 and 9.4. Updated and added detail to example instrument sequence, BFB performance check, and calibration procedures. Section 10.1 and 10.2. Add %RSD requirements for DOC. Add repetition to -MRL, -CCV and LCS/BS, MS/MSD and performance check. Section 11.1, 11.3, and 11.5. Documented injection log procedures, added detail on LIMS report generation, updated recordkeeping and archiving locations. Appendix Table 1. Added molecular ions. Modified primary and qualitative ions for styrene and qualitative ions for 1,4-dichlorobenzene-D4 to reduce interferences. Revised position of 1,1,2,2-tetrachloroethane, inserted placeholders for internal standards at their chromatographic elution orders. Attachment 2. Updated BFB discussion Attachment 3. Further documented initial screening procedure. Attachment 5. Updated based on GEN036 data package components for MS, tailored for MS023 VOC Attachment 6. Updated BS/BSD, RL checks Draft document ID 889
Qualtrax Version 6	R for control chart update	Cover. Replaced LAB logo with ANAB. Global update to rename GEN010 to MS037. Section 1. Updated calibration range and reporting limit for consistency with other sections. Section 7. Updated reporting limit and calibration prep and levels for consistency with other sections. Clarified spike volume for MS prep for convenience. Section 9. Updated reporting limit and quality control criteria consistent with 2019 control charts and review of performance data. Section 10.1. Clarified demonstration of capability procedure to address past issues and future questions. Section 10.2.3. Clarified treatment of blanks to state current evaluation practice. Section 10.2.5. Added a pass-rate criterion for the reporting limit check that is the same as the criterion for the CCV. Section 10 all, Table 2. Updated reporting limits, control limits, and hyperlinked references to performance data and uncertainty statistics per scheduled biennial review & update. Section 11. Updated for consistency with procedures for preparing electronic data packages.

		Attachment 4. Incorporated Pen & Ink 14701 for vial prep to allow flexible acid volume. Attachments 2, 6. Updated control limits consistent with 2019 control charts.
Qualtrax Version 5	R	Improved clarity on procedures and aligned with current lab policy throughout document. Archiving revision history entries for pre-Qualtrax versions of the SOP, v.6-6.2. Sections 1.1 and 7 and Tables 1 and 2, incorporated Pen and Ink changes 4648. Section 7 updated standards and equipment. Section 7, Table 1. Updated treatment of acrolein/acrylonitrile per EPA guidance. Section 9 updated tune, incorporated Pen and Ink change 5052. Section 10 updated RL verification language. Table 2, incorporated Pen and Ink change 5777. Attachment 2, updated language to include explanations. Attachment 5, incorporated Preventative Action 9266. Attachment 6, incorporated Pen and Ink change 7667. Draft document workflow ID 9285.
Qualtrax Version 4	R	Attachment 2. Added deviation concerning the CCV equation.
Qualtrax Version 3	R	Entire document – Revised with editorial changes to improve clarity, readability, and consistency. Sections 1.1, 7.2, and 7.3 and Tables 1 and 2. Methyl tert-butyl ether and 2-chloroethylvinyl ether added as SOP analytes available upon request. Sections 2.1 and 9.2.2.1. Purge gas changed from helium to nitrogen. Section 3.2. Definition of batch updated to emphasize 12-hour analysis window established by BFB evaluation. Added a definition for work order. Sections 5.2.2 and 7.1.1. Reagent water definition altered to remove nitrogen sparging. Sections 6.1 and 6.2. Included equipment related to GC/MS #13. Sections 7.4.6 and 10.2.6. Changed the frequency for preparing MS/MSD QC samples from every batch to every work order. Section 9.2.1. Increased the bake flow of the purge-and-trap operating conditions from 200 mL/min to 250 mL/min. Section 9.5.1.2. Qualitative identification criteria concerning the RT of a peak in a sample vs a reference standard changed from ± 30 seconds to ± 10 seconds. Section 10.1. Demonstration of capability requirements replaced with QMP reference. Demonstration of capability criteria changed from historically-based acceptance limits to CCV recovery limits of 75 – 125%. Sections 10.2.1, 10.2.5, and 10.2.6. Surrogate, BS/BSD, and MS/MSD recovery limits changed from historically-based acceptance limits to CCV recovery limits of 75 – 125%. Section 10.2.3. Method blank criteria updated to specify that, for any analyte, a sample concentration > 5x the blank concentration is considered free of interference from contamination. Section 11.3.3. Added QA policy concerning the naming conventions of electronic data and how they cannot include client information. Table 1. Removed m/z 77 as a qualification ion for fluorobenzene. Updated all analyte RTs.

		Table 2. Updated all the analyte MDLs and blank spike recovery and RPD statistics. Raised the RL for carbon tetrachloride. Clarified that both GC/MS #11 and #13 are eligible to generate data with this SOP. Attachment 2. Removed deviation regarding demonstration of capability criteria for consistency with QMP. Attachment 4. Attachment added to outline procedure for preparing pre-acidified vials for field sampling. Attachment 6. Attachment added to summarize all SOP QC criteria.

R = Revision

16. APPENDICES

Table 1. Method Analyte CAS Numbers, MS Ions, and GC Retention Times

Analyte Type	Compound Name	CAS #	EI Ions#			Retention Time† (min)
			Molecular	Primary	Secondary	
Internal Standard	FLUOROBENZENE	462-06-6	96	96	70, 50	6.661
C mix TC	Dichlorodifluoromethane	75-71-8	120	85	87, 50	1.217
C mix TC	Chloromethane	74-87-3	50	50	52, 49	1.378
C mix TC	Vinyl chloride	75-01-4	62	62	64, 61	1.478
C mix TC	Bromomethane[2]	74-83-9	94	96	94, 81	1.773
C mix TC	Chloroethane	75-00-3	64	64	66, 49	1.883
C mix TC	Trichlorofluoromethane	75-69-4	136	101	103, 66	2.150
Request TC	Acrolein*	107-02-8	56	56	55, 53	2.613
AB mix TC	1,1-Dichloroethene	75-35-4	96	96	63, 61	2.719
D mix TC	Acetone	67-64-1	58	43	58, 42	2.809
D mix TC	Carbon disulfide	75-15-0	76	76	78, 77	2.953
AB mix TC	Methylene chloride	75-09-2	84	84	86, 49, 51	3.346
Request TC	Acrylonitrile*	107-13-1	53	53	52, 51	3.709
AB mix TC	trans-1,2-Dichloroethene	156-60-5	96	96	61, 98	3.751
Request TC	Methyl tert-butyl ether*	1634-04-4	88	73	57, 43	3.815
AB mix TC	1,1-Dichloroethane	75-34-3	98	63	65, 83	4.407
AB mix TC	2,2-Dichloropropane[2]	594-20-7	112	77	79, 97	5.230
AB mix TC	cis-1,2-Dichloroethene	156-59-2	96	96	98, 61	5.243
D mix TC	2-Butanone	78-93-3	72	72	43, 57	5.310
AB mix TC	Bromochloromethane	74-97-5	128	128	49, 130	5.542
AB mix TC	Chloroform	67-66-3	118	83	85, 47	5.667
Surrogate	DIBROMOFLUOROMETHANE	1868-53-7	190	113	111, 192	5.870
AB mix TC	1,1,1-Trichloroethane	71-55-6	132	97	99, 61	5.879
AB mix TC	Carbon tetrachloride	56-23-5	152	117	119, 121	6.082
AB mix TC	1,1-Dichloropropene	563-58-6	110	75	110, 77	6.088
Surrogate	1,2-DICHLOROETHANE-D4	17060-07-0	102	65	67, 51	6.256
AB mix TC	Benzene	71-43-2	78	78	52, 51	6.323
AB mix TC	1,2-Dichloroethane	107-06-2	98	62	64, 49, 98	6.346
	(Fluorobenzene elution order)					
AB mix TC	Trichloroethene	79-01-6	130	95	132, 130, 97	7.072
AB mix TC	1,2-Dichloropropane	78-87-5	112	63	62, 41, 112	7.304
AB mix TC	Dibromomethane	74-95-3	172	93	95, 174, 172	7.423
Request TC	1,4-Dioxane*	123-91-1	88	88	58, 43, 57	7.480

Table 1. Method Analyte CAS Numbers, MS Ions, and GC Retention Times (cont)						
			EI Ions#			Retention Time† (min)
Analyte Type	Compound Name	CAS #	Molecular	Primary	Secondary	
AB mix TC	Bromodichloromethane	75-27-4	162	83	85, 127	7.612
Request TC	2-Chloroethylvinyl ether*	110-75-8	106	63	65, 106, 43	7.960
AB mix TC	cis-1,3-Dichloropropene	10061-01-5	110	75	77, 39	8.088
D mix TC	4-Methyl-2-pentanone	108-10-1	100	58	43, 85, 100	8.278
Surrogate	TOLUENE-D8	2037-26-5	100	98	99, 70	8.365
AB mix TC	Toluene	108-88-3	92	92	91, 65	8.432
Internal Standard	CHLOROBENZENE-D5	3114-55-4	117	82	117, 119	9.812
AB mix TC	trans-1,3-Dichloropropene	10061-02-6	110	75	77, 39	8.664
AB mix TC	1,1,2-Trichloroethane	79-00-5	132	83	97, 85, 99	8.841
AB mix TC	Tetrachloroethene	127-18-4	164	164	129, 131, 166	8.989
AB mix TC	1,3-Dichloropropane	142-28-9	112	76	78, 63	9.008
D mix TC	2-Hexanone	591-78-6	100	57	43, 100, 53	9.127
AB mix TC	Dibromochloromethane	124-48-1	206	129	206, 208	9.230
AB mix TC	1,2-Dibromoethane (EDB)	106-93-4	186	107	109, 188	9.336
	(Chlorobenzene-D5 elution order)					
AB mix TC	Chlorobenzene	108-90-7	112	112	77, 114	9.841
AB mix TC	1,1,1,2-Tetrachloroethane	630-20-6	166	131	119, 133	9.927
AB mix TC	Ethylbenzene	100-41-4	106	106	91, 77	9.966
AB mix TC	m+p-Xylene[1]	108-38-3 /106-42-3	106	106	91, 77	10.085
AB mix TC	o-Xylene	95-47-6	106	106	91, 65	10.474
AB mix TC	Styrene	100-42-5	104	104	78, 103	10.487
AB mix TC	Bromoform[2]	75-25-2	250	173	171, 175, 254	10.651
AB mix TC	Isopropylbenzene	98-82-8	120	105	120, 77	10.850
Surrogate	4-BROMOFLUOROBENZENE	460-00-4	174	95	174, 176	10.988
AB mix TC	Bromobenzene	108-86-1	156	156	77, 158	11.130
AB mix TC	1,1,2,2-Tetrachloroethane	79-34-5	166	83	85, 131	11.133
AB mix TC	1,2,3-Trichloropropane	96-18-4	146	75	61, 110	11.169
AB mix TC	n-Propylbenzene	103-65-1	120	91	120, 121	11.259
AB mix TC	2-Chlorotoluene	95-49-8	126	91	126, 128	11.333
AB mix TC	1,3,5-Trimethylbenzene	108-67-8	120	105	120	11.439
AB mix TC	4-Chlorotoluene	106-43-4	126	91	126, 128	11.442
Internal Standard	1,4-DICHLOROBENZENE-D4	3855-82-1	150	152	150, 154	12.143
AB mix TC	tert-Butylbenzene	98-06-6	134	119	91, 134	11.763
AB mix TC	1,2,4-Trimethylbenzene	95-63-6	120	105	120, 77	11.808
AB mix TC	sec-Butylbenzene	135-98-8	134	105	134, 91	11.982
AB mix TC	1,3-Dichlorobenzene	541-73-1	146	146	111, 148	12.075
AB mix TC	p-Isopropyltoluene	99-87-6	134	119	134, 91	12.130

Table 1. Method Analyte CAS Numbers, MS Ions, and GC Retention Times (cont)						
			EI Ions#			Retention Time† (min)
Analyte Type	Compound Name	CAS #	Molecular	Primary	Secondary	
(1,4-dichlorobenzene-D4 elution order)						
AB mix TC	1,4-Dichlorobenzene	106-46-7	146	146	111, 148	12.165
AB mix TC	1,2-Dichlorobenzene	95-50-1	146	146	111, 148	12.535
AB mix TC	n-Butylbenzene	104-51-8	134	91	92, 134	12.538
AB mix TC	1,2-Dibromo-3-chloropropane	96-12-8	234	155	157, 75	13.304
AB mix TC	1,2,4-Trichlorobenzene	120-82-1	180	180	182, 145	14.143
AB mix TC	Hexachlorobutadiene	87-68-3	258	225	223, 227	14.329
AB mix TC	Naphthalene	91-20-3	128	128	127, 102	14.381
AB mix TC	1,2,3-Trichlorobenzene	87-61-6	180	180	182, 145	14.625

Molecular ions are informational and may not be present in the spectrum with appreciable intensity for some analytes. Primary ions are used for quantitation. Secondary EI ions are used for confirmation of peak identification. Other ions may be equivalent or better for confirmation depending on the compound and the sample matrix/co-eluting interferences.

† Retention times based on 18 min GC oven program using the analytical GC column described in Section 6.2.1.1. Each internal standard is positioned in the table above the set of analytes quantitated against it; chromatographic position of the internal standards are also indicated with placeholder lines in the table for reference.

[1] Under our present chromatographic conditions, m-Xylene is not separable from p-Xylene so we report them as m+p-Xylene

[2] Compounds are subject to thermal decomposition.

* Available to report by request only.

Table 2. MDLs and RLs for Target Compounds in Water by Purge-and-Trap Analysis

Compound Name	MDL[1] (ug/L)	RL[2] (ug/L)
Dichlorodifluoromethane	0.337	2
Chloromethane	0.822	2
Vinyl chloride	0.354	2
Bromomethane	1.178	2
Chloroethane	0.658	2
Trichlorofluoromethane	0.508	2
Acrolein	6.655	12.5
1,1-Dichloroethene	0.676	2
Acetone	2.652	12.5
Carbon disulfide	0.738	2
Methylene chloride	0.409	2
Acrylonitrile	2.513	12.5
trans-1,2-Dichloroethene	0.476	2
Methyl tert-butyl ether*	0.450	2
1,1-Dichloroethane	0.311	2
2,2-Dichloropropane	0.789	2
cis-1,2-Dichloroethene	0.426	2
2-Butanone	2.177	12.5
Bromochloromethane	0.410	2
Chloroform	0.340	2
1,1,1-Trichloroethane	0.706	2
Carbon tetrachloride	0.790	2
1,1-Dichloropropene	0.650	2
Benzene	0.298	2
1,2-Dichloroethane	0.378	2
Trichloroethene	0.470	2
1,2-Dichloropropane	0.285	2
Dibromomethane	0.443	2
1,4-Dioxane*	--	25
Bromodichloromethane	0.359	2
2-Chloroethylvinyl ether*^	1.057	2
cis-1,3-Dichloropropene	0.448	2
4-Methyl-2-pentanone	1.096	5
Toluene	0.350	2
trans-1,3-Dichloropropene	0.362	2
1,1,2-Trichloroethane	0.357	2
Tetrachloroethene	0.491	2
1,3-Dichloropropane	0.262	2

Table 2. MDLs and RLs for Target Compounds in Water by Purge-and-Trap Analysis (cont)

Compound Name	MDL[1] (ug/L)	RL[2] (ug/L)
2-Hexanone	1.163	5
Dibromochloromethane	0.372	2
1,2-Dibromoethane (EDB)	0.368	2
Chlorobenzene	0.329	2
1,1,1,2-Tetrachloroethane	0.284	2
Ethylbenzene	0.379	2
m+p-Xylene	0.562	4
o-Xylene	0.380	2
Styrene	0.288	2
Bromoform	0.411	2
Isopropylbenzene	0.343	2
Bromobenzene	0.382	2
1,2,3-Trichloropropane	0.378	2
n-Propylbenzene	0.343	2
2-Chlorotoluene	0.256	2
1,3,5-Trimethylbenzene	0.247	2
4-Chlorotoluene	0.244	2
1,1,2,2-Tetrachloroethane	0.587	2
tert-Butylbenzene	0.448	2
1,2,4-Trimethylbenzene	0.359	2
sec-Butylbenzene	0.373	2
1,3-Dichlorobenzene	0.472	2
p-Isopropyltoluene	0.369	2
1,4-Dichlorobenzene	0.352	2
1,2-Dichlorobenzene	0.392	2
n-Butylbenzene	0.349	2
1,2-Dibromo-3-chloropropane	0.804	2
1,2,4-Trichlorobenzene	0.398	2
Hexachlorobutadiene	0.678	2
Naphthalene	0.506	2
1,2,3-Trichlorobenzene	0.443	2
DIBROMOFLUOROMETHANE (S)	-	-
1,2-DICHLOROETHANE-D4 (S)	-	-
TOLUENE-D8 (S)	-	-
4-BROMOFLUOROBENZENE (S)	-	-

(footnotes next page)

1. MDLs were determined according to 40 CFR part 136, based on historical data on GC/MS #11 (analyzed May-November 2015, LIMS work order # 1502007, 1511004, 1511001, 1510031, 1509023, 1509013, 1504013, 1505018) and on GC/MS #13 (analyzed October 2015, LIMS work order # E151209). The greater of the two MDLs was selected for publication in this SOP (Qualtrax DOC #5619, 5/9/2016).

2. All RLs, except 1,4-dioxane, methyl tert-butyl ether and 2-chloroethylvinyl ether, were originally determined from the RL study analyzed in October 2012 (LIMS work order # 1301005, GCMS 11). RLs for methyl tert-butyl ether and 2-chloroethylvinyl ether were determined from the RL study analyzed in October 2014 (LIMS work order # 1410013, GCMS 11). The RL for 1,4-dioxane was determined from data analyzed April 2015 (LIMS work order E150320, GCMS 13) and October 2015 (LIMS work order 1510030, GCMS 11). RL's were reviewed in 4/2019 against MDLs and performance data, and were updated (refer to control charts).

3. Uncertainty: Blank and matrix spike recovery statistics were calculated in March 2019 from spike and duplicate QC samples analyzed between 1/26/2017 to 2/28/2019. Surrogate spike recovery statistics were calculated at the same time from field samples analyzed in the same time interval. No outliers were excluded; both instruments were included. Refer to the control charts for these statistics.

4. Control Charts: QC samples from 1/26/2017 to 2/28/2019 were control charted and reviewed in March 2019. Control charts and associated recovery statistics are filed in Qualtrax with the SOP (Qualtrax, Document ID are 17284). Recoveries for target compounds and surrogates were reviewed relative to the SOP's and the reference method's suggested control limits, and RLs were verified or adjusted in response to updated performance data. Surrogate spike recovery statistics were also calculated from field samples analyzed in the same time interval.

(S) = Surrogate

* Available to report by request only.

^If this compound is reported, data will be flagged below 2.5 ug/L until sufficient points are available to verify the reporting limit of 2 ug/L.

The name of this analysis in LIMS is 'VOA – 8260'.

Table 3. BFB Abundance Criteria

Mass (m/z)	Relative Abundance Criteria
50	15 - 40% of m/z 95
75	30 - 60% of m/z 95
95	base peak, 100% relative abundance
96	5 - 9% of m/z 95
173	< 2% of m/z 174
174	> 50% of m/z 95
175	5 - 9% of m/z 174
176	95 - 101% of m/z 174
177	5 - 9% of m/z 176

Attachment 1. Sample Calculations and Equations

Relative Response Factor

Calculate the relative response factor at each calibration level for each target compound and surrogate using Equation 1. The primary characteristic ions used for quantitation are listed in Table 1. Assign the target compounds and surrogates to an internal standard. If an interference prevents the use of a primary ion for a given internal standard, use a secondary ion.

Equation 1:

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

A_x = Area of the primary characteristic ion for the compound to be measured
A_{is} = Area of the primary characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard
C_x = Concentration of the compound to be measured

Average Relative Response Factor

Calculation of the average of the RRFs is shown in Equation 2.

Equation 2:

$$RRF_{avg} = \frac{\sum_{i=1}^{n} RRF_n}{n}$$

RRF_n = Relative response factor for each initial calibration standard
n = Number of calibration standards

Percent Relative Standard Deviation

Equation 3 is used to calculate the %RSD of the RRF values over the working range of the curve.

Equation 3:

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}}$$

x_i = Each individual value used to calculate the mean
n = The total number of values
SD = Standard deviation
$\bar{x}$ = The mean of n value

Percent Difference

The percent difference between the calculated and expected concentration in the continuing calibration verification standard is calculated using Equation 4.

Equation 4:

$$\%D = \left(\frac{calculated\ concentration - expected\ concentration}{expected\ concentration}\right) \times 100$$

Concentration in Water Samples

Calculations for the different calibration models for an analyte in a water sample:

Equation 5:

RRF_{avg} calibration model for water:

$$C_x = \frac{(A_x)(C_{is})(D_f)}{(A_{is})(RRF_{avg})(V_o)}$$

Equation 6:

Linear regression calibration model for water:

$$C_x = \frac{(A_x)(C_{is})(D_f)}{(A_{is} + y_{int})(m)(V_o)}$$

Equation 7:

Quadratic calibration model for water:

$$C_x = \frac{\left(-b \pm \sqrt{b^2 - 4a(c - \frac{A_x}{A_{is}})}\right)(C_{is})(D_f)}{(2a)(V_o)}$$

A_x = Area of the primary characteristic ion for the compound to be measured
A_{is} = Area of the primary characteristic ion for the specific internal standard
a = X-squared coefficient from the least squares fit quadratic regression
b = X coefficient from the least squares fit quadratic regression
c = Constant term from the least squares fit quadratic regression
m = Slope from the least squares fit linear regression
y_{int} = Y-intercept from the least squares fit linear regression
C_{is} = Concentration of the specific internal standard
C_x = Concentration of the compound to be measured (ug/L for water)
V_o = Volume of sample purged in mL
D_f = Dilution factor

Percent Recovery

Calculate the recovery of an analyte using Equation 8.

Equation 8:

$$\%\,recovery = \frac{calculated\ concentration}{spiked\ concentration} \times 100$$

Matrix Spike/Matrix Spike Duplicate % Recovery

Calculate the recovery of each matrix spike compound as follows in Equation 9.

Equation 9:

$$^{MS}/_{MSD}\ \%\,recovery = \frac{SSR - SR}{SA} \times 100$$

SSR = MS/MSD spiked sample result
SR = Unspiked sample result
SA = Spike concentration

Relative Percent Difference

RPD of the concentrations of each compound in the matrix spike and matrix spike duplicate is shown in Equation 10.

Equation 10:

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

MSC = Matrix spike concentration
MSDC = Matrix spike duplicate concentration

Internal Standard Calculations

Percent area response change between the sample and the most recent mid-level calibration standard for each of the internal standards is shown in Equation 11.

Equation 11:

$$\%ARC = \frac{A_s - A_c}{A_c} \times 100$$

%ARC = Percent area response change
A_s = Area response of the internal standard in the sample
A_c = Area response of the internal standard in the most recent mid-level calibration standard

Retention time shifts between the sample and the most recent mid-level calibration standard for each of the internal standards is shown in Equation 12.

Equation 12:

$$RTS = RT_s - RT_c$$

RTS = Retention time shift
RT_s = Retention time of the internal standard in a sample
RT_c = Retention time of the internal standard in the most recent mid-level calibration standard

<u>Regression Equations</u>

Equation 13:

Linear regression:

$$y = mx + b$$

y = Response or relative response
x = Concentration or relative concentration
m = Slope from the least squares fit linear regression
b = Y-intercept from the least squares fit linear regression

Equation 14:

Quadratic regression:

$$y = ax^2 + bx + c$$

y = Response or relative response
x = Concentration or relative concentration
a = X-squared coefficient from the least squares fit quadratic regression
b = X coefficient from the least squares fit quadratic regression
c = Constant term from the least squares fit quadratic regression

Equation 15:

Coefficient of determination for regression:

$$r^2 = \frac{SST_y - SSE_y}{SST_y}$$

r^2 = Coefficient of determination
SST_y = Total sum of squares with respect to y = $\sum(y_i - y_{avg})^2$
SSE_y = Error sum of squares with respect to y = $\sum(y_i - y_{hat})^2$
y_i = Measured response or relative response
y_{avg} = Mean response or relative response
y_{hat} = Calculated response or relative response based on x value and the least-squares fit regression coefficients

Attachment 2. Deviations from SW-846 Method 8260C

Section 1.1 and Tables 1 and 2. This SOP determines only a subset of the target compounds documented in 8260C sections 1.1 and 1.3 as appropriate for analysis by purge & trap of aqueous samples.

Section 9.3.3.1. This SOP provides two choices for demonstrating the BFB tune check: (a) direct injection of 1 uL 2000 ng/uL BFB stock in MeOH at a 400:1 split, or (b) analysis of a 5-mL instrument or vial blank (for initial BFB tune) or any blank or non-MS/MSD QC sample (for continuing BFB checks) containing 10 ng/mL BFB at 150:1 split. Both choices introduce ≤ 50 ng into the MS as specified by 8260C 11.3.1 and as clarified by 8260D 11.3.1.1, and both use identical MS conditions which also match those used for samples. Method 8260C neither allows nor disallows direct injection of BFB from a concentrated stock. This procedure offers the flexibility of two options because direct inject provides more rapid feedback and marginally better tune performance, while use of blanks or QC samples is completely automated and enables continued verification of the BFB tune during extended and overnight sequences.

Sections 9.4.1 and 10.2.9. This SOP does not explicitly check the ICAL response factors of the target compounds against the minimum response factors listed in Table 4 of Method 8260C, as described in 8260C 11.3.4.1. The minimum RRFs in the method are 11 years old and MRL checks are a better, more modern, and more applicable approach to verifying sensitivity. The RRFs for each ICAL point are documented in standard data package reports.

Sections 9.4.2 and 10.2.10. For continuing calibration verification, this SOP uses an acceptance limit for percent difference between calculated vs. expected concentration of 30% or less, not 20% or less as suggested in 8260C 7.7.5.2. The choice of 30% is based on historical performance (see control charts) which show that a 20% difference is too narrow, given the chromatographic conditions in use. A 20% criterion may be more appropriate if ASB was running a longer column with a thicker film and slower temperature program.

Sections 9.4.2 and 10.2.10. For evaluation of the CCV relative to the midrange ICAL, this SOP calculates the concentration-based percent difference per Equation 4 of Attachment 1, regardless of which calibration model is used to calculate a given target compound's concentration. In contrast, 8260C 11.4.5.3 suggests use of RRF-based percent difference, which is functionally equivalent to our calculation, for compounds relying on average RRF, but uses percent drift calculations for compounds using linear or quadratic regressions. ASB uses RPD because the overwhelming majority compounds rely on RRF calibration models, so the calculation is chosen to fit the rule rather than the exception.

Section 9.5.1.2. This SOP does not explicitly monitor relative retention time of each analyte as suggested in 8260C (e.g.,11.3.5). Instead, the absolute retention time of an analyte is to fall within ± 10 seconds of the reference compound's retention time. Absolute RT gives more information sooner about when the system performance begins to degrade; it is a more sensitive measure than relative retention time.

Section 10.2.2. This SOP does not explicitly monitor whether area counts of the internal standard peaks are between 50-200% of the area counts *of the target analytes* in the mid-level ICAL, as suggested in 8260C 7.10. This check is duplicative because, for the midlevel ICAL, the relative response factor itself is a fundamental measure of relative area counts, and RRF's are already monitored as a separate QC check. Area counts of internal standard peaks including midrange as well as all other ICALs, CCV's and individual injections are also monitored in the injection log, which documents whether or not the IS responses in the midlevel ICAL are appropriate.

Attachment 3. Field Sample Screening Procedure

This attachment outlines the procedure for screening samples that are suspected to contain target analytes above the standard ICAL range. The importance of screening cannot be overemphasized. System contamination from high-concentration samples causes down time and can be difficult to remediate. As a rule of thumb, all samples are screened, except trip blanks. A dilution factor of 100x or 50x is a reasonable starting point, unless the sample don't appear like normal water (i.e. oily, smelly, bi-phasic, cloudy, etc.) The data produced by this screening procedure is not intended for official reporting, but, rather, to be used at the discretion of the analyst to determine whether sample dilution is necessary for official analysis.

The described preparation steps below are not absolute requirements. Rather, the prepared calibration concentrations and volumes may be tailored to the needs of the project with consideration given to the nature of the samples and any interferences present therein.

If needed, an initial calibration may be prepared according to Section 7.5, but using the following condensed spiking scheme

	Level 1 *100 ng (20 ppb)*	**Level 2** *500 ng (100 ppb)*	**Level 3** *1000 ng (200 ppb)*
50 ug/mL spiking solutions	40 uL	200 uL	400 uL

The following tables outline the corresponding calibration ranges for the analytes that have variable concentrations in the 50 ug/mL spiking solutions. See the notes under Sections 7.3.3 and 7.3.7 for further details.

250 ug/mL concentration in 50 ug/mL spiking solution	**Level 1**	**Level 2**	**Level 3**
Acrolein **Acetone** **Acrylonitrile** **2-Butanone**	500 ng (100 ppb)	2500 ng (500 ppb)	5000 ng (1000 ppb)

100 ug/mL concentration in 50 ug/mL spiking solution	**Level 1**	**Level 2**	**Level 3**
4-Methyl-2-pentanone **2-Hexanone** **m+p-Xylene**	200 ng (40 ppb)	1000 ng (200 ppb)	2000 ng (400 ppb)

In addition to a method blank, instrument or vial blanks may be needed after each field sample to minimize cross-contamination between screenings. The dilution factor is chosen at the discretion of the analyst. For screening purposes, a higher dilution factor is more ideal. A reagent water reservoir blank will need to be analyzed as part of the method blank QC as well.

The purge-and-trap and GC/MS instruments are prepared as described in Sections 9.2 and 9.3. The calibration curves for each analyte are constructed as described in Section 9.4.1. Once the system has been tuned and calibrated, the field samples may be screened.

Attachment 4. Preparation Procedure of Pre-Acidified Vials for Field Sampling

This attachment outlines the procedure for preparing pre-acidified vials for field sampling. Pre-acidified vials are available to clients upon request.

A solution of 1:1 hydrochloric acid and reagent water may be prepared by carefully adding 20 mL of concentrated HCl to 20 mL of reagent water in a 40 mL VOA vial. A specific, consistent volume of 200-500 µL of the 1:1 solution may be added to each 40 mL VOA vial that is to go into the field. Measurements can be made with mechanical or volumetric pipettes.

Trip blanks may also be prepared, if requested, by filling a pre-acidified VOA vial to zero headspace with reagent water. Label as an acidified Trip Blank from EPA ASB. Prep dates may cause confusion for samplers; they are not recommended to be included. Ensure the Sample Custodian has the vials before the scheduled pick-up or ship-out.

