



# **APPENDIX D-I**

## **Quality Assurance and Surveillance Plan (QASP)**



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Attachment D.I-1 EPA 540-R-20-005, Sampler's Guide: Contract Laboratory Program Guidance for Field Samplers



45031 &amp; 45032

## TITLE AND APPROVAL SHEET

This Quality Assurance and Surveillance Plan (QASP) is approved for use and implementation at Project Minerva. The signatures below denote the approval of this document and intend to abide by the procedures outlined within it.

---

 Signature

DAVID COOK

CHIEF EXECUTIVE OFFICER

---

 Date

---

 Signature

INSERT TYPED NAME

INSERT TITLE

---

 Date

---

 Signature

INSERT TYPED NAME

INSERT TITLE

---

 Date



## FACILITY INFORMATION

Facility Name: Minerva Facility

Injection Wells: Minerva South CCS Well No. 001 (MS CCS 1)  
Minerva South CCS Well No. 002 (MS CCS 2)

Facility Contact: David Cook, CEO  
5599 San Felipe St., Ste. 1450, Houston, Texas 77056  
(713) 419-6808; [dcook@gcscarbon.com](mailto:dcook@gcscarbon.com)

Well Locations: Sec 3, T12S, R13W, Cameron Parish, Louisiana

MS CCS 1 (NAD 1927)  
Surface: 30° 02' 34.10"N, 93° 40' 20.63"W  
Bottom-Hole: 30° 02' 34.10"N, 93° 40' 20.63"W

MS CCS 2 (NAD 1927)  
Surface: 30° 02' 33.84"N, 93° 40' 20.48"W  
Bottom-Hole: 30° 02' 13.74"N, 93° 40' 42.07"W

## 1 INTRODUCTION

This Quality Assurance and Surveillance Plan (QASP) describes how Gulf Coast Sequestration, LLC (GCS) will monitor Project Minerva pursuant to LAC 43.XVII §3625.A.11. Monitoring will be used to demonstrate that Minerva South CCS Well Nos. 001 and 002 (MS CCS 1 and MS CCS 2) are operating as planned, the CO<sub>2</sub> plume and pressure front are moving as predicted, and that there is no endangerment to USDW. Additionally, the monitoring data will be used to validate and adjust the simulation model used to predict the distribution of the CO<sub>2</sub> within the Injection Zone to support Area of Review (AoR) re-evaluations and a non-endangerment demonstration.

## 2 DISTRIBUTION LIST

The participants of the project presented in Table D.I.2-1 will receive the completed QASP and all future updates for the duration of the project. Annual updates of the distribution list will be provided, contingent on staff changes occurring in the preceding 12 months. The GCS Site Manager will be responsible for ensuring that all those on the distribution list will receive the most current copy of the approved QASP. Names and contact details are provided for those positions on the distribution list that have currently been filled. GCS will provide an update to the UIC Director once all positions have been filled, prior to project construction.

## 3 PROJECT/TASK ORGANIZATION

### 3.1 KEY INDIVIDUALS AND RESPONSIBILITIES

Project Minerva is led by GCS and includes the participation of several subcontractors. The testing and monitoring responsibilities will be shared between GCS and designated subcontractors. Key individuals will be appointed before project construction begins; contact details will be provided to the UIC Director at that time.



Designated responsibilities will include:

#### GCS Field Engineering and HSE

- CO<sub>2</sub> stream analysis surface sampling
- Continuous recording of operational parameters
- Corrosion monitoring
- In-zone monitoring - pressure front tracking

#### Oilfield Service and Equipment Consultants (Baker Hughes)

- In-zone monitoring – Pulsed Neutron (PN) logging
- VSP in-direct monitoring of CO<sub>2</sub> plume
- External mechanical integrity testing (MIT) of injection and monitoring wells
- Pressure fall-off testing of injection wells

#### Environmental Consultants (TetraTech)

- Above confining zone groundwater sampling and monitoring

### **3.2 INDEPENDENCE FROM PROJECT QUALITY ASSURANCE (QA) MANAGER AND DATA GATHERING**

The majority of the physical samples collected, and data gathered as part of the Testing and Monitoring Plan will be analyzed, processed, or witnessed by third parties independent from and outside of the project management structure. The term “third parties” is used to refer to any consultancies, contractors, vendors or other businesses contracted to carry out sample collection and analysis, on behalf of GCS. "Third parties" will be given a copy of the QASP within the scope of work during the request for proposal process. GCS will issue an updated copy of the QASP to the Louisiana Department of Conservation and Energy (C&E) Secretary (C&E Secretary) detailing selected third parties once they have been chosen and prior to commencement of work.

### **3.3 QASP RESPONSIBILITY**

GCS is responsible for maintaining and distributing an official, approved QASP. GCS will review, no less than every five years, the QASP and consult with the C&E Secretary to determine whether changes to the plan are warranted.

### **3.4 ORGANIZATIONAL CHART FOR KEY PROJECT PERSONNEL**

Figure D.I.3.4-1 shows the organization structure of the project. GCS will provide the C&E Secretary with a contact list of individuals fulfilling these roles.

## **4 PROBLEM DEFINITION/BACKGROUND**

### **4.1 REASONING**

The primary goal of the Testing and Monitoring Plan is to ensure that the geologic storage



project is operating as permitted and is not endangering USDWs. In compliance with LAC 43.XVII §3625, this QASP was developed and is provided as Appendix D-I of Attachment D, Testing and Monitoring Plan.

## 4.2 REASONS FOR INITIATING THE PROJECT

The goal of Project Minerva is to demonstrate the ability of the upper Frio Formation to accept and retain industrial-scale volumes of CO<sub>2</sub> for permanent geologic storage thus reducing atmospheric concentrations of CO<sub>2</sub>.

## 4.3 REGULATORY INFORMATION, APPLICABLE CRITERIA, ACTION LIMITS

The Class VI rule requires owners or operators of Class VI wells to perform several types of activities during the lifetime of the project to ensure that the injection well maintains mechanical integrity, that fluid migration and the extent of pressure elevation are within the limits described in the permit application, and that USDWs are not endangered. These monitoring activities include CO<sub>2</sub> stream analysis, continuous recording of operational parameters, corrosion monitoring, above confining zone monitoring, MITs, pressure fall-off testing, and tracking of the CO<sub>2</sub> plume and associated pressure changes.

This appendix to the Testing and Monitoring Plan details both the measurements that will be taken as well as the steps to ensure that the quality of all the data can be used with confidence in making decisions during the life of the project, based on requirements stipulated in “EPA Requirements for Quality Assurance Project Plans” (EPA QA R-5).

## 5 PROJECT/TASK DESCRIPTION

### 5.1 SUMMARY OF WORK TO BE PERFORMED

Table D.I.5.1-1 provides a summary of the testing and monitoring activities planned for Project Minerva. The monitoring activities can be divided into the following monitoring categories to achieve objectives at defined locations:

- CO<sub>2</sub> stream monitoring;
- Injection and integrity monitoring; and
- Subsurface monitoring.

### 5.2 GEOGRAPHIC LOCATIONS

Figure D.I.5.2-1 illustrates the locations of the Testing and Monitoring Plan activities and elements.

### 5.3 RESOURCE AND TIME CONSTRAINTS

Project Minerva’s Area of Review (AoR) encompasses land primarily owned/leased by GCS. There are no additional resources or time constraints identified for the Testing and Monitoring Plan based on property ownership issues.

## 6 QUALITY OBJECTIVES AND CRITERIA



The overall objective of the QASP is to provide the standards and procedures necessary to validate the overall integrity of the sampling methods discussed in the Testing and Monitoring Plan and ensure the results of sampling and testing will meet the characterization and non-endangerment goals of Project Minerva. The QASP is the mechanism by which the C&E Secretary has confidence in the rigor of the Testing and Monitoring Plan.

In the following sections, GCS has addressed the applicable quality assurance criteria for the equipment and measurements associated with each of the monitoring areas. An instrumentation summary is provided as Table D.I.6-1.

## 6.1 CO<sub>2</sub> STREAM MONITORING

Grab samples of the CO<sub>2</sub> stream will be obtained for analysis of gases (Table D.I.6.1-1). Samples will be collected at each emitter's outlet and at the comingled inlet pipework to GCS facilities. These grab samples will be obtained using laboratory approved sampling cylinders to collect and transport the samples. Stainless steel tubing or equivalent pressure hoses will be connected to a designated sampling port, with a regulator to control the pressure. The pressure of the CO<sub>2</sub> will be reduced ensuring the CO<sub>2</sub> is collected in the gaseous state as required by the Federal transportation regulations for shipment of compressed gases (CFR 173.301.a.8). Sample cylinders will be purged during sample collection to remove laboratory-added helium gas and ensure a representative sample. Sample containers will have a chain of custody form and will be labeled appropriately. Samples will be sent to and analyzed by a Louisiana Environmental Laboratory Accreditation Program (LELAP) accredited laboratory.

Once the LELAP laboratory is selected, Table D.I.6.1-1 will be updated with the laboratory informed detection limit/range, typical precisions, and QC requirements for each parameter.

## 6.2 INJECTION AND INTEGRITY MONITORING

Injection and integrity monitoring requires equipment used to monitor injection temperature, pressure, rate, and volume; the downhole temperature and pressure in the tubing and tubing-casing annulus of the injection and monitoring wells; and the mechanical integrity logs and tests performed to ensure internal and external integrity of the injection and in-zone monitoring wells. Table D.I.6.2-1 provides a summary of the operating parameters for MS CCS 1 and MS CCS 2 that will be measured with continuous recording devices.

### 6.2.1 Injection Temperature, Pressure, Rate, and Volume Monitoring

Injection temperature, pressure, rate, and volume will be measured at each injection well by volumetric flow meter(s). Each well will be equipped with primary and secondary meters. The volumetric flow meters will also be equipped with electronic pressure and temperature gauges with transmitters to continuously monitor pressure and temperature of the injection stream. The data from the meter station will be collected and recorded in the computerized control system.



GCS is working with vendors to determine the most appropriate flow meter for Project Minerva. The specification sheet of an example flow meter is provided in Appendix D-II of the Testing and Monitoring Plan. The specifications for the example flow meter are summarized in Table D.I.6.2-2 and will be updated with the final values upon completion of detailed design.

### 6.2.2 Temperature and Pressure Wellbore Monitoring

The injection wells, MS CCS 1 and MS CCS 2, will be equipped with the following instrumentation to monitor pressure at the surface and temperature/pressure downhole:

- Wellhead Tubing Pressure Gauge
- Wellhead Annulus Pressure Gauge
- Downhole Annulus Pressure and Temperature Gauge with Transmitter at Surface
- Downhole Pressure and Temperature Gauge with Transmitter at Surface

GCS is still working with vendors to determine the most appropriate gauges and tools for Project Minerva. The specification sheets of an example wellhead pressure gauge and a downhole pressure and temperature gauge are provided in Appendix D-II of the Testing and Monitoring Plan. The specifications, for the example of gauges are summarized in Table D.I.6.2-2 and will be updated with the final values upon completion of detailed design.

### 6.2.3 Mechanical Integrity Logging and Testing

External mechanical integrity will be demonstrated using the following technologies:

- Primary
  - Distributed Temperature Sensing (DTS)
- Alternate
  - Noise Log
  - Oxygen Activation Log
- Supplemental
  - Casing Inspection Log
  - Cement Bond Log
  - Casing Collar Locator Log

GCS will use DTS fiber optic cables installed from surface to total depth of MS CCS 1 and MS CCS 2 as the primary method to demonstrate external mechanical integrity. A baseline temperature survey will be acquired in MS CCS1 and MS CCS2 as part of the Preoperational Testing Program (Attachment C – Table C.2.2-1 and Table C.2.2-3). This survey will provide the data required to corroborate the use of DTS for external mechanical integrity and leak detection. If additional data are deemed necessary, additional temperature surveys will be conducted.

A continuous two-point calibration, using downhole pressure/temperature (P/T) gauges and



wellhead temperature, will allow ongoing verification of DTS temperature measurements.

GCS will employ one of the alternate test technologies should a contingent method be required. Supplemental technologies may be required in conjunction with the primary or alternate technologies.

A summary of the logging technologies with their specifications are provided as Table D.I.6.2-3. The specifications are based on a service provider whose specification sheets for each technology are provided as Appendix D-IV of the Testing and Monitoring Plan. Should a different service provider be selected, the table and appendix will be updated with the required information.

Data comparability expresses the confidence with which one data set can be compared to another. The data sets to be generated by this project will be very comparable to future data sets because of the use of standard methods and the level of QA/QC effort.

#### **6.2.4 Corrosion Monitoring**

GCS will monitor corrosion by analyzing corrosion coupons made from well construction materials exposed to the carbon dioxide stream, as described in the Project Minerva Testing and Monitoring Plan (Attachment D), Section 5.1 Corrosion Monitoring – Surface Monitoring Details.

During scheduled onsite retrieval, corrosion coupons will be visually inspected, documented and properly preserved for transport for detailed analysis. Each coupon will be submitted to a third-party laboratory and assessed using the American Society for Testing and Materials (ASTM) G1-03, Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens (or equivalent industry standard). This ASTM process includes visually inspecting the coupons for evidence of corrosion (e.g., discoloration, pitting), documenting and evaluating any surface deposits or scale prior to cleaning, measuring the weight and size (thickness, width, length) of the coupons, and calculating the corrosion rate based on weight loss during the exposure period divided by the duration (i.e., weight loss method).

All coupons will be newly prepared, uniquely identified, and their initial masses will be recorded.

### **6.3 SUBSURFACE MONITORING**

#### **6.3.1 Above Confining Zone Groundwater Monitoring**

The quality assurance criteria for evaluating the integrity of groundwater sampling protocol include: (1) performance/measurement, (2) precision, (3) bias, (4) representativeness, (5) completeness, (6) and comparability.

##### *Performance/Measurement Criteria*

Groundwater monitoring will be conducted prior to injection to establish a baseline, during the operational lifetime of the project, and during the 50-year post injection site care and site closure (PISC) period. Above Confining Zone (ACZ) monitoring wells (MS USDW 1 and MS USDW 2) will be used to collect water-quality samples from the first confined sand unit



below the base of the Underground Source of Drinking Water (USDW). All the groundwater analytical and field monitoring parameters and outputs are listed in Table D.I.6.3-1. The list of analytes may be reassessed periodically and adjusted to include or exclude analytes based on their effectiveness to the overall monitoring program goals.

### Precision

For groundwater sampling, data accuracy will be assessed by the collection and analysis of field blanks to test sampling procedures and matrix spikes to test lab procedures. Field blanks will be taken no less than one per sampling event to spot check for sample bottle contamination. Laboratory assessment of analytical precision will be the responsibility of the individual laboratories per their standard operating procedures.

### Bias

Field blanks assess potential bias from the combined processes of sample handling, processing, and laboratory analysis. A field blank is prepared in the field by filling a clean container with deionized water and appropriate preservative, if any, using the same techniques as the specific sampling activity being undertaken. A field equipment blank is a sample of analyte-free media that has been used to rinse common sampling equipment to detect potential bias introduced through the usage of the sampling apparatus.

Laboratory bias is determined through the analysis of laboratory control samples and limit of quantitation check samples prepared in the sample matrix (e.g., laboratory grade water, sand, commercially available tissue) using verified and known amounts of all target analytes and by then calculating percent recovery of those analytes. Results are compared to control limits and used during evaluation of analytical performance.

Laboratory assessment of analytical bias will be the responsibility of the individual laboratories per their SOPs and analytical methodologies.

### Representativeness

For groundwater sampling, data representativeness expresses the degree to which data accurately and precisely represents a characteristic of a sample population, parameter variations at a sampling point, a process condition, or an environmental condition. The sampling network has been designed to provide data representative of site conditions. For results of individual groundwater sample analysis, representativeness will be estimated by ion and mass balances. Ion balances with  $\pm 10\%$  error or less will be considered valid. A mass balance assessment will be used in cases where the ion balance is greater than  $\pm 10\%$  to help determine the source of error. For a sample and its duplicate, if the relative percent difference is greater than 10%, the sample may be considered non-representative.

### Completeness

For groundwater sampling, data completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is anticipated that data completeness of 90% for groundwater sampling will be acceptable to meet monitoring goals. For direct pressure and temperature measurements, it is expected that data will be recorded no less than 90% of



the time.

### Comparability

Data comparability expresses the confidence with which one data set can be compared to another. The data sets to be generated by this project will be very comparable to future data sets because of the use of standard methods and the level of QA/QC effort. If historical groundwater quality data becomes available from other sources, their applicability to the project and level of quality will be assessed prior to use with data gathered on this project.

### **6.3.2 Direct Monitoring**

#### Elevated Pressure Monitoring

The baseline in-situ reservoir pressure within the Injection Zone will be established after the completion of MS CCS 1 and MS CCS 2. Once operation commences, reservoir pressure data will be recorded from MS CCS 1 and MS CCS 2 during scheduled shut-ins and compared to the modeled predictions. If pressure increases greater than predicted by the current AoR model, or increases at a greater rate, it may indicate that the AoR model needs to be revised. In this case, GCS will consult with the C&E Secretary to determine whether an AoR reevaluation is necessary.

The specification sheet of an example downhole pressure and temperature gauge is provided in Appendix D-II of the Testing and Monitoring Plan. The specifications for the example gauge are summarized in Table D.I.6.2-2 and will be updated with the final values upon completion of detailed design.

### **6.3.3 Indirect Monitoring**

#### CO<sub>2</sub> Plume Extent Tracking

Four indirect monitoring methods will be used to define the extent of the modeled plume for Project Minerva:

- Walk-Away Vertical Seismic Profile (WA VSP)
- 3D Distributed Acoustic Sensing Vertical Seismic Profile (3D DAS VSP)
- 2D Seismic Survey
- Pulsed Neutron (PN) logging

#### Seismic Surveys

A seismic baseline will be acquired before injection begins using WA VSP, 3D DAS VSP, and 2D seismic surveys simultaneously.

WA VSP and 3D DAS VSP utilize distributed acoustic sensing (DAS) technology installed along MS CCS 1 to receive seismic signals emitted from a source. 2D seismic surveys will utilize geophones to receive seismic signals emitted from a source. Sources include downhole air guns, vibrators, or non-disruptive explosives. The specifications for the proposed DAS and geophone technologies used to conduct indirect plume monitoring are provided in Appendix D-VIII.



Once injection commences, WA VSP will serve as the primary plume monitoring method for approximately 10 years (or until its coverage is surpassed by the leading edge of the plume) and will be supplemented, as necessary, with 3D DAS VSP and/or 2D seismic surveys. After approximately 10 years, 2D seismic surveys will serve as the primary plume monitoring method. These surveys will be conducted along seismic lines or grids described in Section 9.2 (Indirect Monitoring) in the Testing and Monitoring Plan (Attachment D).

Appendix D-IX of the Testing and Monitoring Plan (Attachment D) defines the feasibility of implementing a VSP at Project Minerva.

### *PN Logging*

GCS will utilize Pulsed Neutron (PN) logging to monitor CO<sub>2</sub> saturation changes in the Injection Zone. GCS will conduct time-lapsed PN logging at MS IZ 1 to monitor for CO<sub>2</sub> saturation in the perforated interval. Since MS IZ 1 is located outside of the modeled stabilized CO<sub>2</sub> plume, the CO<sub>2</sub> residual gas saturation should not exceed 20%. The specifications for PNL used in the in-zone monitoring well to conduct indirect plume monitoring are provided in Appendix D-VIII.

PN logging of MS IZ 1 will be conducted annually for the first four years, and every two years thereafter through the operational life of Project Minerva. GCS will continue monitoring MS IZ 1 during the post injection surveillance period defined in the Post Injection Site Care and Closure Plan (Attachment F).

Data comparability expresses the confidence with which one data set can be compared to another. The data sets to be generated by this project will be very comparable to future data sets because of the use of standard methods and the level of QA/QC effort. Direct pressure, temperature, and logging measurements will be directly comparable to previously obtained data.

### *Microseismic Monitoring*

Downhole geophones and accelerometers will be installed in MS IZ 1, located north of the nearest fault. Geophones convert ground motion into electrical signals, while accelerometers measure the acceleration of the ground. The placement of these sensors allows for localized monitoring of seismic activity and fault movement in the surrounding geological formations.

The downhole geophones and accelerometers provide supplementary data by capturing local seismic events and ground motion that may not be detected by DAS alone. This information helps to establish a more comprehensive understanding of fault dynamics.