Attachment 5. VOA Data Package Components
(based on GEN032)

Section	Order	Component
1 (Summary Report)	A	eLDP Cover page [signed]
	B	Data verification checklist [signed; see MS037]
	C	WO .PDF form
	D	Final LIMS Level II report with approved narrative [signed]
	E	Client communications (e.g. CCs, IRs or new P&Is sent to the client)
2 (Instrument Acquisition and Performance Data)	A	Mass spectrometer performance check(s) (BFB tune check; air/water check optional)
	B	Injection log.
	C	Bench sheets (LIMS or printed & scanned)
	D	GCMS and Purge & Trap instrument sequences
2B (Calibration)	A	Calibration summary reports (required for all packages, regardless of whether calibration is original or referenced) • Response Factor report with RF, Avg and %RSDs • Calibration Factor with fit, constants and RSD/Cf • Calibration history with ng at each cal level recommended.
	The following are required for original calibrations. For referenced calibrations, document in the narrative the package (project, WO) where the calibration is reported.	
	B	Quantitation reports for each calibration level and for blank(s) • Quant reports for all cal levels including cal blank • Detailed reports for cal blank, lowest cal level (original or -reprocessed as -MRL1), and the cal level used to set the reference spectra (original or re-processed as -BS1)
	C	ICV evaluation and quant report(s)
	D	CCV1 evaluation and quant report(s) (method performance check)
	E	Reprocessed -MRL evaluation and quant report(s) (ICAL level 1 and additional levels if needed)
	F	Documentation for manual integrations

(con't next page)

3 (QC Data)	A	Method blank quant report(s) [detailed, all analytes, IS & SURR]
	B	Reported storage blank quant report(s) & dilution blanks if needed (e.g, TCLP) (Include instrument & vial blanks only where needed to document a lack of carryover contamination)
	C	BS/BSD evaluation and quant report(s)
	D	Documentation for manual integrations
4 (Sample Data)	A	Quant reports of all reported samples [detailed; all analytes, IS & SURR]
	B	MS/MSD and/or duplicate quant report(s) [detailed; all analytes, IS & SURR]
	C	Documentation for manual integrations
5 (Supporting Information) *If applicable*	A	Supporting instrument data not included in final reporting
	B	Standard preparation records [optional]
	C	Acquisition method instrument control parameters [optional]
	D	Other applicable records of original observation (e.g. balance log, TCLP worksheets, logbook copies etc.)
	E	Analyst notes
	F	Existing Pen & Inks (include other applicable P&Is for the SOP revision used)

Attachment 6. QC Criteria Summary

QC Parameter	Frequency	Criteria
Technical Holding Times & Sample Preservation	N/A	Preservation: ≤ 6°C, pH < 2 Holding time for acidified samples: 14 days Holding time for non-acidified samples: 7 days
GC/MS Instrument Performance Check	Every 12 hours	BFB ion ratios meet criteria in Table 3
Initial Calibration	As necessary, when midlevel CCV criteria not met	RRF_{avg} %RSD ≤ 20% Linear/quadratic regression r^2 ≥ 0.99, weighted 5 points minimum for RRF_{avg} or linear curves 6 points minimum for quadratic curves ≥ 90% of TCs meet ICAL criteria ICV %D ≤ 30%
Mid-level Continuing Calibration Verification (LIMS -BS, -BSD)	Every 12 hours and at the end of a sequence	Limit: concentration is 70-130% of expected ≥ 80% of TCs in opening BS meet criteria
Blank Spikes (LIMS -BS, -BSD)	BS/BSD per batch	See midlevel calibration verification limits as above (70-130% recovery) Relative percent difference RPD ≤60%≥ 80% of TCs meet opening CCV criteria
Internal Standards	Each ICAL standard, field and QC sample	IS area of samples within 50% – 200% of most recent mid-level calibration standard area IS RT of samples within ± 10 seconds of most recent mid-level calibration standard RT IS area of CCVs within 50% – 200% of mid-level ICAL standard area or mean of ICAL standard areas IS RT of CCVs within ± 10 seconds of mid-level ICAL standard RT
Surrogate Spike Recovery	Each field and QC sample	Recovery within 84% - 122% for 1,2-Dichloroethane-d4 84 - 108% for 4-Bromofluorobenzene 73 - 124% for Dibromofluoromethane 88 - 108% for Toluene-d8 2 of 4 surrogates meet recovery limits
Matrix Spikes (LIMS -MS, -MSD)	MS/MSD per batch	Recovery within 60% – 110% Relative percent difference RPD ≤60%
Method Blanks (LIMS -BLK)	At least one per batch	Analyte sample concentration > 5x the concentration in the corresponding blank(s). LIMS “Q” on BLK detections > RL.
RL Checks (LIMS -MRL, -CCV1)	1 each per calibration	-MRL recovery within 50% – 150%, failures result in qualification or a raised reporting limit for all the data produced from the ICAL ≥ 80% of TCs meet criteria -CCV1, chart annually vs 50-150% limits

STANDARD OPERATING PROCEDURE FOR THE MEASUREMENT OF LOW-LEVEL 1,4-DIOXANE IN WATER BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY USING SELECTED ION MONITORING

ASB SOP MS035
(based on SW-846 method 8270E, 8000D, and
EPA Office of Research and Development method 522)

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5 ANALYTICAL SERVICES BRANCH LABORATORY
536 SOUTH CLARK STREET (LAB-10C)
CHICAGO, ILLINOIS 60605

SECTION Page #

1. SCOPE AND APPLICATION 4
2. SUMMARY OF METHOD 5
3. ABBREVIATIONS AND DEFINITIONS 5
4. HEALTH, SAFETY, AND WASTE HANDLING 7
5. CAUTIONS AND INTERFERENCES 8
6. EQUIPMENT AND SUPPLIES 9
6.1. GC/MS system 10
6.2. SmartPrep™ Extractor System 10
6.3. Vacuum Manifold Extractor System 11
6.4. Glassware 11
6.5. Other equipment and supplies 11
6.6. Calibrated support equipment 12
7. REAGENTS AND STANDARDS 12
7.1. Record keeping and labeling 12
7.2. Reagents 12
7.3. Standards 12
7.4. Preparation of standards 13
8. SAMPLE HANDLING AND PRESERVATION 16
9. SAMPLE PREPARATION AND ANALYSIS 16
9.1. Procedural quality controls for sample preparation 16
9.2. Extraction of aqueous samples 18
9.3. Drying extracts 21
9.4. Gas Chromatography / Mass Spectrometry 21
10. QUALITY CONTROL 26
10.1. Surrogate 26
10.2. Method blanks 26
10.3. Reporting Limit verification 27
10.4. Blank spike (BS) 27
10.5. Matrix spike / matrix spike duplicate (MS/MSD) 27
10.6. Instrument performance check 27
10.7. Initial calibration 28
10.8. Continuing calibration verification 28
10.9. Internal standards 28
11. DATA AND RECORDS MANAGEMENT 29
11.1. Data package assembly 29
11.2. Other records 29
11.3. LIMS data entry and reporting 30
12. TROUBLESHOOTING 31
13. PREVENTIVE MAINTENANCE 32
13.1. Gas chromatograph and inlet: 32
13.2. Mass spectrometer: 32
13.3. SmartPrep™ Extractor Module: 33
14. REFERENCES 34
15. REVISION HISTORY 35

16. APPENDICES.....36

Table 1: Method Analyte CAS Numbers, MS Ions, and GC Retention Times.36

Table 2: Spike Recovery Limits and Associated Statistics for 1,4-dioxane (Target Analyte) and 1,4-dioxane-d8 (Surrogate) using SPE 37

Table 3. BFB KEY IONS AND ION ABUNDANCE CRITERIA[a] 38

Table 4. Summary of QC criteria used for this SOP 39

Attachment 1. Sample Calculations and Equations 41
Attachment 2. Deviations from Reference Methods 46
Attachment 3. Data Package Components 47
Attachment 4. SmartPrep™ Extraction Method 49

1. SCOPE AND APPLICATION

1.1. Applications of this SOP

This SOP describes procedures for the preparation and analysis of low-level 1,4-dioxane in water (non-drinking water) matrices using gas chromatography/mass spectrometry (GC/MS) in selected ion monitoring (SIM) mode. Samples are extracted using solid phase extraction (SPE). The extraction and analysis techniques are based on SW-846 method 8270E, 8000D and EPA Office of Research and Development (ORD) method 522. There are minor modifications from the reference methods in this SOP, which are summarized in Attachment 2 of the Appendix. The purpose of this method is to identify, detect, and quantitate the volatile organic compound 1,4-dioxane in water at levels at or below current guidance and regulatory standards. This analysis is outside the scope of ASB's accreditation.

1.2. Target analytes

The target analyte 1,4-dioxane is qualitatively and quantitatively determined by this SOP.

1.3. Sample matrices

This procedure is used for preparation and analysis of water samples, excluding drinking/tap water samples that are chlorinated. If samples are collected from chlorinated sources, then a Pen and Ink will need to be written in order to dechlorinate the water samples before extraction and analysis.

1.4. Calibration range

The calibration range for this procedure may vary depending on the data quality and sensitivity needs for the project, but an initial calibration range is 0.2 - 40 µg/L in a 250 mL sample, and provided in Sec. 7.4.8.

1.5. Reporting limits

The RL for the target analyte extracted from 250 mL of water and 10 mL final volume is listed in Table 2. The RL is set to the first level calibration standard of 5 µg/L which is equivalent to 0.2 µg/L in the sample. The RL for a specific water sample may differ from that listed depending on the amount of sample tested, dilutions, and volume of the extracts. Analytes are not reported to a method detection limit (MDL) unless specifically requested. The MDL study and RL determination for SIM was completed in work order 1702004 from GC/MS#16 in February 2017, Qualtrax workflow 9614.

1.6. Measurement uncertainty

Statistics describing the variability in 1,4-dioxane LCS recoveries are provided in Table 2, and these statistics are used to provide estimates of measurement uncertainty for concentrations generated with this SOP. The statistics included in Table 2 were derived from P&A data and will be updated when more data are available. Uncertainty estimates based on variability in LCS recovery are conservative, however, because some relevant sources of variability are not included (e.g., matrix effects). The uncertainty will be greater near the reporting limit and much greater near the detection limit. Control limits will be updated biennially if sufficient data are available and are initially set to default values herein. The uncertainty was calculated and included in work order 1702004 from GC/MS#16 in February 2017, Qualtrax workflow 9614.

1.7. Use of data generated with this SOP

This SOP will be used for all programs except those subject to regulation under the Clean Water Act (i.e., NPDES permit enforcement and compliance monitoring) and the Safe Drinking Water Act. Although, depending on the data quality objectives for the project, this SOP can be used for screening for these programs.

2. SUMMARY OF METHOD

For this operating procedure, two techniques are used for the extraction of low-level 1,4-dioxane from water: automated SPE and manual (vacuum manifold) SPE. Sample extracts are dried with sodium sulfate. Sample extracts are not concentrated. Extracts are analyzed by gas chromatography / mass spectrometry in SIM mode.

3. ABBREVIATIONS AND DEFINITIONS

ADOC – Analyst demonstration of capability
ASB – Analytical Services Branch
BFB – 4-bromofluorobenzene
BS – Blank spike
CAS – Chemical Abstracts Service
CCV – Continuing calibration verification
CFR – Code of federal regulations
CRL – Chicago Regional Laboratory
DCM – Dichloromethane (methylene chloride)
EDD – Electronic data deliverable
EI – Electron ionization
EPA – Environmental Protection Agency
GC – Gas chromatography or gas chromatograph
GC/MS – Gas chromatography/mass spectrometry
ICAL – Initial calibration
ICV – Initial calibration verification
ID – Identification number
i.d. – Internal diameter
IDOC – Initial demonstration of capability
IS – Internal standard
ISO – International Organization for Standardization
L – Liter
LCL – Lower control limit
LCS – Laboratory control sample
LCSD – Laboratory control sample duplicate
LIMS – Laboratory Information Management System
MB – Method blank
mL – milliliter
MS – Matrix spike
MSD – Matrix spike duplicate
m/z – Mass-to-charge ratio
ORD – EPA's Office of Research and Development
OSHA – Occupational Safety and Health Administration
P&A – Precision and accuracy

%D – Percent difference
%RSD – Percent relative standard deviation
PFTBA – Perfluorotributylamine
p/n – Part number
PPE – Personal protective equipment
ppm – Parts per million
psi – Pound per square inch
PTFE – Polytetrafluoroethylene
QA – Quality assurance
QC – Quality control
QMP – Quality Management Plan
r^2 – Coefficient of determination
RL – Reporting limit
RPD – Relative percent difference
RRF – Relative response factor
RRF_{avg} – Average relative response factor
RT – Retention time
SDS – Material safety data sheet
SIM – Selected ion monitoring
SOP – Standard operating procedure
SPE – Solid phase extraction
TC – Target compound
TIC – Tentatively identified compound
UCL – Upper control limit
µg – microgram

Batch – A group of ≤ 20 samples processed as a unit and prepared using the same reagents, standards, equipment, and method. Each group of 20 samples contains the procedural quality controls outlined in this SOP.

Blank spike (BS) or Laboratory Control Sample (LCS) – An aliquot of reagent water to which known quantities of method analytes are added in the laboratory. The LCS is analyzed exactly like a field sample (including added field preservative), following the same preparation steps. The purpose is to determine whether the methodology is in control and whether the laboratory is capable of accurate and precise measurements.

Continuing calibration verification – A standard prepared near the midpoint of the calibration range and analyzed every 12 hours after initial calibration during analysis to demonstrate the initial calibration is still valid while field samples and batch QC samples were analyzed.

Duplicate – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical sample procedures. Duplicates are not required in this SOP, but may be used at the discretion of the analyst (e.g. used with a single matrix spike when insufficient volume is provided by client to analyze a matrix spike/matrix spike duplicate pair).

Initial calibration – A set of prepared standards diluted from a stock solution in a range of concentrations and used to calibrate the instrument response relative to the selected internal standards with respect to known analyte concentrations.

Initial calibration verification – A standard solution of target analytes from a different vendor than the primary standard used for instrument calibration that is prepared at known concentration analyzed after the initial calibration standards to demonstrate that the calibration functions generate analyte concentrations within a specified tolerance of the certified concentrations.

Matrix spike – An aliquot of an environmental sample to which known quantities of method analytes are added in the laboratory. The matrix spike is analyzed exactly like a sample with all preparation steps that the parent sample received. The purpose is to demonstrate the analytes of interest can be recovered in the sample matrix when a known amount is added. Background concentrations of analytes in the sample matrix determined in the field sample chosen for matrix spiking and used to account for any analyte present prior to calculating recovery.

Method blank – An aliquot of reagent water prepared with a batch of field samples and exposed to all glassware, equipment, solvents, reagents, internal standards, and surrogates used for the batch. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

Method detection limit – The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

Quantitative Transfer – Transfer of a sample or extract from one container to another followed by solvent rinsing of the original container in order to avoid losing any residual chemical that may remain in the original container. The rinse solvent must be of similar composition to the extraction solvent(s) or to the targeted solvent composition of the extracts when they are analyzed. The container rinse is either added to the bulk extraction solvent for the individual sample immediately or is reserved and combined with the sample extract at a later step. Quantitative transfer is only necessary when the entire contents of the original sample container are transferred to a new container, and in some cases, it may be omitted in order to achieve a specified extract volume in a graduated container.

Reporting limit – The lowest concentration of an analyte reported by the ASB except in the case of a special request (see MDL). This is typically the lowest point of calibration.

Reporting limit verification – A low level LCS used to evaluate analyte recovery at target analyte concentrations near or at the RL.

Work order – A group of samples arriving at the ASB on the same day from the same sampling project.

4. HEALTH, SAFETY, AND WASTE HANDLING

For a comprehensive guide to health and safety in the ASB, please see the ASB Chemical Hygiene Plan, with which this procedure complies.

4.1. Safety Data Sheets

SDSs are available online. See the ASB Chemical Hygiene Plan for more information. Review SDSs for specific physical and health hazards including appropriate PPE to be used. SDSs may be accessed at chemical manufacturers' website(s). Alternatively, other vendor websites may be used to locate pertinent information.

4.2. Health hazards

The toxicity and carcinogenicity of each reagent used in this SOP have not been precisely defined. However, each chemical compound and solvent is treated as a health hazard. 1,4-Dioxane and methylene chloride are currently labeled by EPA as likely to be carcinogenic to humans and classified

as Class B2. Exposure to these chemicals should be reduced to the lowest possible level and the appropriate PPE must be utilized.

WARNING – Extreme caution shall be used with all solvents, target analytes, and surrogates as they are likely to be carcinogenic. Safety must be a primary consideration when handling and preparing chemical standards associated with this SOP.

4.3. Waste handling and pollution prevention

4.3.1. Waste streams

For liquid waste carboys, attach the appropriate color tag to identify its contents. Written information must include which waste stream it belongs to ("Green Label"), the date waste collection began, and the date the container was filled. Use a secondary container for carboys and hazardous waste bags in the event that the container fails, and make sure that incompatible wastes (e.g., orange label and green label) are not stored in the same secondary container.

4.3.1.1. Green Label – Organic solvent waste containing PCBs < 50 mg/L, and aqueous solutions that have been in direct contact with dichloromethane. This waste stream is appropriate for all wastes produced with this SOP except for samples/extracts known to contain PCBs > 50 mg/L concentration and for concentrated aqueous acids and bases. If the carboy is used for disposal of vials that still contain organic solvent, write "FOR VIALS ONLY" on the carboy, and do not dispose of bulk liquid wastes in this container.

4.3.1.2. Yellow Label – Appropriate for dilute aqueous waste containing sulfuric acid with pH<2. This waste stream must be used for the sample waste line for the SmartPrep™.

4.3.1.3. Used vials are emptied of their content and disposed of in an approved broken glass box along with all other glass waste. The vial caps are discarded in a regular trash bin. All aqueous organic solvent waste created in this SOP is appropriate for disposal in a green-labeled waste carboy.

4.3.2. Sample and extract disposal

After final data have been generated regarding particular samples and disclosed to the relevant parties, vials containing all field sample and QC sample extracts are stored for sixty days, unless otherwise requested.

LIMS disposal records should be updated for consumed, and/or disposed of standards (e.g. spiking solutions, calibration standards etc). To update records in LIMS, go to Laboratory->standards and check box the disposed of box and set the standard type to inactive.

4.3.3. Additional information on waste reduction can be found in the ASB Environmental Compliance Plan and additional information. Response and reporting guidance on hazardous waste spills can be found in the ASB Hazardous Materials/Hazardous Waste Contingency Plan. Report all major spills according to the guidance found there.

5. CAUTIONS AND INTERFERENCES

5.1. Cautions and warnings

Cautions and warnings are included throughout this SOP near the step in the procedure where they are most relevant. Cautions are included as considerations for the analyst that may be critical for maintaining data quality. Warnings indicate there may be a physical risk to the analyst or equipment.

5.2. Causes of interferences

Interferences can result from contaminants in improper preservation, solvents, reagents, glassware, and other apparatus and can lead to an elevated baseline or qualitatively identifiable peak in the retention time window for one or more analytes of interest. Matrix interferences can also be caused by contaminants that are co-extracted from the samples. Whenever an interference is encountered, the associated method blank and LCS data must be carefully evaluated to determine whether the interference is an artifact of preparation and/or cleanup procedures, or if it is co-extracted from the sample with the target analyte. If the interference is also present in the method blank QC sample(s) in the batch, take corrective action to eliminate the problem, if possible, and re-prepare the affected batch of samples. For matrix interferences, no corrective action is possible, because the interference is an integral part of the sample.

5.3. Minimization

All materials used in sample preparation, including reagents, SPE cartridges, glassware, and equipment, are routinely demonstrated to be free from interferences by analyzing method blanks under the same conditions as the samples. All glassware is cleaned according to ASB SOP GEN008. Prior to use, reusable glassware is solvent rinsed by the analysts. The use of high purity reagents, solvents, and deionized reagent water that has been filtered/purified (ultrapure water) also helps minimize interference problems.

5.4. Preventing carryover contamination

Contamination by carryover can occur whenever high concentration and low concentration samples are analyzed sequentially. After a high concentration sample (i.e. exceeding the calibration range) is analyzed, the following sample(s) must be carefully examined for evidence of cross-contamination. Whenever an unusually concentrated sample is encountered, carryover can be mitigated by the analysis of at least one instrument blank or solvent blank injection to demonstrate lack of contamination.

NOTE: During method optimization, a sample 10x the high level calibration standard was extracted and then followed by the extraction of a method blank. No carryover was detected above the reporting limit. It is recommended that a limit of 5x the high level calibration level is set as the threshold limit to initiate are-extraction of a sample that has <5x RL of 1,4-dioxane in it and follows a contaminated sample extraction. Upon review of the analytical data, the analyst's experience and professional judgment should be used to determine if re-extraction or qualification of the sample is necessary, and documented. Samples analyzed for low-level 1,4-dioxane may also have had VOC analysis requested by purge and trap. The analysts must coordinate the reporting of the 1,4-dioxane depending on sample concentration range detected at screening such that one result for 1,4-dioxane is reported for each sample from one analysis. Purge and trap results can also inform extraction/analysis sequencing by this procedure.

6. EQUIPMENT AND SUPPLIES

All of the vendors and part numbers for the equipment and supplies listed below are what the ASB currently uses as of the date of this SOP and are listed specifically to facilitate the ordering process. The mention of trade names or commercial products in this SOP is for illustrative purposes only and does not constitute an EPA endorsement or exclusive recommendation for use. Analysts are encouraged to seek out equivalent alternatives to the items listed below. These specific vendors and part numbers are provided examples and other materials of equivalent quality may be substituted without altering this SOP. Refer to ASB SOP GEN026 for instructions and analyst responsibilities when purchasing equipment and supplies.

6.1. GC/MS system

6.1.1. Gas chromatograph – Agilent model # 7890B

6.1.1.1. GC column – Restek p/n 13868, Rxi-624Sil MS, 30 m x 0.25 mm x 1.4 µm

6.1.1.2. Inlet liner – Agilent p/n G1544-80700, dual taper direct connect 4.0 mm i.d., deactivated

6.1.1.3. Inlet septum – Supelco p/n 23163, Thermogreen LB-2, 11 mm, conditioned

6.1.1.4. Inlet liner o-ring – Agilent p/n 5188-5365, non-stick, specific for flip-top inlet

6.1.1.5. GC column ferrules – Agilent p/n 5181-3323 (inlet-side) p/n 5062-3508 (mass spectrometer side), 0.4 mm i.d., 85%:15% vespel-graphite

6.1.1.6. Liquid autosampler – Agilent model # 7683B

6.1.2. Mass spectrometer – Agilent model # 5977B

6.1.3. Computer system with the Agilent Chemstation software, a mass spectral library, access to the LIMS database, and a printer.

6.2. SmartPrep™ Extractor System

6.2.1. SmartPrep™ Automated Cartridge Extractor- Horizon p/n SPE-2616 with 6mL plunger and stripper block

6.2.2. 40mL VOA Collection Rack – Horizon p/n 49-2812-02

6.2.3. 6mL Polypropylene tubes packed with 2g coconut charcoal (80-120 mesh, approximately 150 µm) – Restek p/n 26032

6.2.4. Disposable in-line 3.1-micron membrane filters – Horizon p/n 49-2814-01

6.2.5. VOA Septa Seals kit – Horizon p/n 49-2817-02

6.2.6. 6mL Plunger Seal Kit - Horizon p/n 48-2600-01 (Maintenance item)

6.2.7. Waste Block Seal Kit – Horizon p/n 48-28-34-01 (Maintenance item)

6.2.8. Syringe Pump Service Kit – Horizon p/n 48-2826 (Maintenance item)

6.2.9. 12 Port Value Service Kit – Horizon p/n 48-2825 (Maintenance item)

6.2.10. Computer system with the Horizon SmartPrep™ V3 Software and manual

6.3. Vacuum Manifold Extractor System

6.3.1. Vacuum extraction manifold with flow/vacuum control – Supelco p/n 57250-U

6.3.2. Disposable liners for SPE manifold – Supelco p/n 57059

6.3.3. Visiprep™ large volume sampler (transfer lines) – Supelo p/n 57275

6.3.4. 6mL Polypropylene tubes packed with 2g coconut charcoal (80-120 mesh, approximately 150 µm) – Restek p/n 26032

6.4. Glassware

6.4.1. Autosampler vials – 2 mL amber with PTFE-lined caps or equivalent– Agilent p/n 5190-2280 or clear 2 mL vials p/n 5182-0715. Associated blue twist caps – Agilent p/n 5185-5820

6.4.2. Pasteur pipettes – disposable borosilicate glass, 5.75” and 9”, with bulbs – Sigma Aldrich p/n CLS7095B5X and CLS7095B9, Fisher Scientific p/n 22230482.

6.4.3. VOA vials – 40 mL clear with PTFE-lined septa – Thermo p/n SB36-0040

6.4.4. Graduated VOA vials – 40 mL clear with PTFE-lined septa graduated every 5 mL – ESS p/n 0040-0330-PC

6.4.5. Volumetric flasks, class A – 5, 10, 25, or 50 mL with ground-glass stoppers

6.4.6. Sampling bottles – 250 mL amber boston round with PTFE-lined screw caps PC Class — ESS p/n 0250-0150-PC

6.4.7. Beakers – various sizes as needed

6.4.8. Mininert vials – 1.0 and 5.0 mL amber with screw cap valves

6.4.9. Funnels – various sizes

NOTE: Rinse glassware with clean solvent (methylene chloride) before use to minimize interference from contamination.

6.5. Other equipment and supplies

6.5.1. Wide-range pH paper – EMD Chemicals Inc. (Merck) p/n 9590, colorpHast pH-indicator strips (pH 0 - 14)

6.5.2. Gases – nitrogen, helium, and compressed air, ultra-high purity

6.5.3. Metal spatulas

6.5.4. Aluminum foil

6.6. Calibrated support equipment

NOTE: Refer to ASB SOP GEN026 for recertification frequency

6.6.1. Gas-tight graduated syringes – 5, 10, 25, 50, 100, 250, 500, and 1000 uL, certified

6.6.2. Analytical balance accurate to ± 0.1% of sample mass with class S weights to verify calibration.

7. REAGENTS AND STANDARDS

Refer to GEN026 for instructions and analyst responsibilities when purchasing reagents and supplies. Vendors and part numbers for equipment, supplies, and reagents listed below are provided as examples, and other materials of equivalent quality may be substituted without altering this SOP.

7.1. Record keeping and labeling

All solvents and prepared reagents are given a LIMS ID. The LIMS ID is used to track which reagents were used for preparation and analysis of a batch of samples. This LIMS ID must be placed on the container(s) in use in the lab. Reagents that require no preparation are also be tracked by LIMS ID.

7.2. Reagents

7.2.1. Reagent water – deionized water supplied by an in-house reverse osmosis water filtration system. Passing de-ionized water through a Milli-Q filtration system as well is recommended.

7.2.2. Methanol – Honeywell p/n 232-1L (**Purge and Trap grade only for this SOP.**)

7.2.3. Methylene chloride – Burdick and Jackson p/n 300-4 (pesticide quality recommended). The methylene chloride from this source is preserved with cyclohexene. The manufacturer recommends use within 12 months of manufacture to minimize acid decomposition.

7.2.4. Sodium sulfate, anhydrous, granular – used as a drying agent. ACS grade >99% that is suitable for pesticide residue analysis. Sigma Aldrich p/n 239313-2.5KG

7.2.5. Sodium bisulfate, laboratory grade – used as a sample preservative to inhibit microbial growth and potential decay of 1,4-dioxane. It is highly acidic and must be used with appropriate caution. Aqua Solutions Inc. p/n S2535-500G

7.3. Standards

7.3.1. 1,4-Dioxane stock solution – Restek standard p/n 30287, 2000 µg/mL in P/T methanol

7.3.2. 1,4-Dioxane-d8 stock solution – Restek standard p/n 30614, 2000 µg/mL in P/T methanol

7.3.3. Tetrahydrofuran-d8 (THF-d8) – Restek standard p/n 30112, 2000 µg/mL in P/T methanol

7.3.4. Instrument performance check stock solution – Accustandard p/n M-624-SS-03-10X, p-bromofluorobenzene, 2000 µg/mL in P/T methanol

7.3.5. 1,4-Dioxane (2nd source) – Accustandard p/n ALR-062S, 100 µg/mL in P/T methanol.

7.4. Preparation of standards

7.4.1. Allowed flexibility

The described preparations of the standards below are not absolute requirements. The prepared concentrations and volumes may be tailored to the needs of the project. Standard procedure is for the spiking solutions to be targeted toward the middle of the calibration range or higher for the expected final extract volume.

7.4.2. General guidance

Whenever possible, calibration standards, internal standards, and surrogates and target compound spiking solutions should be made from the same stock solution vials to minimize sources of variability.

7.4.3. Storage and shelf life

Unopened stock solutions are stored according to the manufacturer's recommendations. Open stock solutions have a shelf life of one year. Calibration standards and any other prepared solutions containing target analytes are limited to a shelf life of one year in the laboratory unless the integrity of the solution(s) is suspected of being compromised. Calibration standards and tune check standards in autosampler vials are recapped after use to prevent loss. Store prepared standards in the freezer when not in use (≤ 0°C). Store open containers of stock solutions separately from unopened vials.

7.4.4. Record-keeping and labeling

7.4.4.1. Records are required to be maintained for all stock solutions, calibration, and spiking standard preparations. LIMS entries are created prior to or during standard preparation..

7.4.4.2. For LIMS standard entries, include the LIMS IDs of any solvents or stock solutions added, the volume of each solution added, and the preparation date and expiration dates of the standard. For stock solutions, attach the manufacturer's certificate of analysis. When the entry is saved, the LIMS software will assign a unique LIMS ID to the standard and calculate a concentration of each chemical in the solution. Shared standards between ASB and ESAT must have the reciprocal LIMS ID in the LIMS entry.

7.4.4.3. Preparation of individual calibration standards as dilutions of a stock solution is documented in the standards preparation logbook, and these standards may be tracked in the instrument sequence in which the standards are used by recording the calibration standard concentration, the calibration standards stock solution LIMS ID, and the dilution date. Alternatively, each calibration standard is assigned a unique LIMS ID for use in tracking and labelling.

7.4.4.4. If space permits, label containers of prepared solutions with analyte, nominal concentration, solvent matrix, LIMS ID, dates of preparation and expiration, and name of the preparing chemist. At a minimum, include the concentration, expiration date, and LIMS ID for traceability purposes.

NOTE: There are several labelers around the labs (i.e. Dymo™, Thermo™) that are used to keep labels neat, compliant, and legible. Legible handwriting is acceptable also. Be cautious of washing away written labels with solvent during bench work.

7.4.4.5. Include LIMS IDs of the target compound and surrogate spiking solutions used for a batch of samples on the bench sheet(s).

NOTE: It is a good practice to mark the solvent level of the spiking solutions or stock solutions on the side of the vials before storing under refrigeration. That way, the next time the solution is pulled out of storage and warmed back up, it is easy to visually verify whether solvent evaporation has occurred.

7.4.5. Surrogates spiking solution

The compound typically used as a surrogate to monitor performance in this SOP is 1,4-dioxane-d8.

7.4.5.1. A surrogate spiking solution at 20 µg/mL nominal concentration may be prepared by adding a 50 µL volume of the 1,4-dioxane-d8 surrogate's stock solution at 2000 µg/mL in methanol to a 5 mL volumetric flask and diluting to volume with methanol.

7.4.6. Stock spiking solution

A target compound stock spiking solution at 1000 µg/L nominal concentration may be prepared by adding 2.5 µL of the 1,4-dioxane stock solution at 2000 µg/mL in methanol to a 5 mL volumetric flask and diluting to volume with methanol. The concentration of the stock solution may be varied at the analyst's discretion.

7.4.7. Calibration solvent blank

The calibration (solvent) blank is prepared with the methylene chloride (DCM) used for the calibration standards preparation and an aliquot of internal standard. Analysis of this blank controls (checks) for interference from reagents. Add the same amount of internal standard as used for the calibration standards (normally 2.5 µL of the 20 µg/mL standard) to 1000 µL of DCM in an autosampler vial (Section 7.4.10). This check is analyzed before the initial calibration.

7.4.8. Calibration standard solution

A calibration standard stock solution at 1000 µg/L nominal concentration may be prepared by combining 2.5 µL of the 2000 µg/mL 1,4-dioxane-d8 surrogate stock solution and 2.5 µL of the 2000 µg/mL 1,4-dioxane stock spiking solution in a 5 mL volumetric flask and diluting up to volume with methylene chloride (DCM).

The 1000 µg/L stock calibration standard solution is used to make the individual calibration standard concentration levels as described in the example table below. These levels can be adjusted to meet project needs. A minimum of six calibration points is required for a quadratic regression calibration curve, and five points for average response and linear regression models. Therefore, not all of these calibration levels need to be prepared or analyzed, as long as the reporting limit used for the samples is equal to or above the lowest calibration point. Typically, level 5 is used as the continuing calibration standard (CCV) during sample analysis. After individual calibration level preparation, each calibration level autosampler vial receives 2.5 µL of the 20 µg/mL internal standard solution (Section 7.4.10).

Calibration Level	**Calibration Standard Concentration (µg/L)**	**1000 µg/L Stock Calibration Solution Volume (µL)**	**DCM Volume (µL)**	**Equivalent Concentration in an Aqueous Sample (µg/L)**
0 (Cal BLK)	0	0	1000	0
1	5	5	995	0.2
2	10	10	990	0.4
3	15	15	985	0.6
4	20	20	980	0.8
5	25	25	975	1.0
6	50	50	950	2.0
7	100	100	900	4.0
8	250	250	750	10
9	500	500	500	20
10	1000	1000	0	40

7.4.9. Calibration standard solution (second source)

The initial calibration verification (ICV) standard is made from a stock solution containing the target analyte obtained from a separate supplier than the primary source used for instrument calibration. Alternatively, a separate lot number from the same supplier can be used in select circumstances (e.g. availability from alternate suppliers) at the discretion of the analyst. An ICV can be prepared by creating a 1000 µg/L intermediate standard by adding 10 µL of a 100 µg/mL stock solution to 1 mL of methylene chloride in an autosampler vial. The addition of 25 µL of this intermediate standard to 975 µL of methylene chloride using a gas-tight syringe would result in a ICV concentration of 25 µg/L. Add the same amount of internal standard as used for the calibration standards (normally 2.5 µL of the 20 µg/mL standard) to the final ICV standard (Section 7.4.10).

7.4.10. Internal standard

The compound used as an internal standard for both the surrogate and target analyte in this SOP is tetrahydrofuran-d8 (THF-d8). An internal standard spiking solution of THF-d8 at 20

µg/mL nominal concentration is made by adding 50 µL of the internal standard stock solution at 2000 µg/mL in methanol or methylene chloride into a 5 mL volumetric flask and diluting to volume with methanol. Addition of 2.5 µL of this 20 µg/mL solution to each 1 mL calibration standard, CCV and ICV; and 25 µL in each 10 mL sample extract would result in a nominal concentration of 50 µg/L.

7.4.11. Instrument performance check standard

The instrument performance check standard must include 4-bromofluorobenzene (BFB). The mass spectrum of BFB is checked against the tune criteria listed in Table 3. An instrument performance check standard at 1000 µg/L concentration may be prepared by adding 2.5 µL of the instrument performance check stock solution at 2000 µg/mL and diluting to volume in a 5 mL volumetric flask with DCM. An aliquot is then transferred into an auto sampler vial.