GCS has not selected the technology that will be installed in MS IZ 1; however, the specification sheet of an example technology is provided in Appendix D-VIII of the Testing and Monitoring Plan (Attachment D). When GCS is prepared to convert Stream 34 Well No. 001 to MS IZ 1, a detailed conversion plan along with the other required documents will be submitted for approval with the Class V application. This will include the proposed technology that will be installed in MS IZ 1 to monitor microseismic activity.



## 7 SPECIALIZED TRAINING AND CERTIFICATIONS

### 7.1 SPECIALIZED TRAINING AND CERTIFICATIONS

The geophysical survey equipment and wireline logging tools will be operated by trained, qualified, and certified personnel, according to the service company which provides the equipment. The collected data will be processed and analyzed according to industry standards. No specialized certifications are required for personnel conducting groundwater sampling, but field sampling will be conducted by trained personnel. Groundwater sampling will be conducted by personnel trained to understand and follow the specific project sampling procedures outlined in this appendix. Upon request GCS will provide the agency with all laboratory SOPs developed for the specific parameter using the appropriate standard method. Each laboratory technician conducting the analysis on the samples will be trained in the SOP developed for each standard method. GCS will include the technician's training certification with the annual report.

### 7.2 TRAINING PROVIDER AND RESPONSIBILITY

Training for personnel will be provided by GCS or by the subcontractor responsible for the data collection activity.

## 8 DOCUMENTATION AND RECORDS

### 8.1 REPORT FORMAT AND PACKAGE INFORMATION

Reporting will follow the outlines contained within the Testing and Monitoring Plan and will contain all required project data, as specified by the UIC Class VI permit. Data will be provided in electronic or other formats as required by the C&E Secretary.

### 8.2 OTHER PROJECT DOCUMENTS, RECORDS, AND ELECTRONIC FILES

Other documents, records, and electronic files such as, well logs, test results, or other data will be provided as required by the C&E Secretary.

### 8.3 DATA STORAGE AND DURATION

All data and project records will be stored electronically on secure servers and will have routine backups. Reporting will be retained for 10 years after it is collected pursuant to LAC 43:XVII§3625.A.1-9.

### 8.4 QASP DISTRIBUTION RESPONSIBILITY

GCS will be responsible for ensuring that all those on the distribution list will receive the most current copy of the approved QASP.

## 9 DATA GENERATION AND ACQUISITION

This plan will establish a standardized program for data acquisition and validation methods. The program will verify that collected data are reasonable, processed and analyzed correctly, and free of errors. Peer reviews or third-party consultants may serve as a QC



mechanism. Issues identified during a peer review will be addressed and corrected by the data owner. Errors identified in the data via validation will be corrected, and affected users will be notified. Corrective actions will be taken to ensure the impact of any error is fully addressed.

## 9.1 SAMPLING PROCESS DESIGN

### 9.1.1 CO<sub>2</sub> Stream Monitoring

#### Design Strategy

GCS will analyze the CO<sub>2</sub> stream during the operation period to determine its chemical and physical characteristics and to meet the requirements of LAC 43:XVII§3625.A.1. See Section 3.0 (Carbon Dioxide Stream Analysis) of the Testing and Monitoring Plan (Attachment D) for further information.

#### Type and Number of Samples/Test Runs

CO<sub>2</sub> gas stream composition will be analyzed on a quarterly basis to ensure it meets the system specification. This is outlined in Attachment D (Testing and Monitoring Plan).

#### Site/Sampling Locations

CO<sub>2</sub> stream sampling will be conducted at a sample port along the comingled inlet pipework to the Minerva Facility.

#### Sampling Site Contingency

All infrastructure required to implement CO<sub>2</sub> stream analysis and corrosion monitoring will be located on GCS-controlled land, with guaranteed site access. All related facilities will be constructed in a way that allows access during inclement weather.

#### Activity Schedule

Analyzed on a quarterly basis.

### 9.1.2 Corrosion Monitoring

#### Design Strategy

To meet the requirements of LAC 43:XVII§3625.A.3, GCS will monitor well materials during the operation period for loss of mass, thickness, cracking, pitting, and other signs of corrosion to ensure that the well components meet the minimum standards for material strength and performance.

GCS will monitor corrosion by analyzing corrosion coupons. Coupon holders will be constructed from materials suitable for exposure to the CO<sub>2</sub> stream. Final material selection will be completed after detailed engineering. The QASP will be updated with equipment specifications for the chosen technology.

#### Type and Number of Samples/Test Runs

The corrosion coupons will be analyzed on a quarterly basis starting during the first month



of the calendar quarter (i.e., March, June, September, December) following commencement of injection. This is outlined in Attachment D (Testing and Monitoring Plan).

#### Site/Sampling Locations

The corrosion coupons will be installed in parallel with the pipe at the surface facilities that feed CO<sub>2</sub> injectate to the injection wells. The corrosion coupons will be located downstream of all process compression/dehydration/pumping equipment and designed to monitor each injection well independently.

#### Sampling Site Contingency

All infrastructure required to implement CO<sub>2</sub> stream analysis and corrosion monitoring will be located on GCS-controlled land, with guaranteed site access. All related facilities will be constructed in a way that allows access during inclement weather.

#### Activity Schedule

Corrosion coupons will be analyzed quarterly.

### **9.1.3 Above Confining Zone Groundwater Monitoring**

This section is focused on fluid sampling above the confining zone. GCS proposes to drill and complete two ACZ monitoring wells, MS USDW 1 and MS USDW 2, in the first confined sand below the lowermost USDW (Attachment D – Testing and Monitoring Plan). The following section does not address monitoring methods that do not gather physical samples (e.g., logging, seismic monitoring, and pressure/temperature monitoring).

Fluid sampling is planned to include an extensive set of chemical parameters to establish aqueous geochemical reference data. Parameters will include selected constituents that: (1) have primary and secondary EPA drinking water maximum contaminant levels, (2) are the most responsive to interaction with CO<sub>2</sub> or brine, (3) are needed for quality control, and (4) may be needed for geochemical modeling. The full set of parameters is given in Table D.I.6.3-1.

A baseline will be established via testing every 4 months for at least 12 months before the commencement of injection. Following commencement of injection monitoring, monitoring scope may shift to a subset of indicator parameters that are (1) the most responsive to interaction with CO<sub>2</sub> or brine and (2) are needed for quality control. Implementation of a reduced set of parameters would be done in consultation with the C&E. Isotopic analyses will be performed on baseline samples to the degree that the information helps verify a condition or establish an understanding of non-project related variations. For non-baseline samples, isotopic analyses may be reduced in all monitoring wells if a review of the historical project results or other data determines that further sampling for isotopes is unneeded. During any period where a reduced set of analytes is used, if statistically significant trends are observed that are the result of unintended CO<sub>2</sub> or brine migration, the analytical list would be expanded to the full set of monitoring parameters.

Fluid samples will be analyzed using a laboratory meeting the requirements under the LELAP. All other samples will be analyzed by the operator or a third-party laboratory.



Dissolved CO<sub>2</sub> will be analyzed by methods consistent with Test Method B of ASTM D 513-06, “Standard Test Methods for Total and Dissolved Carbon Dioxide in Water” or equivalent. Isotopic analysis will be conducted using established methods.

### Design Strategy

GCS will conduct groundwater quality and geochemical monitoring in the first confined sand unit below the lowermost USDW (Evangeline aquifer). This will be achieved using two groundwater monitoring wells, Minerva South USDW Well No. 001 (MS USDW 1), located at the Minerva South Well Pad, and Minerva South USDW Well No. 002 (MS USDW 2) located at the well pad of the in-zone monitoring well (see Figure D.I.5.2-1).

Mixing models will be constructed to identify trends and constituents that would be diagnostic of either brine or CO<sub>2</sub> leakage into USDW. The baseline monitoring will be completed prior to year three and will be used to 1) modify and optimize the groundwater program to the parameters that are most diagnostic and 2) design a response program such that if an incident of leakage of CO<sub>2</sub> or brine into groundwater occurs, GCS will be ready to assess, report and remediate the area impacted by the leakage.

Throughout the life of the project (injection and post-injection period) monitoring of the ACZ interval will be used for early leakage detection. Fluid sampling in combination with pressure monitoring will be used to determine if leakage is occurring via any pathways through the Confining Zone (faults or artificial penetrations).

Following the three-year characterization and response-planning period, the sampling frequency at the two water wells will be decreased annually during the remaining injection period and every five years during the post-injection monitoring period. This data will be used to demonstrate non-endangerment of the USDW or initiate an immediate project shutdown if a leak of CO<sub>2</sub> is detected.

The number and location of USDW monitoring wells will be adapted to ensure effective monitoring and non-endangerment of the USDW over the injection and post-injection monitoring period. Monitoring data collected during the injection period will be used to history-match the simulation model and define if and where any additional USDW water wells are required.

### Type and Number of Samples/Test Runs

Table D.I.6.3-1 shows the planned monitoring methods, logic behind selecting locations, and frequencies for ACZ groundwater quality and geochemical monitoring in the Evangeline Aquifer. Seasonal sampling including field parameters, dissolved and free gases, and water level monitoring will be conducted four times a year for 3 years for characterization, after which sampling frequency will be decreased and targeted to chemical species that are indicative of leakage. Refer to Section 6 (Above Confining Zone Monitoring) of the Testing and Monitoring Plan (Attachment D) for further details.

### Site/Sampling Locations

Summarized in Figure D.I.5.2-1.



### Sampling Site Contingency

For above zone water sampling, all wells will be drilled on GCS-controlled land and have been assessed for wellsite access. The preliminary locations of these wells are mapped in Figure D.I.5.2-1. No problems of site inaccessibility are anticipated. If inclement weather makes site access difficult, sampling schedules will be reviewed, and alternative dates may be selected that would still meet permit-related conditions.

### Activity Schedule

Sampled quarterly for 3 years to detect seasonal variations and to establish a baseline, then annually, or if an incident occurs.

Continuous temperature monitoring along with the proposed injection wells will be used to detect anomalies attributed to leakage from the Injection Zone. If leakage detected, additional sampling will be added as necessary to ensure non-endangerment of the USDW.

### Critical/Informational Data

During ACZ water sampling and analytical efforts, detailed field and laboratory documentation will be taken. Documentation will be recorded in field and laboratory forms and notebooks. Critical information will include time and date of activity, person/s performing activity, location of activity (wellfield sampling) or instrument (lab analysis), field or laboratory instrument calibration data, field parameter values. For laboratory analyses, much of the critical data is generated during the analysis and provided to end users in digital and printed formats. Noncritical data may include appearance and odor of the sample, problems with well or sampling equipment, and weather conditions.

### Sources of Variability

Potential sources of variability related to monitoring activities include:

- Natural variation in fluid quality, formation pressure and temperature and seismic activity,
- Variation in fluid quality, formation pressure and temperature, and seismic activity due to project operations,
- Changes in recharge due to rainfall, drought, and snowfall,
- Changes in instrument calibration during sampling or analytical activity,
- Different staff collecting or analyzing samples,
- Differences in environmental conditions during field sampling activities,
- Changes in analytical data quality during life of project, and
- Data entry errors related to maintaining project database.

Activities to eliminate, reduce, or reconcile variability related to monitoring activities include:

- Collecting baseline data to observe and document natural variation in monitoring



parameters,

- Evaluating data in a timely manner after collecting to observe anomalies in data that can be addressed by being resampled or reanalyzed,
- Conducting statistical analysis of monitoring data to determine whether variability in a data set is the result of project activities or natural variation,
- Maintaining weather-related data using on-site weather monitoring data or data collected near project site (such as from local airports),
- Checking instrument calibration before, during and after sampling or sample analysis,
- Thoroughly training staff,
- Conducting laboratory quality assurance checks using third party reference materials, and/or blind, and/or replicate sample checks, and
- Developing a systematic review process of data that can include sample-specific data quality checks (i.e., cation/anion balance for aqueous samples).

## 9.2 SAMPLING METHODS

Logging, geophysical monitoring, and pressure/temperature monitoring do not apply to this section and are omitted. The field sampling protocols defined herein are in line with the EPA 540-R-20-005, Sampler's Guide: Contract Laboratory Program Guidance for Field Samplers (Attachment D.I-1).

### 9.2.1 Sampling Standard Operating Procedures (SOPs)

Groundwater samples will be collected primarily using a low-flow sampling method consistent with (ASTM International, 2005c). If a flow-through cell is not used, field parameters will be measured in grab samples. Groundwater wells will be purged to ensure samples are representative of formation water quality. Static water levels in each well will be determined using an electronic water level indicator before any purging or sampling activities begin. Dedicated pumps (e.g., bladder pumps) will be installed in each monitoring well to minimize potential cross contamination between wells. Groundwater pH, temperature, specific conductance, and dissolved oxygen will be monitored in the field using portable probes and a flow-through cell consistent with standard methods (APHA, 2005) given sufficient flow rates and volumes. Field chemistry probes will be calibrated at the beginning of each sampling day according to equipment manufacturer procedures using standard reference solutions. When a flow-through cell is used, field parameters will be continuously monitored and will be considered stable when three successive measurements made three minutes apart fall within the stabilization parameters.

After field parameters have stabilized, samples will be collected. Samples requiring filtration will be filtered through 0.45 µm flow-through filter cartridges as appropriate and consistent with . Prior to sample collection, filters will be purged with a minimum of 100 mL of well water (or more if required by the filter manufacturer). For alkalinity and total CO<sub>2</sub>



samples, efforts will be made to minimize exposure to the atmosphere during filtration, collection in sample containers, and analysis.

### 9.2.2 In-situ Monitoring

Groundwater chemistry monitoring parameters can be found in Table D.I.6.3-1.

### 9.2.3 Continuous Monitoring

GCS will install and use continuous recording devices to monitor, as required per LAC 43:XVII§3621.A.6.a.i-iv, §3627.A.2.a-b, and §3625.A.2, the CO<sub>2</sub> mass delivered to Project Minerva at the transfer point, the volume and temperature of CO<sub>2</sub> allocated to each well, the pressure at the wellhead, the pressure on the injection tubing, the pressure at the wellhead on the annulus between the tubing and the long string casing, and the annulus fluid volume added. Injection pressure and temperature will be continuously measured at the surface via real-time pressure/temperature (P/T) instruments installed at the primary meter station and at the allocation meters at each wellhead. Pressure will be measured using electronic pressure gauges with analog data transmitters, installed at the primary meter station and at each allocation meter/wellhead. Temperature will be measured similarly using electronic temperature sensors with data transmitters installed in close proximity to the pressure gauges both at the primary meter station and at each allocation station.

### 9.2.4 Sample Homogenization, Composition, Filtration

Mixing of the sample is necessary to create a representative sample media. It is extremely important that solid samples be mixed as thoroughly as possible to ensure that the sample is as representative as possible of the sample location. The mixing technique will depend on the physical characteristics of the solid material (e.g., particle size, moisture content, etc.). The mixing container will be large enough to hold the sample volume and accommodate the procedures without spilling. Both the mixing container (generally a bowl or tray) and the mixing implement will be properly decontaminated before use. Samples will be homogenized according to procedures listed in the project-specific sampling and analysis plan (EPA 540-R-20-005 (Attachment D.I-1) or equivalent).

### 9.2.5 Sample Containers and Volumes

The analytical protocol(s) to be used for sample analysis often requires the use of a particular type of sample container. The type of container also may depend on the sample matrix and analysis. The use of borosilicate glass containers, which are inert to most materials, is recommended. Conventional polyethylene is recommended when sampling for metals because of the lower cost and absorption rate of metal ions. Using the wrong container may result in breakage, gathering of an insufficient volume needed to perform sample analysis, or interference of the container material with the analysis. Therefore, the correct sample containers for each sampling event will be identified using Sample Container Type Specifications as outlined in EPA 540-R-20-005 (Attachment D.I-1) or equivalent.



### 9.2.6 Sample Preservation

Degradation of some contaminants may occur naturally. Some water samples will require chemical preservation for certain analytes before the samples are shipped to the laboratory. Any visible reaction between the sample and added chemical preservative will be noted in the field record. Water samples will be preserved and immediately cooled to 4°C ( $\pm 2^\circ\text{C}$ ) upon collection, and samples should remain cooled until the time of analysis (the water samples will not be frozen). Preservation techniques will align with EPA 540-R-20-005 (Attachment D.I-1) or equivalent.

### 9.2.7 Cleaning/Decontamination of Sampling Equipment

Dedicated pumps will be installed in each ACZ monitoring well to minimize potential cross contamination between wells. GCS is still in the process of determining the types of pumps that will be utilized. These pumps will remain in each well throughout the project period except for maintenance. Prior to installation, the pumps will be cleaned on the outside with a non-phosphate detergent. Pumps will be rinsed a minimum of three times with deionized water and a minimum of 1 liter of deionized water will be pumped through pump and sample tubing. Individual clean pumps and tubing will be placed in plastic garbage bags for transport to the field for installation. All field glassware (pipets, beakers, filter holders, etc.) are cleaned with tap water to remove any loose dirt, washed in a dilute nitric acid solution, and rinsed three times with deionized water before use.

### 9.2.8 Support Facilities

For sampling of groundwater, the following are required: air compressor, vacuum pump, generator, multi-electrode water quality sonde, analytical meters (pH, specific conductance, etc.). Field activities are usually completed in field vehicles and portable laboratory trailers located on site. Field gauges will be removed from the injection well and verification well utilizing existing standard industry tools and equipment. Deployment and retrieval of verification well gauges will be done using procedures and equipment recommended by the vendor, subcontractor, or is standard per industry practice. Sampling tubing, connectors and valves required to sample the CO<sub>2</sub> gas stream will be supplied by the analytical lab providing the sampling containers.

### 9.2.9 Corrective Action, Personnel, and Documentation

Field staff will be responsible for properly testing equipment and performing corrective actions on broken or malfunctioning field equipment. If corrective action cannot be taken in the field, then the equipment will be returned to the manufacturer for repair or replaced. Significant corrective actions affecting analytical results will be documented in field notes. Sample holding times will be consistent with those described in (APHA, 2005; ASTM International, 2005a; Wood, 1976). Once the samples are analyzed, the laboratory will be responsible for disposing of the containers and residues properly.



## 9.3 SAMPLING HANDLING AND CUSTODY

### 9.3.1 Maximum Hold Time/Time Before Retrieval

Daily shipment of samples to laboratories will be the preferred method whenever possible. If samples cannot be shipped daily, they will be properly preserved and maintained to meet ASTM Method D6517-00 (or equivalent) temperatures, holding times, and custody requirements (ASTM International, 2005a; ASTM International, 2005b; ASTM International, 2005c). The technical holding times are the maximum time allowed between a sample collection and the completion of the sample extraction and/or analysis. In contrast, contractual holding times are the maximum lengths of time that a laboratory can hold the sample prior to extraction and/or analysis. Samples will be processed, packaged, and shipped to the contracted laboratory, following standard sample handling and chain-of-custody guidance (EPA 540-R-20-005 (Attachment D.I-1), or equivalent). Once the samples are analyzed, the laboratory will be responsible for disposing of the containers and residues properly.

### 9.3.2 Sample Transportation

Samples will be shipped to a LELAP accredited laboratory with chain-of-custody documentation. Shipping and handling shall be in accordance with laboratory recommendations for the specific analytical method. The contracted laboratory will employ EPA-approved or other industry standard analytical methods and have a documented QA/QC program in place.

### 9.3.3 Sampling Documentation

Collected samples will be properly documented for analysis in order to uniquely identify each sample and ensure adequate chain-of-custody procedures. If sampling on privately owned property, the property owner will be provided with a receipt for samples collected and removed from that owner's property. These types of documentation help ensure proper sample identification and provide additional chain-of-custody records.

### 9.3.4 Sample Identification

The use of a sample label, sample tag, or field operations will record documenting information such as daily activities, equipment and materials used, personnel involved, site security, etc. may also be utilized.

### 9.3.5 Sample Chain-of-Custody Record

For groundwater samples, chain-of-custody will be documented using a standardized form. A typical form is shown in Figure D.I.9.3-1, and it or a similar form will be used to document all groundwater sampling. Copies of the form will be provided to the person/lab receiving the samples as well as the person/lab transferring the samples. These forms will be retained and archived to allow simplified tracking of sample status. The chain-of-custody form and record keeping are the responsibility of groundwater sampling personnel.



## 9.4 ANALYTICAL METHODS

### 9.4.1 Analytical SOPs

Analytical SOPs are referenced in Table D.I.9.4-1. Other laboratory specific SOPs utilized by the laboratory will be determined after a contract laboratory has been selected. Upon request GCS will provide the agency with all laboratory SOPs developed for the specific parameter using the appropriate standard method. Each laboratory technician conducting the analysis on the samples will be trained in the standard methods for each SOP developed.