7.4.12. Batch QC sample preparation

Refer to Sec. 9 for detailed description by preparation method.

8. SAMPLE HANDLING AND PRESERVATION

This procedure is based on a 250 mL sample size, but less can be used as long as the project required reporting limit can be met (e.g. using 100 mL initial volume would raise the reporting limit to 0.5 µg/L). Grab samples are collected in 250 mL pre-cleaned amber glass containers with PTFE-lined caps. Conventional sampling practices are followed. Automatic sampling equipment should limit use of Tygon tubing and other potential sources of contamination.

All samples are iced or refrigerated at ≤ 6°C from the time of collection until extraction. Samples arriving at the laboratory on the day of sampling may not have had time to achieve a temperature of less than 6°C. This is acceptable as long as the cooling process has begun.

Field samples should be preserved with sodium bisulfate in the field. This is used as a microbial inhibitor. Approximately 0.25 grams of sodium bisulfate should be added to each 250 mL amber container when provided to the client. This should decrease the pH to ≤ 2. Sample pH are verified during sample login procedures. If the pH is noted at login to be greater than 2, then the holding time will decrease from 28 days to 7 days.

At the laboratory, sample extracts are stored in the refrigerator at ≤ 6°C until they are analyzed but must not be frozen. After injection into the GC/MS, the vial septa are to be replaced on the extract vials before putting them back in refrigerated storage. All extracts must be labelled.

Water samples must be extracted within 28 days of collection and extracts must be analyzed within 28 days after sample extraction, if quantitative data are to be reported unqualified. This 28-28 holding time is only applicable to samples that were properly preserved with sodium bisulfate to pH ≤ 2. If the pH is noted to be greater than 2 at login, then the samples need to be extracted and analyzed within 7 days in order for data to not be qualified.

NOTE: This procedure is for non-chlorinated water samples (e.g. not drinking water collected from municipal water sources). Chlorination breaks down 1,4-dioxane. Therefore, if a client is requesting analysis on chlorinated water, then a pen and ink will need to be written to include procedure for dechlorination during sample collection.

9. SAMPLE PREPARATION AND ANALYSIS

Details on supporting equipment (i.e. SmartPrep™ and GC/MS) are noted in this section.

9.1. Procedural quality controls for sample preparation

Refer to Section 10 of this SOP and to the data review SOP (ASB SOP MS037) for guidance on immediate corrective actions when any of the parameters described below do not meet the performance criteria described in this SOP. Generally sub-sampling should not be required.

9.1.1. Sample preservative: The preservative sodium bisulfate used in the field samples must also be used for batch QC samples. Approximately 0.25 grams of sodium bisulfate must be added to each 250 mL amber container already containing water. This decreases the pH to < 2. Once the preservative has been added, cap the amber bottle and gently invert the bottle to dissolve the preservative. This must be followed by checking that the pH to ≤ 2 using pH paper. Once the pH has been verified, then the surrogate and target analyte spiking solutions can be added.

9.1.2. Surrogate spikes: The surrogate 1,4-dioxane-d8 will be added to all field and QC samples in order to evaluate performance of the sample extraction procedure and (to a lesser extent) to assess whether the sample matrix causes any problems with their recovery. The surrogate must be spiked into each sample prior to extraction. The addition of 12.5 µL of the surrogate spiking solution at 20 µg/mL concentration (Section 7.4.5) to each 250 mL sample with a gas-tight syringe would result in an extract concentration of 25 µg/L assuming 100% recovery which translates to 1.0 µg/L in the sample.

9.1.3. Target compound spikes: Target compounds will be added to all RL verification (MRL), blank spike (BS) and matrix spike (MS/MSD) QC sample types in order to evaluate the integrated performance of the target analyte that is reported through all sample processing steps. The target compound is spiked into the QC samples prior to extraction, just as with the surrogate.

With each batch of 20 or fewer field samples, at least one blank spike (BS/LCS) QC sample is prepared in a clean sample matrix (reagent water) to evaluate how well the target analyte is recovered near the middle of the calibration range throughout sample processing. A graduated cylinder is used to measure out the 250 mL of deionized water used and then poured into a 250 mL amber boston round container (sample used for field samples). The addition of 250 µL of the 1,4-dioxane spiking solution at 1000 µg/L concentration (Section 7.4.6) with a gas-tight syringe would result in an extract concentration of 25 µg/L, assuming 100% recovery which translates to 1 µg/L in the 250 mL sample.

9.1.4. Reporting limit verification: With each batch of 20 or fewer field samples, prepare at least one RL verification QC sample in a clean sample matrix (reagent water) to evaluate how well the target analyte is recovered near the RL throughout all sample processing steps. A L graduated cylinder is used to measure out the 250 mL of deionized water used and then poured into a 250 mL amber. Spike the RL check QC samples with the surrogate (12.5 µL of 20 µg/mL surrogate spiking solution) and target compound (50 µL of 1000 µg/L spiking solution) per 250 mL as written.

9.1.5. Matrix spike/matrix spike duplicate: With each batch of 20 or fewer field samples, at least one MS/MSD pair is extracted and analyzed if sufficient volume is provided. MS/MSDs evaluate how well the target analyte is recovered in the field sample matrix. Spike the

MS/MSD QC samples with the surrogate and target compound at similar concentrations as the blank spike QC samples (Section 9.1.3).

9.1.6. Method blanks: With each batch of 20 or fewer field samples, prepare at least one method blank QC sample in a clean sample matrix (reagent water) to evaluate whether the sample processing steps, equipment, or reagents contribute any target or non-target contaminants. Spike method blanks only with the surrogate spiking solution prior to preparation and at the same concentration as the field samples and batch QC listed above.

9.1.7. Dilution blanks: When a sample is diluted as per section 9.4.9.4 a dilution blank must be made by using a graduated cylinder to collect 10 mL of the methylene chloride used to dilute the extract, pouring it into a clean 40 mL VOA vial and spiking it with internal standard (Section 7.4.10). Then approximately 1 mL is transferred to an auto sampler vial for analysis.

9.2. Extraction of aqueous samples

9.2.1. SPE Extraction using the Horizon SmartPrep™ SPE Cartridge Extractor Module

NOTE: Be sure to include all relevant procedural quality controls (Section 9.1). See Attachment 4 of this SOP for the details of the extraction programming. This extraction programming does not include a sample bottle rinse. The sample bottle rinse set up would have cut the sampling loading capacity in half (from 12 positions to 6). Since the IDOC demonstrated acceptable recoveries, it was apparent that the effects of the lack of quantitative transfer in this step was minimal.

9.2.1.1. Prior to using the extractor, empty any liquid in the solvent and aqueous waste reservoirs into appropriately labeled waste carboys. Fill and label all solvent/reagent bottles to be used. Additionally, rinse off the 12 samples probes with methanol and then DI water. Dry off the sample line probes with chem wipes.

9.2.1.2. Verify that the nitrogen cylinder pressure supplied to the extractor is sufficient (min 40 psi; max 60 psi). The N2P1 (Cartridge purge) pressure should be set to 5 psi and the N2P2 (Sample line purge) pressure should be set to 10 psi.

9.2.1.3. Once the pressure is turned on, turn on the extractor via the switch on the module, then open the program on the computer. The program needs to be run as an administrator (right click **.exe program file→run as administrator**).

9.2.1.4. Verify the reagent port parameters by clicking **advanced options→ set up modul**e. If any adjustments are made, be sure to **save and exit**.

9.2.1.5. A maximum of 12 samples can be set up at a time. Each 250 mL sample extraction takes approximately 55 minutes. Thus, it will likely take two sample loads/cycles for a batch of 20. Although the bench sheet will be split up due to the limited space on the extractor, as long as the reagents used are not changed or refilled, this is acceptable for this method.

9.2.1.6. Place new 6 mL coconut charcoal cartridges in positions 1→12 of the cartridge carousel and a rinse cartridge in position 21.

9.2.1.7. Place labeled 40 mL VOA vials in the collection tube rack. The number of vials needed must correspond to the number of samples/cartridges used. The VOA vials

must have the septa removed and replaced with aluminum foil. Alternately, the Horizon SmartPrep™ VOA vial caps can be used. The vials can be removed after each extraction throughout the sequence and capped in order to minimize volatilization of final extract. Do NOT try to remove the collection rack if a sample extraction is at the elution step.

9.2.1.8. Line up the 250 mL amber sample bottles next to the extractor and remove the cap. Mark the water line on each bottle so the volume can be measured later. Place a sample probe/sip tube with an attached 3.1-micron membrane filter into each sample to be extracted. Make sure everything lines up properly. For example, sample line 1 corresponds to cartridge 1 and elutes into collection tube 1.

NOTE: The SmartPrep™ has the ability to accurately measure the initial volumes. If the extractor is properly calibrated and this calibration is documented, then manually measuring out the initial sample volumes is not necessary. It is recommended though that this calibration is verified according to the user's manual and documented on the bench sheet. Additionally, it is recommended to manually verify at least one sample per batch when this automated function is used.

NOTE: The SmartPrep™ rinses sample lines in the reverse direction at the end of every extraction. This will result in a combined total of approximately 15 mL of methanol and water being squirted back into the sample bottles. This volume must be dumped into the organic waste stream.

9.2.1.9. Build the sequence in the computer software by the following steps:

9.2.1.9.1. Select the "*Prime Sample Lines*" method from the method drop down menu, and then right click pre-run. A sample number box will pop up. Type in "pre-run" in the sample ID field and press save.

9.2.1.9.2. Select the "*522_022117_FINAL 250mL 10mLmin*" method from the method drop down menu and then right click sample 1. A sample number box will pop up. Type in the sample ID according to your bench sheet. Press **Save**. Do this for all samples.

9.2.1.9.3. At the bottom of the screen fill in the report name (typically sequence ID or date) and operator (your initials).

9.2.1.9.4. Once your sequence is complete the right side of the screen tells you the approximated volume of reagents needed. Once again, verify that you have an adequate amount of reagents to proceed.

9.2.1.9.5. On the top left of the screen click **Sample set-up**. Click **Save as** to save the sequence file.

9.2.1.9.6. Once building the sequence is complete, click **Load.**

9.2.1.9.7. Click **Start** when you are ready to ready to initiate the sequence.

9.2.1.9.8. Once the sequence is complete a report is generated in the report subfolder within the program files. Include this report in the data package.

9.2.1.9.9. Discard the used cartridges into the yellow bag waste, and the excess volume in the sample containers into the organic waste stream.

9.2.1.9.10. To turn off the smart prep when a sequence is complete, first close the nitrogen gas tank. Then turn off the SmartPrep™ via the switch on the module and then close the computer program.

NOTE: The user's manual is saved on the desktop computer linked to the SmartPrep™.

9.2.2. SPE Extraction using the Supelco™ vacuum extraction manifold

Precision and accuracy using this extraction was demonstrated with data associated with Qualtrax workflow 10332, work order 1704030 using GC/MS#16.

NOTE: Be sure to include all relevant procedural quality controls (Section 9.1).

9.2.2.1. Verify flow rate

NOTE: Ensure there is a catch reservoir in place within the SPE manifold; 250 mL beakers are recommended.

Attach a rinse cartridge to the manifold, optionally with a disposable liner. Add approximately 10 mL of methylene chloride to the cartridge by continuously topping off the cartridge. Turn on the vacuum to -20 kPa. Pull the solvent through into a 40 mL graduated vial while timing it. All 10 mL should pull through in about 1 minute indicating a flow of 10 mL/min. If the flow is too fast or too slow, adjust the vacuum pressure accordingly and repeat this step.

9.2.2.2. Extraction procedure

9.2.2.2.1. With catch reservoirs in place, attach the cartridge to the SPE manifold using a disposable liner. Add approximately 3 mL methylene chloride to the cartridge, turn on the vacuum, and pull the solvent through, aspirating completely. Repeat once.

9.2.2.2.2. Fill the cartridge with approximately 3 mL methanol and elute with vacuum to just above the top frit - not allowing the cartridge to go dry at the end. From this point forward, do not allow the cartridge to go dry. Repeat twice.

9.2.2.2.3. Fill the cartridge with approximately 3 mL reagent water, turn on the vacuum, and pull the water through. Repeat 5 times, without allowing the cartridge to go dry in between washes or at the end.

9.2.2.2.4. Attach a transfer tube from each sample bottle to each cartridge and then turn on the vacuum.

9.2.2.2.5. Pass each sample through SPE cartridge at approximately -20 kPa to achieve a flow rate of approximately 10 mL/min.

9.2.2.2.6. After the entire sample has passed through each SPE cartridge, shut off the vacuum and vent. Empty the reservoir and then turn the vacuum back on and draw air through the cartridge for 10 minutes at approximately -50 kPa. Turn off and release the vacuum. Proceed immediately with cartridge elution.

9.2.2.3. Cartridge elution

9.2.2.3.1. Lift the extraction manifold top and insert a rack with 40 mL graduated VOA vials into the extraction tank to collect the extracts as they are eluted from the cartridges.

9.2.2.3.2. Fill each cartridge with methylene chloride. Pull enough of the solvent into the cartridge at low vacuum to soak the sorbent.

9.2.2.3.3. Turn off the vacuum and vent the system by opening port or lifting the top of the manifold.

9.2.2.3.4. Allow the sorbent to soak in methylene chloride for approximately 1 minute.

9.2.2.3.5. Apply a low vacuum (approximately -5 kPa) and pull the methylene chloride through the cartridge in a dropwise fashion into the collection vial.

9.2.2.3.6. Continue to add methylene chloride to the cartridge as it is being drawn through until the volume of extract is approximately 9 mL as determined by the markings on the side of the collection tube. Shut off vacuum and vent.

9.2.2.4. Cleaning sample lines

Once the collection VOA vials are removed, place the catch reservoirs back into the manifold. Attach a rinse cartridge to the sample line(s) and use the same disposable liners previously in place. Fill a clean 1 L amber bottle container with 500 mL of DI water and place the end of each sample line used into the container. Turn on the vacuum pressure at -50 kPa and pull the DI water through the transfer line(s). Once all the DI water has been pulled through, repeat this procedure using 250 mL of P/T methanol. Once complete, turn off the vacuum and vent. Sample lines are considered clean at this point. It is recommended to rotate the method blank throughout batches to verify cleanliness of sample transfer lines.

9.3. Drying extracts

9.3.1. Once the extraction is complete, the approximate volume coming off the extractor module is 9 mL. This will primarily be methylene chloride, but there will be some water present. Bring the final volume up to 10 mL by adding methylene chloride to the VOA vial and measuring from the top of the methylene chloride layer. If graduated VOA vials were unavailable, a VOA vial containing 10 mL of methylene chloride that was measured using a graduated cylinder must be used as a reference. Once the final volume is brought up to 10 mL, add 25 µL of the internal standard spiking solution of 20 µg/mL and mix by inverting and swirling the vial a few times. A vortex mixer is NOT recommended. To eliminate residual water, add approximately 2 grams of anhydrous sodium sulfate and mix by inverting and swirling the vial a few times. Transfer an aliquot of the extract to a labeled auto sampler vial and store in the refrigerator maintained at or below 6ºC until analysis.

9.4. Gas Chromatography / Mass Spectrometry

9.4.1. Suggested GC/MS operating conditions

The programs described below have been demonstrated to be acceptable for routine use. The exact GC/MS operating conditions used for a set of samples is documented in every electronic data file generated by the Agilent GC/MS data system under the file name

'acqmeth.txt'. The described inlet pressures and column flows are for a Restek GC column 624Sil MS with dimensions of 30 m x 0.25 mm x 1.4 µm df. Refer to Section 10 of this SOP and to the data review SOP (ASB SOP MS037) for guidance on corrective actions suggested when any of the parameters described below do not meet the performance criteria described in this SOP, and see Section 13 for a brief summary of recommended routine instrument maintenance tasks. The GC/MS user's manual is located on the instrument computer and in the drawer below GC/MS #16.

Suggested GC and MS Operating Conditions:

GC Inlet Program	Splitless injection
Mode	Splitless
Temperature	200°C
Program	9.07 psi (80 mL/min purge flow to split vent at 0.75 min; 1 mL/min septum purge flow)
Split Ratio	None
Gas Saver	20 mL/min at 9 min
MSD Transfer Line Temperature	200°C

GC Oven Program	Expanded List GC Run
Initial	32°C hold for 1 min
Ramp 1	7°C/min to 90°C, hold for 0 min
Ramp 2	50°C/min to 200°C, hold for 3 min
Total Run Time	15 min

MS Settings		Parameters for Expanded List GC Run
Ion source temperature:		200 °C
Gain Factor		5.0
Quadrupole Temperature		150 °C
Solvent Delay		5.3 min
SIM mode	concurrent ions per segment	8
	Dwell time (ms)	40
	spectra / sec	2.89

NOTE: Prior to analyzing extracts, fill the solvent rinse reservoirs in the autosampler and manually prime the syringe with solvent. When not being analyzed, store extracts at < 6^0C with unpierced PTFE-lined septa.

9.4.2. Example autosampler sequence:

With initial calibration in 12-hour sequence:
Instrument performance check standard (BFB)
Calibration solvent blank
Initial calibration standards (low to high concentration)
Initial calibration verification standard (2nd source)
Method blank QC sample(s)
Dilution blank QC sample (if applicable)
RL Verification QC sample(s)
LCS QC sample(s)
Field samples
Matrix spike QC sample(s)
Field samples
Ending CCV

Without initial calibration analyzed in same 12-hour sequence:
Continuing calibration verification (CCV)
Calibration solvent blank
Method blank QC sample(s)
Dilution blank QC sample (if applicable)
RL Verification QC sample(s)
LCS QC sample(s)
Field samples
Matrix spike QC sample(s)
Field samples
CCV

9.4.3. PFTBA system tuning

9.4.3.1. Before performing a new initial calibration, it is recommended to tune the mass spectrometer by running the BFB tune (BFB.u) optimization routine.

9.4.3.2. Tune reports are generated automatically after optimization routines. File these reports in the tuning binder for that instrument.

9.4.4. Instrument performance check (IPC)

Inject 1.0 uL of the instrument performance check standard solution (Section 7.4.11) using the same conditions as will be used for calibration and sample analysis. The Instrument performance check is required each time a major change to the GC/MS system, and prior to establishing and/or reestablishing an initial calibration. Re-establishing the initial calibration means that more than a week has passed since the last injection, and the IPC is reanalyzed and assessed before making additional injections. Daily analysis of the GC/MS tune check solution is not required as part of the CCV. The analyst must, however, closely monitor chromatography as well as target and IS responses in the CCV for deterioration in the system.

9.4.5. Initial calibration

9.4.5.1. The GC/MS will need to be re-calibrated if any major system maintenance is performed (e.g. changing out the column) or if initial or continuing calibration verification standards do not meet QC criteria. When performing calibration analyze calibration standards at 1.0 uL injection volume. Use the same injection volume for

samples and standards. Update the calibration method with these data files, and generate a calibration report showing the average response factors and % RSDs or regression coefficients and r^2 for all target analytes and surrogates. Evaluate the calibration fits against the acceptance criteria in Table 4, changing models or narrowing the calibration range as necessary. When narrowing the calibration range, the lowest, and/or highest calibration levels can be dropped as long as calibration level minimum requirements are still met. Reference spectra are updated per calibration method with a calibration level at or near the midpoint, and a detailed quantitation report is included in the package for the data file used. If the target analyte does not meet the initial calibration criteria, inspect the calibration data and consider either:

1) Re-injecting any individual calibration standards with anomalous results (e.g., low internal standard responses);
2) performing preventative maintenance on the system (Section 13) and re-analyzing the standards;
3) preparing and analyzing a freshly prepared set of calibration standards.

9.4.6. Initial calibration verification

Inject an ICV standard at a concentration close to the midpoint calibration standard (25 µg/L), after the initial calibration standards and before samples are run. Apply the calibration method to this sample and generate an analysis report and an evaluation report. Evaluate the percent difference between the calculated and expected concentration of each analyte against the acceptance criteria in Table 4. If the ICV injection at the beginning of an analytical sequence does not meet the %D criteria for the target analyte, re-injecting, preventive maintenance of the chromatographic system, or recalibration may be necessary.

9.4.7. Continuing calibration verification

Inject a midpoint calibration standard (25 µg/L) once at the beginning of the analytical sequence, and then every 12 hours. The sequence must also include a closing CCV at the end of the analytical run. Apply the calibration method to this sample, and generate an analysis report and a CCV report. Evaluate the percent difference between the calculated and expected concentration of each analyte against the acceptance criteria in Table 4. If the CCV injection at the beginning of an analytical sequence does not meet the %D criteria for the target analyte, re-injection, preventive maintenance of the chromatographic system, or recalibration is necessary.

NOTE: A beginning CCV injection is not needed for any 12-hour analysis window that includes the instrument performance check (BFB) and the suite of initial calibration standards. The closing CCV is not required to be injected within the 12 hours analysis window, but it is injected immediately after the last sample.

9.4.8. Sample data collection

Once acceptance criteria are met for the instrument performance check (Sections 9.4.4, 10.6), initial calibration (Sections 9.4.5, 10.7), and any continuing calibration verification (Sections 9.4.7, 10.8) injections, analyze the field samples and any associated batch QC samples.

9.4.9. Qualitative and quantitative analysis

9.4.9.1. Acquire data for at least the primary m/z and one or two other masses for each compound (see Table 1). The following criteria should be used as guidance to make a qualitative identification:

9.4.9.1.1. Ion traces for characteristic masses maximize within one scan of each other.

9.4.9.1.2. Peak apex falls within the retention time window established during initial calibration.

9.4.9.1.3. When the quantitation ion (QI) of 1,4-dioxane (*m/z* 88) is observed at the correct retention time, verify that it is truly present by verifying that the ratio of the confirmation ion (*m/z* 58) to the QI is within an absolute ± 30% of the ratio observed in a mid-range calibration standard. Ratios must be determined using the integrated areas from the QI and confirmation ion from their respective chromatograms. Using relative abundances obtained directly from the mass spectrum is not acceptable.

9.4.9.1.4. If extraneous ions are noted in the mass spectrum of the target analyte, carefully examine overlaid ion traces to aid in qualitative identification. When analytes co-elute, the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

9.4.9.2. When an analyte has been identified, calculate its amount using the response of the QI and the response and concentration of the designated internal standard. See example calculations in Attachment 1 for more information.

9.4.9.3. Review chromatographic peaks for proper integration. If the peak is integrated unacceptably by the software, manually integrate as necessary, and include screen captures or printouts of the automatic (software-generated) and manual (user generated) peak integrations with the data package, along with appropriate error codes, analyst initials, and the date they were generated. Consult the ASB SOP GEN029 for guidance on appropriate manual integration.

9.4.9.4. When sample results exceed the upper calibration limit, dilute the sample extracts with DCM, add internal standard, and reanalyze so the concentration falls within the calibration range. Since the final 10 mL extract already has internal standard in it, decrease the volume of internal standard added to the diluted extracts proportionally. For example, if you are doing a 10x dilution take 100 µL of the 10 mL extract and dilute it to 1 mL by adding 900 uL of clean methylene chloride and an additional 2.25 µL of the 20 µg/mL internal standard spiking solution (See Section 7.4.10) into an auto sampler vial. When performing dilutions, the re-analysis must include a dilution blank (Section 9.1.7).

9.4.9.5. After processing and reviewing the analytical data, generate detailed analysis reports for all reported field sample data files, and summary reports for calibration standards and all other sample types. Also, generate epatemp.txt files for each field sample and QC sample for subsequent upload into the LIMS data entry table. Epatemp.txt files can also be generated for instrument QC if the analyst is using the sequencing and calibration features in LIMS.

9.4.9.6. Concentrations of the target analyte in field samples are based on amounts of chemicals measured by the instrument, as well as on the amount of sample extracted, and extract volume. Concentrations are typically reported in µg/L for aqueous samples, without correction for recovery data.

10. QUALITY CONTROL

Analysts are responsible for performing data evaluation and verification, including manual verification of any spreadsheet calculations and adding data qualifiers when QA/QC parameters are not met. See ASB SOP MS037 for guidance on data verification and qualification. See the most current version of the ASB QMP for requirements regarding analyst qualifications to be able to use this SOP. Analytes are not reported to a method detection limit (MDL) unless specifically requested. Refer to this section, Sample Preparation and Analysis (Section 9), Troubleshooting (Section 12), and Preventive Maintenance (Section 13) for recommended corrective actions when QC criteria cannot be met. Example calculations and equations are located in Attachment 1 of the appendix.

Following is a summary of the general types of quality controls and immediate corrective actions that are employed with the preparation and analysis procedures described in this SOP, and a list of steps to take if any of the QC types is out of control. The acceptance criteria for these QC types are also summarized in Table 4. More guidance on data qualifiers recommended for exceeding acceptance criteria for various QC parameters is provided in ASB SOP MS037. The current QC limit status update is February 2017.

Refer to the QMP for instructions regarding formal corrective actions for continuing QC issues, intentional and unintentional deviations from the SOP, etc. Note: when QC excursions have occurred multiple times in a row (i.e. more than three times) for reported data, a root-cause analysis must be initiated, and any identified corrective actions must be implemented to address the cause of the non-conformance. The method of tracking continuous QC excursions is documented in ASB SOP MS037. Corrective actions may include (but are not limited to) a re-evaluation of QC limits, standards used, or extraction method modification.

10.1. Surrogate

In order to monitor for unusual matrix effects or gross sample processing errors, each field sample and QC sample is spiked with a surrogate to evaluate method performance. Percent recoveries are calculated and compared to established acceptance criteria (Table 2). If the surrogate is not recovered within acceptance limits, corrective actions include but are not limited to the following:

1) Dilute sample extracts to reduce interferences or improve chromatography and reanalyze them;
2) Qualify affected target analyte data (field samples only); or,
3) Re-prepare and re-analyze affected samples with appropriate batch QC samples.

10.2. Method blanks

In order to evaluate contamination of the reagents, standards and consumables used for preparation and analysis of a batch of samples, at least one method blank QC sample is prepared with each batch of 20 or fewer field samples. The method blank(s) must be free of contamination from the parameters of interest (<RL). If target analyte contamination is observed in the method blank(s) that would

influence reporting of a result in a field sample, corrective actions include but are not limited to the following:

1) Raise the reporting limit of the potentially affected analyte in the sample to a level (the level detected in the blank) at that is confidently reported as a non-detect;
2) Qualify affected field sample data as potentially biased high; or,
3) Re-prepare and re-analyze the affected batch of field samples.
4) Blank contamination is considered to influence sample results if the concentration in the blank is within five times (5x) the level in the sample.

10.3. Reporting Limit verification

The RL is verified in each sample preparation batch by analyzing at least one RL check QC sample, except in instances where concentrations below the LCS level are not of interest to the client (e.g., when their action level is higher than the LCS concentration). The method RL check QC sample (MRL) is a low level LCS intended to evaluate analyte recovery at or near the RL. The recovery of the analytes of interest in the MRL is compared to established acceptance criteria (Table 2). The LIMS will be used to record and track method performance at the RL. If the RL verification criteria are not met, corrective actions include but are not limited to the following:

1) Raise the RL for an analyte not meeting the criteria to the level of a higher concentration RL verification or LCS QC sample that meets the SOP criteria;
2) Qualify field sample data below the measured concentration of a higher level RL verification or LCS QC sample that meets the SOP criteria; or,
3) Re-prepare and re-analyze the affected batch of field samples.

10.4. Blank spike (BS)

In order to evaluate analyte recovery in a clean sample matrix, an LCS sample is prepared with every batch of 20 or fewer field samples. The LCS sample is carried through the entire analytical procedure with the other field samples and QC samples in the batch. The LCS sample is spiked at levels near the midpoint of the calibration, unless a specific concentration of interest is identified by the client, in which case they may be spiked at that level. The recovery of the analytes of interest in the LCS is compared to established acceptance criteria (Table 2). If the LCS criteria are not met, corrective actions include but are not limited to the following:

1) Qualify field sample data for the analyte that does not meet SOP criteria; or,
2) Re-prepare and re-analyze the affected batch of field samples.

10.5. Matrix spike / matrix spike duplicate (MS/MSD) / Duplicates (DUP)

In order to evaluate analyte recovery in the sample matrix, an MS/MSD sample pair is prepared with each batch of 20 or fewer field samples. Alternatively, a MS and DUP may be included in the case of limited sample volumes and/or at the discretion of the analyst. MS/MSD or DUP QC samples are not included if insufficient sample volume is available to the laboratory. MS/MSD and DUP samples are carried through the entire analytical procedure with the batch of field samples. MS/MSD QC samples are spiked at the same concentration as the BS QC sample. Percent recoveries and RPDs are calculated and compared to established acceptance criteria (Tables 2) or to client specified performance criteria. If MS/MSD and/or DUP criteria are not met, corrective actions include but are not limited to the following:

1) Dilute extracts to reduce magnitude of background interferences co-extracted from the sample matrix;
2) Qualify field sample data for the analyte that does not meet SOP criteria; or,
3) Re-prepare and re-analyze the affected batch of field samples, possibly using a smaller amount of field sample.

10.6. Instrument performance check

A GC/MS instrument performance check is analyzed in full scan mode at the beginning of the initial calibration analysis sequence. The instrument performance check contains BFB. The BFB peak in this injection should meet the ion ratio criteria in Table 3. If instrument performance check criteria are not met, corrective actions include but are not limited to performing instrument maintenance (e.g., inlet maintenance, column maintenance, retune mass spectrometer), followed by re-analysis/repreparation of the instrument performance check solution.

10.7. Initial calibration

Calibration curve acceptance criteria are ≤ 20% RSD for the RRF_{avg} calibration model, or $r^2 \geq 0.99$ for linear or quadratic regression models, and ≤ 20% difference from expected concentrations of the target analytes in the initial calibration verification (ICV) second-source standard if one is used (highly recommended)., The simplest calibration model that meets the acceptance criteria should be used, beginning with the RRF_{avg} model, then linear regression, and finally quadratic regression. Regression models should be weighted (in most cases by 1/concentration). The low and/or high point of the calibrations can be dropped in order to achieve acceptable RSDs or r^2 as long as the minimum calibration point requirement is met: Five calibration points for average RRF and linear regression are needed, while a quadratic regression model needs a minimum of six points. A successful calibration is expected to be valid until the CCV and/or internal standard criteria cannot be met. If initial calibration criteria are not met, corrective actions may include but are not limited to performing instrument maintenance (e.g., inlet maintenance, column maintenance, retune mass spectrometer), followed by re-analysis of calibration standards.

10.8. Continuing calibration verification

Instrument calibration is verified to remain valid during analysis of field samples and associated QC samples through CCV injections that are analyzed once every 12 hours after the initial calibration. The acceptance criteria specified for this SOP are for calculated concentrations to be within 20% of the expected values for each analyte of interest and surrogate If continuing calibration verification criteria are not met, corrective actions may include but are not limited to the following:

1) Perform instrument maintenance (e.g., inlet maintenance, column maintenance, retune mass spectrometer), followed by re-analysis of CCV and any affected sample extracts;
2) Qualify affected analyte data.

NOTE: . Since this is a single-analyte method: If an opening CCV fails, perform corrective action and do not proceed with the sequence without a documented case of extenuating circumstances and in consultation with the group leader, supervisor, or Contract Officer's Representative for the task area (in the case of ESAT).

10.9. Internal standards

Sample-specific demonstration of successful instrumental analysis is achieved through addition of a suite of internal standards to each field sample and QC sample extract, monitoring the response of each, and comparison to the response of the same internal standards observed in the calibration standards. Area counts of the internal standard peaks in the extracts are acceptable if they fall within 50-200% of the area of the same internal standards observed in the midpoint calibration standard or in the CCV. Additionally, the retention times of the internal standards in the batch QC and samples must fall within ± 10 seconds.

1) Dilute extracts to reduce magnitude of background interferences co-extracted from the sample matrix;
2) Qualify field sample data for an analyte calculated with internal standards that do not meet SOP criteria; or,
3) Re-prepare and/or re-analyze the affected batch of field samples, possibly using a smaller amount of field sample.

10.10. Demonstration of capability

1) For each analysis system that is not substantially similar to the one described in this method, an IDOC study must be performed. See the ASB QM and work instruction QA-WI-002 for guidance and requirements regarding IDOCs.
2) For an analyst to be qualified to use this SOP, an ADOC study must be performed. See the ASB QM, MS-CHKLST003, and work instruction QA-WI-002 for guidance and requirements regarding ADOCs.
3) The acceptance criteria for P&A samples for an IDOC or an ADOC are equivalent to the QC limits listed in this SOP.
4) A precision & accuracy (P&A) sample is a mid-level LCS. A P&A study for an initial ADOC for this SOP consists of four P&A samples, a blank, and a reporting limit check (MRL). The requirements for a continuing ADOC are the same without the MRL.

11. DATA AND RECORDS MANAGEMENT

Chemstation™ Environmental Data Analysis (Agilent Technologies) is used to process raw GC/MS chromatograms generated with this SOP, and Element (Promium) is used for the LIMS database and report generation. Example calculations and equations used for this SOP are provided in Attachment 1 of the appendix. Results are reported to 2 significant figures, and results are reported in micrograms per liter (µg/L) at the reporting limit. Electronic instrument files are to be backed up daily when running sequence(s).

11.1. Data package assembly

Refer to SOP GEN032 for MS Group data package requirements. See Attachment 3 for an example list of data package components.

11.1.1. Electronic archive

Include the following files, on ASB's archive drive \\204.46.201.26\Root Share\R5CRL\Vol1:

1) Processed data files;
2) Data processing method;
3) Injection log / internal standards summary;
4) Final report containing case narrative;
5) Any EDDs requested by client;

6) Other supporting information necessary to reconstruct the analysis, including but not limited to analyst notes, manual calculation spreadsheets, sequences, SmartPrep™ report etc.

11.2. Other records

11.2.1. Store any Autotune and BFB tune reports, air and water checks, and instrument sequences in the applicable centrally-filed folder on the instrument computer.

11.2.2. Record all maintenance performed in the instrument logbook currently located in LIMS.

11.3. LIMS data entry and reporting

11.3.1. Batch: First, create a batch that contains the analysis name, and select the surrogate spiking standard LIMS ID used.

11.3.2. Bench sheet: Next, create a bench sheet containing all relevant field sample and quality control sample LIMS IDs, sample volumes, surrogate and target compound spike volumes, and preparation dates. By convention, if sample preparation proceeds overnight, the date extraction was started is used for the LIMS preparation date.

11.3.3. Sequence: The LIMS sequence function is optional for this SOP. The LIMS sequence function can be used to build LIMS generated reports (e.g. internal standard recovery sheet, run logs etc). To build a sequence, refer to the Element Users Guide.

11.3.4. Data Entry / Review: Select the appropriate batch or work order and analysis in the data entry / review screen. Create the data entry table, and then click the 'Data Tool' radio button and 'Browse' for the instrument data files. Select the appropriate file folder, and make sure the 'File Type' is set to epatemp.txt. Merge the selected instrument data files with the data entry table in the LIMS. If either 'Unmatched Analytes' or 'Unmatched Units' appear in red, the cross table may need to be updated or repaired prior to saving the merged data entry table. Save the merged file, and then go back to the data entry table and save it as well. Press 'Query' to view the newly populated data entry table, and inspect for discrepancies with the raw data, correcting any errors as necessary. If data are entered manually, include at a minimum the analyte amount, data file name, instrument name, and analysis date/time. Whether data are uploaded automatically or entered manually, be sure to 'lock' the data in the data entry table and update the status to 'analyzed' when evaluation and review is completed by the primary analyst.

11.3.5. Note QC parameters that do not meet the acceptance criteria, and add data qualifiers to the affected samples/analytes following the guidance in ASB SOP MS037.

11.3.6. Write a case narrative using the most current ASB narrative template and following format requirements in the ASB work instruction QA-WI006. Include an explanation of the reason(s) data qualifiers were added or reference key in report with reason codes. Upload this narrative to the LIMS for inclusion in the draft report. For GC/MS, the report template used is typically 'No L.A.B. Logo Analysis v.*[x]*.rpt'.

NOTE: Documentation is not required for initial analysis of samples that are re-extracted and/or re-analyzed as long as all applicable QC criteria are met for re-extraction / re-analysis.

However, it must be discussed in the narrative or analyst note, and provided as supporting information/data not used in the data package and instrument files stored with the final project folder on R5CRL serve Vol1.

11.3.7. Give the data package to the secondary data reviewer and set the LIMS status to “Analyzed” followed by “In-review”. After addressing the data reviewer’s comments and once (s)he has indicated all comments are resolved, send the case narrative for supervisory approval via a Qualtrax workflow. Once the signed narrative is returned, notify the Reviewer who can update the status to ‘Reviewed’ in LIMS. Then generate the final report and final EDD. The work flow for data reporting and transmittal is in GEN032 and GEN018.

12. TROUBLESHOOTING

12.1. Possible causes of inadequate abundance at high masses

12.1.1. Dirty or contaminated ion source, electron multiplier, or quadrupole rod surfaces.

12.1.2. Potentials of ion source elements at wrong values due to open or short circuits.

12.1.3. Faulty ion source electronics, detector electronics, or power supply.

12.1.4. Abnormally high pressure causing ionization difficulties. Check for presence of air at m/z 28 and 32 >10% of the m/z 69 peak of PFTBA, indicating vacuum system leak.

12.2. Possible causes of improper isotope ratios

12.2.1. High background levels of undesired substances (contamination from earlier sample injections) which contribute additional abundance at the isotope mass. Bake out the ion source assembly and condition the column.

12.2.2. Resolution of adjacent masses set improperly: higher than normal ratios due to low resolution (wide peaks) or lower than normal ratios due to high resolution (narrow peaks).

12.3. Probable causes of low overall sensitivity

12.3.1. Dirty or contaminated ion source, electron multiplier, or quadrupole rod surfaces.

12.3.2. Electron multiplier with low gain.

12.3.3. Abnormally high pressure causing ionization difficulties. Check for presence of air at m/z 28 and 32 (CO_2 at m/z 44) indicating vacuum system leak.

12.4. Possible causes of poor reproducibility

12.4.1. Loose or intermittent connection either to a printed circuit or to one or more ion source or quadrupole elements inside the analyzer assembly.

12.4.2. Pressure or flow fluctuations – check using ion gauge. For autotune, check the PFTBA vial. Refill the vial, if necessary.

12.4.3. Variations in breakthrough in the 6 mL coconut charcoal cartridge. Verify that no adjustments were made to the flow rate in the SmartPrep™ method and/or test a cartridge for breakthrough with the same manufacturer lot number.

12.5. Possible causes of high background

12.5.1. Dirty or contaminated ion source, electron multiplier, or quadrupole rod surfaces. Bake out the source.

12.5.2. Column or septum bleed, as evidenced by intense peaks at m/z 73, 147, 207, or 221. Should not have an abundance of greater than 500 counts under autotune conditions. Replace the faulty septum and condition the column.

12.5.3. Contamination in a recently cleaned vacuum system – after any venting of a vacuum system for maintenance, there is the potential for introducing new substances into the vacuum system. Some substances are normal and can be pumped out while others require more cleaning or baking.

12.5.3.1. Solvents used in the cleaning process: these will be present for a while but should be pumped out as heat is applied to the vacuum system.

12.5.3.2. Water absorbed on the metal surfaces while vented. This will pump out with heat.

12.5.3.3. Fingerprints – heavy organic substances from inadequate clean room procedures may not be pumped out and may require source cleaning.

12.6. Possible causes of an unresponsive mass spectrometer

12.6.1. Mass spectrometer electronics are not on – check that the power switch is on.

12.6.2. Secondary fuse blown – check the secondary fuses on the rear of the mass spectrometer and replace the faulty fuse(s).

13. PREVENTIVE MAINTENANCE

The sequence folder contains records of analysis. Logbooks are near the instrument and SmartPrep™. Logbooks in any form are used to record any preventive maintenance performed, hardware and software problems, modifications, method updates, tank changes, etc. It is the responsibility of the analyst to document all maintenance items in the relevant instrument maintenance logbooks.

NOTE: For the SmartPrep™ extractor module, the maintenance logbook is maintained in LIMS at the time of this SOP.

13.1. Gas chromatograph and inlet:

The injector septum and injection port liner are typically replaced and the GC column clipped prior to beginning analysis of a sample set. The liner and injector septum are replaced if worn when the GC

column is replaced. The GC column is changed when chromatographic peak shape becomes poor for select analytes that are more sensitive to the development of active sites in the column that develop with column age, usage, and the degree of interferences in the sample matrix. If replacing the column, liner, and injector septum does not fix the peak shapes or responses of the polar compounds while the peak shapes of the non-polar compounds (e.g. PAHs) remain good, replacing the gold seal in the bottom of the inlet may fix the problem.

13.2. Mass spectrometer:

The ion source is cleaned if the mass spectrum of the system performance check compound (BFB) fails tolerance limits or if sensitivity decreases sharply. Some routine maintenance tasks (e.g. changing the vacuum pump oil) may be covered under the service contract maintained for the instrument and are performed on a routine basis according to the instrument manufacturer's recommended schedule. The user's manual is located on the instrument computer and in the drawer below GC/MS #16.

13.3. SmartPrep™ Extractor Module:

Horizon Technology recommend multiple preventative action items to be completed every 6 months, or more frequently depending on usage. The analyst should keep an eye on the tubing, plunger and syringe of the module and the possibility of any leaks or malfunction that would indicate the need for maintenance. Refer to Part III of the SmartPrep™ Users Guide located on the desktop of the computer linked to the module. These items include:

- Plunger O-ring seal replacement
- Syringe pump replacement
- Port value service
- Changing the plunger assembly
- Homing and calibrating the syringe
- Changing waste block O-rings
- Adjusting the carousel offset position
- Adjusting the syringe liquid sensor position

14. REFERENCES

AGILENT GC 7890B/7683B Operator's Manual

AGILENT Mass Spectrometer 5977B Operator's Manual

Code of Federal Regulations, 40 CFR Part 136, Appendix B.: Definition and Procedure for the Determination of the Method Detection Limit. Revision 1.11. July 2011. Retrieved at: http://law.justia.com/cfr/title40/40-22.0.1.1.1.html#40:22.0.1.1.1.0.1.6.2

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Method 522, Determination of 1,4-Dioxane in Drinking Water by Solid Phase Extraction (SPE) and Gas Chromatography/Mass Spectrometry (GC/MS) with Selected Ion Monitoring (SIM), Version 1.0, National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, September 2018 (EPA Document EPA/600/R-08/101).Retrieved at: https://www.epa.gov/water-research/epa-drinking-water-research-methods

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SW-846 Method 8000D. Determinative Chromatographic Separations, Revision 4, July 2014 Retrieved at: https://www.epa.gov/hw-sw846/sw-846-compendium

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15. REVISION HISTORY

Version #	Status	Description
1	I	This document is first version of the SOP for the measurement of low-level 1,4-dioxane in water by GC/MS. The draft document workflow is Qualtrax ID 9503.
2	R	Entire Document—Replaced/removed CRL. Added ASB. Entire Document—Updated Section number due to edits related to addition of manual extraction method and Calibration Blank QC. Entire Document—Removed passive language such as “generally” and “should” to avoid misconstruing procedural requirements. Section 1.5—Corrected RL calibration level used as the reporting limit. Sections 1.1: 2.0: 6.3: 9.2.2: Attachment 2—Added procedure and additional equipment for the option of manual SPE extractions (Pen & Ink 10181). Sections 3.0: 9.4.2: 9.4.4: 9.4.7: 10.6: Table 4: Attachment 2—Removed the requirement of BFB at the start of each 12hr sequence. Aligning with reference method 522 (9.3.4), 8270E. Section 5.4—Clarified carryover contamination corrective action. Sections 6.4.4: 9.3.1—Added the use of graduated VOA vials. Section 7.4.3—Updated standard expiration dates to bring in line with 8270E and manufacturers’ recommendations. Section 7.4.4.4—Edited the vial label requirements to match SOP MS026. Sections 7.4.7: 7.4.8: 9.4.2—Added Calibration Solvent Blank requirements. Section 7.4.9—Clarified amount of Internal standard added to calibration standards. Sections 9.1: 9.4.1: 10: 11.3.5: Attachment 3—Updated the new SOP numbers for referenced methods. Sections 9.3: 9.4.1: Table 4—Updated extract storage requirements. Section 9.4.5.1—Added reference spectra update to calibration method Section 10.8—Removed CCV every 10 field samples requirement consistent with 8270E (keeps 12-hr frequency.) Added a note stating the opening CCV must pass because this is a single-analyte method. Section 10.10. Updated ADOC definition to conform to current requirements and practice. Section 11. Updated for electronic data packages, file storage, and reporting consistent with current procedures. Section 13.2—Corrected Performance Check compound to match standard in section 7.3.4. Attachment 3—Added reference to SOP GEN032 for preparation of electronic reports. Redline document available in the Document approval workflow, document 9708.

I = Initial, R = Revision, A = Archived

16. APPENDICES

Table 1: Method Analyte CAS Numbers, MS Ions, and GC Retention Times.

Analyte Type	Compound Name	CAS #	EI Ions		Retention Time† (min)
			Primary	Secondary	
Internal Std	Tetrahydrofuran-d8	1693-74-9	46	78, 80	5.83
Surrogate	1,4-Dioxane-d8	17647-74-4	96	62, 64	7.96
TC	1,4-Dioxane	123-91-1	88	58	8.04

Notes: Secondary EI ions are used for confirmation of peak identification.

† Retention times based on 15 min GC oven program described in Section 9.4.

Table 2: Spike Recovery Limits and Associated Statistics for 1,4-dioxane (Target Analyte) and 1,4-dioxane-d8 (Surrogate) using SPE

Compound Name	MDL (ug/L)	RL (ug/L)	Blank Spike Recovery				Matrix Spike Recovery			
			Mean (%)	Standard Deviation (s) (%)	Samples (n)	Estimated Measurement Uncertainty (Mean ± 3s) (%)	Mean (%)	Standard Deviation (s) (%)	Samples (n)	Estimated Measurement Uncertainty (Mean ± 3s) (%)
1,4-Dioxane	0.15	0.20	77	3.1	4	68 - 86	--	--	--	-- -- --
1,4-Dioxane-d8	--	--	85	3.8	4	73 - 96	--	--	--	-- -- --

Notes:
The name of this analysis in the LIMS is ‘1,4-dioxane (low level) by SPE’.

Surrogate spike recovery statistics were calculated from blank spike QC samples used in the IDOC.

Since no historical data has been generated, the QC limits for both the target analyte and surrogate in laboratory control samples and matrix spike samples will default to %R=70-130% until sufficient historical data has been generated. Additionally, RPD criteria will be defaulted to 30%. MRL criteria is defaulted to 50-150% recovery.

RLs will be verified by batch. The MDL study for SIM was completed in work order 1702004 from GC/MS#16 in February 2017. Qualtrax workflow #9614.

Limits set in LIMS were rounded to whole numbers. All data reported will be reported to two significant figures.

Table 3. BFB KEY IONS AND ION ABUNDANCE CRITERIA[a]

Mass (*m/z*)	Ion Abundance Criteria
50	15-40% of *m/z* 95
75	30-80% of *m/z* 95
95	Base peak, 100% relative abundance
96	5-9% of *m/z* 95
173	< 2% of *m/z* 174
174	> 50% of *m/z* 95
175	5-9% of *m/z* 174
176	> 95% but < 101% of *m/z* 174
177	5-9% of *m/z* 176

[a] Criteria taken from EPA Method 522

Table 4. Summary of QC criteria used for this SOP

QC parameter	Frequency	Criteria
Preservation and Holding Times for Samples and Solvent Extracts	For each sample and extract	Preservation criteria upon receipt of aqueous samples: ≤ 6°C Holding time for aqueous samples: 28 days (collection to extraction) Holding time for solvent extracts: 28 days (extraction to analysis) (Must be maintained < 6 °C)
GC/MS Instrument Performance Check	Every initial calibration and major change to system, and when re-establishing the calibration (≥ 1 week since last injection)	BFB ion ratios meet criteria in Table 3
Initial Calibration	As required prior to sample analysis, when ICV/CCV criteria are not met	RRF_{avg} model is acceptable if RSD ≤ 20% linear regression model is acceptable if $r^2 \geq 0.99$, minimum of 5 points quadratic regression model is acceptable if $r^2 \geq 0.99$, minimum of 6 points
Initial Calibration Verification	At the end of every initial calibration	% Difference ≤ 20%
Continuing Calibration Verification	Once every 12 hours and at the end of an analytical sequence	% Difference ≤ 20%
Internal Standards	Each field sample, QC sample, and CCV	For each sample, IS areas within 50 - 200% of CCV or midpoint ICAL standard area IS retention time (RT) of samples ± 10 seconds of most recent CCV For each CCV, each area within 50 - 200% of midpoint ICAL standard or mean of ICAL standards IS RT of CCVs within ± 10 seconds of mid-level ICAL standard RT
Method Blanks	At least 1 method blank for each batch of 20 or fewer samples	Sample concentrations must be > 5x blank concentration
RL Verification	At least one for each batch of 20 or fewer samples	*Default 50%-150%*

QC parameter	Frequency	Criteria
LCS	A blank spike for each batch of 20 or fewer samples	Within initial recovery limits (Table 2) *Default: 70-130%*
Surrogates	Each field sample and QC sample	Within initial recovery limits (Table 2) *Default: 70-130%*
MS/MSD	A pair of matrix spikes for each batch of 20 or fewer samples	Within initial recovery limits (Table 2) *Default: 70-130%; RPD < 30%*
DUP (Optional)	Analyzed with a MS as an alternative to an MS+MSD	RPD < 30%
Qualitative Identification	Target analyte found in field samples	Sample RT within ± 10 seconds of mid-level ICAL or CCV RT Sample ion ratio within ± 30% of mid-level ICAL or CCV standard ion ratio

Attachment 1. Sample Calculations and Equations

Relative Response Factor

Calculate the relative response factor at each calibration level for each target compound and surrogate using Equation 1. The primary characteristic ions used for quantitation are listed in Table 1. Assign the target compounds and surrogates to an internal standard. If an interference prevents the use of a primary ion for a given internal standard, use a secondary ion.

Equation 1:

$$RRF = \frac{(A_x)(C_{is})}{(A_{is})(C_x)}$$

A_x = Area of the primary characteristic ion for the compound to be measured
A_{is} = Area of the primary characteristic ion for the specific internal standard
C_{is} = Concentration of the specific internal standard
C_x = Concentration of the compound to be measured

Average Relative Response Factor

Calculation of the average of the RRFs is shown in Equation 2.

Equation 2:

$$RRF_{avg} = \frac{\sum_{i=1}^{n} RRF_n}{n}$$

RRF_n = Relative response factor for each initial calibration standard
n = Number of calibration standards

Percent Relative Standard Deviation

Equation 3 is used to calculate the %RSD of the RRF values over the working range of the curve.

Equation 3:

$$\%RSD = \frac{\sigma}{\bar{x}} \times 100$$

$$\sigma = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$$

x_i = Each individual value used to calculate the mean
$\bar{x}$ = The mean of *n* value
n = The total number of values
σ = Standard deviation

Percent Difference

The percent difference between the calculated and expected concentration in the continuing calibration verification standard is calculated using Equation 4.

Equation 4:

$$\%D = \left(\frac{\text{calculated concentration} - \text{expected concentration}}{\text{expected concentration}} \right) \times 100$$

Concentration in Water Samples

Calculations for the different calibration models for an analyte in a water sample:

Equation 5:

RRF_{avg} calibration model for water:

$$C_x = \frac{(A_x)(C_{is})(D_f)}{(A_{is})(RRF_{avg})(V_o)}$$

Equation 6:

Linear regression calibration model for water:

$$C_x = \frac{(A_x)(C_{is})(D_f)}{(A_{is} + y_{\text{int}})(m)(V_o)}$$

Equation 7:

Quadratic calibration model for water:

$$C_x = \frac{(-b \pm \sqrt{b^2 - 4a(c - (A_x / A_{is}))})(C_{is})(D_f)}{(2a)(V_o)}$$

A_x = Area of the primary characteristic ion for the compound to be measured
A_{is} = Area of the primary characteristic ion for the specific internal standard
a = X-squared coefficient from the least squares fit quadratic regression
b = X coefficient from the least squares fit quadratic regression
c = Constant term from the least squares fit quadratic regression
m = Slope from the least squares fit linear regression
y_{int} = Y-intercept from the least squares fit linear regression
C_{is} = Concentration of the specific internal standard
C_x = Concentration of the compound to be measured (ug/L for water, ug/kg for soil/sediment)
V_o = Volume of sample extracted in liters
W_s = Mass of sample extracted in kilograms
D_f = Equivalent final extract volume. The final extract volume is the product of the total extract volume before dilution and the dilution factor for any dilutions of the extract made to interpolate the target compound concentrations (e.g. the final volume of the extract is 2 mL and then 10 uL of that extract is diluted to 1 mL, which is then spiked with internal standard and analyzed. The equivalent final volume is 2 x 100 = 200). If there was no dilution and the final extract volume is 1 mL, then D_f = 1.
GPC = GPC factor is generally 2 to account for injecting half of the extract for GPC clean-up D = (% dry solids) / 100

Percent Recovery

Calculate the recovery of an analyte using Equation 12.

Equation 8:

$$\text{\% recovery} = \frac{\text{calculated concentration}}{\text{spiked concentration}} \times 100$$

Matrix Spike % Recovery

Calculate the recovery of each matrix spike compound as follows in Equation 13.

Equation 9:

$$\text{MS \% recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}} \times 100$$

SSR = MS/MSD spiked sample result
SR = Unspiked sample result

SA = Spike concentration

Relative Percent Difference

RPD of the recoveries of each compound in the matrix spike and matrix spike duplicate is shown in Equation 14.

Equation 10:

$$RPD = \frac{|[MS] - [MSD]|}{([MS] + [MSD]) / 2} \times 100$$

[MS] = Measured concentration in the matrix spike QC sample
[MSD] = Measured concentration in the matrix spike duplicate QC sample

Internal Standard Calculations

Percent area response change between the sample/blank analysis and the most recent continuing calibration standard for each of the internal standards is shown in Equation 15.

Equation 11:

$$\%ARC = \frac{A_s - A_c}{A_c} \times 100$$

%ARC = Percent area response change
A_s = Area response of the internal standard in the sample/blank analysis
A_c = Area response of the internal standard in the most recent continuing calibration standard

Retention time shifts between the sample/blank analysis and the most recent continuing calibration standard analysis for each of the internal standards is shown in Equation 16.

Equation 12:

$$RTS = RT_s - RT_c$$

RTS = Retention time shift
RT_s = Retention time of the internal standard in a sample/blank
RT_c = Retention time of the internal standard in the most recent continuing calibration standard

Regression Equations

Equation 13:

Linear regression:

$$y = mx + b$$

y = Response or relative response
x = Concentration or relative concentration
m = Slope from the least squares fit linear regression
b = Y-intercept from the least squares fit linear regression

Equation 14:

Quadratic regression:

$$y = ax^2 + bx + c$$

y = Response or relative response
x = Concentration or relative concentration
a = X-squared coefficient from the least squares fit quadratic regression
b = X coefficient from the least squares fit quadratic regression
c = Constant term from the least squares fit quadratic regression

Equation 15:

Coefficient of determination for regression:

$$r^2 = \frac{SST_y - SSE_y}{SST_y}$$

r^2 = Coefficient of determination
SST_y = Total sum of squares with respect to y = $\sum(y_i - y_{avg})^2$
SSE_y = Error sum of squares with respect to y = $\sum(y_i - y_{hat})^2$
Y_i = Measured response or relative response
Y_{avg} = Mean response or relative response
Y_{hat} = Calculated response or relative response based on x value and the least-squares fit regression coefficients

Attachment 2. Deviations from Reference Methods

522:

Global: Method 522 utilized manual SPE. This SOP uses either manual or automated SPE.

Global: QC limits designed in method 522 were not used. Default laboratory limits of 70-130% with be used for LCS and MS recoveries, and 50-150% will be used for MRL recoveries until adequate data is generated enabling ASB to control chart in-house limits. These limits were established following guidance in SW-846 8000D. Additionally, calibration limits and internal standard recovery limits were defaulted to limits specified in Method 8000D.

Section 3. Batch is not defined by a 24-hr period as in 522 section 3, but a 12-hr period.

Section 8. No sodium sulfite is used in this SOP since this SOP is for non-drinking water, and is therefore not expected to be chlorinated.

Section 8.2.4. Sample pH will be verified at pH≤2 instead of pH<4.

Section8.4. Sample extracts are stored at < 6^0C

Section 10. Internal standard criteria vary from 522:9.3.5, 522: 10.3.2. Calibration acceptance criteria vary from 522: 10.2.4. Continuing calibration check criteria are more narrow than 522:10.3.3. LCS criteria don't widen near the reporting limit as in 522:9.3.3.

Section 12.2 The confirmation ion ratio criteria for m/z 58 is absolute ± 20% of the ratio in method 522. This SOP uses ± 30% which is consistent with organic SW-846 methods.

8000D:

Section 11.7 This SOP performs single-analyte calibration verification by evaluating only the percent
difference regardless of the type of calibration model used (e.g. average RF, average CF, linear regression
etc.). This deviates from the reference method SW-846 8000D by 1) the definition of percent difference
according to this SOP, and 2) evaluating the percent difference only.

Attachment 3. Data Package Components

NOTE: The following is a checklist that may be used to assemble a data package. It is to be viewed only as an example. See ASB SOP GEN032 for current requirements and instructions on how to assemble an electronic data package.

1. Checklist from Mass Spectrometry (MS) Checklists MS-1CHKLST001
2. LIMS report (with case narrative)
3. Summary spreadsheets (as necessary)
4. LIMS work order
5. Applicable pen and inks sent to client, client contacts, non-conformances affecting samples, etc.
6. LIMS bench sheet
7. LIMS sequence report (optional)
8. GC/MS instrument sequence (required)
9. Injection log and internal standards summary
10. SmartPrep™ Report
11. Instrument performance check [Chemstation]:
    i. BFB ion ratio report
12. Initial calibration and continuing calibration verification reports [Chemstation]:
    i. Response Factor Report
    ii. Calibration Report
    iii. Calibration History
    iv. Evaluate continuing calibration report(s)
    v. Quantitation Reports
    vi. Manual integrations*
13. QC samples/field samples [Chemstation]:
    i. Manual integrations*
    ii. Quantitation Report
    iii. Labeled chromatogram
14. Supporting Information
    i. Copy of laboratory notebook pages

*Include print-outs or images of both the automatic integration and manual integration (see SOP GEN029). Annotate all manual integrations (reason code, date, initials).

Attachment 4. SmartPrep™ Extraction Method

Procedure Name: 522 022117 FINAL 250mL 10mLmin
Cartridge: Coconut Charcoal
Cartridge Size: 6
Number of Steps: 17

1. Condition cartridge using 3 mL of Methylene Chloride at 20 mL/min Soak 10 sec Dry 2 sec
2. Condition cartridge using 3 mL of Methanol at 20 mL/min Soak 10 sec Dry 2 sec
3. Condition cartridge using 3 mL of Methanol at 20 mL/min Soak 10 sec Dry 2 sec
4. Condition cartridge using 3 mL of Reagent Water at 20 mL/min Soak 10 sec
5. Condition cartridge using 3 mL of Reagent Water at 20 mL/min Soak 10 sec
6. Condition cartridge using 3 mL of Reagent Water at 20 mL/min Soak 10 sec
7. Condition cartridge using 3 mL of Reagent Water at 20 mL/min Soak 10 sec
8. Condition cartridge using 3 mL of Reagent Water at 20 mL/min Soak 10 sec
9. Load all of Sample at 40 mL/min. Deliver at 10 mL/min.
10. Dry for 10 minutes
11. Elute cartridge using 3 mL of Methylene Chloride at 10 mL/min into 1st Collection Tube Soak 20 sec
12. Elute cartridge using 3 mL of Methylene Chloride at 10 mL/min into 1st Collection Tube Soak 20 sec
13. Elute cartridge using 3 mL of Methylene Chloride at 10 mL/min into 1st Collection Tube Soak 20 sec Dry 20 sec
14. Clean sample line using 6 mL of Methanol at 40 mL/min Dry 2 sec
15. Clean sample line using 6 mL of Reagent Water at 40 mL/min Dry 2 sec
16. Clean sample line using 6 mL of VENT at 40 mL/min
17. Clean plunger using 6 mL of Methylene Chloride at 40 mL/min Soak 10 sec Dry 10 sec

Standard Operating Procedure
For the Analysis of Metals by ICP
EPA Method 200.7/6010D
Using the Thermo 6500 Duo

United States Environmental Protection Agency
Region 5 LSASD Analytical Services Branch
536 S Clark Street (LAB-10C)
Chicago IL, 60605

Table of Contents

1. SCOPE AND APPLICATION 3
2. SUMMARY OF METHOD 6
3. ABBREVIATIONS AND DEFINITIONS 7
4. HEALTH, SAFETY AND WASTE HANDLING 10
5. CAUTIONS AND INTERFERENCES 11
6. EQUIPMENT AND SUPPLIES 15
7. REAGENTS AND STANDARDS 16
8. SAMPLE HANDLING AND PRESERVATION 26
9. SAMPLE PREPARATION AND ANALYSIS 27
10. QUALITY CONTROL 32
11. DATA AND RECORDS MANAGEMENT 42
12. TROUBLESHOOTING 43
13. PREVENTATIVE MAINTENANCE 45
14. REFERENCES 47
15. REVISION HISTORY 50
APPENDIX A 52
APPENDIX B 56
APPENDIX C 59
APPENDIX D 64

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) is applicable to the analysis of 31 elements in waters (ground water, surface water, and drinking water), and wastewaters and 30 elements in soils and sediments using the hot block digestion procedure METALS025 (EPA Method 200.2/200.7/200.8) or drinking water without acid digestion if the samples have been properly acid preserved and have turbidity <1 NTU (direct analysis). Other digestion methods may be used such as microwave digestion or hotplate/beaker digestion (METALS034), but performance data must be gathered for the new sample preparation method(s). The analysis method is based on SW-846 Method 6010D and EPA Method 200.7 with minor acceptable deviations. EPA Method 200.7 is approved for NPDES monitoring in 40 CFR part 136.3, Table 1B for 27 of these elements. Twelve of these elements are priority pollutants per 40 CFR part 401.15. Other subsets of these elements may be reported for various programs based on the data quality objectives (DQOs) of the project.

Please note: Drinking water is covered under the ASB Scope of Accreditation for the following analytes: aluminum, barium, beryllium, cadmium, calcium, chromium, iron, magnesium, manganese, nickel, selenium, silver, and sodium.

1.2. The instrument used for metals analysis by ICP-OES as described here is the Thermo 6500 Duo, using axial and radial plasma views.

1.3. The elements determined by this SOP include the following:

Analyte	Symbol	Registry Number (CASRN) Chemical Abstract Services	P.P.*	Table IB
Aluminum	Al	7429-90-5		Y
Antimony	Sb	7440-36-0	Y	Y
Arsenic	As	7440-38-2	Y	Y
Barium	Ba	7440-39-3		Y
Beryllium	Be	7440-41-7	Y	Y
Boron	B	7440-42-8		Y
Cadmium	Cd	7440-43-9	Y	Y
Calcium	Ca	7440-70-2		Y
Cerium[a]	Ce	7440-45-1		
Chromium	Cr	7440-47-3	Y	Y

Analyte	Symbol	Registry Number (CASRN) Chemical Abstract Services	P.P.*	Table IB
Cobalt	Co	7440-48-4		Y
Copper	Cu	7440-50-8	Y	Y
Iron	Fe	7439-89-6		Y
Lead	Pb	7439-92-1	Y	Y
Lithium	Li	7439-93-2		
Magnesium	Mg	7439-95-4		Y
Manganese	Mn	7439-96-5		Y
Molybdenum	Mo	7439-98-7		Y
Nickel	Ni	7440-02-0	Y	Y
Phosphorus[b]	P	7723-14-0		Y
Potassium	K	7440-09-7		Y
Selenium	Se	7782-49-2	Y	Y
Silica[b,c]	SiO_2	7631-86-9		Y
Silver	Ag	7440-22-4	Y	Y
Sodium	Na	7440-23-5		Y
Strontium	Sr	7440-24-6		
Thallium	Tl	7440-28-0	Y	Y
Tin	Sn	7440-31-5		Y
Titanium	Ti	7440-32-6		
Vanadium	V	7440-62-2		Y
Zinc	Zn	7440-66-6	Y	Y

***P.P.** refers to Priority Pollutant listing in 40 CFR part 401.15. **Table IB** refers to 200.7 as an allowable method under 40 CFR part 136, Table IB.

[a]Cerium is monitored due to potential spectral interferences but is not routinely reported.

[b]Phosphorus and silica are not routinely reported except by client request.

[c]This method is not suitable for the determination of silica in soil/sediment.

1.4. The method detection limits (MDLs) and reporting limits (RLs) are found in Appendix A. MDLs for this procedure were determined using ICAP 3 for digested waters (January 2020, DOC workflow ID #20849) and soils/sediments (May 2017, DOC workflow ID #10378, work orders 1704023 and 1705012). The RLs were verified using ICAP 3 for soils/sediments (February 2020, DOC workflow ID #20959). A valid MDL should be on file for each matrix/preparation. Annual MDLs are calculated using RL verification and digestion blank data that has been collected throughout the year per 40 CFR Part 136 Appendix B or prior to sample analysis if required by regulation (waters by EPA Method 200.7). The MDL

procedure provides for calculation of an initial MDL if adequate historical data is not available to perform the Ongoing Annual Verification. Determination of digested water MDLs fulfills the annual MDL requirement for undigested water. If results are requested below the RL for undigested water samples, MDLs will be determined on undigested water prior to reporting out the data. RL verification is performed per batch and is evaluated annually as an alternative assessment of low level analyte performance for matrices/methods not required by regulation to determine annual MDLs (soil/sediment).