### 9.4.2 Equipment/Instrumentation Needed

Equipment and instrumentation are specified in the individual analytical methods referenced in Table D.I.9.4-1.

### 9.4.3 Method Performance Criteria

Non-standard method performance criteria are not anticipated for this project.

### 9.4.4 Analytical Failure

Each laboratory conducting analyses in Table D.I.9.4-1 will be responsible for analytical failure according to their SOPs.

### 9.4.5 Sample Disposal

Each laboratory conducting the analyses will be responsible for appropriate sample disposal.

### 9.4.6 Laboratory Turnaround

Laboratory turnaround will vary by laboratory, but generally turnaround of verified analytical results within one month will be suitable for project needs.

### 9.4.7 Method Validation for Nonstandard Methods

Nonstandard methods are not anticipated for this project. If nonstandard methods are needed or proposed in the future, the C&E will be consulted on additional appropriate actions to be taken.

## 9.5 QUALITY CONTROL

As part of the quality control (QC) process during testing and surveillance, most of the samples collected and the data gathered will be analyzed, processed, validated, or witnessed by third parties independent of the operations staff. For specialized data such as seismicity and DTS, Project Minerva will have additional support from the providers of the selected technologies in quality control, verification of the data, and system calibration.

QC of the sampling and results will follow the protocols established in the standard analytical method used for testing. The operator reserves the right to audit the laboratory procedures and protocols to validate that the methods are being followed and the results are accurate.



## 9.5.1 QC activities

### 9.5.1.1 Blanks

Trip blanks for QA/QC purposes will be collected and used to validate test results and ensure samples are free of contamination.

### 9.5.1.2 Duplicates

For each water sampling round, a duplicate water sample is collected from a well from a rotating schedule, yet to be determined. Duplicate samples are collected from the same source immediately after the original sample in different sample containers and processed as all other samples. Duplicate samples are used to assess sample heterogeneity and analytical precision.

## 9.5.2 Exceeding Control Limits

If the sample analytical results exceed control limits (i.e., ion balances > ±10%), further examination of the analytical results will be done by evaluating the ratio of the measured total dissolved solids (TDS) to the calculated TDS (i.e., mass balance) per American Public Health Association (APHA) method (or equivalent) (APHA, 2005). The method indicates which ion analyses should be considered suspect based on the mass balance ratio. Suspect ion analyses are then reviewed in the context of historical data and interlaboratory results, if available. Suspect ion analyses are then brought to the attention of the analytical laboratory for confirmation and/or reanalysis. The ion balance is recalculated, and if the error is still not resolved, suspect data are identified and may be given less importance in data interpretations.

## 9.5.3 Calculating Applicable QC Statistics

### 9.5.3.1 Charge Balance

Titration methods will be based on standard protocols from Standard Methods 2320B-2011. Laboratories shall have standard operating procedures for the analytical methods performed.

The analytical results are evaluated to determine correctness of analyses based on anion-cation charge balance calculation. Because all potable waters are electrically neutral, the chemical analyses should yield equally negative and positive ionic activity. The anion-cation charge balance will be calculated using the formula:

$$\% \text{ difference} = 100 \frac{\sum \text{cations} - \sum \text{anions}}{\sum \text{cations} + \sum \text{anions}} \quad \text{Equation -1}$$

where the sums of the ions are represented in milliequivalents (meq) per liter and the criteria for acceptable charge balance is ±10%.

### 9.5.3.2 Mass Balance

The ratio of the measured TDS to the calculated TDS will be calculated in instances where the charge balance acceptance criteria are exceeded using the formula:

$$1.0 < \frac{\text{measured TDS}}{\text{calculated TDS}} < 1.2 \quad \text{Equation 16.4.2-1}$$



Equation 3.4.2-2 is an example equation, where the anticipated values are between 1.0 and 1.2.

### 9.5.3.3 Outliers

Outliers may result from random variation or may indicate something scientifically interesting. In any event, we typically do not want to simply delete the outlying observation. However, if the data contains significant outliers, we may need to consider the use of robust statistical techniques, which may include: (1) Probability Plots, (2) Box Plots, (3) Dixon's test, and (4) Rosner's test. The EPA-1989 outlier test may also be used as another screening tool to identify potential outliers.

## 9.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Logging-tool equipment will be maintained as per the wireline industry best practices.

For groundwater sampling, field equipment will be maintained, factory serviced, and factory calibrated per manufacturer's recommendations. Spare parts that may be needed during sampling will be included in supplies on-hand during field sampling.

For all laboratory equipment, testing, inspection and maintenance will be the responsibility of the analytical laboratory per standard practice, method-specific protocol, or LELAP requirement.

## 9.7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

All field equipment will be visually inspected and tested prior to use. Spare instruments, batteries, etc., will be stored in the field support trailer.

Pressure gauges used to conduct fall-off tests will be calibrated in accordance with the manufacturers' recommendations. In lieu of removing the injection tubing to recalibrate the downhole pressure gauges, their accuracy will be demonstrated by comparison with a second pressure gauge with current certified calibration, which will be lowered into the well to the same depth as the permanent downhole gauge. Calibration curves for the downhole gauge, based on annual calibration checks using the second calibration gauge, can be used for the fall-off test. These calibration curves (showing all historic pressure deviations) will accompany the fall-off test data.

During laboratory analysis, the calibration, testing, maintenance, and inspection of laboratory equipment shall be the responsibility of the contracted analytical laboratory. Equipment calibration shall be in accordance with the approved method-specific protocols for the analytical method and the laboratory's QA program. The laboratory QA program shall be reviewed and approved prior to the contract award.

### 9.7.1 Calibration and Frequency of Calibration

Equipment used for field sampling will be calibrated, serviced, and maintained according to the manufacturer's recommendations.



### 9.7.2 Calibration Methodology

Equipment used for field sampling will be calibrated, serviced, and maintained according to the manufacturer's recommendations.

### 9.7.3 Calibration Resolution and Documentation

Equipment used for field sampling will be calibrated, serviced, and maintained according to the manufacturer's recommendations.

## 9.8 INSPECTION/ACCEPTANCE FOR SUPPLIES AND CONSUMABLES

Both field operations and laboratory operations need supplies and consumables. The focus of this section is the management of laboratory and field sampling supplies and consumables. For information on the actual field/lab supplies and consumables needed for any specific method, see the reference method in 40 CFR Part 50, the general guidance methods and technical assistance documents on AMTIC and the manufacturer's operations manuals. From this information, monitoring organizations, as part of the QASP requirements, will develop specific SOPs for its monitoring and analytical methods. One section of the SOPs requires a listing of the acceptable supplies and consumables for the method. Pollutant parameters are measured using electronic (e.g., continuous emission monitors, FTIRs, etc.), wet chemical techniques, or physical methods. Chemical analysis always involves the use of consumable supplies that must be replaced on a schedule consistent with their stability and with the rate at which samples are taken. Currently used physical methods require adequate supplies of chemicals for operation for three months so that the supplier can comply with the delivery schedules. In some cases, analytical reagents for specific air contaminants deteriorate rapidly and need protective storage. The following information may be helpful when considering the use of these consumable items. Much of the information presented below is derived from the document *Quality Assurance Principles for Analytical Laboratories* from (Garfield, Klesta, & Hirsch, 2000).

## 9.9 NONDIRECT MEASUREMENTS

### 9.9.1 Data Sources

For time lapse seismic surveys, repeatability is paramount for accurate differential comparison. Therefore, to ensure survey quality, the locations for the shots and acquisition methodology of sequential surveys will be consistent. Once these surveys are conducted, they will be compared to a baseline survey to track and monitor plume development.

### 9.9.2 Relevance to Project

Time lapse seismic surveys will be used to track changes in the CO<sub>2</sub> plume in the subsurface. Processing and comparing subsequent surveys to a baseline will allow project managers to monitor plume growth, as well as to ensure that the plume does not move outside of the intended storage reservoir. Numerical modeling will be used to predict the CO<sub>2</sub> plume growth and migration over time by combining the processed seismic data with the existing geologic model. In-zone pressure monitoring data will be used in numerical modeling to predict plume and pressure front behavior and confirm the plume stage within



the AoR.

### 9.9.3 Acceptance Criteria

Following standard industry practices will ensure that the gathered seismic data will be used for accurate modeling and monitoring. Similar ground conditions shot points located within tolerable limits, functional geophones, and similar seismic input signal will be used from survey to survey to ensure repeatability. When processing seismic data, several QA checks will be done in accordance with industry standards including reformatting to structured files, geometry application, amplitude compensation, predictive deconvolution, elevation statics correction, root mean square (RMS) amplitude gain, velocity analysis every 1.24 miles (2 km), normal moveout (NMO) correction application using picked velocities, common midpoint (CMP) stacking, random noise attenuation, and instantaneous gain.

### 9.9.4 Resources/Facilities Needed

GCS will subcontract all necessary resources and facilities for the seismic monitoring, in-zone pressure monitoring, and groundwater sampling.

### 9.9.5 Validity Limits and Operating Conditions

For seismic surveys and numerical modeling, intraorganizational checks between trained and experienced personnel will ensure that all surveys and numerical modeling are conducted conforming to standard industry practices.

## 9.10 DATA MANAGEMENT

### 9.10.1 Data Management Scheme

GCS will be responsible for ensuring that all electronic and hard copy data, forms and logs are accounted for, properly completed and stored in project files.

Documentation will be sufficient that a third party can reproduce the results from the raw data. This requires that all necessary information will be documented, and that the documents are organized and maintained such that the information may be practically retrieved and made use of.

Documentation will consist of instrument and other digital files, hard copy field log sheets, calibration certificates, laboratory reports, etc. All of these documents will ultimately be stored in electronic form; however, hard copy log sheets will be retained on file. An electronic data package will be compiled containing project documentation sufficient to allow a third party to reproduce the results and organized in such a manner that this may be done without undue effort.

### 9.10.2 Recordkeeping and Tracking Practices

All reports, submittals and notifications will be submitted to both the EPA and the C&E. All records will be retained by GCS throughout the life of the project and for 10 years following site closure. Data on the nature and composition of all injected fluids collected will be retained as well for 10 years after site closure. The records will be delivered to the UIC Program Director after the retention period if required by the director. Monitoring data as



described in the Testing and Monitoring Plan will be retained for 10 years after it is collected. Well plugging reports, post-injection site care data and the site closure report itself will be retained for 10 years following site closure.

### 9.10.3 Data Handling Equipment/Procedures

GCS will be responsible for ensuring that all electronic and hard copy data, forms and logs are accounted for, properly completed and stored in project files. All data and project records will be stored electronically on secure servers and will have routine backups. Reporting will comply with Class VI UIC requirements.

### 9.10.4 Responsibility

GCS will be responsible for ensuring that all electronic and hard copy data, forms and logs are accounted for, properly completed and stored in project files. All data and project records will be stored electronically on secure servers and will have routine backups. Reporting will comply with Class VI UIC requirements.

### 9.10.5 Data Archival and Retrieval

GCS will be responsible for ensuring that all electronic and hard copy data, forms and logs are accounted for, properly completed and stored in project files. All data and project records will be stored electronically on secure servers and will have routine backups. Reporting will comply with Class VI UIC requirements. All reports, submittals and notifications will be retained by GCS throughout the life of the project and for ten (10) years following site closure. Data on the nature and composition of all injected fluids collected will be retained as well for ten (10) years after site closure. The records will be delivered to the UIC Program Director after the retention period if required by the director. Monitoring data as described in the Testing and Monitoring Plan will be retained for 10 years after it is collected. Well plugging reports, post-injection site care data and the site closure report itself will be retained for ten (10) years following site closure.

### 9.10.6 Hardware and Software Configurations

All GCS and vendor hardware and software configurations will be appropriately interfaced.

### 9.10.7 Checklists and Forms

Checklists and forms will be procured and generated as necessary.

## 10 ASSESSMENT AND OVERSIGHT

### 10.1 ACTIVITIES TO BE CONDUCTED

Assessments of each of the QASP elements:

- CO<sub>2</sub> stream analysis surface sampling
- Continuous recording of operational parameters
- Corrosion monitoring
- External MIT
- ACZ monitoring



- Pressure fall-off testing
- In zone CO<sub>2</sub> plume and pressure from tracking

After completion of sample analysis, results will be reviewed for QC criteria as noted in Section 1.4 (Quality Objectives and Criteria). If the data quality fails to meet criteria set in Section 1.4, samples will be reanalyzed, if still within holding time criteria. If outside of holding time criteria, additional samples may be collected, or sample results may be excluded from data evaluations and interpretations. Evaluation for data consistency will be performed according to procedures described in *Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities* a USEPA Unified Document (USEPA, 2009).

## 10.2 RESPONSIBILITY FOR CONDUCTING ASSESSMENTS

GCS or its designated subcontractors gathering data will be responsible for conducting their internal assessments. All stop work orders will be handled internally within individual organizations.

## 10.3 ASSESSMENT REPORTING

GCS will coordinate reporting of assessments.

## 10.4 CORRECTIVE ACTION

All corrective action affecting only an individual organization's data collection responsibility should be addressed, verified, and documented by the individual project managers and communicated to the other project managers as necessary. Corrective actions affecting multiple organizations should be addressed by all members of the project leadership and communicated to other members on the distribution list for the QASP. Assessments may require integration of information from multiple monitoring sources across organizations (operational, in-zone monitoring, above-zone monitoring) to determine whether correction actions are required and/or the most cost-efficient and effective action to implement. GCS will coordinate multi-organization assessments and corrective actions as warranted.

## 10.5 REPORTS TO MANAGEMENT

### 10.5.1 QA Status Reports

QA status reports should not be needed. If any testing or monitoring techniques are changed, the QASP will be reviewed and updated as appropriate in consultation with C&E. Revised QASPs will be distributed by GCS to the full distribution list at the beginning of this appendix.

## 11 DATA VALIDATION AND USABILITY

### 11.1 DATA REVIEW, VERIFICATION, AND VALIDATION

#### 11.1.1 Criteria for Accepting, Rejecting, or Qualifying Data

As part of the QC process, during testing and surveillance, most of the samples collected and data gathered will be analyzed, processed, validated, or witnessed by third parties



independently, outside of the operator staff.

For specialized data such as seismicity, the project will have additional support from the provider of the selected technologies to perform QC and verification of the data as well as calibration of the systems as needed.

All C&E-reportable data will be formatted appropriately and uploaded to the GSDT at the required frequency.

## **11.2 VERIFICATION AND VALIDATION METHODS**

### **11.2.1 Data Verification and Validation Processes**

The project will establish a standardized program to validate the data and acquisition methods. The program will verify that collected data are reasonable, were processed and analyzed correctly, and are free of errors. Peer reviews or third-party consultant will be used as a QC mechanism to verify the information.

### **11.2.2 Data Verification and Validation Responsibility**

If an error is identified in data under validation, in addition to correcting the error, affected work products and management decisions will be identified, affected users will be notified, and corrective actions will be coordinated to an extent of the error's impact is fully addressed.

### **11.2.3 Issue Resolution Process and Responsibility**

GCS or its designee will overview the data handling, management, and assessment process. Contractors involved in these processes will consult with GCS to determine actions required to resolve issues.

### **11.2.4 Checklist, Forms, and Calculations**

Checklists and forms will be developed specifically to meet permit requirements. These will be detailed as site-specific design advances.

## **11.3 RECONCILIATION WITH USER REQUIREMENTS**

### **11.3.1 Evaluation of Data Uncertainty**

Statistical software will be used to determine data consistency using methods consistent with the C&E Secretary's guidance.

### **11.3.2 Data Limitations Reporting**

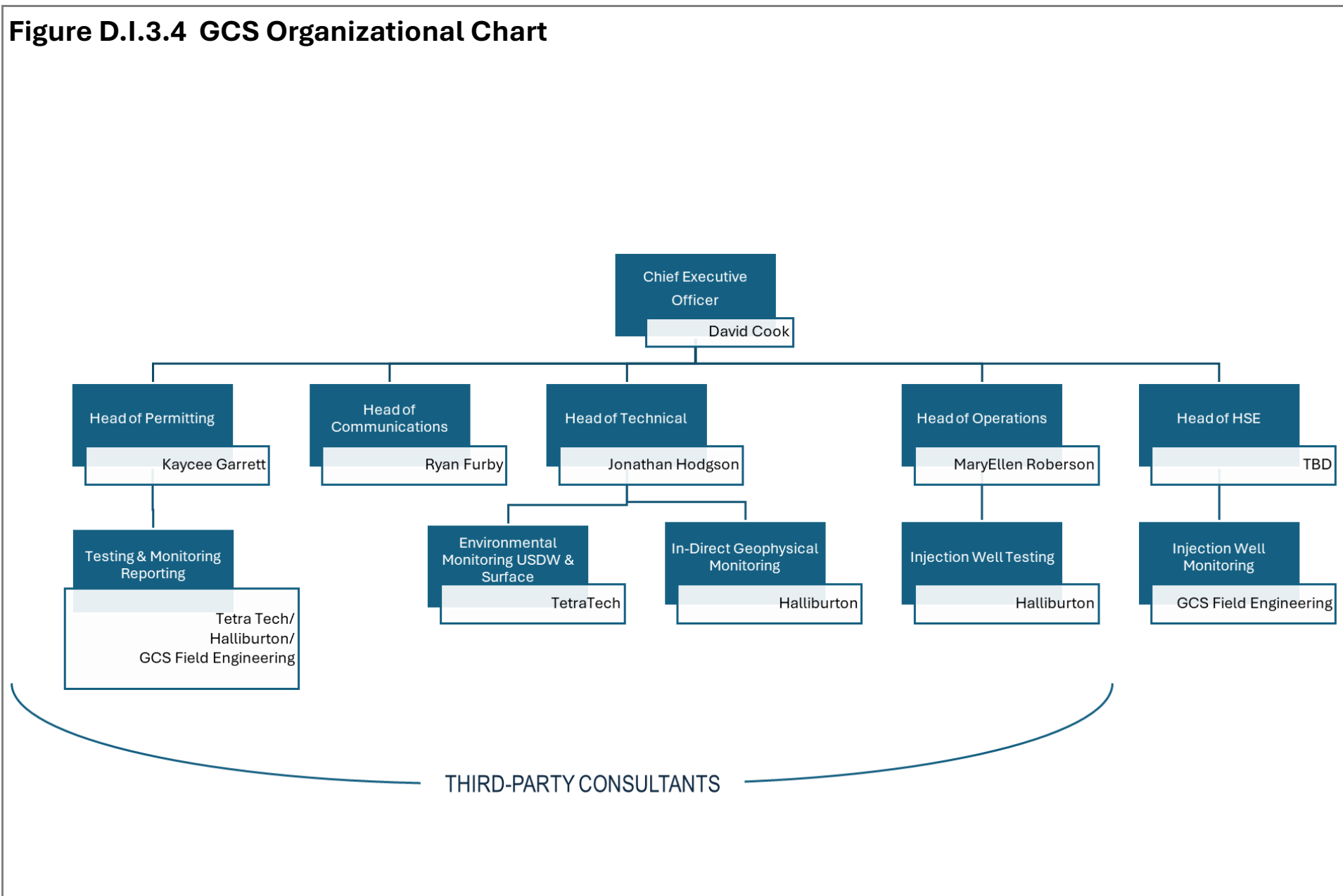
The organization-level project managers will be responsible for ensuring that data developed by their respective organizations is presented with the appropriate data-use limitations. GCS will use the current operating procedure on the use, sharing, and presentation of results and/or data for Project Minerva. This procedure has been developed to ensure quality, internal consistency and facilitate tracking and record keeping of data end users and associated publications.




## 12 REFERENCES

- American Petroleum Institute (2023). *API Recommended Practice 580: Elements of a Risk-Based Inspection Program*, 4<sup>th</sup> Edition. Washington, DC: API Publishing Services
- APHA. (2005). *Standard Methods for the Examination of Water and Wastewater, Volume 21* (Vol. 21). (A. D. Eaton, & M. A. Franson, Eds.) American Public Health Association.
- ASTM International. (2005a). Standard Guide for Field Preservation of Groundwater Samples: Designation: D6517 – 00 (Reapproved 2005). *ASTM Standard Methods*, 5.
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- ASTM International. (2005c). Standard Guide for Purging Methods for Wells Used for Ground-Water Quality Invesetigations: Designation: D 6452 – 99 (Reapproved 2005). *ASTM Standard Methods*, 6.
- Garfield, F. M., Klesta, E., & Hirsch, J. (2000). *Quality Assurance Principles for Analytical Laboratories* (3rd Edition ed.). Gaithersburg: AOAC International; Association of Official Analytical Chemist.
- USEPA. (2009). *Unified Documnet: Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities*. US Environmental Protection Agency.
- Wood, W. W. (1976). Chapter D2: Guidelines for Collection and Field Analysis of Ground-Water Samples for Selected Unstable Constituents. In U. S. Survey, *Techniques of Water-Resources Investigations of the United States Geological Survey* (p. 24). Alexandria: United States Geological Survey.

**Figure D.I.3.4 GCS Organizational Chart**



 <p>Gulf Coast Sequestration, LLC (G1037) 5599 San Felipe, Suite 1450, Houston, TX 77056 (713) 997-3145   www.gcscarbon.com</p>	<b>Minerva South CCS Well Nos. 001 &amp; 002</b>	
	Project: <b>Minerva South</b>	Project ID: <b>R06-LA-0002</b>
	Location: <b>Cameron Parish, LA</b>	LDENR Appl Nos: <b>45031 &amp; 45032</b>



Gulf Coast Sequestration, LLC (G1037)  
5599 San Felipe, Suite 1450, Houston, TX 77056  
(713) 997-3145 | www.gcscarbon.com

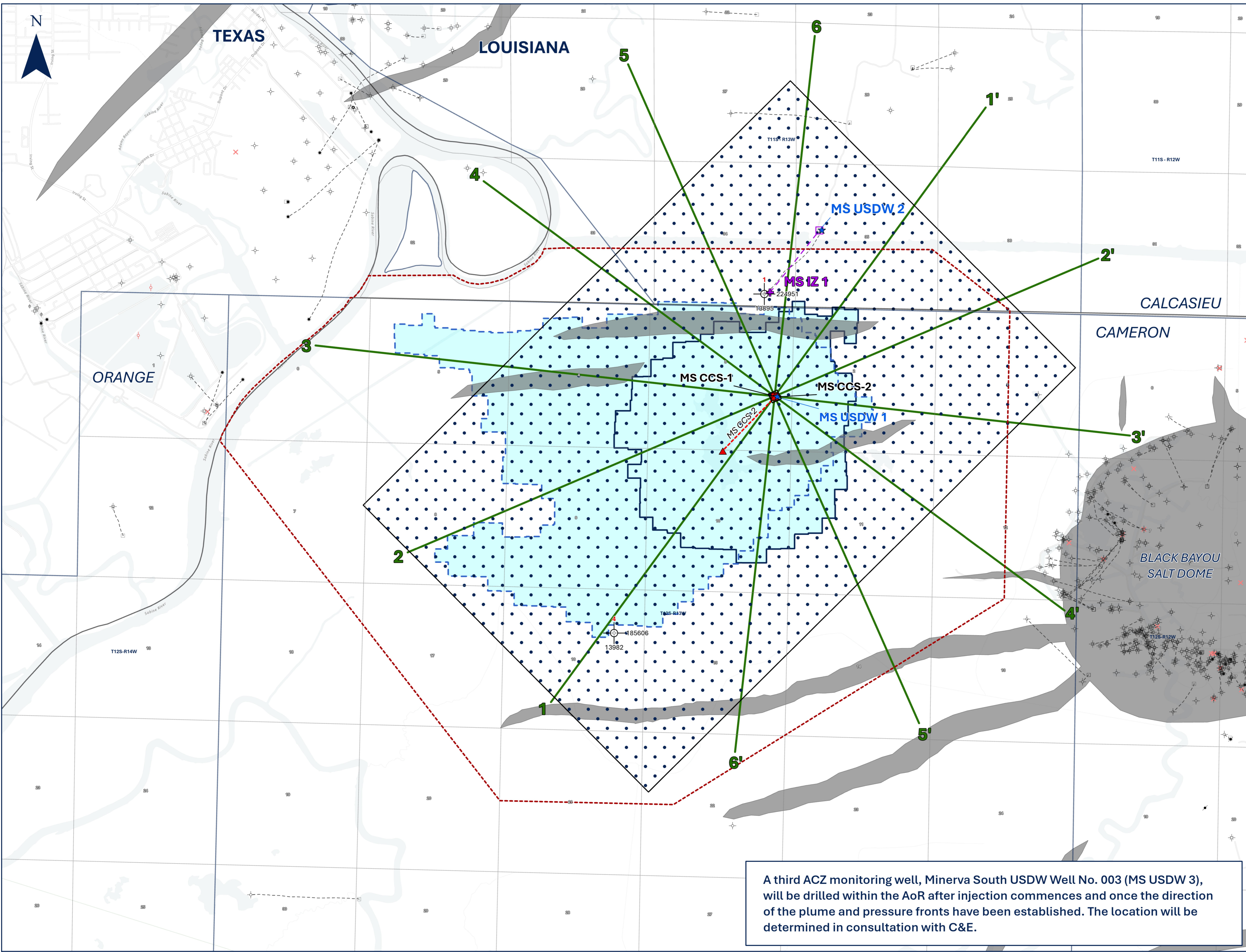
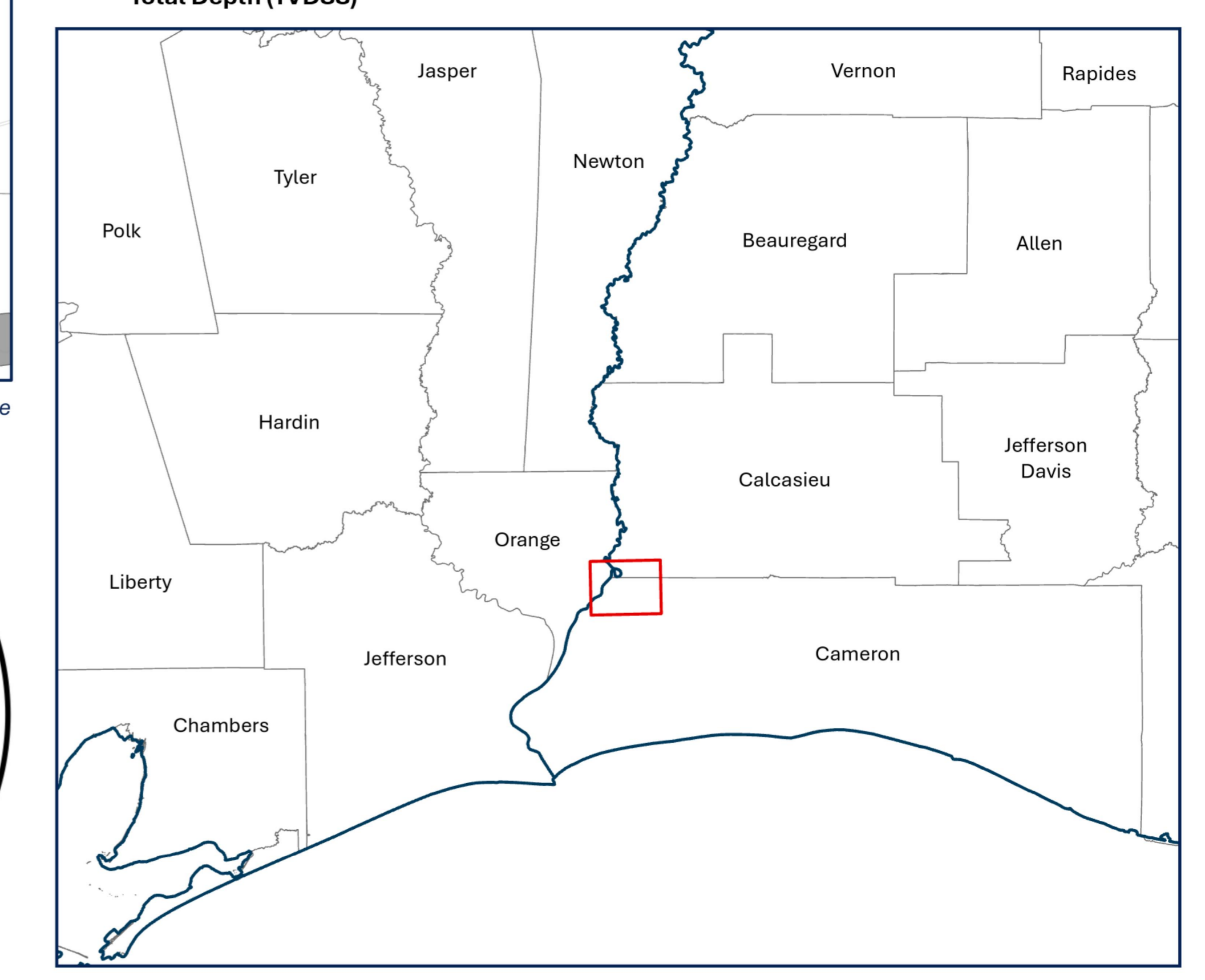
### FIGURE D.I.5.2-1 Monitoring Network for Project Minerva

#### MAP LEGEND

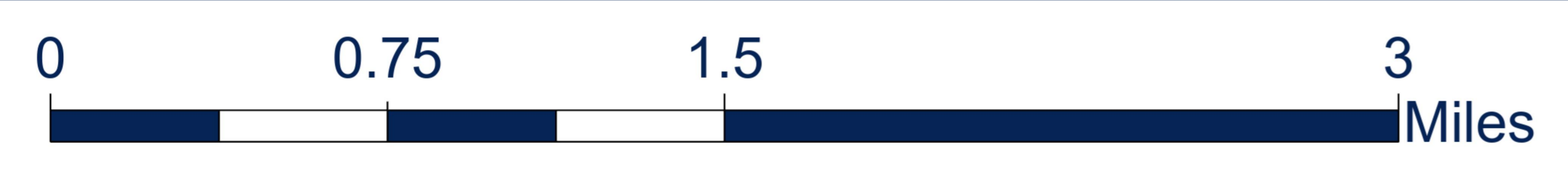
- ▲ Injection Well (Bottom Hole)
  - Injection Well (Surface)
  - - - Injection Well Trajectory (Proposed)
  - Walk-Away VSP / 2D Seismic Lines
  - 3D VSP Sources
  - - - Magnetic Survey Flight Area
  - Delineated Area of Review
  - Max Extent of Pressure Front
  - - - Max Extent of Stable CO2 Plume
  - Top Upper Frio Faults
  - Proposed Well Pad (Not to Scale)
  - Parishes
  - Townships & Ranges
  - Sections
- Monitoring Wells**
  - ⊕ In-Zone Bottom Location
  - In-Zone Surface Location
  - - - In-Zone Well Trajectory
  - ★ USDW Monitoring Well (Vertical)
- Well Status**
  - Other
  - ★ ACTIVE-PRODUCING
  - DRILLED
  - DRY & PLUGGED
  - INACTIVE
  - ★ ORPHAN
  - × PERMIT EXPIRED
  - ⊕ PLUGGED & ABANDONED
  - ⊕ REVERTED
  - ★ SHUT\_IN
  - ⊕ TEMPORARILY ABANDONED
  - ★ UNABLE TO LOCATE
  - Surface Location
  - - - Well Trajectory

#### Well Labels

- Map ID
- Serial Number
- Total Depth (TVDSS)
- Monitoring Well Name



A third ACZ monitoring well, Minerva South USDW Well No. 003 (MS USDW 3), will be drilled within the AoR after injection commences and once the direction of the plume and pressure fronts have been established. The location will be determined in consultation with C&E.



Culture Data Sources: Louisiana Department of Conservation and Energy (LDCE) Strategic Online Natural Resources Information System (SONRIS) and Cameron Parish Tax Assessors Office

MAP DETAILS			
PREPARED BY	R WENCEL	DATE	4/23/2026 3:58 PM
APPROVED BY	K GARRETT	DATE	04/23/2026
MAP SCALE	1:16,000		
PAGE SIZE	A0 (46.81 WX 33.11 H)		

SPATIAL REFERENCE	
PCS	NAD 1927 State Plane Louisiana South (FIPS 1702)
GCS	GCS North American 1927
DATUM	North American 1927
PROJECTION	Lambert Conformal Conic

PROJECT MINERVA DETAILS	
WELLS	Minerva South CCS Well Nos. 001 and 002
LOCATION	Cameron Parish, Louisiana
EPA PROJECT ID	R06-LA-0002
LDENR APPL NOS	45031 & 45032



# Chain of Custody Record

TAT(Check One): Standard 24hr 48hr 72hr Other\_\_\_\_\_   
 (Additional charges may apply for rush TAT. If not specified, standard TAT will apply)

Order ID: \_\_\_\_\_

Client Name: _____ Address: _____ Phone: _____ _____ Email: _____ Contact Name: _____ P.O. Info: _____	Project Name: _____ Address: _____ _____ Regulatory ID (SDWA/Permit #): _____
---	--

Comments:

STL Sample Number	Sample Description / Site ID:	Date Sampled	Time Sampled	Samplers Initials	Test(s) Requested:	Bottle Quantity	See Codes Below				Comments / Field Data:
							Matrix	Sample Type	Bottle Type	Preservative	

Relinquished By:	Count	Date: _____	Temp °C: _____	<b>Sample Conditions</b> Submitted with COC? Y / N Number of containers match number on COC? Y / N All containers in tact? Y / N Tests within holding times? Y / N 40 mL VOA vials free of headspace? Y / N	<b>Matrix Key</b> NPW = Non-Potable Water Solid = Raw Sludge, Dewatered sludge, soil, etc. (reported as mg/kg) PW = Potable Water (not for SDWA compliance) SDWA = Safe Drinking Water Act Potable Sample	<b>Bottle Type Key</b> P = Plastic G = Glass GA = Glass Amber VOA = 40mL G or GA PP = Sterile Polypropylene PS = Sterile Polystyrene HDPE = High Density Polyethylene O = Other
		Time: _____				
Received By:		Date: _____	Temp °C: _____			
		Time: _____	Acceptable: Y / N			
Relinquished By:		Date: _____	Temp °C: _____			
		Time: _____	Acceptable: Y / N			
Received in Lab By:		Date: _____	Temp °C: _____			
		Time: _____	Acceptable: Y / N			

**Table D.I.2-1 QASP Distribution List**

Title	Name	Phone	Email	Address
Chief Executive Officer	David Cook	1 (713) 419-6808	<a href="mailto:dcook@gcscarbon.com">dcook@gcscarbon.com</a>	5599 San Felipe Dr., Suite 1450 Houston, Texas 77056
Field Manager	David Richard			2417 Shell Beach Dr. Lake Charles, LA 70601
Site Manager	TBD	TBD	TBD	2417 Shell Beach Dr. Lake Charles, LA 70601
24-7 Control Room Operations	TBD	TBD	TBD	5599 San Felipe Dr., Suite 1450 Houston, Texas 77056
Safety Manager	MaryEllen Roberson	1 (713) 997-3296	<a href="mailto:mroberson@gcscarbon.com">mroberson@gcscarbon.com</a>	5599 San Felipe Dr., Suite 1450 Houston, Texas 77056
Audit/Risk Manager	TBD	TBD	TBD	5599 San Felipe Dr., Suite 1450 Houston, Texas 77056

**Table D.I.5.1-1 Summary of Testing and Monitoring**

Activity	Location(s)	Method	Analytical Technique	Lab/Custody	Purpose
<b>CO<sub>2</sub> STREAM MONITORING</b>					
<b>Carbon Dioxide Stream Analysis</b>	Comingled inlet pipework to Minerva facility	Sampling Port	Standard gas analysis at lab	Provided by lab	Monitor injectate for accounting
<b>INJECTION AND INTEGRITY MONITORING</b>					
<b>Injection Pressure</b>	Distributed meters at MS CCS 1 & 2	Electronic volumetric flow meter with P/T measurement	Direct measurement	NA	Pressure of CO <sub>2</sub> stream
<b>Injection Rate</b>	Distributed meters at MS CCS 1 & 2	Electronic volumetric flow meter with P/T measurement	Direct measurement	N/A	Rate of injection
<b>Injection Volume/Mass</b>	Distributed meters at MS CCS 1 & 2	Electronic volumetric flow meter with pressure & temperature (P/T) measurement	Volume measurements	NA	Volume of CO <sub>2</sub> distributed to each well; calculation of mass
<b>Annular Pressure/Temperature</b>	Downhole gauge with transmitter at each wellhead	Electronic P/T gauge in the annulus	Direct measurement	NA	Pressure maintenance in tubing-casing annulus
<b>Annular Volume</b>	Downhole gauge with transmitter at each wellhead	Electronic P/T gauge in the annulus	Calculated measurement	NA	Pressure maintenance in tubing-casing annulus
<b>Downhole Pressure/Temperature</b>	Downhole gauge with transmitter at each wellhead of MS CCS 1 & 2	Downhole P/T gauge run on tubing	Direct measurement	NA	P/T downhole near injection zone
<b>Wellhead Pressure</b>	Pressure gauge on the tubing and annulus of MS CCS 1 & 2	Surface pressure gauge at the wellhead	Direct measurement	NA	Wellhead pressure safety
<b>External Mechanical Integrity</b>	MS CCS 1 & 2	DTS fiber optic cables installed in the wellbore annulus	Direct Continuous monitoring	NA	Detect fluid or pressure migration from the injection zone and leakage in casing
<b>Wireline Logging</b>	Conducted at each injection well	Injection profile log	Provided by vendor	NA	Demonstrate External Mechanical Integrity
<b>Corrosion Monitoring</b>	Corrosion Loop	Non-Destructive Testing: Ultrasonic and Radiography equipment	ASTM G1-03	NA	Monitor system integrity
<b>SUBSURFACE MONITORING</b>					
<b>Above Confining Zone Monitoring</b>	MS USDW 1 & 2	Water sampling	Laboratory analysis	Provided by lab	Document groundwater chemistry
	MS CCS 1 & 2	DTS fiber optic cables installed along the long-string	Direct measurement	NA	Monitor for fluid migration from the injection zone
<b>Pressure Fall-Off Testing</b>	MS CCS 1 & 2	EPA Region 6 UIC Pressure Falloff Testing Guideline-Third Revision (August 8, 2002)	Direct Measurement	NA	Assess injectivity
<b>Pressure Front Tracking (Direct)</b>	MS CCS 1 & 2	Downhole P/T gauge run on tubing	Direct measurement	NA	Monitor pressure influence from injection
<b>CO<sub>2</sub> Plume Tracking (Indirect)</b>	MS CCS 1	Walk Away VSP	VSP	NA	Defining the edge of the plume and ensuring it remains within the permitted AoR.
	MS CCS 1	Vertical seismic profile (VSP)	VSP	NA	
	MS CCS 1	2D Seismic Lines	2D Seismic	NA	
	MS IZ 1	PN logs	Wireline logging	NA	Monitor carbon dioxide saturation

**Table D.I.6-1 Instrumentation Summary**

Instrument Type	Monitoring Location	Monitoring Target	Data Collection Location(s)	Explanation
<b>CO<sub>2</sub> STREAM MONITORING</b>				
Sampling port	CO <sub>2</sub> Stream	Monitor injectate for accounting	Collection at sampling port; analysis via lab	Identify unacceptable impurities, CO <sub>2</sub> accounting
<b>INJECTION AND INTEGRITY MONITORING</b>				
Electronic volumetric flow meter with pressure & temperature (P/T) measurement	MS CCS 1 & 2	Monitor injectate for each well	Computerized control system	Injection volume, rate, and pressure (with mass and density calculations) for surveillance and input to models
Wellhead pressure gauge on tubing	MS CCS 1 & 2	Pressure in injection well tubing	Manual reading of gauge	Safety and compliance
Wellhead pressure gauge on annulus	MS CCS 1 & 2	Pressure in annulus	Manual reading of gauge	Safety and compliance
Annulus pressure & temperature gauge	MS CCS 1 & 2	Pressure and temperature in tubing-casing annulus	Computerized control system	Monitoring, safety, and compliance
Downhole pressure & temperature gauge	MS CCS 1 & 2	Pressure and temperature in injection interval	Computerized control system	Monitoring, safety, and compliance
DTS fiber optic cable	MS CCS 1 & 2	Surface to Total Depth	Computerized control system	Well integrity
Ultrasonic and Radiography equipment	MS CCS 1 & 2	Corrosion of materials of construction	Loop	Safety and compliance, Well Integrity
<b>SUBSURFACE MONITORING</b>				
Dedicated pump & groundwater level monitoring system	MS USDW 1 & 2	Evangeline Aquifer	Environmental data storage	Installed system or relocated to each of the wells
Downhole pressure & temperature gauge	MS CCS 1	Injection Zone – Pressure	Computerized control system	Track lateral pressure front
Pulse Neutron logging	MS IZ 1	Injection Zone - Plume	Wireline Log	Monitor carbon dioxide saturation within the injection zone
Downhole Geophones or Accelerators	MS IZ 1	Surface to Total Depth	Microseismic Monitoring	Detect seismic events associated with pressure influence along faults
DAS fiber optic cable	MS CCS 1	Surface to Total Depth	Dedicated server, designed as part of VSP array, support microseismic monitoring	CO <sub>2</sub> Plume Tracking, Microseismic Monitoring
Walk-Away VSP	MS CCS 1	Injection Zone – Plume	DAS in MS CCS 1 Sources along seismic lines in star pattern	Define the edge of the plume and ensure it remains within the permitted AoR.
3D DAS VSP	MS CCS 1	Injection Zone - Plume	DAS in MS CCS 1 Sources along 500 x 500 foot Grid	Define the edge of the plume and ensure it remains within the permitted AoR.
2D Seismic Lines	Surface	Injection Zone - Plume	Sources and receivers along seismic lines in star pattern	Define the edge of the plume and ensure it remains within the permitted AoR.