1.5. The practical range of determination is based on the RL and linear range (LR). Refer to Appendix A for a summary of the linearity limits. Linearity verification for all analytes was performed in April 2019 (Qualtrax ID #10125).

1.6. The estimate of minimum laboratory contribution to the measurement of uncertainty of this method for each analyte is the mean ± 3*SD and is expressed in Appendix B. These uncertainties were calculated for digested waters in January 2020 (Qualtrax ID #20865) and for soils/sediments in September 2020 (Qualtrax ID #22929). This uncertainty will vary by analyte, will be greater near the RL, and will be much greater near the MDL. Usually the sampling component of the uncertainty will be far greater than the laboratory uncertainty.

1.7. Samples digested by ASB SOP METALS025 with silver values greater than 0.1 mg/L in water digests or 0.5 mg/L in soil/sediment digests will be redigested after dilution and reanalyzed due to the 0.5% HCl concentration in the 200.2/200.7/200.8 digestion. The solubility of silver is dependent on the formation of the $[AgCl_2]^-$ complex.

1.8. ASB SOP METALS025 hot block digestion will solubilize and hold in solution only minimal concentrations of barium in the presence of free sulfate. If barium is a requested analyte in samples having varying and unknown concentrations of sulfate, analysis should be completed as soon as possible after sample preparation. Such samples may be prepared with multiple dilutions prior to digestion to obtain a more accurate barium result. Precipitation in the digest over time is an indication the samples should be redigested for barium.

1.9. If reporting between the MDL and RL is requested, the results will be rounded to one significant figure.

1.10. For elements having high and low channels, the high channel is used for water when the sample exceeds the linear range of the low channel except when failure of quality control audits would preclude the use of either channel. The high channels are the default lines reported for soil/sediment.

1.11. Dissolved samples are prepared just as are the corresponding total samples to ensure proper matrix matching. It is recommended that both are analyzed together.

1.12. Deviations from the reference method are found in Appendix D.

2. SUMMARY OF METHOD

2.1. This method describes the determination of 31 elements dissolved and stabilized in aqueous acidic media which are then analyzed using an ICP-OES instrument, namely the Thermo 6500 Duo. As a matter of routine, representative sample sub-aliquots are taken and digested with mineral acids using ASB hot block digestion or beaker/hotplate digestion procedures. These digestion procedures are designed to ensure that as much of the analytes that are available for recovery (i.e. total recoverable) are rendered soluble and relatively stable in aqueous acidic medium. For description of the digestion methods, see ASB SOP METALS025 hot block digestion or METALS034 hotplate/beaker digestion. It is noted that the acid strength of the standards described in this SOP are matched only by digestion method METALS025 hot block digestion. For direct analysis total recoverable determination of analytes in drinking water where sample turbidity is <1 NTU, the sample is prepared for analysis by addition of an appropriate amount of nitric acid.

2.2. The resulting solutions are peristaltically pumped and pneumatically aspirated into aerosol mists which are conveyed in an argon gas stream through an inductively RF coupled region whereby a plasma is formed. Within the plasma, final desolvation, ionization, excitation, and characteristic radiative emission for the analytes take place. The resultant emitted radiation is directed through the optics of the spectrometer where it is passed through a narrow angle order-splitting prism before and after it is dispersed via a grating into component wavelengths that are indicative of specific elements present in the plasma. The intensity of the characteristic radiation is measured using a charge injection device array detector.

2.3. The plasma can be viewed radially (from the side) and axially (down the central channel). If low sample concentrations are expected, axial will yield greater sensitivity due to its longer path length. If high concentrations are expected, radial views will extend the linear range to greater concentrations. Since most elements have multiple analytical lines in this method, some were chosen to have one or more lines with axial view for sensitivity but with a radially viewed line to extend the concentration range. The major elements are all viewed radially because low concentrations of these elements are not needed for environmental decision-making.

2.4. The Thermo 6500 Duo ICP-OES optics are purged with argon. MgF_2 optics along with the argon gas purge facilitates the measuring of analyte lines in the far UV portion of the electromagnetic region of the spectrum. As the purge gas exits the optical system through the purged optical pathway (POP) tube, it is used to remove constituents in the plasma interface that would absorb UV light intensity, and it provides a counter flow of argon that can eliminate environmental factors such as dust and soot, which can cause interferences.

2.5. Emission intensities are counted at analyte peak locations and compared to counts obtained from calibration standards containing known amounts of specific analytes. The sample concentrations are calculated via the Qtegra ISDS ICP-OES software using a first order regression analysis. For a given spectral line, one to three pixels of the detector are used to define the area under the peak. This integrated intensity is corrected for interferences by means of an interelement correction (IEC) model.

2.6. The concentration results and the integrated data are printed to pdf. Integrations are variable length based upon intensity at the subarray. The average of several integrations is used for the reported concentration.

3. ABBREVIATIONS AND DEFINITIONS

3.1. Abbreviations

BLK: Digestion Blank (Laboratory Reagent Blank, LRB)
BS: Digestion Blank Spike (Laboratory Fortified Blank, LFB)
CCB: Continuing Calibration Blank
CCV: Continuing Calibration Verification
DQO: Data Quality Objective

DUP: Duplicate (Laboratory Duplicate, LD)
ICP-OES: Inductively Coupled Plasma-Optical Emission Spectrometry
ICB: Initial Calibration Blank
ICV: Initial Calibration Verification
IEC: Interelement Corrections
LCB: Laboratory Control Blank (same as CCB)
LCM: Laboratory Control Solution Measured (same as ICV or CCV)
LCS: Laboratory Control Standard (same as BS)
LIMS: Laboratory Information Management System
LLCCV: Low Level Continuing Calibration Verification
LR: Linear Range
MDL: Method Detection Limit
MS: Matrix Spike (Laboratory Fortified Sample Matrix, LFM)
NPDES: National Pollutant Discharge Elimination System
QAPP: Quality Assurance Project Plan
QMP: Quality Management Plan
RCRA: Resource Conservation and Recovery Act
RF Radio Frequency
RL: Reporting Limit
RLC: Reporting Limit Check (same as LLCCV)
RLV: Reporting Limit Verification
SDS: Safety Data Sheet
SIC: Spectral Interference Check

3.2. Definitions

3.2.1. Axial: End-on plasma viewing; i.e. spectrometer looks down the central channel of the plasma and collects analyte emission over the plasma length

3.2.2. Continuing Calibration Blank (CCB)/Laboratory Control Blank (LCB): A calibration blank analyzed throughout the analytical run to demonstrate that the instrument continues to be capable of acceptable performance.

3.2.3. Continuing Calibration Verification (CCV)/Laboratory Control Solution Measured (LCM): A control standard analyzed throughout the analytical run to demonstrate that the initial calibration remains valid through the course of the run.

3.2.4. Digestion Blank (BLK): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, reagents, and internal standards that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

3.2.5. Digestion Blank Spike (BS)/Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrices to which known quantities of method analytes are added in the laboratory. The BS is analyzed exactly like a sample with all preparation steps. The purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

3.2.6. Duo View: System used in the Thermo 6500 Duo ICP-OES instrument which employs software-controlled mirrors to allow plasma to be viewed either radially or axially.

3.2.7. Duplicate (DUP): Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. The relative percent differences between the DUP and parent sample indicate the precision associated with the laboratory procedures.

3.2.8. Initial Calibration Blank (ICB): A calibration blank analyzed at the beginning of the analytical run to demonstrate that the instrument is capable of acceptable performance.

3.2.9. Initial Calibration Verification (ICV): A control standard, generally from a second source or lot than the calibration standard, analyzed at the beginning of the analytical run to demonstrate that the instrument is capable of acceptable performance.

3.2.10. Linear Range (LR): Range for which data behaves in linear manner

3.2.11. Low Level Continuing Calibration Verification (LLCCV)/Reporting Limit Check (RLC): A standard analyzed throughout an analytical run to demonstrate that the initial calibration remains valid at the RL listed in the SOP through the course of a run.

3.2.12. Matrix Spike (MS): An aliquot of an environmental sample to which known quantities of method analytes are added in the laboratory. The MS is analyzed exactly like a sample with all preparation steps that the parent sample received. The purpose is to determine whether the sample matrix contributes bias to the analytical results.

3.2.13. Method Detection Limit (MDL): The minimum concentration of analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is distinguishable from method blank results.

3.2.14. Radial: Side-on plasma viewing; i.e. the spectrometer views the analyte emission from the side of the plasma

3.2.15. Reporting Limit (RL): The lowest concentration reported by ASB except by special request.

3.2.16. Reporting Limit Verification (RLV): A low level blank spike/laboratory control solution used to evaluate analyte recovery at the reporting limit. The RL verification is analyzed exactly like a sample with all preparation steps.

3.2.17. Subarray: Subsection of the detector, consisting of a group of pixels on an array detector which are positioned to measure a small, specific wavelength range

3.2.18. Super Q: Laboratory distilled water is passed through a mixed bed resin column before use

4. HEALTH, SAFETY AND WASTE HANDLING

4.1. Health and Safety

4.1.1. Users of this method should operate a formal safety program. Perform this procedure in accordance with the ASB Chemical Hygiene Plan (CHP). Consult the Master List ID #16512 for the CHP's current location.

4.1.2. Each chemical should be considered as a potential health hazard and individual exposure should be minimized. Review safety data sheets (SDS)

for specific physical and health hazards including appropriate personal protective gear (safety glasses, gloves, lab coats) to be used. SDS's may be accessed at www.sigmaaldrich.com or from another qualified vendor.

4.1.3. **WARNING –** Intense ultra-violet (UV) and radio frequency (RF) radiation is present when operating the ICP-OES instrument. Reasonable precautions to avoid exposure to these emitted radiation sources should be taken. This would include not looking directly at the ignited plasma or its reflection. The Thermo 6500 Duo is equipped with interlocks on the torch compartment door that will not allow the plasma to be ignited if the door is ajar. Unless routine maintenance is required in the torch compartment, the door should always be closed.

4.1.4. **WARNING --** Lethal high voltages are present within the spectrometer. Routine maintenance that would require the analyst to access high voltage areas should not be performed unless the instrument is powered down and locked and tagged out. Only trained, professional service engineers should perform complex repairs (i.e. non-routine) and maintenance.

4.2. Waste Handling

4.2.1. Report all injuries and major spills.

4.2.2. Unused portions of stock solutions, acids, calibration and analytical standard solutions, nebulizer waste, and digested samples must be poured into the RED labeled waste containers. Acidic waste containing high levels of hydrochloric acid, nitric acid, and/or metals with mercury (corrosive) are poured into the ORANGE waste. The used sample tubes are disposed of in the trash after rinsing and defacing the label.

5. CAUTIONS AND INTERFERENCES

5.1. Cautions

5.1.1. Due to ionization enhancement effects, potassium may be potentially biased high by 10 to 20% in a typical sample with 50-100 mg Na/L.

5.1.2. The intensities of the internal standard for both axial and radial measurements should be monitored. The intensities are affected by the condition of the purge windows as well as the sample delivery system.

5.1.3. The RSD's of intensity replicates for the internal standard and analyte wavelengths should be monitored especially during calibration, as elevated RSD's can indicate emerging poor performance.

5.1.4. If high suspended solids are apparent in the samples, the samples should be prepared with dilutions prior to analysis as well as analyzing directly.

5.1.5. High dissolved solids can contribute to salting out on the tip of the nebulizer, and this will affect plasma performance. If high dissolved solids are suspected in the samples, monitor the nebulizer and take corrective action if necessary.

5.2. Interferences

5.2.1. A certain amount of background is intrinsic to the plasma source itself. One component of this background is black-body radiation, which is the light emitted by any heated body. Another component is Bremsstrahlung, which results from collisions of the accelerated electrons and charged particles. The third component of the light emitted from the plasma is cyclotron radiation, resulting from electrons being accelerated in a magnetic field and is much stronger near the work coil. The sum of these effects is a broad emission band with the maximum at about 460 nm.

5.2.2. An important contribution to the background of the plasma is the result of reactions between components of the sample matrix or surrounding atmosphere. Nitrogen and oxygen combine in the envelope around the plasma to give excited NO molecules which emit a structured spectrum from about 180 to 230 nm. Water from the sample gives rise to OH emission mostly in the 280 to 310 nm region. NH emits at about 360 nm; CO emission contributes to the background from 180-200 nm; CN emits a spectrum from 388 to 420 nm.

5.2.3. Recombination of electrons with ionic species can give continuum emission when the excited atom falls to a metastable or ground state. The emission is

continuum rather than line because the energy emitted is related to both the atomic energy and that held by the electron prior to collision. This type of emission is most often seen for high concentrations of alkaline earths.

5.2.4. Another source of continuum background is the reaction of excited atoms from the sample with atmospheric oxygen. An example of this is the AlO emission from the oxygen cutoff from about 180 nm to 230 nm.

5.2.5. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak.

5.2.6. Line spectra are a very rich source of interference in plasma emission spectroscopy. Every component of the sample emits to some degree, and even the argon itself has a number of emission lines. Of the latter, only one analyte wavelength (Na 588.995) has a serious argon line interference.

5.2.6.1. Line spectral interferences can be grossly classified into two categories: overlapping lines, either through direct coincidence or within the resolving power of the instrument, and broadened lines. Directly overlapping lines of comparable strength are relatively rare. Strong lines for Cd at 228.80 nm and As at 228.81 nm are an exception. More often the analyte line is very near to a line from a major constituent. For example, the Be 234.86 nm line is within about twice the full width at half maximum of the Fe line at 234.81 nm.

5.2.6.2. Elemental emission lines have an intrinsic natural line width which is quite narrow, resulting in the large number of significant figures found in wavelength tables. The plasma is a very complex system. Therefore, these lines are broadened by a number of means. Doppler broadening results from the distribution of velocities of the emitting particles in the hot environment of the plasma. Collisional broadening has several forms: Van der Waals broadening by collision with neutral argon atoms, Stark broadening by collision of electrons with ions, and resonance broadening. The latter form of line broadening is most important when the interfering

element is at high concentration. If an element has two lines which are close enough to be in resonance and is present at high concentrations, collisions of the form

$$X^*1 + Xg \text{ ---> } Xg + X^*2$$

can take place, where Xg refers to ground state X and X*1 and X*2 are the two resonance excited states. The Al lines gain a significant background from the Ca lines which are well separated at low levels.

5.2.7. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated for using IEC equations. Interelement correction is a spectral interference correction technique in which emission contributions from interfering elements that emit at the analyte wavelength are subtracted from the apparent analyte emission after measuring the interfering element concentrations at other wavelengths.

5.2.8. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they may be ameliorated by use of an internal standard. Yttrium is the most common choice of internal standard. If it is used, the same amount must be added to all standards and samples. The instrument is equipped with a 3-channel peristaltic pump with one channel dedicated to the addition of the internal standard.

Please note: Caution must be used with internal standards. It is assumed that there is no difference in chemical behavior or physical transport efficiency between the internal standard element and the analyte elements. For this reason, the intensity of the internal standard must be monitored throughout the run. If later in the run there is a significant (>20%) deviation from the intensities obtained on the standards, appropriate action should be taken. For example, both yttrium and scandium have insoluble fluorides.

5.2.9. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP-OES technique. The most common interference of this

type can be seen in the analysis of an alkali metal (e.g. K) in the presence of a high concentration of another alkali metal (e.g. Na). Alkali metals are easily ionized but are determined by emission from the neutral species. A high concentration of one alkali metal will supply an excess of electrons to the plasma which boosts the neutral atom population of the less concentrated alkali metal, causing an enhancement relative to the standards.

Please note: This phenomenon was studied during method development on the Perkin Elmer 3300 DV. The study showed that those elements that are being determined axially are not affected. However, the transition elements that are determined radially will be affected in a highly alkali matrix.

5.2.10. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system thoroughly between samples.

6. EQUIPMENT AND SUPPLIES

6.1. Refer to ASB SOP GEN026 for purchasing services and supplies instructions.

6.2. Re-Certified Supporting Equipment

6.2.1. Pipets (Eppendorf or equivalent) are re-certified at least semi-annually.

6.2.2. Balances are re-certified at least annually.

6.3. The following brand names, suppliers, and part numbers are stated in this SOP for illustrative purposes. No endorsement is implied.

6.4. Thermo 6500 Duo inductively coupled argon plasma-optical emission spectrometer, including computer and Qtegra ISDS ICP-OES software

6.4.1. Thermoflex recirculating chiller

6.4.2. ESI SC FAST autosampler

6.4.3. PFA Microflow nebulizer

6.4.4. Cyclonic spray chamber

6.4.5. Charge injection device array detector

6.5. Peristaltic pump tubing (CPI part #)

6.5.1. PVC, flared, 2-stop, black/black, 0.76 mm i.d. (Part # 4062-430F)

6.5.2. PVC, flared, 2-stop, orange/green, 0.38 mm i.d. (Part # 4062-415F

6.5.3. PVC, flared, 2-stop, red/red, 1.14 mm i.d. (Part # 4062-445F)

6.6. Argon Supply (BLKMB Liquid Argon Micro Bulk)

6.7. Volumetric flasks, Class A glass or plastic (100 mL ± 0.2 mL and other volumes as required for dilutions)

Please note: Low level zinc contamination (~10 µg/L) has been observed by ICP-MS for new unused plastic volumetric flasks. Before using new plasticware, rinse with nitric acid followed by Super Q water.

6.8. 30 mL and 60 mL polystyrene cups

6.9. 50 mL centrifuge tubes

7. REAGENTS AND STANDARDS

7.1. Refer to ASB SOP GEN026 for purchasing services and supplies instructions.

7.2. Nitric Acid - Baker Instra-Analyzed (or equivalent). Each lot received is assigned a LIMS ID. Expiration date is five years from receipt.

7.3. Hydrochloric Acid - Ultrex, Baker Instra-Analyzed (or equivalent). Each lot received is assigned a LIMS ID. Expiration date is five years from receipt.

Please note: Concentrated HNO_3 and HCl may be found in LIMS under UNASSIGNED because any group may use them.

7.4. Nitric acid (1+1) - Add 250 mL conc. HNO_3 to 200 mL of Super Q water and dilute to 0.5 L. Solution expires after one year.

7.5. Hydrochloric acid (1+1) - Add 250 mL conc. HCl to 200 mL of Super Q water and dilute to 0.5 L. Solution expires after one year.

7.6. Water - Laboratory distilled water is passed through a mixed bed resin column before use. A "Super-Q" column or other deionization filtration system that produces 18 MΩ water may be used. All water used for ICP-OES analysis and standards is Super Q.

7.7. Carrier Solution

7.7.1. Water Carrier: 1% HNO_3/0.5% HCl carrier solution (LIMS description **ICP (w) FAST carrier**) – Acid matrix is matched to water samples. Add 20 mL conc. HNO_3 and 10 mL conc. HCl to Super Q water in a 2 L Nalgene container. Solution expires after one year.

7.7.2. Soil/Sediment Carrier: 2% HNO_3/2% HCl carrier solution (LIMS description **ICP (s) FAST carrier**) – Acid matrix is matched to soil/sediment samples. Add 40 mL conc. HNO_3 and 20 mL conc. HCl to Super Q water in a 2 L Nalgene container. Solution expires after one year.

7.8. 2% HNO_3/0.05% Triton-X 100 rinse solution (LIMS description **ICP FAST Rinse**) – Add 40 mL conc. HNO_3 and 1 mL Triton-X 100 to Super Q water in a 2 L Nalgene container. Sonication will speed dissolution of the Triton-X 100. Triton-X 100 is added to improve rinse-out characteristics. This solution is poured into the gallon jug labeled Rinse 1 below each instrument. Solution expires after one year.

Please note: 2% HNO_3/0.05% Triton-X 100 is no longer used as the default rinse solution. However, if inadequate washout is observed when using Super Q water, this rinse solution may be used.

7.9. Plasma-grade certified 10,000 and 1000 mg/L standards - These standards must be of sufficient purity that mixing standards will not result in addition of unknown amounts of other elements, possibly affecting the final concentration of the analyte. For this reason High-Purity, VHG Labs, and Inorganic Ventures standards (or equivalent) are recommended for mixed standards. Each standard is entered into LIMS upon its arrival. The expiration date by convention is the end of the month given by the vendor unless a specific date is given.

7.10. Reagents and Standard Preparation

7.10.1. All standard solutions must be entered in LIMS as soon as possible after preparation. All solution bottles shall be properly labeled with the contents, preparer's initials, date of preparation, expiration date, LIMS standard number, and acid matrix. The expiration date for all such working solutions is one year from the date of preparation.

7.10.1.1. When entering a new instance of a standard already in LIMS, select one that is already made and then click **Copy**. The relevant information is updated. Check that **Solvent/Solvent Lot** contains the acid matrix, usually 1% HNO_3/0.5% HCl (water) or 2% HNO_3/2% HCl (soil/sediment). Enter the **LIMS IDs** of acids used. Maintain a current list of standards by pruning expired ones. At this point, remove at least one expired standard by editing the **Department** field to read **Expired Standards**. Alternatively, check the **Inactive** box. Inactive standards are viewable when the **All** box is checked. If an expired standard is the only one of its type, retain the standard in the active list so that others may have the convenience of copying the record when preparing it again.

7.10.1.2. A uniform system of naming standards makes them easier to sort by the **Description** column in the **Metals Standards** screen and allows for labeling consistency. The naming convention is based on Technique,

Matrix, Standard. It is suggested that standards be named according to the **Standard ID** column in the preparation tables below.

7.10.2. The acid matrix of all calibration and analytical standards is matched to the final acid matrix of the sample preparation method. The acid matrix for standards prepared for undigested water analysis is 1% HNO_3.

7.10.3. The following table outlines the standards used for calibrating the instrument. The standards are prepared from High-Purity and VHG Labs EPA Methods standards (or equivalent).

Standard ID	Stock ID	Element(s)	Stock Conc. (mg/L)	Final Conc. (mg/L)
ICP CAL BLANK			0	0
ICP CAL 1	VHG Labs 44CS1Y-100 VHG Labs 44CS2Z-100	Al Ca Fe K Mg Mo Na P SiO_2 Ti As Li Sb Se Tl Zn B Ba Be Cd Ce Co Cr Cu Mn Ni Pb Sn Sr V Ag*	1000 500 200 50	10 5 2 0.5
ICP CAL 2 Ag	High-Purity Silver 10 51-1	Ag	10	0.1

*Present in the standard but not currently used for calibration

Please note: Calibration standards are prepared in 100 mL volumetric flasks. Add 50 mL Super Q and appropriate volumes of 1:1 HCl, and 1:1 HNO_3 per matrix type to each flask. Add the required volume of stock standard(s) and bring to volume. Transfer the working standard to the designated 125 mL Nalgene calibration standard bottle. During analysis, the calibration standards are transferred to 50 mL centrifuge tubes placed in the autosampler. Standards in use may be left in the centrifuge tubes. The CAL BLANK is the exception and is made fresh on the day of analysis in a plastic volumetric flask to avoid introducing contaminants into the CAL BLANK, such as zinc, which could bias subsequent results low.

Please note: When preparing standards, always transfer the stock standards to a fresh 30 mL polystyrene cup to avoid contamination. Never pipet directly from a stock standard bottle. Always close the bottle immediately after pouring to maintain the stock standard concentration. Do not leave

standards or acids in the polystyrene cups for extended periods of time, as they will pick up contaminants such as zinc.

7.10.4. The following table outlines the analytical standards used during an analysis. The standards are prepared from a second source (Inorganic Ventures), which is separate from the sources used to prepare the calibration standards.

Standard ID	Stock ID	Element(s)	Stock Conc. (mg/L)	Final Conc. (mg/L)
ICP LCB			0	0
ICP LCM 1	Inorganic Ventures QCP-QCS-1 QCP-QCS-2	K P SiO_2 Sn Tl As Hg* Pb Sb Al B Ba Be Ca Cd Ce Co Cr Cu Fe Li Mg Mn Mo Na Ni Se Sr Ti V Zn Ag*	500 200 100 25	5 2 1 0.25
ICP LCM 2 Ag	Inorganic Ventures Silver MSAG-10PPM	Ag	10	0.05
ICP LCM HI	Inorganic Ventures EPA-V-3C	Al Ca Fe K Na Mg	1000 600	100 60

*Present in the standard but not used in this standard for QC purposes

Please note: Analytical standards are prepared in 200 mL volumetric flasks. Add 100 mL Super Q and appropriate volumes of 1:1 HCl, and 1:1 HNO_3 per matrix type to each flask. Add the required volume of stock standard(s) and bring to volume. Transfer the working standard to the designated analytical standard bottle. The LCB is made fresh on the day of analysis in a plastic volumetric flask. LCM1, LCM2 Ag and HiLCM may be prepared at a volume of 200 mL or 500 mL at the analyst's discretion since there are at least two QC sets per run.

7.10.5. The following table outlines the Reporting Limit Check (RLC) multielement stock and working RLC standard. The RLC multielement stock is a vendor-prepared custom stock (High-Purity #SM-2985-002) but may be prepared from High-Purity (or equivalent) single-element standards in a 100

mL plastic flask if the custom stock is unavailable or project-specific reporting limits are requested. The working RLC standard is prepared in a 100 mL plastic flask by diluting 2 mL RLC multielement stock (Low Level) in an acid matrix of 1% HNO_3/0.5% HCl (water) and 2% HNO_3/2% HCl (soil/sediment).

Stock ID	Elements	RLC Stock Conc (mg/L)	Working RLC Conc (µg/L)
Low Level	K	40	800
	Al Ca	25	500
	Na	20	400
	Mg P SiO_2	10	200
	Fe	4.0	80
	Tl	3.0	60
	B Se	2.5	50
	As	2.0	40
	Pb Zn	1.5	30
	Li	1.25	25
	Cu Sb Sn	1.0	20
	Mo Ni	0.60	12
	Ag	0.50	10
	Mn	0.40	8.0
	Co Ti	0.30	6.0
	Cr Sr V	0.25	5.0
	Ba	0.15	3.0
	Be Cd	0.10	2.0

Please note: Any elements inadvertently omitted from the low level spike solution may be prepared as a single element stock and added to the working RLC standard or may be analyzed in a single element working standard.

7.10.6. The following table outlines the Spectral Interference Check (SIC) standards. SICs are analyzed at least once each analysis day directly following instrument calibration or anytime during a run if high analyte concentrations are observed. Individual SICs may be omitted from a run if prior information is known about the field samples such that high concentrations of interfering analytes are not expected or if high concentrations are not observed during the run. The components of these standards were chosen based on spectral evidence found during method

development which points to possible areas of weakness in the IEC models. The standards are prepared from High-Purity (or equivalent) standards.

SIC Solution	Concentration (mg/L)
ICP SIC Al	200
ICP SIC Ba	10
ICP SIC Ca	200
ICP SIC Cr	10
ICP SIC Cu	10
ICP SIC Fe	300
ICP SIC Mg	200
ICP SIC Mn	20
ICP SIC Mo	10
ICP SIC Ni	10
ICP SIC Si	30
ICP SIC Sn	5
ICP SIC Ti	10
ICP SIC V	10

Please note: SICs other than the concentrations and solutions listed above can be analyzed if based on the analyte concentrations observed during a run or if the linearity of the instrument is exceeded.

Please note: The SIC standards are prepared in 100 mL volumetric flasks. Add 50 mL Super Q and appropriate volumes of 1:1 HCl, and 1:1 HNO_3 per matrix type to the flask. Add the appropriate amount of the concentrated stock(s) to the flask. For example, for 10 mg/L, add 1 mL of 1000 mg/L stock to the flask. For 200 mg/L Fe, add 2 mL of 10,000 mg/L Fe stock to the flask.

7.10.7. The following tables outline the spike solutions used to prepare the digestion blank spike and sample designated as the matrix spike. AB and solid spikes are purchased as custom spike mixes from High-Purity (or equivalent) standards. Alternatively, these may be prepared from single-element standards if the purchased custom spike mix is unavailable or a project requires a spike concentration other than what is described below.

7.10.7.1. Water Spikes

Spike Solution	Element(s)	Stock Conc. (mg/L)	Final Conc. (mg/L)*
AB	Be	0.5	0.01
	Ag	1.25	0.025
	Cd	2.5	0.05
	Co Cr Cu Li Mo Ti V	5	0.1
	As Ba Mn Ni	10	0.2
	Ce Pb Sb Se Sn Tl	25	0.5
	Al B Fe Sr P Zn	50	1
	SiO_2	500	10
	Mg	1000	20
	K	1250	25
	Ca Na	2500	50

*of analyte in digest, assuming 1 mL of Spike Solutions AB is added to 50 mL sample aliquot

Please note: Ag has been observed to give increasingly lower recoveries over time in the AB spike. If Ag begins to show consistently low recovery, Ag may be spiked using a single element standard into a separate Ag only blank/matrix spike or Ag may be omitted from the AB spike and spiked using a single element standard into the multielement blank/matrix spike.

7.10.7.2. Soil/Sediment Spike

Spike Solution	Element(s)	Stock Conc. (mg/L)	Final Conc. (mg/L)*
Solid	Mn Zn	100	10
	Ba Cr Cu Pb	50	5
	As Ni Sb Se Tl	20	2
	B Cd Mo Sn Sr	10	1
	Co Li V	5	0.5
	Ti	2	0.2
	Ag	1	0.1
	Be	0.5	0.05

*of analyte in digest, assuming 5 mL solid spike solution is added to a 0.5 g sample; after digests are brought up to 50 mL; Values in mg/L are multiplied by the solids correction factor to yield mg/kg in the original sample.

Please note: Mo, Sb, and Sn were previously prepared daily as a separate unstable spike solution at the concentrations listed above, but they were included in the most recent solid spike solution. If any element contained in

the solid spike solution begins to show consistently low recovery, that element may be omitted from the solid spike solution and prepared as an unstable spike solution.

7.10.7.3. The water spikes can also be used for soil/sediment at double the concentration.

7.10.8. The following table outlines the TCLP spike solutions. These solutions are used to prepare an additional type of matrix spike for TCLP extracts and are added prior to preservation of the extract. The solutions are prepared from High-Purity (or equivalent) standards in an acid matrix of 3% HNO_3/3% HCl for TCLP spike As Ba Cd Cr Pb Se and 2% HNO_3 for TCLP spike Ag.

Spike Solution	Element(s)	Volume High-Purity Stock Used (mL)†	Stock Conc. (mg/L)	Final Conc. (mg/L)*
TCLP spike As Ba Cd Cr Pb Se	As Ba Cr Pb Cd Se	10 2	100 20	0.5 0.1
TCLP spike Ag	Ag	0.5	5	0.01

*of analyte in digest, assuming 250 µL of TCLP spike As Ba Cd Cr Pb Se and 100 µL of TCLP spike Ag are added to 5 mL sample aliquot and diluted to 50 mL

†High-Purity stock standard concentrations are 1000 mg/L for TCLP spikes.

7.10.9. The following table outlines the low level spike solution used to prepare the reporting limit verification sample. The low level spike solution is purchased as a custom spike mix from High-Purity (or equivalent) standards. Alternatively, this solution may be prepared from single-element standards if the purchased custom spike mix is unavailable or a project requires reporting limits other than what is described in the section pertaining to the preparation of the RLC solution.

Stock ID	Elements	Stock Conc (mg/L)	Final Conc (µg/L)*
Low Level	K Al Ca Na Mg P SiO_2 Fe Tl	40 25 20 10 4.0 3.0	800 500 400 200 80 60

Stock ID	Elements	Stock Conc (mg/L)	Final Conc (µg/L)*
	B Se	2.5	50
	As	2.0	40
	Pb Zn	1.5	30
	Li	1.25	25
	Cu Sb Sn	1.0	20
	Mo Ni	0.60	12
	Ag	0.50	10
	Mn	0.40	8.0
	Co Ti	0.30	6.0
	Cr Sr V	0.25	5.0
	Ba	0.15	3.0
	Be Cd	0.10	2.0

*of analyte in digest, assuming 1 mL of the low level spike solution is added to 50 mL sample aliquot or 0.5 g soil/sediment sample, digests are brought up to 50 mL

7.10.10. Should a consistent bias be discovered in a standard, usually by comparison to a second source, a third source, usually NIST, is employed to determine which is more correct. The vendor(s) are contacted to negotiate a replacement, or the vendor is changed.

7.10.11. The traceability of both the single-element standards and mixed-element standards is given on the Certificate of Analysis sheet that is provided by the manufacturer. Hardcopy certificates (when provided) are kept in the ICP-OES laboratory, and electronic certificates are uploaded into LIMS.

7.10.12. Yttrium internal standard, 10 mg/L (LIMS description **ICP FAST ISTD Y**) - Add 2 mL of 10,000 mg/L Y stock standard to a 2 L Nalgene container to which has been added 20 mL conc. HNO_3 and 10 mL conc. HCl in Super Q water.

7.10.13. Pre-made stocks and solutions may be purchased from a vendor instead of being prepared in-house.

7.10.14. The preparation of digestion QC samples may be found in Section 10 Quality Control.

8. SAMPLE HANDLING AND PRESERVATION

8.1. All samples must have been collected using an approved/appropriate sampling plan.

8.2. Water samples should be collected in polyethylene bottles with solid polypropylene caps. It is recommended that the bottles be pre-treated. The bottles may be soaked for 24 hours with dilute HNO_3 before use. The usual contaminants have been found to be lead and zinc. At least 200 mL of sample is requested to allow a duplicate and spike to be analyzed. More would be necessary if reanalyses were needed.