**Table D.I.6.1-1 Summary of Analytical Parameters for CO<sub>2</sub> Stream**

Parameter	Analytical Methods <sup>1</sup>
Carbon Dioxide (CO <sub>2</sub> )	ISBT <sup>2</sup> 2.0 Caustic absorption Zahm-Nagel ALI method SAM 4.1 subtraction method (GC/DID) GC/TCD
Water (H <sub>2</sub> O)	ISBT 3.0
Oxygen (O <sub>2</sub> )	ISBT 4.0 (GC/DID) GC/TCD
Nitrogen (N <sub>2</sub> )	ISBT 4.0 (GC/DID) GC/TCD
Hydrogen Sulfide (H <sub>2</sub> S)	ISBT 14.0 (GC/SCD)
Argon (Ar)	ISBT 4.0 (GC/DID) GC/TCD
Sulfur dioxide (SO <sub>2</sub> )	ISBT 14.0 (GC/SCD)
Methane (CH <sub>4</sub> )	ISBT 10.1 (GC/FID)
Total hydrocarbons (C <sub>2</sub> H <sub>6</sub> , C <sub>3</sub> H <sub>8</sub> +) )	ISBT 10.1 (GC/FID)
Hydrogen (H <sub>2</sub> )	ISBT 4.0 (GC/DID) GC/TCD
Carbon Monoxide (CO)	ISBT 5.0 (Colorimetric) ISBT 4.0(GC/DID)
Nitrogen Oxides (any (NO <sub>x</sub> ))	ISBT 7.0 Colorimetric

**Note**

<sup>1</sup> An equivalent method may be employed with the prior approval of the Commissioner

<sup>2</sup> International Society of Beverage Technologists (ISBT) Carbon Dioxide Guidelines MBAA TQ vol. 39, no. 1, 2002, pp. 32-35 as cited in ISO/TR 27921:2020(en). Carbon dioxide capture, transportation, and geological storage—Cross Cutting Issues—CO<sub>2</sub> stream composition



**Table D.I.6.2-1 Summary of Operational Parameters Measured with Continuous Recording Devices**

Parameter	Device(s)	Location
Annular Pressure and Temperature	Electronic pressure & temperature (P/T) gauge in the annulus with transmitter at the surface	Downhole gauge with transmitter at the wellhead
Annular Fluid Volume	Fluid level gauge	Annular Fluid Reservoir
	Electronic P/T gauge in the annulus with	Downhole gauge with transmitter at the wellhead
Injection Pressure	Electronic volumetric flow meter with pressure measurement	Meter with transmitter at wellhead
Injection Rate	Electronic volumetric flow meter with rate measurement	Meter with transmitter at wellhead
Injection Volume/Mass	Electronic volumetric flow meter with P/T measurement	Meter with transmitter at wellhead
Downhole Pressure	Downhole P/T gauge run on tubing	Downhole gauge with transmitter at the wellhead
Downhole Temperature	Downhole P/T gauge run on tubing	Downhole gauge with transmitter at the wellhead



## Table D.I.6.2-2 Continuous Recording Device Specifications

Parameter	Value
<b>VOLUMETRIC FLOW METER</b>	
Calibrated working flow rate range	1 - 33 ft/s (0.3 -10 m/s)
Initial flow rate accuracy	< ± 0.15% of measured volume
Flow rate resolution	0.01 m/s
Flow rate zero stability	< 0.003 ft/s (0.0009 m/s)
<b>DOWNHOLE PRESSURE AND TEMPERATURE GUAGE (Reservoir &amp; Annulus)</b>	
<b>Calibrated working pressure range</b>	200 - 10,000 psi
Pressure accuracy	±0.015% 1.5 psi (at full scale)
Pressure resolution	0.0001
Pressure drift stability	±0.02% full scale, 2.0 psi/year
<b>Calibrated working temperature range</b>	77 to 302°F (25 to 302°C)
Temperature accuracy	0.27°F (0.15°C)
Temperature resolution	0.0001°F
Temperature drift stability	0.018°F (<0.01°C) per year
Temperature rating	-99.4 to 302°F (-73 to 302°C)
<b>Instrument calibration frequency</b>	Annual
<b>WELLHEAD PRESSURE GUAGE (Tubing &amp; Annulus)</b>	
<b>Calibrated working pressure range</b>	10-20,000 psi
Pressure accuracy	0.05% full scale accuracy
Pressure resolution	0.001
Pressure drift stability	Limit of 5% full scale for pressure stability


**Table D.I.6.3-1 Groundwater Analysis Parameters and the Analytical Methods**

Parameters	Analytical Methods <sup>1</sup>
<b>FIELD MEASUREMENTS</b>	
Dissolved Oxygen (field)	APHA 2005
Specific Conductance (field)	APHA 2510
Temperature (field)	Thermocouple
Water Density (field)	Oscillating body method
Static Water Level	Sounder
pH (field)	EPA 150.1
<b>LABORATORY ANALYSIS</b>	
Cations (Al, Ba, Mn, As, Cd, Cr, Cu, Pb, Sb, Se, Zn, Tl)	SW846 6010/6020
Cations (Ca, Fe, K, Mg, Na, Si)	SW846 6010/6020
Anions (Br, Cl, F, NO <sub>3</sub> , SO <sub>4</sub> )	SM 300
Total Dissolved Inorganic Carbon	Coulometric titration, ASTM D 1067
Dissolved CH <sub>4</sub> (Methane)	SM 6211 B or 6211 C
Hydrogen Sulfide (H <sub>2</sub> S)	ISBT 14.0 (GC/SCD)
Total Dissolved Solids (TDS)	Gravimetry; Method 2540 C
Oxygen, Argon, and Hydrogen	ISBT 4.0 (GC/DID) GC/TCD
Alkalinity	Method 2320B

**Notes:**
<sup>1</sup> Or similar method

- The listed analytical methods may be revised based on input from the laboratories selected to do the work, and field instruments used during a given event.
- Field duplicates to be collected at rate of 10% (1 for every 10 samples) or 1 per event.
- MS/MSDs to be collected at a rate of 5% (1 for every 20 samples) or 1 per event.
- Field blanks and equipment blanks will be collected as per Section 6.3 of the QASP, Appendix I.



## Table D.I.9.4-1 Summary of Analytical Standard Operating Procedures (SOPs)

Parameters	Analytical Methods <sup>(1)</sup>	Detection Limit/ Range	Typical Precisions	QC Requirements
<b>Cations</b> Al, Ba, Mn, As, Cd, Cr, Cu, Pb, Sb Se, & Tl	ICP-MS, EPA Method 6020	0.001 to 0.1 mg/L (analyte, dilution and matrix dependent)	±15%	Daily calibration; blanks, duplicates and matrix spikes at 10% or greater frequency
<b>Cations</b> Ca, Fe, K, Mg, Na, & Si	ICP-OES, EPA Method 6010B	0.005 to 0.5 mg/L (analyte, dilution and matrix dependent)	±15%	Daily calibration; blanks, duplicates and matrix spikes at 10% or greater frequency
<b>Anions</b> Br, Cl, F, NO <sub>3</sub> , & SO <sub>4</sub>	Ion Chromatography, EPA Method 300.0	0.02 to 0.13 mg/L (analyte, dilution and matrix dependent)	±15%	Daily calibration; blanks and duplicates at 10% or greater frequency
<b>Total Dissolved Inorganic CO<sub>2</sub></b>	Coulometric titration, ASTM D 1067	25 mg/L	±15%	Duplicate measurement; standards at 10% or greater frequency
<b>TDS</b>	Gravimetry, APHA 2540C	12 mg/L	±10%	Balance calibration, duplicate analysis
<b>Water Density</b> (field)	Oscillating body method	0.0000 to 2.0000	±0.0002 g/mL	Duplicate measurements
<b>Alkalinity</b>	APHA 2320B	4 mg/L	±3 mg/L	Duplicate analysis
<b>pH</b> (field)	EPA 150.1	2 to 12 pH units	±0.2 pH unit	User calibration per manufacturer recommendation
<b>Specific conductance</b> (field)	APHA 2510	0 to 200 mS/cm	±1% of reading	User calibration per manufacturer recommendation
<b>Temperature</b> (field)	Thermocouple	-5 to 50°C	±0.2°C	Factory calibration
<b>Mercury</b>	ASTM D5954 [18]	0.5 ng/m <sup>3</sup>	±5%	Adherence to reagent water spec, contamination control
<b>Dissolved Hydrocarbon</b> CH <sub>4</sub> , C <sub>2</sub> H <sub>6</sub>	ASTM D8028	Methane: 10 µg/L to 11,000 µg/l Ethan: 10 µg/L to 5,000 µg/L	11 to 23%	Establish valid calibration, samples collected under nominal ambient pressure
<b>Oxygen, Argon, and Hydrogen</b>	ISBT 4.0 (GC/DID) GC/TCD	1–30 µL/L (ppm v/v)	±10% of reading	Daily standard w/in 10% of calibration; secondary standard after calibration
<b>Hydrogen Sulfide (H<sub>2</sub>S)</b>	ISBT 14.0 (GC/SCD)	0.01–0.1 µL/L (ppm v/v)	5–10% of reading	Daily standard w/in 10% of calibration; secondary standard after calibration

### Notes

- 1 An equivalent method may be employed with the prior approval of the Commissioner.
- 2 Gas evolution technique by Atekwana and Krishnamurthy (1998), with modifications made by Hackley et al. (2007)



# Sampler's Guide



Contract  
Laboratory  
Program Guidance  
for Field Samplers



Office of Superfund Remediation and Technology Innovation (OSRTI)  
United States Environmental Protection Agency (EPA)  
Washington, DC 20460

OLEM 9240.0-51  
USEPA 540-R-20-005  
November 2020

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**LIST OF ACRONYMS**


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<b>ARO</b>	Aroclor
<b>ASB</b>	Analytical Services Branch
<b>BTEX</b>	Benzene, Toluene, Ethylbenzene, and Xylene
<b>CBC</b>	Chlorinated Biphenyl Congener
<b>CDD</b>	Chlorinated Dibenzo- <i>p</i> -dioxins
<b>CDF</b>	Chlorinated Dibenzofurans
<b>CERCLA</b>	Comprehensive Environmental Response, Compensation, and Liability Act
<b>CLP</b>	Contract Laboratory Program
<b>CLPSS</b>	Contract Laboratory Program Support System
<b>COC</b>	Chain of Custody
<b>CRQL</b>	Contract Required Quantitation Limit
<b>CVAA</b>	Cold Vapor Atomic Absorption
<b>CWA</b>	Clean Water Act
<b>DMC</b>	Deuterated Monitoring Compound
<b>DOT</b>	Department of Transportation
<b>DQO</b>	Data Quality Objectives
<b>EPA</b>	United States Environmental Protection Agency
<b>ERT</b>	Environmental Response Team (EPA)
<b>ET</b>	Eastern Time
<b>FSP</b>	Field Sampling Plan
<b>GC/MS</b>	Gas Chromatograph/Mass Spectrometer or Gas Chromatography/Mass Spectrometry
<b>GPS</b>	Global Positioning System
<b>HAZWOPER</b>	Hazardous Waste Operations and Emergency Response
<b>HASP</b>	Health and Safety Plan
<b>HCN</b>	Hydrocyanic Acid
<b>HTML</b>	Hypertext Markup Language
<b>IATA</b>	International Air Transport Association
<b>IC</b>	Ion Chromatography
<b>ICP-AES</b>	Inductively Coupled Plasma - Atomic Emission Spectroscopy
<b>ICP-MS</b>	Inductively Coupled Plasma - Mass Spectrometry
<b>MA</b>	Modified Analysis
<b>MS</b>	Matrix Spike
<b>MSD</b>	Matrix Spike Duplicate
<b>NIOSH</b>	National Institute for Occupational Safety and Health
<b>NPL</b>	National Priorities List
<b>OLEM</b>	Office of Land and Emergency Management
<b>OSC</b>	On-scene/On-site Coordinator
<b>OSHA</b>	Occupational Safety and Health Administration
<b>OSRTI</b>	Office of Superfund Remediation and Technology Innovation
<b>PCBs</b>	Polychlorinated Biphenyls
<b>PE</b>	Performance Evaluation
<b>PHMSA</b>	Pipeline and Hazardous Materials Safety Administration
<b>ppb</b>	Parts-Per-Billion (e.g., µg/L or µg/kg)
<b>PPE</b>	Personal Protective Equipment

<b>ppm</b>	Parts-Per-Million (e.g., mg/L or mg/kg)
<b>ppt</b>	Parts-Per-Trillion (e.g., ng/L or ng/kg)
<b>PRP</b>	Potentially Responsible Party
<b>PTFE</b>	Polytetrafluoroethylene
<b>PVC</b>	Polyvinyl Chloride
<b>QA</b>	Quality Assurance
<b>QAPP</b>	Quality Assurance Project Plan
<b>QATS</b>	Quality Assurance Technical Support
<b>QC</b>	Quality Control
<b>RPM</b>	Remedial Project Manager
<b>RSCC</b>	Regional Sample Control Coordinator
<b>SAM</b>	Site Assessment Manager
<b>SAP</b>	Sampling and Analysis Plan
<b>SARA</b>	Superfund Amendments and Reauthorization Act
<b>SDG</b>	Sample Delivery Group
<b>SIM</b>	Selected Ion Monitoring
<b>SMC</b>	System Monitoring Compound
<b>SMO</b>	Sample Management Office
<b>SOP</b>	Standard Operating Procedure
<b>SOW</b>	Statement of Work
<b>SPLP</b>	Synthetic Precipitation Leaching Procedure
<b>SPP</b>	Site Project Plan
<b>SVOA</b>	Semivolatile Organic Analyte
<b>TCLP</b>	Toxicity Characteristic Leaching Procedure
<b>TIFSD</b>	Technology Innovation and Field Services Division
<b>TOC</b>	Total Organic Carbon
<b>TR/COC</b>	Traffic Report/Chain of Custody
<b>UN</b>	United Nations
<b>UPS</b>	United Parcel Service
<b>USCG</b>	United States Coast Guard
<b>USDA</b>	United States Department of Agriculture
<b>VOA</b>	Volatile Organic Analyte
<b>XML</b>	eXtensible Markup Language

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## 1.0 INTRODUCTION

The *Contract Laboratory Program Guidance for Field Samplers* (also referred to as the *Sampler's Guide*) describes the organizational roles and responsibilities for those who plan and conduct environmental sample collection for analysis through the U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP).



The following lists the sections of this Guide:

- Section 1, *Introduction*, introduces the structure and purpose of this document.
- Section 2, *General Sampling Information*, describes the general activities associated with environmental sampling.
- Section 3, *CLP Statements of Work*, describes the Statements of Work (SOWs) that define the requirements for CLP sampling.
- Section 4, *CLP Sample Documentation*, lists the types of documentation used to track CLP samples.
- Section 5, *The Scribe Documentation Software Tool*, provides information about Scribe, a software tool used to create sample documentation.
- Section 6, *CLP Sample Containers*, describes the types of containers required for CLP samples.
- Section 7, *CLP Sample Collection*, describes the process by which CLP samples are collected.
- Section 8, *CLP Sample Transportation and Shipping*, outlines the requirements for the packing and shipping of CLP samples.
- Section 9, *Sampler Resources*, provides links to additional information for sampling organizations.

The following lists the appendices of this Guide:

- Appendix A, *Functions within a Sampling Project*, describes the functions within a sampling project which are taken from the Quality Assurance Project Plan (QAPP) requirements.
- Appendix B, *General CLP Sample Collection Guidelines for Aqueous VOA Samples*, provides guidelines for Volatile Organic Analyte (VOA) water samples.
- Appendix C, *CLP Sample Collection Guidelines for Soil VOA Samples by SW-846 Method 5035A, TCLP Extraction by EPA SW-846 Method 1311, and SPLP Extraction by EPA SW-846 Method 1312* provides guidelines for VOA soil samples.
- Appendix D, *CLP Sample Collection Requirements by Analysis Type*, contains the sample collection requirements by SOW.
- Appendix E, *Sampling Techniques and Considerations*, recommends sampling techniques.
- Appendix F, *International Shipping*, contains information regarding shipping samples to laboratories outside the United States.
- Appendix G, *Sampling Checklists*, contains checklists used to help the sampler ensure that all necessary steps are completed.
- Appendix H, *Glossary*, provides definitions for terms used in this document.



If the field sampling team is planning to use the CLP, they should use this Guide to develop the Site Project Plan (SPP), QAPP, and Field Sampling Plan (FSP) documents.

## 1.1 Overview of the CLP

The CLP is a national network of EPA personnel, commercial laboratories, and support contractors whose fundamental mission is to provide environmental sample collection and analysis under the Superfund program. The Superfund program was established under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 and presently exists under the Superfund Amendments and Reauthorization Act (SARA) of 1986. The CLP is directed by the EPA Analytical Services Branch (ASB) of the Technology Innovation and Field Services Division (TIFSD) within the Office of Superfund Remediation and Technology Innovation (OSRTI) under the Office of Land and Emergency Management (OLEM).

The primary responsibility of the CLP is to provide analytical data of known and documented quality to CLP customers through its routine and modified chemical analytical services. The CLP provides a framework that allows data to be produced in a cost-effective and efficient manner. In addition, the CLP has established strict Quality Control (QC) procedures and detailed documentation requirements to ensure the consistent quality of the data. Current CLP data users include the EPA Regions, other EPA Headquarters programs, State and Tribal governments, and other Federal agencies.

### 1.1.1 Key Participants within the CLP Sampling Process

In coordinating Superfund sampling efforts, ASB is supported by the Sample Management Office (SMO) contractor, Regional Sample Control Coordinators (RSCCs), Site Assessment Managers (SAMs), On-scene/On-site Coordinators (OSCs), and Remedial Project Managers (RPMs). Samplers may work directly with the RSCC, and/or an OSC from the Site Support Personnel during a sampling event. Refer to Table 1-1 for a description of the functions performed by key participants (functions may vary by Region).

**Table 1-1. Participants in the CLP Sampling Process**

Participants	Responsibilities
<b>Analytical Services Branch (ASB)</b>	<p>ASB directs the CLP within the TIFSD of the OSRTI in the OLEM. ASB's responsibilities include:</p> <ul style="list-style-type: none"> <li>• Development of the SOWs that define required analytical methods (including QC, detection/quantitation limits, and holding times) for the analytical services procured under the CLP</li> <li>• Development and implementation of policies and budgets for Superfund analytical operations</li> <li>• Development of information management policies and products for analytical data</li> <li>• Management of SMO and Quality Assurance Technical Support (QATS) contracts</li> <li>• National administration, evaluation, and management of the CLP</li> <li>• Direction of CLP Quality Assurance (QA) activities in coordination with overall OLEM QA activities</li> </ul> <p>To obtain the most current ASB contact list, refer to the following website: <a href="https://www.epa.gov/clp/forms/contact-us-about-superfund-analytical-services-or-contract-laboratory-program#tab-2">https://www.epa.gov/clp/forms/contact-us-about-superfund-analytical-services-or-contract-laboratory-program#tab-2</a></p>
<b>CLP Sample Management Office (SMO)</b>	<p>The contractor-operated SMO provides management, operations, and administrative support to the CLP. SMO receives Regional analytical requests, coordinates and schedules sample analyses, and tracks sample shipments. SMO also receives and checks data for completeness and compliance, processes laboratory invoices, and maintains a repository of sampling records and program data.</p>

Table 1-1. (Continued) Participants in the CLP Sampling Process

Participants	Responsibilities
<b>CLP Contract Laboratories</b>	The contractor-operated laboratories within the CLP provide analytical services for the separation, detection, and quantitation of the CLP's target analytes.
<b>Environmental Response Team (ERT)</b>	The ERT is responsible for the development, implementation, and management of the Scribe software system. In addition, the ERT oversees the development of Scribe training webinars and on-site training.
<b>Environmental Response Team (ERT) Support Contractors</b>	The ERT Support Contractors provide technical and administrative support for the development, implementation, and management of the Scribe software system. In addition, the ERT Support Contractors support the development of Scribe training webinars and on-site training.
<b>Regional Sample Control Coordinator (RSCC)</b>	In most Regions, the RSCC coordinates sampling efforts and serves as the central point-of-contact for sampling questions and problems. The RSCC works with SMO to request analytical services and receive laboratory assignments. In addition, the RSCC's activities may include informing SMO of sample shipment, cancellations, special instructions, and sampling issues. To obtain the most current RSCC contact list, refer to the following website: <a href="https://www.epa.gov/clp/forms/contact-us-about-superfund-analytical-services-or-contract-laboratory-program#tab-3">https://www.epa.gov/clp/forms/contact-us-about-superfund-analytical-services-or-contract-laboratory-program#tab-3</a>
<b>Site Support Personnel</b>	The Site Support Personnel consist of the EPA personnel and contractors responsible for developing the QAPP and Sampling Plan for the sampling event at the site. It includes such personnel as the sampling team, QA personnel, OSC, SAM, and RPM. In most Regions, the Site Support Personnel develop Standard Operating Procedures (SOPs) for field sampling and related procedures, and assist sampling teams in adhering to the SOPs. The sampling team determines what type(s) of CLP services will be required for a particular sampling event. The Site Support Personnel review Sampling and Analysis Plans (SAPs) prepared by sampling teams and oversee sampling teams in the field. In addition, the state or territorial environmental protection agency for the location of the site provides support for the sampling event.

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## 2.0 GENERAL SAMPLING INFORMATION

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### 2.1 Goals of the Sampling Process

Once the U.S. Environmental Protection Agency (EPA) has determined that physical, chemical, and/or biological testing of a site is necessary, samples of material from the site area must be collected. The type of material that must be collected and the analytical method to be used depends upon the physical location of the site, detection level(s), site history (previous sampling), and known or unknown conditions and contaminants.



Samples should be collected according to the approved project and site-specific Quality Assurance Project Plan (QAPP) and Sampling and Analysis Plan (SAP). This Guide does not define specific sampling procedures, as these depend upon individual site conditions, Regional requirements, and acceptance and performance criteria. Since Regions may have their own specific requirements for individual sampling programs, they are responsible for generating Region-specific sampling Standard Operating Procedures (SOPs). EPA personnel shall also follow the procedures in CIO 2105-P-02.0 (*EPA QA Field Activities Procedures*).

#### 2.1.1 Follow the Required Sampling Procedures

It is imperative that samplers be aware of the minimum Contract Laboratory Program (CLP) and Regional requirements that directly impact and define how a sampling event will take place. It is important to note that the procedures and guidelines set forth in this document are considered minimum CLP requirements.

The purpose of sampling is to collect representative portions from a suspected contaminated site. Sample collection is critical to determining the presence, type, concentration, and extent of environmental contamination by hazardous substances; thus, it is a crucial part of every sampling and environmental testing effort. Sampling procedures must be consistently followed to mitigate risk of error and the expense of resampling.

Failure to follow proper sampling and shipping procedures could result in samples that are contaminated, in broken containers, mislabeled, lost during shipping, compromised due to improper preservation, or unusable because of a missed holding time. If procedures are inconsistently or improperly followed, any resultant analytical data may be inaccurate and may not be legally defensible.

#### 2.1.2 Maintain Chain of Custody of Samples and Data

Acquiring accurate and legally defensible data is the CLP's primary objective; therefore, the samplers must collect samples according to strict sampling procedures, plans, and guidelines. The Chain of Custody (COC) Records must be generated and the sample COC maintained. EPA and

many other Federal agencies use data resulting from analytical testing of samples for the following activities:

- Site Characterization
- Source Evaluation
- Identification of Potentially Responsible Parties (PRPs)
- Remedial Design
- Assessment of response and remedial priorities
- Remedial Action
- Remedial Investigation/Feasibility Studies
- Assessment of risk to human health and the environment
- Determination of appropriate cleanup actions
- Determination of cleanup achievements

### 2.1.3 Field Operation Records

Samplers must maintain complete, accurate, and legible field operations records as they perform a sampling activity. The following records are included:

- Field logbooks
- Corrective Action reports
- Sampling trip reports
- Supplemental standardized forms
- Records such as maps or photographs that document each step of each activity performed in the field

Samplers must refer to their project plans for Region-specific field operations record requirements. These records are an important tool because they are considered a part of the official project file when legal issues arise.

### 2.1.4 Comply with Safety Procedures

Care must be taken to maintain the safety of personnel collecting and handling CLP samples. If sampling requires digging in soil, utility lines (gas, oil, cable, etc.) must be marked to prevent injury or utility outage. Samples must be handled, packed, and shipped in accordance with all applicable Federal [Occupational Safety and Health Administration (OSHA) and Department of Transportation (DOT)] regulations for hazardous materials. Refer to the Health and Safety Plan (HASP) for detailed site safety requirements.

## 2.2 Obtain Municipal Permits, Licenses, and Clearances

Before starting a sampling event, samplers must obtain the proper municipal permits, access to the property, and any government clearances, if required. Samplers must also contact any appropriate utility companies to ascertain where any underground pipes, cables, etc., may be located.

### 2.2.1 Request Access to County, State, Tribal, Military, and/or Federal Property

Proper access to perform sampling activities is important not only for legal reasons, but also to eliminate delays in work and possible refusal to allow sampling to take place. It is crucial that the appropriate permits, licenses, and clearances be secured to obtain access for sampling activities that will be performed on County, State, Tribal, military, and/or Federal property. The sampler must contact the appropriate government offices or personnel well in advance to determine what kinds of approval are required. Pre-approval may be required for specific types of sample collection such as drilling or excavation. For example, drilling on a military base requires pre-

approval. Base security may require clearances for all members of the sampling team, including subcontractors. This process may take two or more days.

## 2.2.2 Contact Private Property Owners

Samplers must obtain written permission from the private property owner(s) before sampling on his/her property, even if verbal permission has been granted. It is recommended that samplers obtain verbal permission prior to their arrival at the sampling location, but written permission can be obtained on the day of sampling. If a property owner refuses to grant access to his/her property, it may be necessary for the sampling organization to contact the appropriate authorities for assistance. A sampler who enters private property without permission may be subject to a charge of trespassing, and samples may be considered part of an illegal search and invalid for legal proceedings.

## 2.2.3 Contact Utility Companies

The sampler must contact local utility companies (e.g., power, phone, gas, cable, sanitation, etc.) at least one week prior to the sampling event to have underground cables, lines, and pipes flagged and marked. This is required by law. A national one-call directory can be found at: <https://call811.com>

It may be necessary to turn off the utilities (i.e., electrical wires or gas lines) in order to obtain samples. The utility service(s) disruption dates should be confirmed at least two days prior to sampling activities. Samplers should follow Regional or other appropriate program procedures for the disruption of utilities.



Pre-payment of survey fees to local utility companies may be required.

## 2.3 Analytical Services Request

To prepare for the sampling event, the samplers should review the “Analytical Services Request Regional Notification” for samples from the CLP. This information may be in the form of the “Analytical Services Request Regional Notification” obtained from the Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal, supplied by the Regional Sample Control Coordinator (RSCC), or in other forms of communication from the Region. Information for Modified Analyses may also be obtained from the SMO CLPSS Portal. Field team leaders should contact their RSCC, Remedial Project Manager (RPM), or On-Site Coordinator (OSC) to review this information prior to going into the field, and ensure that this information matches information in the Site Project Plan (SPP), SAP, and/or QAPP. An eXtensible Markup Language (XML) file is provided as part of the laboratory assignment from the SMO CLPSS Portal which can be used for uploading site information into Scribe.

Review the following sources of information for planning:

- **Sample information:** Provides the number of samples requested, the sample matrix, the analysis types, and if Preliminary Results are required. This information is used to determine the equipment and supplies needed for the sampling event.
- **Site location:** Determines whether there are any specific requirements for accessing/exiting the site, or for working at the site.
- **Shipping period:** Establishes the time frame during which the samples are to be shipped to the laboratory/laboratories. This helps determine when sampling should occur.
- **Laboratory information:** Specifies where the samples will be shipped.

### 2.3.1 Review Sample Request

Review the “Analytical Services Request Notification” and other Regional communication for the following information:

- The sample request determines some of the preparatory activities for the sampling event.
- Review and evaluate the sample request to determine the number and types of samples to be collected.
- Review the required sample collection method(s).
- Review decontamination procedures necessary for site.
- Make note of sample holding times and conditions.
- Determine Performance Evaluation (PE) and Quality Control (QC) sample requirements.
- Determine whether shipping container temperature blanks are required.

## 2.4 Review Project Plans

Project plans describe, in detail, the requirements for the sampling event. All field team members should be familiar with the applicable project plans prior to beginning field sampling. These plans may include the following documents.

### 2.4.1 Site Project Plan (SPP)

The SPP describes the requirements for any activity taking place at the site. It contains information such as site history, potential contaminants, topographical information, etc. that may be integrated into the QAPP.

### 2.4.2 Health and Safety Plan (HASP)

The HASP describes the measures necessary to maintain the health and safety of the sampling team during the sampling event. It can include topics such as the following:

- Organization structure
- Job hazard analysis
- Site control
- Training
- Medical surveillance
- Personal protective equipment (PPE)
- Exposure monitoring
- Thermal stress
- Decontamination
- Emergency response
- Location of and directions to the nearest Emergency Room or hospital
- Standard Operating Procedures
- Confined space operations
- Spill containment

### 2.4.3 Quality Assurance Project Plan (QAPP)

The QAPP describes the data quality objectives and data requirements for the project, and is used by samplers to develop any subsequent plans such as the SAP or the Field Sampling Plan (FSP). Refer to Appendix A for more information on QAPP sampling requirements.

## 2.5 Assemble Sampling Materials

Samplers should prepare for a sampling project by assembling the appropriate sampling materials (equipment, supplies, sample containers, packing materials, and shipping materials). The equipment and supplies must be properly cleaned, calibrated, and tested as necessary to meet the needs of the sampling project prior to sampling.

### 2.5.1 Equipment and Supplies

Samplers should review the project plans to determine the equipment necessary for sample collection.

The following should be obtained prior to a sampling event:

- Sample containers
- Shipping containers
- Packing material
- Reagent water for decontamination and for preparation of field/equipment blanks
- Access to the Scribe software for creating sample labels, stickers, Traffic Report/COC (TR/COC) Records, and Region-specific Scribe templates
- Custody seals
- Sampling equipment such as bowls, augers, pumps, etc.
- PPE
- Internet access (either at the time of sampling or soon after the samples are shipped)

The QAPP, the SAP, or the CLP Statement of Work (SOW) may also require field samplers to provide the following:

- Shipping container temperature blanks
- Trip blanks for Volatile Organic Analyte (VOA) analysis
- Preservation supplies (e.g., ice or acid)
- Specially prepared sample vials or bottles (e.g., VOA or hexavalent chromium)
- Utensils or equipment for handling tissue samples requested by Modified Analysis

## 2.6 Perform Readiness Review/Dry Run

A readiness review/dry run is a test run of the proposed sampling event. It is a recommended practice as it affords samplers a chance to review all plans, documentation software (i.e., Scribe), and equipment lists for accuracy and completeness prior to sampling activities. It also provides an opportunity to consult with sampling team members to make sure that all the elements are in place and that everyone understands their tasks before actually going out into the field. Sampling project managers should provide the readiness review or dry run dates and schedules to samplers so that they can prepare accordingly.

## 2.7 Assess the Status of the Site and the Team

Samplers should consider taking the following actions prior to starting a sampling event:

- Communicate personnel roles and lines of authority.
- Verify that HAZWOPER training is up to date for all field staff as required.
- Ensure that permission has been granted to enter the site and collect samples.
- Confirm that utility work has been completed (if required).
- Review local weather forecast to be aware of possible dangerous weather conditions. Ensure that sampling staff are prepared for weather conditions.

- If the sampling location is inaccessible, contact the appropriate field or Regional personnel for instructions.
- Verify that the correct sampling equipment is on site.
- Ensure that personal safety measures are in place.
- Ensure that a site HASP is in place and includes procedures for emergency medical treatment and first aid, evacuation procedures, emergency contacts, and location of emergency medical facilities.
- Identify and mark the sampling location with buoys, flags, or stakes according to the sampling plans, maps, and grids.
- Park the car/van away from the sampling site and turn off the engine. Be aware of car exhaust (BTEX) contamination to volatile organic samples through all procedures, including loading and unloading the containers during shipping.

## 2.8 Initiate Site Control Measures

The sampling team is responsible for implementing the necessary site control measures during the sampling event, which may include the following:

- Maintaining a log of authorized personnel entering the site.
- Preventing unauthorized persons from entering the site.
- Ensuring that any decontamination equipment or procedures to prevent sample cross-contamination required in the QAPP and/or FSP are in place and carried out.

## 2.9 Maintain Field Logbook

Samplers must maintain a field logbook that documents the field activities. The information in the field logbooks may be used as evidence in court. The field logbook should meet the following criteria:

- Use waterproof ink to record all entries.
- Record the date and time of all entries, and the name of the person recording the information.
- Correct all errors by drawing a line through the error, initialing and dating the error, and then adding the correct information.
- Document sampling project information such as:
  - Project name, Project ID, and location
  - Names of samplers
  - Geological observations, including maps and Global Positioning System (GPS) information
  - Atmospheric conditions
  - Field measurements
  - Sampling dates, times, locations, and sample location coordinates
- Record sampling activity information such as:
  - Sampling dates and times
  - Name(s) of person(s) recording the information
  - Sample identifications
  - Sample matrices
  - Sample descriptions (e.g., odors and/or colors)
  - Number of samples collected
  - Sampling methods/equipment
- Record any and all deviations from the sampling plan.
- Record any and all difficulties in sampling and/or any unusual circumstances.

## 2.10 Preventing Errors

Errors in the sampling process can result in additional costs and delayed sampling results. The following section lists some of the steps that can be taken to avoid common sampling errors.

### Document samples correctly:

- Use the CLP Sample Number and SMO CLPSS Portal-generated CLP Case Number correctly (sample number on each sample).
- Submit the signed TR/COC Record with the sample(s).
- Ensure that the TR/COC Record and the sample container information match each other and are accurate.
- Accurately and legibly complete and attach a custody seal to each shipping container. The project QAPP may also require that custody seals be attached to each sample container or plastic sample bag. Refer to the project QAPP for specific instructions.

### Collect and preserve samples correctly:

- Collect a sufficient volume of sample so that the laboratory can perform the requested analysis and quality controls, such as Matrix Spike (MS), Matrix Spike Duplicate (MSD), and laboratory Duplicate.
- Ensure that required preservatives have been added to samples.

### Ship samples correctly:

- Pack bottles and containers to prevent breaking or spilling during shipping.
- For iced samples, evenly distribute bags/packets of ice throughout the cooler and between the sample containers to ensure that all samples are sufficiently cooled to a temperature of  $\leq 6^{\circ}\text{C}$ , but not frozen, and include a sealed container of water to be used as a temperature blank.
- Verify that the TR/COC Records have been included prior to sealing the shipping container.
- Verify that custody seals are attached to the shipping containers.
- Ensure that the shipping containers are labeled with the designated laboratory address.
- Include shipping information for United States Department of Agriculture (USDA) Quarantine samples if applicable.
- If samples are to be shipped internationally, assemble the required additional paperwork or customs authorizations. Refer to Appendix F, International Shipping, for additional information.
- Upload the electronic TR/COC Record as soon as possible after shipping.

### Communicate effectively:

- It is extremely important that all parties involved in a sampling event remain in contact during the sampling process.
- The key elements of communication for a sampling event include the relationship between the RSCC, SMO, the samplers in the field, and the laboratories that will be accepting the samples.
- In instances where there are any changes to the sampling event due to a cancellation or an increase or decrease in the number of samples to be shipped, the sampler should contact the RSCC as soon as possible. The RSCC will work with SMO to resolve any potential capacity, availability, or overbooking issues with the CLP laboratories.

## 2.11 Exiting the Site

The following activities should be performed before leaving the sampling site:

- Ensure that all equipment has been collected and removed.
- Follow Regional guidance regarding decontamination and doffing of PPE, if used.
- Follow Regional guidance for waste removal and disposal.
- Ensure that all sampling personnel have cleared the site.
- If sampling on private property, provide a sample receipt to the property owner for all samples collected and removed from the site.

## 3.0 CLP STATEMENTS OF WORK

The overall requirements for sample collection, analysis, and handling under the Contract Laboratory Program (CLP) are described in the CLP Statements of Work (SOWs).

### 3.1 The CLP SOWs

The CLP SOWs are available on the U.S. Environmental Protection Agency (EPA) website at: <https://www.epa.gov/clp/superfund-clp-analytical-statements-work-sows>. Table 3-1 lists the CLP SOWs.

**Table 3-1. CLP Statements of Work**

Statement of Work	Analysis Types	Matrix Types
<b>High Resolution</b>	Chlorinated Dibenzo- <i>p</i> -Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Chlorinated Biphenyl Congeners (CBCs)	Soil, sediment, biosolids, oil, sludge, ash, tissue, water, and wipe
<b>Organic/Inorganic</b>	Trace Volatile Organic Analytes (Trace VOAs) Trace VOAs Selected Ion Monitoring (SIM) Volatile Organic Analytes (VOAs) Semivolatile Organic Analytes (SVOAs) SVOAs SIM Pesticides Aroclors Metals by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP - AES), Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) Mercury by Cold Vapor Atomic Absorption (CVAA) Spectrometry Cyanide by Spectrophotometry Anions by Ion Chromatography (IC)* Hexavalent Chromium by IC* Total Organic Carbon (TOC)*	Soil, sediment, water, Toxicity Characteristic Leaching Procedure (TCLP)/ Synthetic Precipitation Leaching Procedure (SPLP) leachate , waste, and wipe

\*Anions, Hexavalent Chromium, and TOC will be available by Modified Analysis (MA).

### 3.2 CLP Sample Definition

A CLP sample is defined as a discrete portion of material to be analyzed from one location for each individual or set of analyses.

A sample consists of all sample aliquots (portions), provided that the analyses are all requested from the same CLP analytical program:

- for each individual or set of analytical methods
- from one location
- for one sample matrix
- for one laboratory

### 3.2.1 Mixed-matrix Samples

In some instances, a mixed-matrix sample which contains either a supernate (for a sediment/soil sample) or a precipitate (for a water sample) may be collected. The decisions made with regard to the different matrices in such samples can have profound impacts on data usability. In this event, samplers should consult their sampling plans and/or discuss the required procedures with the Remedial Project Manager (RPM), On-Site Coordinator (OSC), or designee.

In general, it is recommended that two individual samples be collected by separating the aqueous layer from the solid/precipitate layer at the point of collection if possible. If the phases or layers cannot be separated effectively in the field at the point of collection, direction should be provided to the receiving laboratory to separate the layers under controlled conditions. In this case, additional sample numbers will be needed for the separate phases. They should be assigned two different sample IDs (e.g., Sample IDs ABC124 and ABC125 for Sample ID ABC123), along with notes in the field sample log and in the Special Instructions section of the Chain of Custody (COC) Record, indicating that the sample IDs are derived or related to the same sample. Refer to Section 5.1.5, Using Scribe for Mixed-matrix Samples, for information on how to use the Scribe software to track mixed-matrix samples.



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When samples are collected from several locations to form a composite sample, the sample should be assigned either a number from one of the locations used during collection, or a unique number that represents the composite sample, for tracking purposes. The numbering scheme used internally at a sampling event for identifying composite samples should also be documented appropriately (e.g., in the field logbooks).