8.3. Water samples are preserved in the field to a pH<2. Use 5 mL of 1:1 HNO_3 per liter of sample or blank. More acid may be used if necessary. If the sample is not acidified for any reason, especially due to an anticipated hazard, an indication of this condition should accompany the samples and be directed to the attention of the Metals Analyst. If pH is >2, either the pH must be adjusted with HNO_3 and time allowed to re-solubilize the analyte(s) that may have adsorbed onto the container walls (24 hour minimum), or if the sample is extremely basic or highly buffered and addition of acid would cause precipitation of the analytes of interest, this fact and the pH of the sample must be documented. All pH adjustments should be made within two weeks of sampling.

8.4. Solid samples require no preservation prior to analysis. There is no established holding time limitation for solid samples other than what is recommended by the Office of Solid Waste and Emergency Response (OSWER) in SW-846 or required by OSWER for its Contract Laboratory Program (CLP).

8.5. Samples can be held for six months prior to analysis. Digests are retained for at least 2 ½ months after results have been reported.

8.6. Ground samples and digests are labeled with LIMS number.

8.7. Dilutions are labeled with LIMS number and “1/N” for “dilution 1/N”, where N = dilution factor.

8.8. Field filtered (dissolved metals) samples are digested and treated the same as total metals samples.

8.9. Digestion is not required for drinking water where sample turbidity is <1 NTU.

9. SAMPLE PREPARATION AND ANALYSIS

9.1. Sample Preparation

9.1.1. Refer to the appropriate preparation SOP for sample preparation procedures and user manuals for the use of appropriate supporting equipment. Generally, METALS025 hot block digestion is used to prepare samples to be analyzed by this analytical SOP.

9.1.2. Refer to the appropriate preparation SOP and/or Section 10.3 of this SOP for preparation of digestion/batch quality control audit samples.

9.2. Sample Analysis

9.2.1. Instrument Preparation

9.2.1.1. Turn on exhaust fan and chiller and check the level of waste in the waste carboy.

9.2.1.2. Turn on the computer and open the Qtegra ISDS software. The software opens on the **Dashboard** page and shows the current instrument and autosampler configuration.

9.2.1.3. Perform the following routine daily before turning on the plasma.

- Check the pressure and volume of argon in the tank.
- Inspect the peristaltic pump tubing for damage or deterioration and change if necessary. Engage pump tubing. Flared-end polyvinyl chloride orange/green and black/black tubing are used to pump the internal standard and carrier solutions, respectively. Santoprene gray/gray or PVC red/red tubing may be used for the drain. Record tubing changes in the logbook.
- Check autosampler rinse supply and refill if necessary.

- Place uptake tubes into Super Q water once the plasma has been lit and verify the correct direction of liquid flow.

9.2.2. Plasma Ignition

9.2.2.1. On the **Dashboard** page verify the interlock indicators are green. If an interlock indicator is red, correct the problem.

9.2.2.2. Click the **Get Ready** button (shown below) to light the plasma and perform the spectrometer optimization after a 15 minute warm-up.

- When the **Get Ready** button is clicked, verify that **Warm-up** (15 minutes), **Spectrometer Optimization**, and **Use Manual Sampling** are selected in the dialog box and click **OK**.

- For manual sampling, click **OK** when the two dialog boxes listed below are opened. These boxes open sequentially once the plasma is lit and after the spectrometer optimization is completed.

  - Move the probe to wash position.
  - Move the probe to the vial containing the solution used for warm-up/after completion.

9.2.2.3. Allow the instrument to warm up for at least 1 hour.

9.2.3. LabBook Creation and Sample List Setup

9.2.3.1. Click on the **LabBook** page to create a new LabBook from an existing Template or LabBook. A new LabBook can also be created from the **Template** toolbar of an open template. Consult the instrument logbook for changes made to the current Template (data acquisition or

analysis methods, operating conditions, calibration, interelement correction factors, etc.).

- Type a name for the new LabBook and select a location for the LabBook to be saved.

- Choose one of the options below and select the name of the existing Template or LabBook from the drop-down list. When creating a new LabBook from a template, enter the number of samples to be included in the sample list.

  - Create a new LabBook from an existing Template.
  - Create a new LabBook from an existing LabBook.

- Click **Create LabBook**.

9.2.3.2. On the **Content** pane of the new LabBook, click on **Sample List** to edit the list of samples to be analyzed.

- Calibration standards and instrument QC samples are automatically inserted into the sample list based on the **Sample Definition** page of the Template from which the LabBook was created. Verify that the appropriate calibration standards and QC samples have been inserted at the frequency specified in this SOP and confirm the sample types (BLK/STD/QC/UNKNOWN) and autosampler positions.

- The number of samples specified when the LabBook was created is automatically inserted into the sample list between QC samples. Enter the sample IDs, autosampler positions, and sample types (QC/UNKNOWN) for the field samples and insert additional samples as needed.

- Additional sample information can be entered into the sample list to enable automatic QC evaluation by the software.

- Under the **Standard** column, select the **Quality Control Standard** (Reporting Limit Check, Blank Spike, LCM1, etc) that corresponds to the concentrations by which the sample should be evaluated.
- Under the **QC Action** column, select the **Quality Control Test** (CCB, RLC, LCS, CCV, etc) that corresponds to the limits by which the sample should be evaluated.
- To evaluate a QC sample that refers to another sample (duplicate, matrix spike, dilution test), enter the same designation ("a", "1", etc) under the **QC Reference** column for the QC sample and the sample to which it refers.

- A typical analytical sequence is as follows:

  - Instrument calibration standards
  - Initial QC checks (i.e. LCB, RLC, LCM1, LCMHI, and LCM2 Ag).
  - SIC solutions, generally followed by the QC checks.
  - Series of field sample digests (10 or fewer) not including digestion QC (i.e. blanks, blank spikes, reporting limit verifications, laboratory duplicates, matrix spikes, and standard reference material).
  - QC checks (LCB, RLC, LCM1, LCMHI, and LCM2 Ag).
  - Another group of 10 field samples or less, if present, and QC checks until finished.
  - Final QC checks are analyzed at the end after all the samples and digestion QC.

9.2.4. Calibration and Sample Analysis

9.2.4.1. Insert the carrier probe into the carrier solution (1% HNO_3/0.5% HCl for water or 2% HNO_3/2% HCl for soil/sediment) and the internal standard probe into the yttrium internal standard. Allow at least 15 minutes for the internal standard to reach the plasma and stabilize before starting the data acquisition.

9.2.4.2. Load the standards and samples into the correct autosampler locations.

9.2.4.3. Click the green arrow button on the **LabBook** toolbar to schedule the LabBook and then click the green arrow button on the **Scheduler** toolbar to begin the analysis.

9.2.4.4. Unattended instrument shutdown can be enabled on the **Scheduler** toolbar > **Options** > **System Options** > Close down on an empty queue.

9.2.5. Instrument Shutdown

9.2.5.1. Insert the carrier and internal standard probes into Super Q water and flush the system while the plasma is still on.

9.2.5.2. Extinguish the plasma by clicking on the **Dashboard** page > **Get Ready** button > **Shut Down** and allow the plasma to shutdown before continuing instrument shutdown.

9.2.5.3. Turn off chiller and exhaust fan.

9.2.5.4. Disengage peristaltic pump tubing.

9.2.6. Data Export

9.2.6.1. Data should be uploaded to the R5CRL server as soon as possible after completion of the run. Raw data to be uploaded include the XML LIMS export file and pdf report export file. See ASB SOP METALS005 for data upload to R5CRL.

9.2.6.2. Create the export file that is amenable to Data Tool upload on the **Content** pane of the LabBook > **Concentration** view > **LabBook** toolbar > **Export**. Select the XML export file type in the dropdown list and verify that all content boxes are checked. Choose a file name and location and click **Export**.

9.2.6.3. Create the pdf data report export file on the **Content** pane of the LabBook > **Report**. Generate and save the following report using the associated template.

- Chicago EPA Sample Report with Calibration and Sample List

10. QUALITY CONTROL

10.1. Operator Requirements

10.1.1. Before using this procedure to analyze samples, data should be available documenting the initial demonstration of performance. The required data should document the determination of the linear ranges; demonstration of the desired method sensitivity and instrument detection limits; and determination and verification of the appropriate correction factors or other routines for correcting spectral interferences. These data should be generated using the same instrument, operating conditions, and calibration routine to be used for sample analysis. These data should be kept on file and be available for review by the data user or auditor. The linear ranges should be verified annually thereafter or when there have been significant instrument modifications that may affect linearity, a full linearity study, consisting of a minimum of three different concentration standards across the range, should be performed. Verification is accomplished by analyzing one standard at the previously determined linearity limit. The recovery should be no less than 10% below the nominal standard concentration to confirm that the linearity has not changed. The current linearity study for all analytes is located in the Qualtrax Document Tree under the METALS003 SOP (Qualtrax ID #10125).

Please note: If an annual linearity verification is not performed, samples that exceed the analyte concentration in the calibration standards, should be diluted and rerun or a linearity check should be run as a one-day linearity.

10.1.2. Sensitivity, instrumental detection limit, precision, linear range, and interference corrections need to be established for each individual target analyte on each particular instrument. All measurements (both target analytes and constituents which interfere with the target analytes) need to be within the instrument linear range where the correction factors are valid.

10.1.3. The instrument is calibrated daily using a blank and one non-zero standard, which is verified with mid- and low-level initial calibration verification

(ICV) standards analysis using the limits specified in the section below. No more than 10% of the reported target analytes or 1, whichever is greater, may fail the mid-level ICV to continue with analysis. When the ICV fails despite immediate corrective action attempts, the affected data may be reported semi-quantitatively (qualified).

10.2. Instrument Check Audits

10.2.1. A set of instrument checks (LCB, LCM1, LCM2 Ag, LCMHI) should be analyzed for every 10 field samples or fraction thereof. The RLC should be analyzed at least as frequent as the beginning and end of a run. As such, it is advisable to include an additional RLC check(s) within a long run in case some circumstance occurs to prevent analysis of the concluding RLC. Audit limits are given in the table below. Any values beyond these limits are flagged.

Analytical QC Audit ID	Audit Limits
LCB	± method detection limit
LCM1, LCM2 Ag, LCMHI	100 ± 5% recovery, immediately following calibration 100 ± 10% recovery, subsequent analyses
RLC*	100 ± 30% recovery

*The low level initial/continuing calibration verification is no longer included in the most recent version of SW-846 Method 6010 (D). However, it is still used by this SOP to monitor the instrument's low level performance throughout the run. Consideration will be given to removal of this instrument check audit in future revisions of this SOP.

Drinking Water Compliance note: For drinking water compliance, the instrument must be recalibrated if the LCM falls outside the audit limit for a requested analyte. The instrument does not require recalibration if the analyte concentrations in the affected samples are within ten times the reporting limit check with acceptable bracketing RLC audits.

10.2.2. The solutions analyzed as SIC standards are those with which the IEC model has been shown to be imperfect relative to the RL of the analytes that are interfered with. In an ideal model, the contribution from the interfering element would be completely removed, and noise in the signal would be the only component remaining affecting the analyte reading. However, contribution from an interferent may result in a false positive or negative.

10.2.2.1. SIC results are compared to RLs. If any SIC result is outside the RL for a primary line but inside the RL for an alternate line, the IEC factor on the primary line should be re-evaluated. The IEC factor is recalculated by accessing the **Content** pane of the LabBook > **Intensities** view. Right-click on the SIC result to be used for IEC recalculation > **Inter-Element Correction** > **Enable** check box next to the interfering element and click the green refresh button in the **Formula** column > **OK**. All data will be recalculated using the newly determined IEC factor.

10.2.2.2. If the concentration of an analyte in a sample exceeds the concentration of an existing SIC or the calibration if no SIC is routinely run for the analyte, an additional SIC may need to be analyzed.

- For analytes with sample results above the RLs and comparable RLs for the primary and alternate lines, the lines should agree within about 20%. If there is disagreement, an SIC should be analyzed for the high concentration element and IEC factors adjusted if the SIC results exceed the RLs.

- If there is still disagreement between the primary and alternate lines after adjustment of the IEC factors, the **Subarray Plot** in the **Details** window should be examined to see if there is an additional interference on the primary line. The **Subarray Plot** is accessed on the **Content** pane of the LabBook > **Intensities view** by double-clicking an intensity value. The **Subarray Plot** can be viewed as a **Smoothed Plot** or a **Bar Chart** by right-clicking the graph and selecting/deselecting **Smoothed Plot**.

- If it becomes apparent through examination of the alternate lines and **Subarray Plots** that there in an additional interference that is inadequately corrected on the primary line, an alternate line may be used for reporting if it is of comparable sensitivity.

10.3. Digestion/Batch Audits

10.3.1. A complete set of digestion/batch QC are required for every 10 field samples or less except the digestion blank, digestion blank spike, and

standard reference material. One blank/blank spike pair and SRM are required to accompany every 20 samples in the batch. Additional blanks and blank spikes may be prepared with the sample batch at the analyst's discretion. One reporting limit verification per LIMS batch number is required.

Please note: If insufficient sample volume is provided to perform sample, duplicate, and matrix spike preparation, these QC samples may be omitted or the sample volume, sample mass, and/or spike volume adjusted accordingly.

10.3.2. Digestion Blank (BLK) - The same volume of Super Q water as used for the samples treated as a sample. All steps used for the samples (e.g., filtration or centrifugation) are also performed on the BLK. The BLK demonstrates that there is no contamination in the reagents or glassware. The limit for this audit is ± MDL. Any values beyond these limits are flagged.

Drinking Water Compliance note: For drinking water compliance, re-analysis of the BLK and affected samples must be performed when the BLK result for a requested analyte exceeds 10% or more of the analyte concentration for a sample. If acceptable BLK results are not obtained after re-analysis, then the affected samples should be re-prepared and analyzed after the source of contamination has been corrected.

10.3.3. Duplicate (DUP) - This is a second aliquot of a field sample treated as a separate sample. The DUP gives an indication of sample homogeneity and consistency of subsampling. The limit for this audit is an absolute difference of ± MDL when near the detection limit or a relative percent difference (RPD) of ≤ 20%. Any values beyond these limits are flagged. RPD is defined as

$$RPD = \frac{|D - S|}{(\frac{D + S}{2})} X\ 100$$

where S is the sample result, and D is the duplicate result. The results used for calculation are in units of mg/L for water and mg/kg for soil/sediment.

10.3.4. Matrix Spike (MS) - This is a second aliquot of a field sample to which the appropriate spike solutions per matrix type have been added. Current practice is to add 1.0 mL of spike solution AB to a 50 mL aliquot for water, although less sample volume may be used if 50 mL is not provided or 5.0 mL of solid spike solution to a 0.5 g sample for solids. The MS is then processed the same as a regular sample. Low spike recovery on three or more elements may indicate poor digestion results. For a complex sample, low spike recovery may indicate interferences; or when the DUP result is considered, it may indicate a lack of homogeneity or a failure to obtain a representative subsample. A low spike may also be caused by instrument drift. The instrument QC checks will confirm any instrument drift or nebulizer problem. The limit for this audit is a percent recovery defined as

$$\%\ Recovery = \frac{(Sp - S)}{A} X\ 100$$

where Sp is the result for the MS, S is the result for the sample, and A is the added spike. The results used for calculation are in units of mg/L for water and mg/kg for soil/sediment. The limit for this audit is 100 ± 25% recovery and is valid up to the point where the spike concentration is less than 30% of the sample concentration. Any values beyond these limits are flagged. If the accuracy audit is invalidated by high source concentration, the BS is used as accuracy audit. Matrix spikes that exceed the linear range for a given channel should be diluted and rerun just as any sample would be diluted and rerun for that element. Alternatively, a linearity check can be performed as a one-day linearity.

10.3.5. Digestion Blank Spike (BS) - This is a blank to which the appropriate spike solutions per matrix type have been added and then digested. The percent recovery is defined as

$$\%\ Recovery = \frac{BS}{A} X\ 100$$

where BS is the concentration measured for the BS and A is the added spike. The limit for this audit is 100 ± 15% recovery. Any values beyond these limits are flagged.

Drinking Water Compliance note: For drinking water compliance, failure of the blank spike audit for a requested analyte should be investigated and resolved before continuing analysis.

10.3.6. Reporting Limit Verification (RLV) –This is a blank to which the low level spike solution has been added and then digested. The percent recovery is defined as

$$\% \, Recovery = \frac{RLV}{A} x \, 100$$

where RLV is the concentration measured for the RLV and A is the added spike. The limit for this audit is 100 ± 35% recovery. Any values beyond these limits are not flagged, but repeated failure of the RLV may indicate the need to raise the RL to a level at which the percent recovery criteria can be met.

10.3.7. Standard Reference Material (SRM) – The SRM is applicable to solid samples only. The limits for this audit are given in the manufacturer's Certificate of Analysis, under "QC Performance Acceptance Limits". The Certificate of Analysis is subordinate to this SOP in the Qualtrax document tree. Any values beyond these limits are flagged. As enough data points are generated (at least 20-30), a laboratory average will be calculated.

Please note: The soil SRM may exceed 0.5 mg/L of silver in the digest for a 0.5 g aliquot. Therefore, a smaller aliquot may be needed.

10.3.8. If the field sampler has designated a sample for matrix QC, that is the sample to be used as the DUP and MS. Additional samples may be chosen at the analyst's discretion except designated field blanks. A field blank may not be used for the DUP or MS.

Please note: If a field blank is the only water sample in a work order, the DUP/MS pair may be omitted in the digestion batch for that work order.

10.3.9. A complete set of batch QC are required for undigested drinking water following the same type and frequency as specified for digestion QC.

10.3.10. An alternate spectral line may be used for reporting if failure of instrument or digestion/batch QC audits would preclude the use of the primary analytical line and if the alternate line is of comparable sensitivity to the primary line. This also applies to analytes with low and high channels.

10.3.11. Dilution Test

10.3.11.1. A dilution test should be performed if the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrument detection limit in the original solution but <90% of the linear limit), an analysis of a 1+4 dilution should agree (after correction for the fivefold dilution) within ± 10% of the original determination. If not, a chemical or physical interference effect should be suspected, and the associated data flagged accordingly.

10.3.11.2. When a new or unusual matrix is encountered, a dilution test may be performed prior to reporting concentration data for samples of that matrix to ensure that neither positive nor negative interferences are operating on any of the analytes to distort the accuracy of the reporting value. However, for low level samples, a dilution test may not be useful if analyte concentrations are diluted below the reporting limit.

10.3.11.3. If a high concentration sample is not available for the dilution test, then perform the dilution test on the matrix spike since reasonable concentrations of most analytes are present.

10.3.12. Quality Control Summary

Audit	Frequency	Limit
LCB	Beginning, end, and once every 10 samples	± MDL
RLC	Beginning, end, and more frequently as needed	70 - 130% Recovery
LCM	Beginning, end, and once every 10 samples	95 - 105% Recovery (immediately following calibration) 90 - 110% Recovery (subsequent analyses)

Audit	Frequency	Limit
SIC	Per calibration as needed based on sample conc	± RL
RLV	One for each LIMS batch ID number	65 – 135% Recovery; Nominal Spike Conc = RL per analyte
BLK	One for each group of 20 samples or fewer	± MDL
BS	One for each group of 20 samples or fewer	85 - 115% Recovery
DUP	Per site and one for each group of 10 samples or fewer	RPD ≤ 20% or absolute difference ± MDL when near the MDL
MS	Per site and one for each group of 10 samples or fewer	75 - 125% Recovery
SRM	One for each group of 20 samples or fewer (soil/sediment only)	acceptance limits given in manufacturer's Certificate of Analysis
Dilution Test	One per batch and more as needed per new or unusual matrix	RPD ≤ 10% if analyte conc is greater than 50x MDL after dilution

10.3.13. The QC audit limits given in this section are derived from the reference methods, as these methods allow for fixed limits. Historical limits were reviewed in January 2020 (digested waters) and September 2020 (soil/sediment) for BLK, BS, RLV,SRM (soil/sediment only), DUP, and MS and are available in the Qualtrax document tree subordinate to this SOP (Qualtrax ID #'s 20865 and 22929, respectively).

10.4. Acceptance of Run/Element Data.

10.4.1. When analyzing simultaneously for many elements, it is statistically predictable that some audits will be outside the stated limits due to random error. However, there is no generally accepted statistical method for determining that an analysis is in control when an audit has exceeded control limits. If an element of interest to the client is out of control for an audit, the samples shall be re-analyzed, or re-prepared and re-analyzed, as needed. These steps may be avoided if, after discussion with the client, the failed audit is found to have little effect on the intended use of the data.

Data quality objectives should be part of a quality assurance project plan received with the samples. These should give action limits and intended use of the data. If these are not present, discussion with the sampler or the project manager can help with the decision whether reanalysis is necessary. For example, if an element is found in the blank above the detection limit, or the spike recovery for the element is high, but all results for the element are below the action level for the Program, the data are usable.

10.4.2. The run must be reviewed for systematic errors. Systematic errors may be found in at least three categories: contamination, which may be random or consistent; drift of baseline; or change in the calibration. This by no means covers all types of error, rather only the most common. Evidence of systematic error would be cause for rejection of an element from a run or, in extreme cases, rejection of the run.

10.4.2.1. Systematic error from low level contamination is difficult to spot but can be diagnosed by examination of blanks and field blanks. Duplicates and spikes may also show low level contamination if the concentration in the sample itself is low. Field blanks should be confirmed to show that the contamination does not arise from the laboratory. One member of a DUP/MS pair may be elevated by contamination, throwing the audit out of control. The pattern of all the audits from a digestion run must be examined for evidence of contamination. The Metals Group Leader or QC Coordinator may choose to have the run redigested for that element.

10.4.2.2. Systematic errors from baseline drift, if positive, may appear to be similar to the contamination mentioned above. It is easily confirmed during analysis by aspirating the calibration blank. It is less easily diagnosed after the run is over. Negative drift is equally to be avoided because false negatives can result. It is possible that other QC audits may be in control even if the blanks show unacceptable drift.

10.4.2.3. A change in the calibration may occur as a result of a nebulizer clog or change in plasma temperature during the course of a run. These problems will affect different elements differently and may cause changes in different directions. The instrument QC should be examined

not only to show if they are in or out of control but for trends. For example, a low LCM that is within control but biased low may indicate why a spike for that element becomes flagged. Only a portion of a run may be affected, in which case only that portion needs to be rerun.

10.4.3. Corrective action when the QC is outside limits depends on the severity of the violation and on the statistical nature of multianalyte analysis.

10.4.3.1. If the QC limit excursion affects the determination of a violation of Program limits or the DQOs of the project, the first recourse is for reanalysis. If re-preparation is necessary, the presence of sufficient sample to carry this out and holding time are factors. If reanalysis is not possible, this must be explained to the client.

10.4.3.2. If the QC limit violation is not critical, the corrective action hinges on the statistics. Typically, 5% of the determinations may exceed the limits. If the number of violations exceeds this amount, an examination must be made as to whether the samples were overly difficult or the instrument requires maintenance. If the former, repreparation may be needed to verify this. If the latter, the digests may need to be reanalyzed after the maintenance is performed.

10.4.3.3. Any QC limit violations shall be flagged on the data as described in Section 4 of ASB SOP METALS005.

10.4.3.4. Refer to ASB Policy 024 (doc #16310) for formal corrective action instructions.

10.4.4. All QC violations should be logged into the QC excursion spreadsheet per work order by analyte. Any consistent (three times in a row per final reported analysis) QC failure should be investigated, findings documented, and corrective actions taken as appropriate.

10.5. The measurement of the method detection limit (MDL) is performed by the procedure specified in 40 CFR Part 136 Appendix B. The reporting limit (RL) is determined to be 3 to 10 times the MDL value, and this becomes the concentration of the RLC solution. Frequency of MDL determination is a function of instrument

conditions and historical QC data. The current ASB policy is for MDLs to be renewed annually if results are reported below the RL or if required by regulation.

10.5.1. If the instrument has had major work done to the spectrometer, then a measurement of a new MDL must be performed.

10.5.2. If there is a change to the SOP or the instrument method, such as a change in the RF power, wavelengths, correction scheme, mode of viewing, or calibration range, then a measurement of a new MDL must be performed.

10.5.3. If there is a trend observed in the historical data obtained for the BLK or recovery of the RLC, as displayed in the control charts or range charts, then consideration is given to recalculation of the MDL depending on the effect on reported data and/or the length of time until the next annual MDL determination.

11. DATA AND RECORDS MANAGEMENT

11.1. No calculations are required for water sample results. Concentrations from the instrument output file are uploaded to LIMS in units of mg/L and are reported in units of mg/L to 3 significant figures but no more than 4 decimal places. Results are reported down to the reporting limit.

11.2. Soil and sediment digest concentrations from the instrument output file are uploaded to LIMS in units of mg/L and are reported to 2 significant figures in units of mg/kg on a dry weight basis. Results are reported down to the reporting limit. The calculation to convert concentration in mg/L in the digest to concentration in mg/kg dry weight is:

$$\frac{C_{mg}}{kg} = \frac{C_{mg}}{L} x \, {}^{V_L}/_{m_g} \, x \, {}^{10^3 \, g}/_{kg}$$

where $C_{mg/kg}$ is the analyte concentration in mg/kg; $C_{mg/L}$ is the analyte concentration in the digest in mg/L; V_L is the final volume of digest in L (for METALS025 hot block digestion, the final volume is normally 0.05 L); and m_g is the initial sample mass in g.

11.3. ASB uses Element DataSystem with DataTool for information management and reporting. Refer to Appendix C for detailed instructions on bench sheet creation, data entry, and report generation.

11.4. The instrument raw data file(s) and all other electronic files related to the sample batch are uploaded to R5CRL. Refer to ASB SOP METALS005 for upload of inorganic data to R5CRL.

12. TROUBLESHOOTING

12.1. Analytical performance cautions and interferences are discussed in Section 5.

12.2. Equipment problems are discussed in Section 13.

12.3. Corrective actions when QC is outside limits are given in Section 10.

12.4. If the plasma does not initiate, check the following:

12.4.1. Verify argon is flowing to the torch.

12.4.2. Verify that bubbles are exiting through the clear tubing on the spray chamber drain side soon after the peristaltic pump starts. The plasma will not initiate if the spray chamber is flooded. This can occur if the drain tubing was misaligned or improperly plumbed. If the spray chamber is flooded, recheck tube plumbing and tube clamps at the peristaltic pump. Turn the pump on for several minutes to empty the spray chamber. If several attempts to initiate the plasma fail, examine the torch for signs of water. If there is water on the torch, remove the torch assembly from the instrument, dry, and remount.

12.4.3. Consider replacing the o-rings. Any air leak into the argon flow will prevent the plasma from forming.

12.5. Monitor the intensities of the internal standard. This will be affected by the condition of the radial and axial windows and the performance of the nebulizer. During the run, a lowered intensity of the internal standard may be a sample matrix effect. The RSD of the internal standard should be less than 5%. This will be

affected by the pump tubing, the nebulizer, and any flickering of the plasma. A high solid sample will also raise the RSD and lower the internal standard intensity. A very high count can indicate the probe did not withdraw sample. A consistently high RSD will indicate that the either the sample delivery or the plasma torch or generator requires maintenance.

12.6. If an analytical line of interest shows behavior such as strong negative concentrations for blanks and other low samples, access the **Subarray Plot** on the **Content** pane of the LabBook > **Intensities** view and double-click the intensity value. If a peak is present in the blank and is there for the other lines of comparable sensitivity, the calibration blank was contaminated, or the sample introduction lines were dirty. The system should be pumped with 20% HCl and the calibration restarted.

12.7. Any time an analytical line of interest shows odd behavior; either strongly negative or there is serious difference (>20% RPD) between lines of comparable sensitivity; and the result is more than five times the RL, consult the **Subarray Plots** to see if there is a previously undocumented spectral interference or if the background correction point is misplaced. If background correction points are moved, the IECs should be recalculated.

12.8. Check and readjust the pixels surrounding the highest point of the peak in the **Subarray Plot** as necessary per instruction of Thermo and the Metals Group Leader.

12.9. If the autosampler is not responding or does not appear to initialize upon opening the Qtegra ISDS ICP-OES software, double-click the **ESI SC-4DX** window on the **Dashboard** page and click **Tools** > **Initialize** on the toolbar.

12.10. If the autosampler probe does not locate properly within the autosampler positions, the probe should be recalibrated. This may be caused, for instance, if the probe strikes an object while it is moving. The calibration routine is accessed on the **Dashboard** page > **ESI SC-4DX** window > **Tools** > **Calibrate** on the toolbar. The probe is calibrated with respect to an indented dot on the autosampler deck. When calibrating, adjust the Y component first and then X because Y has an X component.

12.11. The percent RSD of the three replicates readings for each sample can be affected by pulsations in the internal standard and carrier liquid flow. If the peristaltic pump tubing tension is misadjusted, especially with smaller internal diameter tubing and smaller rollers, pulsations may result. Adjust the internal standard and carrier tubing tension separately as follows. Turn off the nebulizer gas on the **Dashboard** page because the PFA microflow nebulizer self-aspirates. Put an air bubble in the line and loosen the tube tension until the bubble stops. Tighten until it restarts motion and then adjust the tube tension a quarter turn tighter. Turn the nebulizer gas back on.

12.12. If the nebulizer becomes plugged, the obstruction removal kit contained in the Consumables/Spares kit may help.

13. PREVENTATIVE MAINTENANCE

13.1. Refer to the User Guide which is located on the bench near the instrument for detailed maintenance instructions.

13.2. Inspect the pump tubing before operating. Tubing can be stretched gently 10 times by hand before installation. When pump is not in use, release the pressure plate and release the tubing to prevent flat spots from forming. It is recommended that the internal standard and carrier pump tubing be replaced once per week if the instrument is run every day or sooner if flat spots develop. The long-wearing Santoprene drain tubing should last for several months.

13.3. The following should be regularly checked:

13.3.1. The gas filters attached to the purge and plasma gas inlets should be checked for cleanliness. If the filters appear dirty, replace the filters and check the quality of the gas supply.

13.3.2. The level and condition of the fluid in the chiller should be inspected and the fluid replaced as necessary. If the water filters fitted to the chiller inlets appear dirty, replace the filters and flush the chiller.

13.3.3. The nebulizer should be inspected for clogs, and the sample capillary tubing should be inspected for build-up and wear.

13.3.4. The torch, glassware, and aerosol injector tube should be clean with no trace of deposits or signs of melting. Devitrification of the torch will occur, and over time in severe cases, cracking will take place. If the torch cracks, the shape of the plasma will change, and the operating characteristics will deteriorate. The torch should be replaced if cracking occurs.

13.3.5. The Purged Optical Path (POP) windows (axial and radial) should be inspected and cleaned as necessary. The radial window is subject to collection of sample material if very high solids samples are run. The need to clean the windows can be monitored by examination of standard absolute intensities for selected elements.

13.3.6. If the argon supply is turned off or allowed to deplete, at least two hours should be allowed for the optical components to purge once the argon supply is restored before turning on the chiller and running samples.

13.3.7. It is important that the FAST valve be maintained monthly because shavings from the rotor can plug and damage the valve. Refer to autosampler manual for detailed valve cleaning instructions.

13.3.7.1. Keep the interior and exterior surfaces of the valve clean. Open ports and fittings can allow particulate matter to enter the valve and scratch the sealing surfaces, which can cause the valve to fail.

13.3.7.2. Take apart the valve using the Allen wrench supplied. Squirt Super Q water in the rotor grooves and wipe the rotor dry with a Kimwipe. Examine stator orifices for valve shavings, which may be removed with Super Q water or compressed air. When reassembling the system, the rotor's grooved side must face outwards. Tighten the valve screws finger tight and then a slight bit beyond that.

13.4. If maintenance has been done on the torch compartment, an alignment of the torch should be performed. Ignite the plasma and aspirate a 2 mg/L Zn solution. To aspirate the solution, place the internal standard and carrier probes into the Zn solution and wait until it has traveled up to the nebulizer. Click on the **Dashboard** page > **Torch Alignment**. Confirm that the Zn solution is being aspirated and click **OK** in the dialog box to begin a torch alignment.

13.5. If the instrument has been shut down for an extended period or if new analyte wavelengths are added to the method, perform an **AutoPeak**. Ignite the plasma and perform the routine warm-up and spectrometer optimization. Open or create a LabBook. On the **Content** pane of the LabBook > **Method Parameters** > **Acquisition Parameters**, click **AutoPeak** on the toolbar. Select analytes on which to perform **AutoPeak** in the dialog box > **OK**. Confirm that a high concentration standard solution is being aspirated and click **OK** to begin an **AutoPeak**. Multiple standards may be used if all analytes are not present in one standard.

13.6. All maintenance must be recorded in the instrument logbook, which is located on the bench near the instrument. This includes simple daily functions such as replacing pump tubing.

14. REFERENCES

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14.14. iCAP 6000 Series ICP-OES Spectrometer Hardware Manual, Thermo Scientific, 2012.