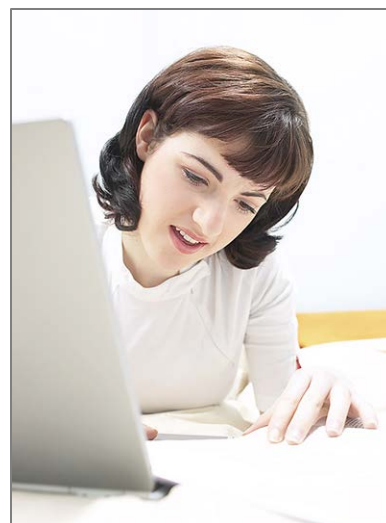
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### 3.3 CLP Analyses

CLP analysis is generally used for Superfund sites and includes the target analytes. The matrices can be water, leachates derived from the TCLP or SPLP, soil, sediment, waste, wipe, or tissue (non-human) for high resolution analyses. Additional matrices requested under Modified Analysis (MA) for inorganics and organics may include oil, sludge, ash, construction waste, biosolids, or tissue (non-human).

## 4.0 CLP SAMPLE DOCUMENTATION

The U.S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) is required to produce accurate and legally defensible data. Therefore, in order to produce legally defensible data, control of the samples must be maintained to ensure that the samples accurately represent the site and location from which they were collected. Sample documents are tools that allow EPA to maintain the chain of custody of the samples from collection, through shipping, to analysis. The documentation also associates the sample to the sample data. Samplers should review their site-specific project plans and Quality Assurance Project Plans (QAPPs) to determine other types of documentation that must be completed for a sampling project. The following section describes the documents used to maintain the chain of custody and the tools used to create these documents.



The following table summarizes CLP sample documentation.

**Table 4-1. CLP Sample Documentation Tracking**

Type	Source	Purpose
CLP Sample Number	Assigned by sampling software (Scribe); ranges are supplied by the Regional Sample Control Coordinator (RSCC)	Identifies sample data. Associates the sample to the sample data.
CLP Case Number	Generated by the Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal	Identifies groups of samples collected during a single sampling event.
Traffic Report/Chain of Custody (TR/COC) Record	Created in Scribe	Tracks chain of custody of the sample and sample data.
Custody seals	Supplied by the RSCC or field sampling team	Maintains sample integrity; may indicate sample tampering or contamination if broken.
Sample labels	Created in Scribe	Affixed to the sample container to identify an individual sample.
Field operations records (as necessary)	Created and maintained by sampling team	Maintains a record of activities at the site.
Shipping container label (to the laboratory)	Carrier standard form	Used by the carrier to ship the samples to the laboratory.
Shipping container return label (return from the laboratory)	Carrier standard form	Used by the carrier to have the laboratory return the container to the Region.

The documentation required by a Region for a sampling event is outlined in project plans such as the QAPP, Sampling and Analysis Plan (SAP), and Field Sampling Plan (FSP).



EPA recommends that a dedicated field team member be responsible for all sample documentation steps, including reviewing laboratory scheduling information, creating sample labels and TR/COC Records in Scribe, maintaining a field operations log, and relinquishing control of the samples to the laboratory. This person should be identified in the Site Project Plan (SPP) or QAPP.



Under no circumstances should the site name or address appear on any documentation that is sent to the laboratory (for the CLP).

## 4.1 CLP Sample Numbers

A sample number is a number that is unique per sampling location and identifies each CLP sample. It is used to track samples throughout the sampling and analytical processes, and is recorded on several types of sample and sample documentation (e.g., TR/COC Records and sample labels).

**Table 4-2. CLP Sample Number Letter Codes**

Region	Letter Code
1	A
2	B
3	C
4	D
5	E
6	F
7	G
8	H
9	Y
10	J

According to the CLP guidelines, each individual inorganic water sample may be analyzed for total metals or filtered metals, but not both. Therefore, water samples collected for total metal and filtered metal analyses from the same sampling location must be assigned separate and unique CLP Sample Numbers. Samples for Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) may also require separate CLP Sample Numbers.

### 4.1.1 Requesting Sample Numbers

CLP Sample Numbers are created in Scribe with ranges supplied by the RSCC. The sampler should request a range of sample IDs from the RSCC. Once the initial number is entered into Scribe, the software will auto-generate the additional sample numbers required for the project. Do not re-use CLP sample numbers, as this may create difficulties for Laboratory Information Management Systems.

## 4.2 CLP Case Numbers

SMO CLPSS Portal-generated Case Numbers are used to track groups of samples from a sampling event throughout the sampling and analytical processes, and are recorded on several types of sample and sample documentation (e.g., TR/COC Records, sample labels). Samplers should correctly assign the Case Number to the appropriate sample bottle or container. CLP Case Numbers are obtained from the “Analytical Services Request Regional Notification” in the Access Assignment Information task in the SMO CLPSS Portal. Samplers should correctly assign the Case Number to the appropriate sample bottle or container.

### 4.3 CLP TR/COC Records

A TR/COC Record is used as physical evidence of sample custody and as a permanent record for each sample collected. A chain of custody record documents the exchange and transportation of samples from the field to the laboratory.

To meet CLP sample documentation and chain of custody requirements, samplers must attach a separate, signed TR/COC Record to each container shipped. The sampler should consider TR/COC documentation needs when shipping directly from the site.

- The TR/COC Record must document each sample within the shipping container.
- The Carrier Name and Airbill Number should be on each COC.
- Each TR/COC Record **must be signed by the designated field sampler**, documenting that they have relinquished control of the samples.
- TR/COC Records should be separated and shipped in the containers with the samples listed on them. Attach the TR/COC Record to the inside of the lid of the shipping container. Samples may not be shipped in a container without the corresponding TR/COC Record. This practice maintains the chain of custody for all samples in case of incorrect shipment.
- The electronic TR/COC Record should be uploaded as soon as possible after shipping.

If more than one TR/COC Record is used for the samples within one shipping container, all of the records must have complete header information and original signatures. Samplers are responsible for the care and custody of samples from the time of collection to the time of shipment to the laboratories for analysis. A sample is considered under custody if the following conditions are met:

- It is in possession or in view after being in possession
- It was in possession and then secured or sealed to prevent tampering
- It was in possession when placed in a secured area

Each time the custody of samples is turned over to another person, the TR/COC Record must be signed off by the former custodian and accepted by the new custodian.

### 4.4 Chain of Custody Seals

A chain of custody seal is any adhesive label or tape that can be used to seal a sample bottle, container, plastic bag, or shipping container. In the event that a container is opened or tampered with, the seal will be broken. The custody seal is used to maintain the chain of custody, as well as guard against possible sample contamination or tampering during shipping.

- Custody seals must be placed on each shipping cooler or container, and if required by the project's QAPP or FSP, on each sample bottle, container, or bag (as appropriate).
- The CLP does not provide custody seals. Custody seals should be obtained from the RSCC or supplied by site personnel.

### 4.5 Sample Labels

A sample label is a sticker that is attached to a sample bottle or container that contains a field sample or quality control (QC) sample.

- Sample labels are affixed to each sample container as samples are collected in the field or prior to going in the field.
- A sample label must contain, at a minimum, the sample number so that the sample can be associated with, and listed on, the associated TR/COC Record.



## 5.0 THE SCRIBE DOCUMENTATION SOFTWARE TOOL

The U.S. Environmental Protection Agency (EPA) Analytical Services Branch (ASB) requires samplers to use the Scribe software to create documentation for all Contract Laboratory Program (CLP) sampling efforts. The EPA recommends that a dedicated member of the sampling team be trained in the Scribe software, and be responsible for all uses of Scribe including the sample labels and the Traffic Report/Chain of Custody (TR/COC) Records at the sampling location. For



assistance with obtaining or using the Scribe software, contact the Environmental Response Team (ERT) Software Support Help Desk at 800-999-6990 from 9:00 AM – 5:00 PM ET. For additional information regarding Scribe use and training materials, refer to the following website: <https://response.epa.gov/scribe>. Scribe allows users to create one or more sampling projects, enter data, and create sample documents for that project. Some of the capabilities of Scribe include:

- Tracking sample numbers and Case Numbers
- Associating analysis information to sample numbers
- Creating sample labels
- Setting label size and printing labels
- Selecting sample numbers to add to the chain of custody record
- Printing chain of custody records
- Filtering lists of samples
- Exporting sample data in the following formats: text file (.txt, .csv), spreadsheet (.xls, .wb3), HTML (.htm), XML (.xml), or QuickMap (.kml, .kmz)

The Scribe software tool allows users to track samples electronically. It can be downloaded at no charge from the EPA On-Scene Coordinator (OSC) website at <https://www.ertsupport.org/downloads.htm>

### 5.1 Setting Up a Sampling Event in Scribe

Scribe allows the sampler to enter much of the information prior to the event in order to facilitate processing on the day of the event. The following sections describe how to set up the sampling event in Scribe.

#### 5.1.1 Set Up Project

The initial step when using Scribe is to set up the project as follows:

- Access the Scribe **New Project Wizard** to set up the sampling project.
- Enter the project **Site Name**, **Site #**, and **Region #** (required).
- Enter additional project information, if available. The eXtensible Markup Language (XML) file for the Case can be imported from the Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal and will customize the analysis and lab lists.

## 5.1.2 Create Analysis Types

The analysis types to be used for the sampling event must be defined for the project. Refer to the site sampling plan to determine which analyses are to be used.

- Use the **Analyses** table to display a list of all the analysis types available.
- Only analyses with the Program Type of “CLP,” such as “CLP ICP-AES Metals,” or “CLP Semivolatiles” should be selected for CLP Cases.
- If the required analysis type is missing, it can be added manually using the **Add** button.

## 5.1.3 Set Default Sample Number Information

Set up the default values for sample number. This allows the samplers to let the system increment sample numbers, rather than hand entering each one.

- Select **File**→**Options**→**CLP/Tag Settings** to display the **CLP/Tag Settings** window.
- Enter the new default values and click **OK**.

## 5.1.4 Indicate Modified Analysis (MA) on Scribe COC Records

When completing a TR/COC Record in Scribe, indicate an MA as follows:

- Identify the samples that will be analyzed using (a) CLP MA(s) by creating a new analysis within the Scribe Analyses table or at the time of entering the Analyses for the sample.
- The MA analysis should contain the Modification Reference Number within the name assigned to the analysis. For example, if a Region submits an MA for an additional analyte, and the SMO assigns the Modification Reference Number 3001.0, the Scribe Analyses could be named “CLP VOA by MA 3001.0.”. The associated abbreviation for this analysis could be “VOA MA”.

## 5.1.5 Using Scribe for Mixed-matrix Samples

The Scribe **LinkSampleNo** field links the original sample to the split samples and numbers. Use this field to link to the sample IDs used for the different sample phases as follows:

- Add two (2) additional samples in Scribe indicating in the matrix field which one is the liquid/aqueous phase and which one is the solid phase (i.e., ABC124 and ABC125).
- Tie the two additional samples to the original sample number using the “LinkSampleNo” field.
  - **In Scribe**, in the **Samples** tab, click the **View** button; the **Select Columns** drop-down menu displays. Put a checkmark next to **LinkSampleNo** to make that column visible.
  - Add the “parent” or the original field sample # in the **LinkSampleNo** column (i.e., ABC123).
  - **On the COC Record**, indicate in the **Special Instructions** which of the two new sample numbers the laboratory is to use for the liquid/aqueous phase and which sample number is to be used for the solid phase.

An example of a Scribe COC Record with mixed-matrix samples is shown in Figure 5-1.



**Table 5-1. (Continued) Scribe CLP Analysis Codes**

<b>Analysis Name</b>	<b>Abbreviation</b>
CLP Cadmium	Cd
CLP Calcium	Ca
CLP Chromium	Cr
CLP Cobalt	Co
CLP Copper	Cu
CLP Cyanide	CN
CLP Hardness	Hardness
CLP Hexavalent Chromium	Cr+6
CLP ICP-AES Metals	ICP-AES
CLP ICP-MS Metals	ICP-MS
CLP Iron	Fe
CLP Lead	Pb
CLP Magnesium	Mg
CLP Manganese	Mn
CLP Mercury	Hg
CLP Nickel	Ni
CLP Potassium	K
CLP Selenium	Se
CLP Silver	Ag
CLP Sodium	Na
CLP SPLP Aluminum	SPLP Al
CLP SPLP Antimony	SPLP Sb
CLP SPLP Arsenic	SPLP As
CLP SPLP Barium	SPLP Ba
CLP SPLP Beryllium	SPLP Be
CLP SPLP Cadmium	SPLP Cd
CLP SPLP Calcium	SPLP Ca
CLP SPLP Chromium	SPLP Cr
CLP SPLP Cobalt	SPLP Co
CLP SPLP Copper	SPLP Cu
CLP SPLP Cyanide	SPLP CN
CLP SPLP ICP-AES Metals	SPLP ICP-AES
CLP SPLP Iron	SPLP Fe
CLP SPLP Lead	SPLP Pb
CLP SPLP Magnesium	SPLP Mg
CLP SPLP Manganese	SPLP Mn
CLP SPLP Mercury	SPLP Hg
CLP SPLP Nickel	SPLP Ni
CLP SPLP Potassium	SPLP K
CLP SPLP Selenium	SPLP Se
CLP SPLP Silver	SPLP Ag
CLP SPLP Sodium	SPLP Na

**Table 5-1. (Continued) Scribe CLP Analysis Codes**

<b>Analysis Name</b>	<b>Abbreviation</b>
CLP SPLP Thallium	SPLP TI
CLP SPLP Vanadium	SPLP V
CLP SPLP Zinc	SPLP Zn
CLP TCLP Arsenic	TCLP As
CLP TCLP Barium	TCLP Ba
CLP TCLP Cadmium	TCLP Cd
CLP TCLP Chromium	TCLP Cr
CLP TCLP ICP-AES Metals	TCLP ICP-AES
CLP TCLP Lead	TCLP Pb
CLP TCLP Mercury	TCLP Hg
CLP TCLP Selenium	TCLP Se
CLP TCLP Silver	TCLP Ag
CLP Thallium	TI
CLP Total Organic Carbon Water	TOC Water
CLP Total Organic Carbon Soil	TOC Soil
CLP Vanadium	V
CLP Zinc	Zn
<b>Organics</b>	
CLP PAH+PCP	PAH
CLP PAH+PCP by SIM	PAH SIM
CLP Semivolatiles	SVOA
CLP SPLP Semivolatiles	SPLP SVOA
CLP SPLP Volatiles	SPLP VOA
CLP TCLP Semivolatiles	TCLP SVOA
CLP TCLP Volatiles	TCLP VOA
CLP Trace Volatiles	TVOA
CLP Trace Volatiles by SIM	TVOA SIM
CLP Volatiles	VOA
<b>Pesticides</b>	
CLP Pesticides	PEST
CLP SPLP Pesticides	SPLP PEST
CLP TCLP Pesticides	TCLP PEST



To avoid issues with interpretation, ensure that Total Metals and Dissolved Metals are labeled with separate sample IDs.

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## 6.0 CLP SAMPLE CONTAINERS

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The analytical protocol(s) for sample analysis often requires the use of a specific type of sample container. The type of container will also depend on the sample matrix.

It is recommended that samplers use borosilicate glass containers which are inert to most materials when sampling for pesticides and/or other organics, and for Total Organic Carbon (TOC). Borosilicate glass is also recommended for sampling soils for metals. Conventional polyethylene is recommended when sampling for metals and anions in water because of the lower cost and absorption rate of metal ions.



Have extra containers readily available for each sampling event in case of breakage, loss, or contamination.

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Containers procured for a sampling event are usually pre-cleaned and shipped ready for use from the manufacturer to the sampling site. Regardless of the type of container used, samplers must ensure that the containers have been analyzed or certified clean to levels below concern for the project based on the specifications in the Quality Assurance Project Plan (QAPP). Certificates must be kept on record. These containers must meet the U.S. Environmental Protection Agency (EPA) container type specifications listed in Tables D-1 through D-4 in Appendix D.



Samplers should document the lot numbers for every lot of cleaned containers used for each project and maintain corresponding certificates of analysis on file and available upon request.

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## 7.0 CLP SAMPLE COLLECTION

Samplers should determine the types of samples or aliquots to be collected, the volume required for each aliquot, and the preservation requirements by referring to the Site Project Plan (SPP) and Contract Laboratory Program (CLP) sample requirements tables in Appendix D. The following sections describe the types of samples that may be required to be collected.



### 7.1 Requesting the Scheduling of the Laboratory

Samplers must request that the Regional Sample Control Coordinator (RSCC) schedule the laboratory to be used for the analysis. This should be done as far in advance of the sampling event as possible.

- If one or more Modified Analysis (MA) will be required, scheduling requests should be submitted at least four weeks prior to the start of the sampling event to allow the solicitation process to be completed.
- Samplers should provide information regarding the number of samples, analyses, etc. and if Saturday delivery is likely. If known, additional information regarding weekly shipping frequency is helpful to include.
- When scheduling a sampling event that will last for more than one week, it is recommended that the sampler contact the RSCC (or designee) on a weekly basis to provide updates. Communication between the sampler, the RSCC (or designee), and the Sample Management Office (SMO) is important as it ensures better availability of laboratory capacity.
- If the time frame or number of samples for a sampling event changes, the RSCC and SMO should be notified as soon as possible to maintain capacity at the scheduled laboratory.
- The Region should contact the Analytical Services Branch (ASB) in advance if it is suspected that the samples may contain asbestos.



The CLP has the capability to schedule sampling on an emergency basis; however, the sampler must contact the RSCC (or designee) to obtain details regarding how to handle such a situation.

### 7.2 Preparing for the Shipping of Samples

Once the samples are collected, they will be shipped to the CLP laboratory for analysis. Samplers must have the necessary shipping supplies on site.

#### 7.2.1 Procure Shipping Supplies

Samplers should refer to the appropriate project plans to determine the types of samples that will be collected during the sampling project to determine the necessary packaging materials to have at the site for all pertinent sample container types and sample matrices.

Samplers should also obtain the appropriate shipping paperwork (e.g., shipping forms required by the delivery service).



The CLP strongly discourages the use of vermiculite and cat litter as sources for packing material. These materials interfere with labeling and documentation and are difficult to remove from sample containers and shipping containers.

## 7.2.2 Laboratory Assignment Notification

Prior to beginning fieldwork, the samplers may either download an “Analytical Services Request Regional Notification” file via the Access Assignment Information task in the SMO Contract Laboratory Program Support Services (CLPSS) Portal, or obtain it from the RSCC.



The “Analytical Services Request Regional Notification” applies only to activities being performed under the CLP Statements of Work (SOWs).

## 7.2.3 Verify Laboratory Shipping Information

Samplers should verify the laboratory contact information, including the following:

- Laboratory name
- Laboratory address
- Contact name
- Laboratory phone number

This information, which is provided on the “Analytical Services Request Regional Notification”, is used to complete both the Traffic Report/Chain of Custody (TR/COC) Records and shipping paperwork such as address labels and airbills. This file may be accessed through the SMO CLPSS Portal, or can be obtained from the RSCC prior to sampling.

## 7.2.4 Obtain Shipping Company Information

Samplers should also verify the shipping company information, including the following:

- Company name
- Telephone number
- Account number
- Pickup schedule



Additional guidance will be provided by the U. S. Environmental Protection Agency (EPA) if samples are to be shipped internationally. Also, see Appendix F, International Shipping.

## 7.2.5 Prepare Sample Container Return Documentation

CLP laboratories must routinely return sample shipping containers to the appropriate sampling office within 14 calendar days following receipt of shipment from the sampler. To ensure that empty sample shipping containers are returned to the appropriate sampling office, samplers must complete the applicable container documentation and work with Regional personnel and government agencies to provide a cost-effective mechanism for this process. The sampling container return documentation should be prepared in advance and provided to samplers before field activities begin.



The sampler (not the CLP laboratory) is responsible for the cost of the return or disposal of the container and should also include shipping airbills bearing the sampler's account number, as well as a return address, to allow for container return.

To maintain consistency across shipping container transportation programs, samplers should proceed as follows:

- Minimize the use of multiple transportation carriers.
- Use multiple-copy labels so the laboratory and the sampling team can each retain a copy for their records.
- Prepare labels in advance so that the laboratory can simply affix a completed shipping label on the container.
- Include third-party billing information (i.e., their shipping account number) on labels so the laboratory will not be billed by the transportation carrier.
- Confirm that the laboratory has been notified of the transportation carrier being used.
- Include the SMO CLPSS Portal-generated CLP Case Number on the return information.

### 7.3 Collecting Samples

The CLP requirements for sample volumes, preservation, and contractual holding times are defined by the applicable CLP SOWs, and outlined in the tables in Appendix D.

Samplers should follow the sample collection requirements for analyses as provided in the following tables in Appendix D:

- Organic methods (Tables D-1 and D-2)
- Inorganic methods (Table D-3)
- High Resolution methods for Dioxins/Furans and Chlorinated Biphenyl Congeners (CBCs) (Table D-4).