14.15. iCAP 6000 Series ICP-OES Spectrometer User Guide, Thermo Scientific, 2012.

14.16. iCAP 7000 Plus Series ICP-OES Software Manual, Qtegra ISDS Software 2.10 SR5, Thermo Scientific, 2019.

14.17. SC-4 DX FAST System for Thermo iCAP Manual, Version 2.6.2.

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14.19. Standard Operating Procedure for the Procurement and Management of Equipment and Supplies, U.S. Environmental Protection Agency, Region 5 Chicago Regional Laboratory, 536 South Clark Street (ML-10C) Chicago, Illinois 60605. GEN026.

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14.24. Standard Operating Procedure for Electronic Data Packages, U.S. Environmental Protection Agency, Region 5 Chicago Regional Laboratory, 536 South Clark Street (ML-10C) Chicago, Illinois 60605. GEN032.

15. REVISION HISTORY

V= Version, S = Status, I = Initial, R = Revision, A = Archive

V	S	Location of Change History
8	R	Throughout document – Updated dates/Qualtrax ID #'s/values for MDL & RL studies (Section 1.4 & Appendix A), uncertainty determination (Section 1.6 & Appendix B), and control charts (Section 10.3.13) to reflect the 2020 studies
		Sections 1.5, 10.1.1, and 10.1.1 note – Added "for all analytes" after "linearity verification" to differentiate between the 2019 linearity verification that was performed for all analytes and within run linearity checks that are analyte specific; added note allowing within run linearity checks in lieu of an annual verification if it cannot be performed
		Section 7.10.5 & 7.10.9 – Updated Sr concentration in RLC multielement stock/low level spike to reflect current MDL & RL studies
		Section 7.10.7.1 – Added note regarding use of Ag single element standard as a spike solution if Ag begins to give consistently low recoveries in the blank/matrix spike
		The revision history for this SOP's version 4 has been archived with version 7.
		For the redline (tracked changes) copy of this SOP, which includes minor changes, refer to Qualtrax Draft Document workflow ID #23002.
7	R	Sections 2.5/6.4/9.2/10.2.2.1-10.2.2.2/12.6/12.9-12.11/13.4-13.5/14.16, Appendix C C.1.4.2-C.1.4.3, and Appendix D #3 and #4 – Replaced references to iTEVA software with Qtegra ISDS software and updated sample analysis instructions to reflect use of new data acquisition software
		Sections 7.10.5 & 7.10.9 – Corrected concentration of low level multielement stock solution to reflect Al and Ca RL change in v6
		Section 7.10.6 and note – Updated SIC concentrations to be within the current instrument linearity study; added option to change SIC concentration based on linearity or analyte concentration
		The revision history for this SOP's version 3 has been archived with version 6.
6	R	Global change – Replaced printing hardcopies with printing to pdf based on the electronic data package procedure in GEN032
		Global change – Replaced CRL with ASB to reflect EPA realignment
		Section 1.1 note – Updated list of certified drinking water analytes per June 2018 certification letter.
		Section 1.3 – Corrected phosphorus CAS number (CAR #11669)
		Sections 1.4 & 1.5 – Updated dates and Qualtrax ID # of MDL/RL/linearity studies
		Section 7.10.6 – Removed/added SIC solutions and changed concentrations based on SICs common to both EPA 200.7 and SW-846 6010D
		Section 7.10.7.1 – Combined spike A and B into AB based on new custom AB spike solution
		Section 7.10.7.2 – Removed unstable spike and added Ag/Mo/Sn into solid spike based on new custom solid spike solution; gave option to prepare unstable spike if needed.
		Section 7.10.11 – Added upload of electronic CoAs for standards to LIMS per requirement in GEN026
		Section 10.3.1 – Added SRM frequency which was previously missing
		Sections 10.3.3 & 10.3.4 – Clarified units used in DUP & MS calculations for water and soil/sediment; updated MS spike scheme based on new custom spike solutions

V	S	Location of Change History
		Appendix A – Updated dates and Qualtrax ID # of MDL/RL/linearity studies; updated digested water MDLs/RLs/linearities and soil/sediment RLs based on 2019 studies
		Appendix C.2 thru C.4 & C.6 – updated report generation procedure and data package components to reflect change to electronic data package procedure in GEN032
		For the redline (tracked changes) copy of this SOP, which includes minor changes, refer to Qualtrax Draft Document workflow ID #18464.
5	R	Cover page and throughout document – Replaced L-A-B logo with ANAB logo and replaced references to L-A-B with ANAB
		Section 1.1 and note –Added EPA Methods 200.7 and 200.8 as additional reference methods for METALS025
		Sections 1.4 and 10.5 (and subsections therein) – Added/updated language to incorporate changes to the MDL procedure as referenced in the Method Update Rule of 40 CFR Part 136 Appendix B; incorporated language from METALS001 (previously pen&ink 9939) to harmonize digested and undigested water MDLs/RLs (§1.4)
		Section 3.2.13 updated MDL definition to better match Revision 2 of 40 CFR Part 136 Appendix B.
		Section 7.10.5 – Updated to reflect the use of a vendor-prepared custom RLC stock and single-element silver stock to make the RLC
		7.10.9, former 10.3.13, 10.3.6 – RLV no longer applies to soils only
		10.3.7 and 10.3.12- incorporated Pen&Ink 14528 to change SRM audit limits from 100% +/- 20% to the manufacturer's established acceptance criteria.
		Sections 10.3.11.3, 10.3.12, former 10.3.13 – Added use of matrix spike for the dilution test (10.3.11.3); deleted post-digestion MS section (10.3.12) based on addition of 10.3.11.3; deleted post-digestion MS from QC Summary table (former 10.3.13)
		11.4/C.5 – Replaced GEN001 with METALS005 - the latter has superseded the former
		Section 14 – Removed reference to GEN001 since it was incorporated into METALS005; added reference to 40 CFR Part 136 Appendix B
		Appendix B- incorporated Pen&Ink 10413 for MDL edits to Table II: soils/sediments
		Appendix D – Added post-spike MS deviation; deviation regarding determination of digested water MDLs fulfills the annual MDL requirement for undigested water.
		(SOP-wide)- references to, and values of, MDLs, linearities, and uncertainties have been updated
		For the redline (tracked changes) copy of this SOP, which includes minor changes, refer to Qualtrax Draft Document workflow ID 14384.

APPENDIX A

Method Detection Limits, Reporting Limits, and Linearity Limits

The MDLs, RLs, and linearity limits per matrix for the Thermo 6500 Duo are listed in the tables below. Linearity limits apply to all matrices, whereas MDLs and RLs are matrix-specific. The plasma view refers to the instrument method setup for the Thermo 6500 Duo. Only the primary lines for reporting are given.

Table I. MDLs, RLs, and Linearity Limits for digested waters

Element/Wavelength (nm)	MDL (µg/L)	RL (µg/L)	Linearity Limit (mg/L)	Plasma View
Ag 328.068**	2.1	10	0.10 (0.50 soil)	Axial
Ag 338.289	11	25	0.10 (0.50 soil)	Axial
Al 308.215[HI]	270	500	200	Radial
Al 396.153**	81	500	750	Radial
As 189.042**	4.6	40	25	Axial
As 193.696	4.4	40	25	Axial
B 249.677	10	50	40	Radial
Ba 455.403	0.79	3	40	Radial
Be 313.042	0.20	2	20	Axial
Ca 315.887	133	500	750	Radial
Cd 214.440**	0.27	2	10	Axial
Cd 226.502	0.22	2	10	Axial
Co 228.616	0.87	6	8	Axial
Cr 267.716	0.79	5	25	Axial
Cu 324.752**	5.0	20	80	Axial
Cu 327.393	3.2	20	80	Axial
Fe 259.939**	21	80	500	Radial
Fe 273.955[HI]	39	80	750	Radial
K 766.490	97	800	750	Radial
Li 670.784	2.5	25	25	Radial
Mg 279.077[HI]	57	200	500	Radial
Mg 280.271**	19	200	100	Radial
Mn 257.610	3.5	8	20	Axial
Mo 202.031**	1.1	12	40	Axial
Mo 204.597	1.4	12	40	Axial
Na 589.592	42	400	750	Radial
Ni 231.604	3.3	12	8	Axial
P 177.4	25	200	50	Axial

Pb 220.353	5.3	30	10	Axial
Sb 206.836	4.9	20	25	Axial
Se 196.026	12	50	25	Axial
Si 251.611	77	200	50	Axial
Sn 189.927	6.2	20	8	Axial
Sr 407.771	2.1	5	20	Radial
Sr 421.552**	2.1	5	8	Axial
Ti 334.940**	0.69	6	20	Axial
Ti 336.121	1.0	6	20	Axial
Tl 190.801	12	60	25	Axial
V 292.402	1.3	5	75	Axial
Zn 213.857	6.2	30	25	Axial

*For water analysis the high channels for Al, Fe and Mg are used when the linear range of the low channels are exceeded. For the current linearity study, the Al high channel has a lower linearity limit than the Al low channel. Therefore, the high Al line is used as an alternate line for reporting in the case of QC failure on the low Al line.

**Default line

[HI]High channel

MDLs for digested waters were determined January 2020 (DOC workflow ID #20849) under multiple work orders (ICAP 3). Linearities were verified April 2019 (Qualtrax ID #10125).

Table II. MDLS and RLs for soils/sediments

Element/Wavelength (nm)	MDL (mg/L)	RL (mg/L)	MDL (mg/kg)	RL (mg/kg)
Ag 328.068**	0.001	0.01	0.1	1
Ag 338.289	0.004	0.025	0.4	2.5
Al 308.215[HI]	0.02	0.5	2	50
Al 396.153	0.008	0.5	0.8	50
As 189.042 (ICAP 3)**	0.003	0.04	0.3	4
As 193.696	0.004	0.04	0.4	4
B 249.677	0.003	0.05	0.3	5
Ba 455.403	0.001	0.003	0.1	0.3
Be 313.042	0.001	0.002	0.1	0.2
Ca 315.887	0.02	0.5	2	50
Cd 214.440**	0.001	0.002	0.1	0.2
Cd 226.502	0.001	0.002	0.1	0.2
Co 228.616	0.001	0.006	0.1	0.6
Cr 267.716	0.001	0.005	0.1	0.5
Cu 324.752**	0.001	0.02	0.1	2
Cu 327.393	0.001	0.02	0.1	2
Fe 259.939	0.008	0.08	0.8	8
Fe 273.955[HI]	0.01	0.08	1	8
K 766.490	0.06	0.8	6	80
Li 670.784	0.001	0.025	0.1	2.5
Mg 279.077[HI]	0.02	0.2	2	20
Mg 280.271	0.005	0.2	0.5	20
Mn 257.610	0.001	0.008	0.1	0.8
Mo 202.031**	0.001	0.012	0.1	1.2
Mo 204.597	0.001	0.012	0.1	1.2
Na 589.592	0.02	0.4	2	40
Ni 231.604	0.001	0.012	0.1	1.2
Pb 220.353	0.002	0.03	0.2	3
Sb 206.836	0.003	0.02	0.3	2
Se 196.026	0.003	0.05	0.3	5
Sn 189.927	0.001	0.02	0.1	2
Sr 407.771	0.001	0.005	0.1	0.5
Sr 421.552**	0.003	0.005	0.3	0.5
Ti 334.940**	0.001	0.006	0.1	0.6
Ti 336.121	0.001	0.006	0.1	0.6
Tl 190.801	0.003	0.06	0.3	6
V 292.402	0.001	0.005	0.1	0.5
Zn 213.857	0.01	0.03	1	3

*For soils/sediments analysis the high Al, Fe, and Mg lines are the default lines for reporting.

The values are given in mg/L (since sample masses will vary) and in mg/kg (based on 0.5 g sample).

MDLs for soils/sediments were determined May 2017 (DOC workflow ID #10378) under work orders 1704023 and 1705013 (ICAP 3 and ICP 4). RLs for soils/sediments were verified February 2020 (DOC workflow #20959) under multiple work orders (ICAP3).

APPENDIX B

Uncertainty Calculation

Uncertainty in the method for determination of metals in waters and wastewaters by ICP-OES is estimated by statistical evaluation of the blank spike results as described in ASB SOP GEN006. Method uncertainties were determined in January 2020 (Qualtrax ID #20865) using data acquired by the Thermo 6500 Duo ICP-OES from April 2018 – January 2020.

Table III. Uncertainties for digested waters

Element	Spike Conc. (µg/L)	Number of Points	Mean % Recovery	% Uncertainty
Aluminum	1000	28	102	10.3
Antimony*	500	19	101	9.99
Arsenic	200	28	100	11.6
Barium	200	28	101	9.30
Beryllium	10	28	101	9.84
Boron	1000	28	101	10.7
Cadmium	50	28	101	8.58
Calcium	50,000	28	96.7	8.28
Chromium	100	28	101	9.03
Cobalt	100	28	97.7	10.5
Copper	100	28	104	12.2
Iron	1000	28	99.2	7.02
Lead	500	28	99.2	8.46
Lithium	100	28	103	10.4
Magnesium	20,000	28	96.0	6.78
Manganese	200	28	100	8.58
Molybdenum*	100	19	104	9.21
Nickel	200	28	99.8	12.8
Phosphorus	1000	26	111	16.9
Potassium	25,000	28	99.1	9.87
Selenium	500	28	100	12.5
Silica	10,000	28	101	10.6
Silver	25	28	99.3	22.5
Sodium	50,000	28	98.2	9.93
Strontium	1000	28	98.3	11.2
Thallium	500	28	99.2	8.04
Tin	500	28	100	10.1
Titanium	100	28	107	14.1
Vanadium	100	28	102	12.2
Zinc	1000	28	101	8.79

*Due to persistent instrument issues with antimony and molybdenum during part of the time frame over which the uncertainty was calculated, data points collected during the affected time frame were rejected.

Uncertainty in the method for determination of metals in soils/sediments by ICP-OES is estimated by statistical evaluation of the standard reference material or blank spike (if analyte is not present in the SRM) as described in ASB SOP GEN006. Method uncertainties were determined in September 2020 (Qualtrax ID #22929) using data acquired by the Thermo 6500 Duo ICP-OES from May 2018 – January 2020.

Table IV. Uncertainties for soils/sediments

Element	Nominal Conc. (mg/kg)	Number of Points	Mean % Recovery	% Uncertainty
Aluminum**	8000	11	90.9	30.9
Antimony	65.1	22	145	49.8
Arsenic	40.7	23	91.3	22.5
Barium	314	23	91.6	16.1
Beryllium	53.4	23	93.4	17.3
Boron	74.2	23	94.7	42.6
Cadmium	193	23	91.7	16.4
Calcium	4580	23	88.5	22.9
Chromium	82.6	23	90.4	21.1
Cobalt	81.3	23	93.6	15.9
Copper	171	23	93.3	18.1
Iron**	14100	11	93.4	46.2
Lead	92.3	23	90.6	23.9
Lithium	50	15	98.3	7.6
Magnesium**	2240	11	94.2	21.4
Manganese	222	22	92.6	13.8
Molybdenum	145	23	97.2	16.5
Nickel	137	23	95.6	16.6
Potassium	2000	23	88.7	20.8
Selenium	187	23	95.3	23.9
Silver	40.7	22	86.6	38.1
Sodium	216	23	92.1	17.2
Strontium	102	23	94.2	16.7
Thallium	153	23	93.5	17.4
Tin	96.2	23	89.9	22.8
Titanium	371	23	87.7	38.4

Element	Nominal Conc. (mg/kg)	Number of Points	Mean % Recovery	% Uncertainty
Vanadium	86.6	23	96.1	18.2
Zinc	189	23	94.4	17.3

*Analyte not present in SRM; BS used to calculate uncertainty
**Uncertainty calculated from high line (default for soils/sediments)

As stated in Section 1.6 of this SOP, this uncertainty will be greater near the RL and will be much greater near the MDL. Usually, the sampling component of the uncertainty will be far greater than the laboratory uncertainty.

APPENDIX C
LIMS Entry and Reporting

C.1. LIMS Data Entry

C.1.1. All LIMS data entry is based on first creating a bench sheet describing the sample preparation. This bench sheet describes the samples prepared and the digestion/batch QC samples. The analyst must make certain that the preparation date in LIMS matches the actual preparation date and that the preparation time occurs before the analysis time. By convention, the sample preparation date to be recorded is the date on which the last preparation step has been completed.

C.1.1.1. The bench sheet is created under **Laboratory** > **Batch**. Click the **Add** button and then choose **Batch Department**. Select **Preparation Method**, **Batch Matrix**, and **Analyses** associated with this batch. Click **Save**.

C.1.1.2. Alternatively, the parameters from a previous batch can be copied into a new batch under **Laboratory** > **Batch**. Click the **Copy** button and then click **Save**.

C.1.1.3. Go to **Bench Sheet** page, select the **Batch** number, and click **Edit**. Click **Add** and select **Client Sample**. Locate the samples in the **All Sample Containers** dialog box and then right-click to view the shortcut menu. Click **Include Selection** to insert the selection into the **Bench Sheet**.

C.1.1.4. Verify that the numbers of matrix QC samples are correct or add/delete as appropriate. Click **Save** once all edits to the bench sheet have been made.

C.1.1.4.1. Highlight the DUP or MS and right-click to select the **Source** sample.

C.1.1.4.2. Highlight the BS, MS, or RLV and right-click to add the **Spike Solution LIMS ID** and **Spike Amount** in microliters. The stock spike solutions described in Section 7.10.7 are used here for

the BS and MS, and the stock spike solution described in Section 7.10.9 are used here for the RLV.

C.1.1.4.3. Highlight the SRM and right-click to add the **Spike LIMS ID** and **Spike Amount** in microliters. Although no spike is actually added to the SRM, the data is entered as it it were an addition of a spike volume numerically equal to 1000 times the SRM mass.

C.1.1.5. The bench sheet can be printed from the **Print** menu. The bench sheet is to be initialed and dated with the actual date of preparation. By convention, if the sample preparation proceeds overnight, the date to be recorded on the bench sheet is the date the preparation began with a notation that the digestion proceeded overnight. In contrast, the date to be recorded in LIMS is the date on which the last preparation step was completed. Once the bench sheet is completed, it is scanned as a PDF for inclusion into the electronic data package.

C.1.2. When the data are ready to be entered into LIMS, the batch is called up into the **Data Entry/Review** module. All analyses or selected analyses can be included. If only a few analyses are to be entered, data may be entered manually. Otherwise, it may be more practical to use Data Tool.

C.1.3. Manual Data Entry

C.1.3.1. When performing manual data entry, enter the results in the **Result** column in mg/L. For each result, enter the date of analysis in the **Analyzed** column. This column has a calendar feature as do other date fields in LIMS. If dilutions were necessary for the analysis, enter the dilution in the **Dilution** column. The sample result should be the one measured and not corrected for the dilution factor.

C.1.3.2. When all data are entered, click the **Save** button. After saving, proceed to the **Review** tab and click the **Query** button. Verify that the correct initials and instrument identification are present in the **Analyst** and **Instrument** columns, respectively. Verify that all conversions to reporting units and dilutions have been calculated correctly. Verify that

RLs have been correctly applied. Flags may be added at this stage, following the guidance given in ASB SOP METALS005. Before second review, lock the data and update the status to **Analyzed**.

C.1.4. Data Entry Using Data Tool

C.1.4.1. If Data Tool is to be used to input data, once the batch is called up in **Data Entry/Review**, click the **Export** button to create an Excel file in the User Directory. Name this file in a manner that it can be easily associated with that analysis. If totals and dissolved pairs are analyzed in the same batch, create a separate Excel export file for each of the two analyses.

C.1.4.2. The file created in Section 9.2.6.2 (.xml file) contains the instrument readings for the samples. If multiple measurements for a given sample are present, such as for dilutions, Data Tool allows for changing the names identified at the instrument. In the main Data Tool page, click **Merge Files**. Click the **Instrument Data** tab, right-click **Lab_Number**, and click the **Replace** button. Before saving the merged file, click the **Refresh** button.

C.1.4.3. Once in Data Tool, click **Browse** for the **Element Data Entry Table** and select the .xls file created in C.1.4.1. Click **Browse** to search for the instrument data file. Set the **File Type** to **Thermofisher ICAP QTegra v2.2 XLS (*.xls,*.xml)**. If unneeded sample entries remain in the lower left-hand box, click **Clear**. Double-click on the desired .xml file. Either click **Auto Select** or highlight individual samples and then click **Include**.

Please note: If totals and dissolved pairs are analyzed in the same batch, be careful to manually select only the total or dissolved samples, as needed.

C.1.4.4. Once the samples and batch/digestion QC are selected, click **Done**, and it will return to the main Data Tool page. Click **Merge Files**. If either **Unmatched Analytes** or **Unmatched Units** appear in red, repair the cross table with the assistance of the Metals Group Leader, if

necessary. Verify that the results in **Initial Result** are correct and click **Save**, which will create an Excel file. Name this one differently from the name chosen in C.1.4.1 and click **Done**.

C.1.4.5. Return to the **Data Entry/Review** module in LIMS. Click **Open** and select the .xls file created in C.1.4.4. Verify that all items are correct as in the manual data entry and click **Save**. Query the data and proceed as in C.1.3.2.

C.1.5. Note that only one analytical line is used per element for Data Tool. In Data Tool, the **Analyte Cross Table** can be set with upper concentration limits for each analytical wavelength. For analytes with high and low channels, such as aluminum, iron, and magnesium, this information allows Data Tool to select the correct wavelength. These limits can be set in the **Data Range** column. For example, Data Tool can be made to select the Fe 259.939 nm line for digest concentrations up to the low channel's linearity limit and Fe 273.955 nm for concentrations up to the high channel's linearity limit. If there is a concentration over the high channel's linearity limit, Data Tool will import nothing.

C.2. LIMS Report Generation

C.2.1. Once all data are entered with the status of **Analyzed**, prepare a draft report. In LIMS, select **Project Management** > **Reports**. Choose the work order, analyses, and report format. The report format is **Analysis_v14.rpt**. Choose **Draft** to generate the report. If QC is to be omitted from the report, such as with a preparation batch shared with ICP-MS, including separate spikes for the two methods, choose **Modified Draft** and uncheck the QC samples that were not analyzed.

C.2.2. After the second reviewer has updated the status of the LIMS entries to **Reviewed**, the final report may be generated. The report format chosen is **Analysis_no QC_v13.rpt** for the client report if no QC is requested and **Analysis_v14.rpt** for the final report that is included in the official electronic data package. Choose either the **Final Report** or **Modified Final Report** to generate the report. The analyst's signature is automatically applied to the cover page of the final report by LIMS.

<u>Please note:</u> The ANAB logo is automatically applied to LIMS reports and a list of accredited analytes is included in the reports for analytes/matrices/analyses in which a certification has been added under **QA Admin** > **Analysis Matrix** > **Certification** based on the ASB Scope of Accreditation.

C.2.3. Once the final report has been generated, correct use of the ANAB logo is verified by the second reviewer.

C.3. For any analysis requested for a work order there are a standard components to the electronic data package. See the Data Package Requirements Section of ASB SOP METALS005 and Group-Specific Components of Electronic Laboratory Data Packages Section of ASB SOP GEN032.

C.4. After second review, the completed electronic data package and electronic data deliverable are placed in the **Data for Transmittal to EDMS** folder on the i drive and notification is sent to the Data Coordinator through the Qualtrax Data Reporting workflow.

C.5. The instrument raw data file(s) and all other electronic files related to the sample batch are uploaded to R5CRL. Refer to ASB SOP METALS005 for data upload instructions.

C.6. The electronic client data is emailed to the Data Coordinator for transmittal. The client typically receives a PDF of the final LIMS report and an electronic data deliverable, but additional report options are available to the client upon request.

APPENDIX D

Differences Between SOP METALS003A and EPA Method 200.7 and SW-846 Method 6010D

1. Mercury is not currently part of this SOP. Phosphorus and silica are only reported by request.

2. The matrix for the digested water standards is 1% HNO_3 and 0.5% HCl to match the samples, which are prepared by EPA Method 200.2/200.7/200.8 without the 2-fold concentration step in 200.7. This limits the silver concentration to 0.1 mg/L.

3. Interelement correction factors are used as the Qtegra ISDS software generates them. They are not evaluated by the procedure given in 7.13 of EPA Method 200.7. The spectral interference checks (SICs) given in Section 7.10.6 of this SOP include single element interference checks for analytes that are common to both EPA Method 200.7 Section 7.13 and SW-846 6010D Section 9.9.1, and/or the listed elements have been observed in field samples at high concentrations.

4. The torch plasma viewing positions are evaluated using the Qtegra ISDS software using 2 mg/L Zn, as recommended by the manufacturer.

5. All QC checks are done using solutions from a different source from the calibration standards. Thus, the IPC, QCS, ICV, and CCV are the same in this procedure.

6. QC check limits (Section 10.3) are set based on the method with the more stringent requirement between SW-846 Method 6010D or EPA Method 200.7.

7. Blank spike percent recovery is calculated without subtraction of the blank (Section 10.3.5) unlike the calculation in EPA Method 200.7 Section 9.3.2. This decision was based on discussion with other laboratories currently performing these methods and current in-house blank spike recovery calculations for other analyses.

8. The post-digestion matrix spike described in SW-846 6010D Section 9.11.2 is not performed since the pre-digestion matrix spike is used for the dilution test if a high level sample is not available (Section 10.3.11.3).

9. Determination of digested water MDLs fulfills the annual MDL requirement for undigested water. If results are requested below the RL for undigested water samples,

MDLs will be determined on undigested water prior to reporting out the data. (Section 1.4)

U.S. Environmental Protection Agency – Region 5

Superfund & Emergency Management Division Analytical Request Form and ASB Chain-of-Custody

U.S. ENVIRONMENTAL PROTECTION AGENCY—REGION 5
SUPERFUND & EMERGENCY MANAGEMENT DIVISION (SEMD)
ANALYTICAL REQUEST FORM (ARF)

This form must be completed and emailed to the Analytical Services Branch (ASB) Lab Sample Coordinator (wroble.amanda@epa.gov) and the Regional Sample Control Coordinator (RSCC) (R5RSCC@epa.gov) with as much advanced notice as possible. Please copy (cc) the EPA project contact on all analytical requests.

SITE AND CONTACT INFORMATION

Site Name:		**City, State:**	
EPA Project Contact:		**Site/Spill ID:**	**EPA ID:**
QAPP Approval Date:		**QAPP SEMS Document ID:**	
Investigation Type:		**Operable Unit:**	
Sampling Organization:		**Sampling Organization Email & Phone Number:**	
Sampling Description:		**Sampling Dates:**	

Note: The laboratory (Region 5 Analytical Services Branch [ASB] or Contract Laboratory Program [CLP]) selected to provide analytical services is determined based on the FASTAC tiered strategy and site-specific needs.

The sampling event is: ☐ **One-time** or ☐ **Weekly** or ☐ **Monthly** or ☐ **Quarterly** or ☐ **Semiannual** or ☐ **Annual**

Saturday delivery? ☐ **Yes** ☐ **No** **NOTE: Only available for CLP deliveries.**

All work scheduled must be included in an approved quality assurance project plan or sample planning document. This is required before sampling. Please submit one to the RSCC as soon as available. Alternatively, note the EPA SEMS document ID number in the field above. Analytical requests for ASB services should be submitted as early as possible. Analytical requests that include CLP modified, specialized, and SIM analyses must be submitted 3 weeks in advance.

ANALYTICAL OPTIONS

CLP target analytes and reporting limits can be found at:

https://www.epa.gov/clp/what-superfund-clp-analytical-services-are-available

ASB target analytes and reporting limits can be found at:

https://usepa.sharepoint.com/sites/R5/CRL/Lists/SOPs/AllItems.aspx

CLP OPTIONS FOR WATERS	CLP OPTIONS FOR SOILS	ASB OPTIONS
Volatile Organic Compounds (VOCs): CLP TVOA (Trace), CLP VOA-LOW	**Volatile Organic Compounds (VOCs):** CLP VOA-LOW, CLP VOA-MED, CLP VOA TCLP	**See attached ASB request form**
Semivolatile Organic Compounds: (SVOCs): CLP SVOA-LOW-SIM, CLP SVOA-LOW, CLP PAHs	**Semivolatile Organic Compounds: (SVOCs):** CLP SVOA-LOW-SIM, CLP SVOA-LOW, CLP SVOA-MED, CLP SVOA TCLP	
Pesticides: CLP PEST **PCBs:** CLP AROCLORS, CLP CBCS **DIOXIN AND FURANS**: CLP DIOXIN/FURANS	**Pesticides:** CLP PEST, CLP PEST TCLP **PCBs:** CLP AROCLORS, CLP CBCS **DIOXIN AND FURANS**: CLP DIOXIN/FURANS	
Inorganics: CLP ICP-AES (total or dissolved), CLP ICP-MS (total or dissolved), CLP HARDNESS, CLP MERCURY by CVAA (Total or dissolved), CLP CYANIDE	**Inorganics:** CLP ICP-AES, CLP ICP-MS, CLP ICP-AES TCLP, CLP MERCURY by CVAA, CLP MERCURY TCLP, CLP CYANIDE, CLP pH	

NOTE: Modified CLP analyses (MAs) may be available on request. Contact the RSCC for additional details.

ANALYTICAL REQUEST

Analysis Required (See options above and attached)	Matrix (e.g. Soil, sediment, groundwater, surface water, drinking water, fish tissue, waste)	Number of Samples (per matrix, including field QC)

DATA TURN AROUND TIME (TAT) AND DELIVERABLE INFORMATION

TAT Requested:	Standard (45 days for ASB, 21 days for CLP)
CLP Data Validation Level Needed:	
Non-Standard Electronic Data Deliverable Needed? **Standard Electronic Data Deliverables: (Standard Excel for ASB, SEDD Stage 3 for CLP)**	
Preliminary Data Needed?	
Additional Comments:	

SHIPPING DETAILS

Shipping protocols for CLP should follow the *Contract Laboratory Program Guidance for Field Samplers* found at: https://www.epa.gov/sites/production/files/2015-03/documents/samplers_guide.pdf

Shipping protocols for ASB should follow instructions included at the end of the ASB request form (attached).

Prior to shipping every day, notify the CLP Sample Management Office (SMO) or ASB (as appropriate).

CLP - Casey Marston (703) 461-2134, casey.marston@gdit.com **ASB - Robert Snyder (312) 353-9083, snyder.robert@epa.gov**

Sampling Requestor:	
Date Submitted:	

THE REMAINING PAGES ARE ONLY TO BE FILLED OUT FOR ASB REQUESTS

General Chemistry	Instructions: Select the checkbox to request an analysis and enter the proposed # of samples per matrix type in the box underneath the appropriate matrix.			
Analysis	**Check to Request**	**soil/sediment**	**water/liquid**	**other/waste***
acidity	☐		_____	_____
alkalinity	☐		_____	_____
ammonia-N	☐	_____	_____	_____
anions**	☐	_____	_____	_____
biochemical oxygen demand-5 day (BOD)	☐		_____	_____
carbonaceous BOD-5 day (CBOD)	☐		_____	_____
chemical oxygen demand (COD)	☐		_____	_____
corrosivity by pH	☐		_____	_____
cyanide, amenable to chlorination	☐		_____	_____
cyanide, total	☐	_____	_____	_____
fluoride	☐	_____	_____	_____
grain size	☐	_____		_____
ignitability by flashpoint	☐		_____	_____
nitrate-nitrite-N	☐		_____	_____
paint filter liquid test	☐		_____	_____
percent solids	☐		_____	_____
pH	☐	_____	_____	_____
residue, filterable (TDS)	☐		_____	_____
residue, non-filterable (TSS)	☐		_____	_____
total Kjeldahl nitrogen (TKN)	☐	_____	_____	_____
total organic carbon (TOC)	☐	_____	_____	_____
total phosphorus (TP)	☐	_____	_____	_____
turbidity	☐		_____	_____
water content	☐		_____	_____

Metals	Instructions: Select the checkbox to request an analysis and enter the proposed # of samples per matrix type in the box underneath the appropriate matrix.			
Analysis	**Check to Request**	**soil/sediment**	**water/liquid**	**other/waste***
chromium (VI)	☐	_____	_____	_____
hardness	☐		_____	_____
In-Vitro Bioaccessibility (IVBA indicate Pb, As)	☐	_____		
mercury (Hg)	☐	_____	_____	_____
total metals** (except Hg & Cr (VI))	☐	_____	_____	_____ wipe/filter

ONLY APPLICABLE TO ASB REQUESTS

Organics	Instructions: Select the checkbox to request an analysis and enter the proposed # of samples per matrix type in the box underneath the appropriate matrix.			
Analysis	**Check to Request**	**soil/sediment**	**water/liquid**	**other/waste***
air toxics**	☐			_____ air
1,4-dioxane, low level	☐		_____	_____
microcystins (drinking water only)	☐		_____	_____
oil & grease	☐		_____	_____
per- and polyfluoroalkyl substances**(PFAS)	☐	_____	_____	_____
pesticides, chlorinated**	☐	_____	_____	_____
pesticides, low level**	☐		_____	_____
PCB aroclors**	☐	_____	_____	_____
semi-volatiles** (SVOCs)	☐	_____	_____	_____
total petroleum hydrocarbons (TPH as DRO/ORO)	☐	_____	_____	_____
volatiles** (VOCs)	☐	_____	_____	_____

Toxicity Characteristic Leaching Procedure (TCLP)	Instructions: Select the checkbox to request an analysis and enter the proposed # of samples per matrix type in the box underneath the appropriate matrix.			
Analysis	**Check to Request**	**soil/sediment**	**water/liquid**	**other/waste***
TCLP Hg	☐	_____	_____	_____
TCLP metals	☐	_____	_____	_____
TCLP pesticides	☐	_____	_____	_____
TCLP SVOCs	☐	_____	_____	_____
TCLP VOCs	☐	_____	_____	_____

***Additional Matrix Description**	
Please describe *other/waste* matrix, if not specified above:	

****Specific Analyte Class/Subset Request**	
Please list or attach specific class/subset for multi-analyte test, if requested:	

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NON-STANDARD REQUESTS

For analyses/matrices not listed above or to obtain analyte lists, quality control limits, and/or reporting limits, please contact the ASB Sample Coordinator Amanda Wroble (wroble.amanda@epa.gov, 312-353-0375) to discuss.

ASB DATA FORMAT

The ASB standard data deliverable includes: 1) a pdf of the final Level II report that includes the work order and 2) an electronic data deliverable (EDD) that includes batch quality control sample data. EDD typically refers to an Excel spreadsheet of the data, but EDDs are available in a variety of formats and can be customized upon request. A full data package (Level IV) is also available upon request and will be transmitted electronically via the ASB SharePoint. Contact the ASB Data Coordinator Gabriel Omotayo (omotayo.gabriel@epa.gov, 312-353-2964) for additional details.

ASB SAMPLE DISPOSAL POLICY

Due to space limitations in a controlled temperature environment, samples are relocated to secure room temperature storage six months after the analysis completion of the project. Notification of the intent to relocate the samples is given to the customer with sufficient time for the customer to respond with any objections. Samples remain in secure room temperature storage until the case/project is completed and the samples are no longer needed. Notification is given to the customer with sufficient time for customer response prior to sample disposal.

ASB SAMPLE SHIPMENT REQUIREMENTS

Before collecting samples, please refer to the sample requirements table for sample sizes, containers, and preservatives. Notify the ASB Sample Custodian Robert Snyder (snyder.robert@epa.gov, 312-353-9083) and the ASB Sample Coordinator Amanda Wroble (wroble.amanda@epa.gov, 312-353-0375) before shipping any samples and to arrange for sample receipt.

When packing samples for shipment:

- ✓ Seal individual samples in plastic bags, preferably Ziploc bags.
- ✓ The temperature of samples requiring refrigeration during transport MUST be maintained at or below 6°C.
- ✓ Ice in a sealed plastic bag or reusable ice substitute freeze packs are acceptable cooling media.
- ✓ Chain of custody forms MUST be sealed in a large Ziploc bag and taped to the inside of the cooler lid.
- ✓ Include the address to which the cooler should be returned.

After items are packed for shipment, secure the cooler with tape and attach a custody seal across the seam of the cooler lid.

All samples MUST be shipped overnight to arrive Monday thru Friday or hand-delivered. No deliveries are accepted on weekends or Federal holidays. Exceptions may be made on a case by case basis depending on sampling priority/emergency status.

Send all samples to:

Robert Snyder
US EPA Region 5
Analytical Services Branch
536 S. Clark Street, 10th Floor
Chicago, IL 60605

ONLY APPLICABLE TO ASB REQUESTS

U.S. EPA ANALYTICAL SERVICES BRANCH
HOLDING TIME AND CONTAINER REQUIREMENTS FOR WATER / AQUEOUS SAMPLES

DISCLAIMER: This table represents the Analytical Services Branch's (ASB) recommended guidelines. Additional containers may be required for laboratory quality control samples (see notes section). There are non-routine analytes (reported upon request) that may require modification to the specifications detailed in this table. It is the client's responsibility to confirm container, preservation, and holding time requirements for a project prior to initiating sampling. This includes any equipment procurements, if applicable. No brand endorsements are made or implied.

General Chemistry	ASB SOP(s)	Reference Method	Holding Time (days)	Min. Volume (mLs)[l]	Container[e]		Preservation
Acidity	AIG004A	SM 2310	14	50	500 mL Poly		≤6 C
Alkalinity	AIG005	SM 2320 B	14	50	500 mL Poly		≤6 C
Ammonia (Nitrogen, NH3) Distilled	AIG029B	SM 4500-NH_3 B/H	28	50	500 mL Poly		pH<2, H_2SO_4, ≤6 C
Anions (Br, Cl, F, NO_3, NO_2, $P0_4$, SO_4)[a]	AIG045A	EPA 300.0	2[b] or 28	10	250 mL Poly		≤6 C
Biochemical Oxygen Demand (BOD) 5day	AIG006, A	SM 5210 B	2	60	1 L Poly		≤6 C
BOD, Carbonaceous (cBOD)	AIG006, A	SM 5210 B	2	60	1 L Poly		≤6 C
Chemical Oxygen Demand (COD)	AIG007A	EPA 410.4	28	2	250 mL Poly		pH<2, H_2SO_4, ≤6 C
Corrosivity	AIG003	EPA 9040C	365	20	250 mL Amber		≤6 C
Cyanide, Amenable	AIG025A	SM 4500 CN^- G	14	50	500 mL Poly		dechlorinate[c] NaOH, pH>10, ≤6 C
Cyanide, Total	AIG025C	EPA 335.4	14	50	500 mL Poly		dechlorinate[c] NaOH, pH>10, ≤6C
Ignitability (Flashpoint)	AIG048A, B	EPA 1010A , 1020B	365	100	250 mL Clear		ambient
Nitrogen, Nitrate+Nitrite	AIG031B	NECi N07-0003	28	10	500 mL Poly		pH<2, H_2SO_4, ≤6 C
Nitrogen, Total Kjeldahl (TKN)	AIG035B	EPA 351.2	28	10	500 mL Poly		pH<2, H_2SO_4, ≤6 C
Organic Carbon, Dissolved (DOC)	AIG021D	EPA 5310B	28	20	500 mL Poly		field filtered[d] pH<2, H_2SO_4, ≤6 C
Organic Carbon, Total (TOC)	AIG021D	EPA 5310B	28	20	500 mL Poly		pH<2, H_2SO_4, ≤6 C
Paint Filter Liquid Test	AIG010	EPA 9095B	30	100	250 mL Amber		≤6 C
pH	AIG002	SM 4500-H^+ B	15 min	50	250 mL Poly		≤6 C
Phosphorus, Total Dissolved (TDP)	AIG034B	EPA 365.4	28	10	500 mL Poly		field filtered[d] pH<2, H_2SO_4, ≤6 C
Phosphorus, Total (TP)	AIG034B	EPA 365.4	28	10	500 mL Poly		pH<2, H_2SO_4, ≤6 C
Solids, Total Dissolved (TDS)	AIG017	SM 2540 C	7	50	500 mL Poly		≤6 C
Solids, Total Suspended (TSS)	AIG018	SM 2540 D	7	100	500 mL Poly		≤6 C
Turbidity	AIG054	EPA 180.1	2	30	250 mL Clear		≤6 C
Water Content	AIG015A	EPA 9000	365	10	250 mL Amber		≤6 C
Metals	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Volume (mLs)[l]**	**Container**		**Preservation**
Chromium (VI)	AIG032A	EPA 218.6	28	50	250 mL Poly		pH 9.3-9.7, ≤6 C NaOH/$(NH4)_2SO_4$
Hardness	Metals026	SM 2340 B	180	50	500 mL Poly		pH<2, HNO_3
Mercury (Hg)	AIG044D, E	EPA 245.1/7470A	28	20	500 mL Poly		pH<2, HNO_3
Metals, Total	Metals001, 003, 003A	EPA 200.7/200.8 EPA 6010D/6020B	180	50	500 mL Poly		pH<2, HNO_3
Metals, Dissolved	Metals001, 003, 003A	EPA 200.7/200.8 EPA 6010D/6020B	180	50	500 mL Poly		field filtered[d] pH<2, HNO_3
Organics	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Volume (mLs)[l]**	**Container**	**MS[m] MSD**	**Preservation**
1,4-Dioxane (low-level)	MS035	EPA 522/8270E	28[g]	250	2 - 250 mL Amber	2	pH<2, Na_2SO_4, ≤6 C
Chlorothalonil	MS033	EPA 525.3/8270D	7[f]	40	3 - 40 mL Amber VOA	2	≤6 C
Microcystins (drinking water only)	OM26	EPA 544	28[g]	500	2 - 500mL Amber narrow-mouth	4	pH 6.5-7.5, ≤10 C
Oil and Grease	GC030	EPA 1664B	28	1 L	2 - 1L Clear wide-mouth	2	pH<2, H_2SO_4, ≤6 C
Polychlorinated Biphenyls (PCBs)	GC002, 003	EPA 608/8082A	7[f,k] or 365[f]	1 L	2 - 1L Amber narrow-mouth	2	≤6 C
Per- and Polyfluoroalkyl Substances (PFAS)	OM21	NA	28	5	2 - 15 mL Polypropylene tube (preweighed)	4	≤6 C
Pesticides (low level)	OM019	NA	28	8	2 - 40 mL amber VOA	2	≤6 C
Pesticides, Chlorinated	GC001	EPA 608/8081B	7f	1 L	2 - 1L Amber narrow-mouth	2	≤6 C
Petroleum Hydrocarbons (TPH as DRO/ORO)	GC034	EPA 8015C	7f	1 L	2 - 1L Amber	2	≤6 C
Semi-Volatile Organic Compounds (SVOCs)	MS026, 27	EPA 625.1/8270E	7f	1 L	2 - 1L Amber narrow-mouth	2	≤6 C
Volatile Organic Compounds (VOCs)	MS023, 24	EPA 624.1/8260C	7 (unpreserved) 14 (Preserved)	40	3 - 40mL VOA no headspace	2	pH<2, HCl,≤6 C
Waste Characterization	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Volume (mLs)[l]**	**Container**		**Preservation**
Toxicity Characteristic Leaching Procedure (TCLP)[h]	GEN019	EPA 1311	Varies[i]	Varies[j]	Varies		≤6 C

Notes:

[a] Orthophosphate must be field filtered
[b] Nitrite, nitrate, and ortho-phosphate have a 48 hour holding time
[c] Dechlorinate with ascorbic acid
[d] Field filtering should use a 0.45 µm filter
[e] All containers must be filled completely and maintained on ice at ≤ 6 C
[f] 40 day holding time post extraction
[g] 28 day holding time post extraction
[h] Can be requested for metals, Hg, Pesticides, SVOCs and VOCs
[i] Field collection->TCLP ext. (in days): 14 for organics, 28 for Hg, 180 for metals
[j] Contact ASB for additional details and/or options
[k] Applicable to method 608.3 only
[l] Does not include amount needed for QC samples or excess needed for dilutions/reanalysis
[m] Extra containers needed for MS/MSD location. Frequency= 1/20 field samples

ONLY APPLICABLE TO ASB REQUESTS

U.S EPA ANALTYICAL SERVICES BRANCH
HOLDING TIME AND CONTAINER REQUIREMENTS FOR SOIL / SOLID SAMPLES

DISCLAIMER: This table represents the Analytical Services Branch's (ASB) recommended guidelines. Additional containers may be required for laboratory quality control samples (see notes section). There are non-routine analytes (reported upon request) that may require modification to the specifications detailed in this table. It is the client's responsibility to confirm container, preservation, and holding time requirements for a project prior to initiating sampling. This includes any equipment procurements, if applicable. No brand endorsements are made or implied.

General Chemistry	ASB SOP(s)	Reference Method	Holding Time (days)	Min. Mass (g)[k]	Container[c]	Preservation[d]
Ammonia (Nitrogen, NH_3)	AIG029B, 22A	SM 4500-NH_3 B/H	28	1	4 oz. jar	≤6 C
Anions (Br, Cl, F, NO_3, NO_2, PO_4, SO_4)	AIG039, 45A	EPA 300.0	2[a,b] or 28[b]	10	4 oz. jar	≤6 C
Chemical Oxygen Demand (COD)	AIG007A, 22A	410.4	28[b]	10	4 oz. jar	≤6 C
Cyanide, Total	AIG025B, C	EPA 335.4	14	1	4 oz. jar	≤6 C
Nitrogen, Total Kjeldahl (TKN)	AIG022A, 35B	EPA 351.2	28[b]	1	4 oz. jar	≤6 C
Organic Carbon, Total (TOC)	AIG009A	ASA-SSSA	28[b]	1	4 oz. jar	≤6 C
Particle Size	AIG038, 38A	ASTM D2487-93	365	100	16 oz. jar	≤6 C
pH	AIG008	EPA 9045D	365	20	4 oz. jar	≤6 C
Phosphorus, Total (TP)	AIG022A, 34B	EPA 365.4	28[b]	1	4 oz. jar	≤6 C
% Solids	AIG019	SM 2540 G	7	10	4 oz. jar	≤6 C
Metals	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Mass (g)[k]**	**Container**	**Preservation[d]**
Chromium (VI)	AIG033A	EPA 7199/3060A	30	2.5	4 oz. jar	≤6 C
Mercury (Hg)	AIG043C,D,E	EPA 245.5/7471B	28	1	4 oz. jar	≤6 C
In-Vitro Bioaccessibility (Lead and/or Arsenic)	Metals035	EPA 1340	180	100	4 oz. jar	≤6 C
Metals, Total	Metals001, 003, 003A	EPA 200.7/200.8 EPA 6010C,D/6020B	180	100	4 oz. jar	≤6 C
Organics	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Mass (g)[k]**	**Container**	**Preservation[d]**
Pesticides, Chlorinated	GC001	EPA 8081B	14[l]	10	8 oz. jar	≤6 C
Polychlorinated Biphenyls (PCBs)	GC002, 003	EPA 8082A	365[l]	10	8 oz. jar	≤6 C
Per- and Polyfluoroalkyl Substances (PFAS)	OM022	NA	28	2	50 mL Polypropylene Tube[k]	≤6 C
Petroleum Hydrocarbons (TPH as DRO/ORO)	GC034	EPA 8015C	14[l]	30	8 oz. jar	≤6 C
Polycyclic Aromatic Hydrocarbons, Alkylated	MS026	NA	14[l]	30	8 oz. jar	≤6 C
Semi-Volatile Organic Compounds (SVOCs)	MS026	EPA 8270E	14[l]	30	8 oz. jar	≤6 C
Volatile Organic Compounds (VOCs)	MS001	EPA 8260C	2	5	3 Encores™[e] or 3 VOA vials w/ stir bar[e,f,j]	≤6 C
Waste Characterization	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Min. Mass (g)[k]**	**Container**	**Preservation[d]**
Toxicity Characteristic Leaching Procedure	GEN019	EPA 1311	Varies[h]	Varies[i]	16 oz. jar	≤6 C
HOLDING TIME AND CONTAINER REQUIREMENTS FOR FILTERS / WIPE SAMPLES						
Organics	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Num. of Wipes**	**Container**	**Preservation**
Polychlorinated Biphenyls (PCBs)	GC002, 003	EPA 8082A	365[l]	1 wipe w/hexane	4 oz. jar	≤6 C
Semi-Volatile Organic Compounds (SVOCs)	MS026	EPA 8270E	14[l]	1 wipe w/ isopropanol	4 oz. jar	≤6 C
HOLDING TIME AND CONTAINER REQUIREMENTS FOR AIR / VAPOR SAMPLES						
Volatiles	**ASB SOP(s)**	**Reference Method**	**Holding Time (days)**	**Pressure**	**Vessel**	**Preservation**
Air Toxics	MS005	TO-15	30	approx. -7 "Hg	2.7 L Summa[i]	Ambient

Notes:

[a] Nitrite, nitrate, and ortho-phosphate have a 48 hour holding time

[b] Holding time after extraction

[c] All jars should be wide mouthed and have a Teflon lid

[d] All containers must be filled completely and maintained on ice at ≤ 6 C

[e] If no additional organics are requested, a 4 oz. jar must be submitted for % solids. For MS/MSD locations, 3 extra encores/VOA vials are need. Frequency = 1/20 field samples

[f] Dispensed in preweighed 40 mL VOA vials with stir bar. Preferred over Encore™ or similar

[g] Can be requested for metals, Hg, Pesticides, SVOCs and VOCs

[h] Field collection->TCLP ext. (in days): 14 for organics, 28 for Hg, 180 for metals

[i] Contact ASB for additional details and/or options

[j] Collected w/ a 5 gram coring device (e.g. Terracore™ or similar

[k] Per sample. Does not include amount needed for QC samples or excess needed for dilutions or reanalysis

[l] 40 day holding time post extraction

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 5
77 West Jackson Boulevard
Chicago, Illinois 60604

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME													Activity Code:
SAMPLERS: *(Print Name and Sign)*						NO. OF CON-TAINERS	Analyte:							
STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION									TAG NUMBERS

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*		Ship To:
Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*		ATTN:
Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Airbill Number
				Chain of Custody Seal Numbers

Distribution: White - Accompanies Shipment; Pink - Coordinator Field Files; Yellow - Laboratory File

Printed on Recycled Paper/Printed with Soy-Based Ink

5-32569

Appendix C

Example Data Validation Forms

BELOW IS AN EXAMPLE DATA VALIDATION REPORT. WHEN VALIDATION IS PERFORMED, ALL SECTIONS WILL BE UPDATED BASED ON ANALYTICAL METHODS AND LABORATORY QUALITY CONTROL RESULTS PER DATA PACKAGE.

Southeast Rockford Area XX - XXXX Groundwater Samples
Data Validation Report

Sample Delivery Group (SDG) Number: XXXXX
Laboratory: XXXXX

Matrix: Groundwater
Collection date: 11/12/2019 & 11/13/2019
Analysis/Methods:

Voaltile Organic Compounds (VOCs) 8260
1,4-Dioxane - SW-846 8000D SIM

Samples in SDG:

ESAT Lab ID	Sample Number	ESAT Lab ID	Sample Number
XXXXX	XXXXX	XXXXX	XXXXX

Data validation was performed in accordance with the specific analytical methods and National Functional Guidelines for Organic Superfund Methods Data Review (EPA January 2017).

Volatile Organic Compounds 8260 / 1,4-Dioxane 8000D

Precision:	**Yes No N/A**
Are the field duplicate relative percent differences (RPD) ≤30% (aqueous)?	**Yes**
Were the Matrix Spike Duplicate RPDs ≤ 20%? (Or lab defined limits)	**Yes**
Laboratory Control Spike Duplicates RPD within limits?	**Yes**
Laboratory Duplicate RPDs within limits?	**N/A**

Comments (note deviations):

Field Duplicates	**Sample**	**Duplicate**	**%RPD**	**Codes**	**Qualifiers**	**Associated Samples**
	XXXXX	**XXXXX**				
1,4-Dioxane			Acceptable			
VOCs			Acceptable			

MS/MSD	**%RPD**	**Limit**	**Codes**	**Qualifiers**	**Associated Samples**
1,4-Dioxane					
E19K007-MS1 / MSD1	Acceptable				
E19K007-MS2 / MSD2	Acceptable				

LCS/LCSD	**%RPD**	**Limits**	**Codes**	**Qualifiers**	**Associated Samples**
1,4-Dioxane					
E19K007-BS1 / BSD1	Acceptable				

Laboratory Duplicate	**%RPD**	**Limits**	**Codes**	**Qualifiers**	**Associated Samples**
N/A					

Accuracy:	**Yes No N/A**
Was the Matrix Spike/Matrix Spike Duplicate criteria met? (frequency ≥ 5% and laboratory determined control limits)	**No**
Laboratory Control Sample criteria met?	**No**
Were the Laboratory Method Blank results all < RL?	**Yes**
Were the Field Blanks results all < RL?	**Yes**
Was the ICAL criteria met?	**Yes**
Was the CCV criteria met?	**Yes**
Was the Tuning criteria met?	**Yes**
Were the Surrogate % recoveries within laboratory determined control limits?	**Yes**
Were the Internal Standard areas within ± 50 - 150%?	**N/A**

Comments (note deviations):

Blanks		Concentration	MDL /PQL	Codes	Qualifiers	Associated Samples
1,4-Dioxane						
E19K007-BLK1		Nondetect				
VOCs						
E19K008-BLK1		Nondetect				
E19K014-BLK1		Nondetect				
E19K014-BLK2		Nondetect				
E19K014-BLK3		Nondetect				

Field Blank		Concentration	MDL / PQL	Codes	Qualifiers	Associated Samples
A11-FB001-191112		Nondetect				
A11-TB001-191112		Nondetect				

Surrogates		%R	Limit	Codes	Qualifiers	Associated Samples
		Acceptable				

MS/MSD		%R	Limits (%)	Codes	Qualifiers	Associated Samples
1,4-Dioxane						
E19K007-MS1 / MSD1		Acceptable				
E19K007-MS2 / MSD2		Acceptable				
VOCs						
E19K014-MS1/MSD1		Acceptable				
E19K014-MS2						
(E191105-03)	Toluene	257 / 143	60-140		**J**	**XXXXX**
	Ethylbenzene	156 / 112	60-140		**J**	**XXXXX**
	m.p-xylene	165 / 113	60-140		**J**	**XXXXX**

LCS/LCSD	Analyte	%R	Limits	Codes	Qualifiers	Associated Samples
1,4-Dioxane						
E19K007-BS1 / BSD1		Acceptable				
E19K007-MRL1		Acceptable				
VOCs						
E19K008-BS1		Acceptable				
E19K008-MRL1		Acceptable				
E19K014-BS1/BSD1						
	2,2-Dichloropropane	117 / 65.1	70-130		**J/UJ**	**XXXXX**
E19K014-BS2						
	Dichlorodifluoromethane	57.9	70-130		**J/UJ**	**XXXXX**
	Chloromethane	65.1	70-130		**J/UJ**	**XXXXX**

ICAL	RRF	%RSD	Codes	Qualifiers	Associated Samples
1,4-Dioxane					
September 20, 2019	Acceptable	Acceptable			
VOCs					
November 22, 2019	Acceptable	Acceptable			

CCV	RRF	%D	Limits	Codes	Qualifiers	Associated Samples
1,4-Dioxane						
September 20, 2019	Acceptable	Acceptable				
VOCs						
November 22, 2019	Acceptable	Acceptable				

ICV / CCV	%R	Limits	Codes	Qualifiers	Associated Samples
N/A					

Tune
Acceptable

Internal Standards	Area	Area Lower / Upper Limit	Codes	Qualifiers	Associated Samples
N/A					

Representativeness:	Yes No N/A
Were sampling procedures and design criteria met?	Yes
Were holding times met?	Yes
Was preservation criteria met? (0° C - 6° C)	Yes
Were Chain-of-Custody records complete and provided in data package?	Yes

Comments (note deviations): The cooler temperature was 3.6 ° C.

Preservation	Cooler Temperature (Degrees C)	Preservation Criteria	Codes	Qualifiers	Associated Samples
	Acceptable				

Holding Times	Analyte	Days to Extraction	HT Criteria	Codes	Qualifiers	Associated Samples
		Acceptable				

Comparability:	Yes No N/A
Were analytical procedures and methods followed as defined in the QAPP or field change documentation?	Yes

Comments (note deviations):

Completeness (90%):	Yes No N/A
Are all data in this SDG usable?	Yes

Comments (note deviations):

Sensitivity:	Yes No N/A
Are MDLs present and reported?	Yes
Do the reporting limits meet project requirements?	Yes

Comments (note deviations):

Comment:
Data is usable with appropriate qualifers applied.

Data Validator: XXXXX Date: 2/20/2020

Data Reviewer: XXXXX Date: 2/21/2020

BELOW IS AN EXAMPLE DATA VALIDATION REPORT. WHEN VALIDATION IS PERFORMED, ALL SECTIONS WILL BE UPDATED BASED ON ANALYTICAL METHODS AND LABORATORY QUALITY CONTROL RESULTS PER DATA PACKAGE.

Southeast Rockford Area XX - XXXX Groundwater Samples
Metals Data Validation Report

Sample Delivery Group (SDG) Number: XXXXX
Laboratory: XXXXX

Matrix: Water
Collection date: XXXXX
Analysis/Methods:

ICP MS Metals (Total) - EPA CLP Method ISM02.4
ICP MS Metals (Dissolved) - EPA CLP Method ISM02.4

Samples in SDG:

Sample Number	CLP Number
XXXXX	XXXXX

Data validation was performed in accordance with the specific analytical methods and the National Functional Guidelines for Inorganic Superfund Methods Data Review (EPA January 2017) .

ICP MS Metals (Total / Dissolved) - EPA CLP Method ISM02.4

Precision:	**Yes**	**No**	**N/A**
Are the field duplicate relative percent differences (RPD) ≤ 30% for water ≤35% for soils or within CRQL criteria?			**N/A**
Are the laboratory duplicate RPDs ≤ 20% for water ≤35% for soils or within CRQL criteria?	**Yes**		
Are the matrix spike duplicates RPD ≤ 20%?			**N/A**
Are the laboratory control spike duplicates RPD ≤ 20%?			**N/A**

Comments (note deviations):

Field Duplicates	**Sample**	**Duplicate**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
N/A						

MS/MSD	**%R**	**Limits**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
N/A						

LCSD	**RPD %**	**Limits**		**Codes**	**Qualifiers**	**Associated Samples**
N/A						

Laboratory Duplicate	**Sample**	**Duplicate**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
			Acceptable			

Accuracy:	**Yes**	**No**	**N/A**
Were serial dilutions analyzed and within control limits of ±10% for waters (± for 15% for soils) or initial sample result less than 50x MDL?	**Yes**		
Was matrix spike criteria met (frequency 20% and % recovery 75-125%)?	**Yes**		
Was post digestion spike criteria met (if applicable)?			**N/A**
Was laboratory control sample criteria met?	**Yes**		
Was laboratory blank criteria met (within control limits)?		**No**	
Were ICV/CCV % recoveries within 90-110%?	**Yes**		
Were the Detection Limit PQL Standards within 70-130?			**N/A**
Was the %D on form 15-IN for the initial calibration instrument response and concentration data <30%?	**Yes**		
Were ICSA/ICSAB % recoveries acceptable or within CRQL criteria?	**Yes**		
Was the tune %RSD <5% ?	**Yes**		
Was internal standard criteria met?	**Yes**		

Comments (note deviations):

Serial Dilution

Analyte	Initial Sample Result	%D	50 x MDL	Codes	Qualifiers	Associated Samples
		Acceptable				

Matrix Spike

Analyte	%R	Limits	Post Digestion % R	Codes	Qualifiers	Associated Samples
	Acceptable					

LCS

Analyte	%R	Limits	Codes	Qualifiers	Associated Samples
.	Acceptable				

ICV/CCV

%R	Limits	Codes	Qualifiers	Associated Samples
Acceptable				

Blanks

Note: ICBs and Prep blanks are associated with all samples. Individual CCBs are associated with specific samples.

Prep Blank

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples
Associated Samples: All samples					
PBW685	Nondetect				
PBW686	Nondetect				

ICB

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples
Associated Samples: All samples					
	Nondetect				

CCBs

Prep Blank and ICBs were evaluated and qualifiers were applied to their associated results where applicable. CCB qualifications were applied to associated sample results if not previously accounted for in the prep blank/ ICB review. The following CCB qualifications were required:

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples
	Nondetect				

Field Blank

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples
N/A					

ICSA/AB

Analyte - Solution A	%R	Found Sol. A / True A	CRQL	Codes	Qualifiers	Associated Samples
	Acceptable					

PQL Standard Check

	%R	Codes	Qualifiers	Associated Samples
N/A				

Tune

Analyte	%RSD	Limits	Codes	Qualifiers	Associated Samples
	Acceptable				

Internal Standard (IS)

Standard / Element	%R	Affected Analyte	Limits	Codes	Qualifiers	Associated Samples
	Acceptable					

Initial Calibration %D (Form 15)

Analyte	%D	Limits	Codes	Qualifiers	Associated Samples
	Acceptable				

Mercury - CCVA ISM02.4

Precision:	**Yes**	**No**	**N/A**
Are the field duplicate relative percent differences (RPD) ≤ 20% for water ≤35% for soils or within CRQL criteria?			**N/A**
Are the laboratory duplicate RPDs ≤ 20% for water ≤35% for soils or within CRQL criteria?	**Yes**		
Are the matrix spike duplicates RPD ≤ 20%?			**N/A**
Are the laboratory control spike duplicates RPD ≤ 20%?			**N/A**

Comments (note deviations):

Field Duplicates	**Sample**	**Duplicate**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
N/A						

MS/MSD	**%R**	**Limits**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
N/A						

LCSD	**RPD %**	**Limits**	**Codes**	**Qualifiers**	**Associated Samples**
N/A					

Laboratory Duplicate	**Sample**	**Duplicate**	**RPD %**	**Codes**	**Qualifiers**	**Associated Samples**
			Acceptable			

Accuracy:	**Yes**	**No**	**N/A**
Were serial dilutions analyzed and within control limits of ±10% for waters (± for 15% for soils) or initial sample result less than 50x MDL?			**N/A**
Was matrix spike criteria met (frequency 20% and % recovery 75-125%)?	**Yes**		
Was post digestion spike criteria met (if applicable)?			**N/A**
Was laboratory control sample criteria met?			**N/A**
Was laboratory blank criteria met (within control limits)?	**Yes**		
Were ICV/CCV % recoveries within 90-110%?	**Yes**		
Were the Detection Limit PQL Standards within 70-130?			**N/A**
Was the %D on form 15-IN for the initial calibration instrument response and concentration data <30%?	**Yes**		
Were ICSA/ICSAB % recoveries acceptable or within CRQL criteria?			**N/A**
Was the tune %RSD <5% ?			**N/A**
Was internal standard criteria met?			**N/A**

Comments (note deviations):

Serial Dilution

Analyte	**Initial Sample Result**	**%D**	**50 x MDL**	**Codes**	**Qualifiers**	**Associated Samples**
		Acceptable				

MS

Analyte	**%R**	**Limits**	**Post Digestion % R**	**Codes**	**Qualifiers**	**Associated Samples**
Mercury	Acceptable					

LCS

Analyte	**%R**	**Limits**	**Codes**	**Qualifiers**	**Associated Samples**
N/A					

ICV/CCV

%R	**Limits**	**Codes**	**Qualifiers**	**Associated Samples**
Acceptable				

Blanks **Note: ICBs and Prep blanks are associated with all samples. Individual CCBs are associated with specific samples.**

Prep Blank

Associated Samples: All samples

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples
	Nondetect				

ICB

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples

Prep Blanks were evaluated and qualifiers were applied to their associated results where applicable. ICB qualifications were applied to associated sample results if not previously accounted for in the prep blank/ ICB review. The following CCB qualifications were required:

None Required

CCBs

Prep Blank and ICBs were evaluated and qualifiers were applied to their associated results where applicable. CCB qualifications were applied to associated sample results if not previously accounted for in the prep blank/ ICB review. The following CCB qualifications were required:

None Required

Field Blank

Analyte	Result (µg/L)	MDL/CRQL	Codes	Qualifiers	Associated Samples

N/A

ICSA/AB

Analyte - Solution A	%R	Found Sol. A / True A	CRQL	Codes	Qualifiers	Associated Samples

N/A

PQL Standard Check

%R	Codes	Qualifiers	Associated Samples

N/A

Tune

Analyte	%RSD	Limits	Codes	Qualifiers	Associated Samples

N/A

Internal Standard (IS)

Standard / Element	%R	Affected Analyte	Limits	Codes	Qualifiers	Associated Samples

N/A

Initial Calibration %D (Form 15)

Analyte	%D	Limits	Codes	Qualifiers	Associated Samples
	Acceptable				

Representativeness:	**Yes No N/A**
Were sampling procedures and design criteria met?	**Yes**
Were holding times met?	**Yes**
Were preservation criteria met? (0 - 6°C)	**Yes**
Were Chain-of-Custody records complete and provided in data package?	**Yes**
Was the raw data present for drying logs, preparation logs, analytical instrument real-time printouts and laboratory bench sheets?	**Yes**
Were results less than MDL reported with a "U" and values less than the CRQLs but greater than MDL reported with a "J?"	**Yes**

Comments (note deviations): Cooler temperatures were 2.8 °C.

Preservation

Cooler Temperature (Degrees C)	Preservation Criteria	Qualifier	Associated Samples
Acceptable			

Holding Times

Analyte	Days to Analysis	HT Criteria	Codes	Qualifiers	Associated Samples
	Acceptable				

Completeness (90%):	**Yes No N/A**
Are all data in this SDG useable?	**Yes**

Comments (note deviations):

Sensitivity:	**Yes No N/A**
Are MDLs present and reported?	**Yes**
Do the reporting limits meet the project requirements?	**Yes**

Comments (note deviations):

Overall Comments:

Data is usable with appropriate qualifiers applied.

Data Validator: XXXXX Date: 7/27/2020

Data Reviewer: XXXXX Date: 7/28/2020