For an explanation of the various sample types and the requirements for collecting and submitting each particular type, refer to Table 7-1.

**Table 7-1. Sample Types and CLP Submission Requirements**

Sample Type	Purpose	Collection <sup>1</sup>	CLP Sample Number
Field Sample	To analyze for target analytes of interest	Collect from areas that are known or suspected to be contaminated. Collect at the frequency specified in the Quality Assurance Project Plan (QAPP) and Sampling and Analysis Plan (SAP).	Assign CLP Sample Numbers to the sample.
Field Duplicate	To check reproducibility of laboratory and field procedures To indicate non-homogeneity	Collect from areas that are known or suspected to be contaminated. Collect at the frequency specified in the QAPP and SAP.	Assign two separate (unique) CLP Sample Numbers (i.e., one number to the field sample and one to the duplicate). Submit single blind to the laboratory.

Table 7-1. (Continued) Sample Types and CLP Submission Requirements

Sample Type	Purpose	Collection <sup>1</sup>	CLP Sample Number
Field Blank	To check cross-contamination during sample collection, preservation, and shipment, as well as in the laboratory Also to check sample containers and preservatives	Collect for each group of samples of similar matrix at the frequency specified in the QAPP and SAP. Organics - Use water (demonstrated to be free of the contaminants of concern). Inorganics - Use metal-free (deionized or distilled) water or a single clean wipe.	Assign separate CLP Sample Numbers to the field blanks. Submit single blind to the laboratory.
Filter Blank	To check contamination of samples from filtering procedure	Collect when water samples are filtered by filtering blank water using the same procedure and filtering equipment that is used for samples. Use blank water (water demonstrated to be organic-free, deionized or distilled for inorganics) and collect into sample containers.	Assign separate CLP Sample Numbers to the filter blanks. Submit single blind to the laboratory.
Temperature Blank	To provide an accurate measurement of field sample temperature upon arrival to the laboratory Also to establish whether the temperature range has been maintained while in transit	Collect for each shipping container with the frequency specified in the QAPP and SAP.	Ship together with samples from the field to the laboratory. A CLP Sample Number is not required.
Trip Blank [Volatile Organic Analyte (VOA) analysis Only]	To check contamination of VOA samples during handling, storage, and shipment from field to laboratory	Prior to going into the field, prepare and seal one trip blank sample per shipment per matrix. Trip blanks should be matched with respect to matrix and volume of the preservatives used. Prepare trip blank samples with the same preservatives and reagent water used for the corresponding samples. Carry each through the same sampling and handling protocols used for field samples. Aqueous trip blank samples should be prepared using water demonstrated to be free of the contaminants of concern (deionized water is appropriate). Place one trip blank sample for each matrix in each container used to ship VOA samples.	Assign separate CLP Sample Numbers to the trip blanks. Submit single blind to the laboratory.
Equipment Blank or Rinsate Blank	To check field decontamination procedures	Collect when sampling equipment is decontaminated and reused in the field or when a sample collection vessel (bailer or beaker) will be used. Use blank water (water demonstrated to be organic-free, deionized or distilled for inorganics) and pour rinse water into the sample containers.	Assign separate CLP Sample Numbers to the equipment blanks/rinsate. Submit single blind to the laboratory.

Table 7-1. (Continued) Sample Types and CLP Submission Requirements

Sample Type	Purpose	Collection <sup>1</sup>	CLP Sample Number
Matrix Spike (MS) and Matrix Spike Duplicate (MSD) (Organic Analysis Only)	To check accuracy and precision of organic analyses in specific sample matrices	Collect from areas that are known or suspected to be contaminated. MS/MSD are optional per Regional direction.	Assign the same CLP Sample Number to the field sample and the extra volume for MS/MSD. Identify the sample designated for MS/MSD on the TR/COC Record.
Matrix Spike (MS) and Laboratory Duplicate (Inorganic Analysis Only)	To check accuracy and precision of inorganic analyses in specific sample matrices	Collect from areas that are known or suspected to be contaminated. It is recommended that MS and Duplicates be collected in the first round of sampling and included in the first shipment of samples to the laboratory.	Assign the same CLP Sample Number to the field sample and extra volume (if collected). Identify the sample(s) designated for MS and Duplicates on the TR/COC Record.
Performance Evaluation (PE) Samples	Specially-prepared Quality Control (QC) samples used to evaluate a laboratory's analytical proficiency	The PE samples contain analytes with concentrations unknown to the laboratory. Designated Regional or authorized personnel (depending on Regional policy) arrange for Case-specific CLP PE samples to be prepared and shipped by the Quality Assurance Technical Support (QATS) contractor. The PE samples can be shipped to the site, or shipped per Regional direction. QATS provides the appropriate preparation instructions and chain of custody materials.	Samplers must order PE samples and ship them to the laboratory if required by the Region.  Assign separate CLP Sample Numbers to the PE samples. Include PE ampule numbers on the TR/COC Record.

<sup>1</sup> Consult Regional or Project Manager Guidance for field QC sample frequencies; laboratory QC sample frequencies are generally fixed in the laboratory contracts or specified in analytical methods.

### 7.3.1 Field QC Samples

Field QC samples are designed to assess variability of the media being sampled and to detect contamination and sampling errors in the field. The types of field QC samples that are generally collected include the following:

- Field duplicates
- Field blanks (such as equipment, trip, or rinse blanks)

Unless otherwise instructed, field duplicate samples should remain “blind” to the laboratory (i.e., they should have separate CLP Sample Numbers).

### 7.3.2 Laboratory QC Samples

A laboratory QC sample is an additional analysis of a field sample, as required by the laboratory's contract. There are three types of such samples:

- MS (for organic and inorganic samples)
- MSD (for organic samples only)
- Duplicates (for inorganic samples only)

Samplers should observe the following guidelines when collecting laboratory QC samples:

- Follow Regional guidance regarding the collection of laboratory QC samples.

- Regarding Gas Chromatography/Mass Spectrometry (GC/MS) organic analyses, a Region may decide to reference Deuterated Monitoring Compounds (DMCs) rather than MS/MSD for accuracy and precision.
- Wipe samples do not require laboratory QC samples.
- When laboratory QC is scheduled for CLP analyses, samplers should select one sample per matrix per Sample Delivery Group (SDG). An SDG closes when 20 samples are received for a Case or one week (3 days for 7-day turnaround samples) has elapsed, whichever comes first. Designate MS/MSD for Pesticides, Aroclors, and GC/MS analyses as directed. For GC/MS, MS/MSD are optional per Regional direction. Designate MS and laboratory Duplicate for metals, mercury, cyanide, anions, hexavalent chromium, and Total Organic Carbon (TOC). Samplers should not select a field blank or a PE Sample for laboratory QC.
- Designated samples should be identified on the TR/COC Record; the sample(s) designated for laboratory QC should be noted in “Sample Type” column.
- QC samples should be shipped in the same container as the field samples when possible.



Field QC samples should not be designated as laboratory QC samples.



In the event of multiple sample shipments during a sampling event, it is recommended that the sampler submit laboratory QC samples in the first sample shipment, and as necessary in subsequent shipments to meet laboratory contract requirements.

## 7.4 Recording Samples

Samplers must use Scribe to record the samples that are collected. To record the samples:

- Access the Scribe **Sampling** tab to select the type of sampling (Water Sampling, Soil/Sediment Sampling, Wipe Sampling).
- Enter the detailed information for the sample. Include laboratory analysis for each sample; see tabs for sample details and for analysis.
- When all information has been entered, click the **Close** button at the bottom of the page to save the entries and close the window.

Refer to Table 5-1 for the CLP analysis codes.

For assistance while using the Scribe software, contact the Environmental Response Team (ERT) Software Support Help Desk at 800-999-6990 from 9:00 AM – 5:00 PM ET. Refer to the following website for information on the use and training of Scribe:

[https://response.epa.gov/site/site\\_profile.aspx?site\\_id=ScribeGIS](https://response.epa.gov/site/site_profile.aspx?site_id=ScribeGIS)

### 7.4.1 Hardcopy Recording

In the event that Scribe is unavailable, samplers must have backup hardcopy Scribe TR/COC Records. This should be done only in cases of power/equipment failure, and not as a matter of routine during a sampling event.

## 7.5 Meeting Volume, Preservation, and Holding Time Requirements

Samplers should refer to Tables D-1 through D-4 in Appendix D to obtain the specific sample volumes to be collected, the preservation needed for those samples, and the technical holding times under which they must submit samples to the scheduled CLP laboratory.

### 7.5.1 Collect Required Sample Volumes

Samplers should ensure that a sufficient volume is collected for each sample. If the sample volume does not meet the requirement set by the project plan, the laboratory may not be able to analyze the sample correctly. Questions regarding collecting lesser volumes than those listed in Tables D-1 through D-4 in Appendix D should be directed to ASB.

Refer to Appendix B for information regarding the collection of aqueous VOA samples. When collecting samples for VOA in soils, samplers must use the SW-846 Method 5035A guidelines included in Appendix C.



If tissue samples are homogenized or processed, it should be performed at a sample processing facility under clean room conditions to reduce potential contamination. Tissue samples should be packed and cooled on ice immediately. Tissue samples should never be sent on Friday for Monday delivery.

### 7.5.2 Preserve Samples

Without preservation, some samples (e.g., VOAs) may degrade to the point that they will not provide accurate results for the site. The sampler must chemically preserve some water samples for certain analytes prior to shipping them to the laboratory.

Samplers should observe the following procedures:

- Note any visible reaction between the sample and added chemical preservative in the field record.
- Preserve and immediately cool all organic, cyanide, hexavalent chromium, and anions (nitrate, nitrite, orthophosphate, and sulfate) water samples to  $\leq 6^{\circ}\text{C}$ , but not frozen, upon collection.
- Keep samples cooled until shipping (do not freeze water samples).
- Preservation techniques vary among Regions, so samplers should obtain Region-specific instructions and review the appropriate project plans and Standard Operating Procedures (SOPs).
- Refer to Appendix B for information regarding the collection of VOA water samples.

### 7.5.3 Ship Samples within Holding Times

There are two types of holding times: technical and contractual.

- **The technical holding time** is the maximum amount of time allowed between sample collection and the completion of sample extraction and/or analysis.
- **The contractual holding time** is the maximum amount of time that the CLP laboratory can hold a sample prior to extraction and/or analysis. The contractual holding time is the elapsed time expressed in days from the date of receipt of the sample by the laboratory until the date of its extraction and/or analysis, as specified in the applicable CLP SOW.



Contractual holding times are generally set to be two days less than the technical holding times to allow for sample packing and shipping.

**Samplers should ship samples to scheduled CLP laboratories as soon as possible after collection.**

- Ship samples daily to CLP laboratories whenever possible.
- Samples for certain anions (nitrate, nitrite, and orthophosphate) must ship the same day to meet holding times.
- If samples cannot be shipped on a daily basis, they must be properly preserved and maintained to meet CLP-specified temperatures, holding times, and custody requirements.
- Uploading the electronic TR/COC Record is mandatory and should be performed as soon as possible after shipping.



If samplers are shipping samples after 5:00 PM ET, they should notify the RSCC (or designee) and SMO by 8:00 AM ET on the following business day. When making a Saturday delivery, samplers should notify the RSCC (or designee) and SMO as soon as possible so that SMO will receive the delivery information (including shipping airbill information) by 3:00 PM ET on the Friday prior to delivery. Uploading the electronic TR/COC Record to the SMO CLPSS Portal sends a notification to SMO and to the laboratory that the samples have been shipped.

## 7.6 Completing the Documentation

The sample documentation required is defined by the project plan. It is highly recommended that samplers provide documentation, even if the Region does not require it.

In general, samplers must complete the following documentation for the samples collected:

- CLP Sample Number (on the sample container or bottle)
- Sample label
- Chain of custody seals (as appropriate)
- TR/COC Record
- Field operations records (as necessary)



Under no circumstances should the site name or address appear on any documentation being sent to the laboratory, unless the laboratory is a Regional EPA laboratory.

An example of a packaged sample is shown in Figure 7-1.



**Figure 7-1. Packaged Sample with Identification and Chain of Custody Documentation (Excluding TR/COC Record)**

### 7.6.1 Record and Label the Samples

The sample labels created in Scribe must be affixed to each sample container. A sample label contains the following information:

- Associated CLP Sample Number (either written or pre-printed)
- SMO CLPSS Portal-generated CLP Case Number
- Preservative used
- Analysis
- Additional information such as the location or the date/time of collection

Samplers should record and label the samples collected as follows:

- Using Scribe, select the **Sampling** tab to select the type of matrix (e.g., Soil/Sediment, Water Sampling).
- Access the Scribe **Sample Details** page to enter the analysis method, CLP Sample Number, and SMO CLPSS Portal-generated CLP Case Number for each sample.
- Enter samples associated with an MA by using the MA analysis previously created in Scribe. If the MA does not exist, refer to Section 5.1.2, Create Analysis Types, to create the analysis type for the MA.
- Print the sample label and place it on the sample container or bottle.
- If handwriting a sample label, complete the information using waterproof ink, place the label on the outside of the sample bottle or container, then cover the label with clear packaging tape to protect it and maintain legibility.
- Avoid wrinkles in the tape and labels.
- Sample tags are NOT required by the CLP.
- Refer to Figure 7-1 for an example of sample label placement.

Use the following guidelines for these special conditions:

- **Water samples collected for total metals and filtered metals analyses from the same sampling location** - must assign separate (unique) CLP Sample Numbers.
- **Tared VOA sample vials** - must not attach labels to tared VOA sample vials.

## 7.6.2 Complete the COC Records in Scribe

Samplers should use the Scribe format for CLP projects. Complete the Scribe COC Record as follows:

### Access the Scribe COC Page

- Select the **Chain of Custody** link under the **Sample Management** header. The *Chain of Custody* page displays.

### Create the COC Record

- Click the **Add a Chain of Custody** button at the bottom of the page. The *COC Details* pop-up window displays.
- Enter the information for the COC, including selecting the **CLP format** (Inorganic, Organic, or High Resolution).

**Note:** It is important that the correct **COC Format** is selected when the COC Record is created. The user should choose the CLP format for the type of samples being submitted, as shown in Figure 7-2.

**Figure 7-2. COC Details Pop-up Window**

- Ensure that the **Case #** is also filled in. (If it was entered in the CLP/Tag Settings, it will automatically be filled in.)

### Assign Samples to the COC Record

- Assign samples to the **COC Record** (it will filter based on selected COC format SOW).
- Ensure that all sample information has been entered.
- Enter any additional information, such as sampler name, matrix, and preservation.
- Indicate any samples that will be analyzed using an MA.
- Scribe generates a laboratory and a Regional copy of the COC Record (see Figures 7-3 and 7-4).

### Print the COC Record

- Print the **COC Record** by selecting either the **Lab Copy** or **Region Copy**. There will be a QC check; ensure that all information is filled in.
- Print as many copies of the **COC Record** as is necessary.
- Sign and submit original copies of the **COC Record**.



Sampler information, etc., is not added when creating the COC Record; it is added when editing the sample itself.

### 7.6.3 Making Manual Edits to Printed Scribe COC Records

If a Scribe COC Record has been printed and deletions or edits need to be made, samplers must use the following procedures:

- If making a deletion, delete the incorrect information in Scribe, reprint the COC Record, and discard the original.
- If making an addition, enter the new information in Scribe, reprint the COC Record, and discard the original.
- If corrections occur after shipment, adhere to the Regional procedures and guidelines for handling hardcopy COC Records.

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USEPA CLP COC (LAB COPY)

CHAIN OF CUSTODY RECORD

No: 2-010614-124708-0001

Date Shipped: 1/3/2020

Lab: EPA Labs

Carrier Name: FedEx

Case #: 21490

Lab Contact: John Smith

Airbill No: ABC12345

Cooler #:

Lab Phone:

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	For Lab Use Only
12345-0001	B0AA0	Soil/ EPA	Grab	SVOA(21)/PR, SVOA 1723.3(21)/PR, PEST(21), ARO(21), VOA(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	ABC	01/03/2020 08:00	
12345-0002	B0AA1	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	ABC	01/03/2020 08:00	
12345-0003	B0AA2	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	DEF	01/03/2020 09:00	
12345-0004	B0AA3	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	GHI	01/03/2020 10:00	
12345-0005	B0AA4	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	JKL	01/03/2020 11:00	
12345-0006	B0AA5	Soil/ EPA	Grab	SVOA(21), SVOA 1723.3(21), PEST(21), ARO(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	DEF	01/03/2020 09:00	
12345-0007	B0AA6	Soil/ EPA	Grab	SVOA(21), SVOA 1723.3(21), PEST(21), ARO(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	GHI	01/03/2020 10:00	
12345-0008	B0AA7	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	JKL	01/03/2020 11:00	

Sample(s) to be used for Lab QC: 12345-0001, 12345-0001, 12345-0001, 12345-0001, 12345-0001, 12345-0001	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: SVOA-CLP Semivolatiles, SVOA 1723.3-CLP SVOA MA 1723.3, PEST-CLP Pesticides, ARO-CLP Aroclors, VOA-CLP Volatiles, VOA MA-CLP VOA (MA 1722.4)	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt

Figure 7-3. Scribe Chain of Custody Record (Laboratory Copy)

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USEPA CLP COC (REGION COPY)

CHAIN OF CUSTODY RECORD

No: 2-010614-124708-0001

Date Shipped: 1/3/2020

Field Sampler's Guide

Lab: EPA Labs

Carrier Name: FedEx

Case #: 21490

Lab Contact: John Smith

Airbill No: ABC12345

Cooler #:

Lab Phone:

Sample Identifier	CLP Sample No.	Matrix/Sampler	Coll. Method	Analysis/Turnaround (Days)	Tag/Preservative/Bottles	Location	Collection Date/Time	Sample Type
12345-0001	B0AA0	Soil/ EPA	Grab	SVOA(21)/PR, SVOA 1723.3(21)/PR, PEST(21), ARO(21), VOA(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	ABC	01/03/2020 08:00	Lab QC
12345-0002	B0AA1	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	ABC	01/03/2020 08:00	Field Sample
12345-0003	B0AA2	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	DEF	01/03/2020 09:00	Field Sample
12345-0004	B0AA3	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	GHI	01/03/2020 10:00	Field Sample
12345-0005	B0AA4	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	JKL	01/03/2020 11:00	Field Sample
12345-0006	B0AA5	Soil/ EPA	Grab	SVOA(21), SVOA 1723.3(21), PEST(21), ARO(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	DEF	01/03/2020 09:00	Field Sample
12345-0007	B0AA6	Soil/ EPA	Grab	SVOA(21), SVOA 1723.3(21), PEST(21), ARO(21), VOA MA(21)	(0 C), (0 C), (0 C), (0 C), (0 C), (0 C) (6)	GHI	01/03/2020 10:00	Field Sample
12345-0008	B0AA7	Soil/ EPA	Grab	ARO(21), PEST(21), SVOA(21)	(0 C), (0 C), (0 C), (0 C) (4)	JKL	01/03/2020 11:00	Field Sample


Sample(s) to be used for Lab QC: 12345-0001, 12345-0001, 12345-0001, 12345-0001, 12345-0001, 12345-0001	Shipment for Case Complete? Y
	Samples Transferred From Chain of Custody #
Analysis Key: SVOA=CLP Semivolatiles, SVOA 1723.3=CLP SVOA MA 1723.3, PEST=CLP Pesticides, ARO=CLP Aroclors, VOA=CLP Volatiles, VOA MA=CLP VOA (MA 1722.4)	

Items/Reason	Relinquished by (Signature and Organization)	Date/Time	Received by (Signature and Organization)	Date/Time	Sample Condition Upon Receipt

Figure 7-4. Scribe Chain of Custody Record (Region Copy)

## 7.6.4 Complete and Attach Custody Seals

Custody seals are usually pre-printed stickers that are signed (or initialed) and dated by the samplers after sample collection and placed on sample bottles or containers and/or shipping containers (see Figure 7-5).

 UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL	SAMPLE NO.	DATE	SEAL BROKEN BY	DATE
	SIGNATURE			
	PRINT NAME AND TITLE			

**Figure 7-5. Custody Seal**

The custody seal documents the individual who sealed the sample container and verifies that the sample has not been tampered with. Custody seals can also be used to maintain custody of other items such as envelopes.

The use and type of custody seals can vary by Region or collecting organization. Samplers should obtain the appropriate custody seals and specific instructions for their use from the RSCC. Note that some Regions require the sampling team to provide their own custody seals.

- Custody seals shall be placed in such a manner that they will break if the sample bottle or container, or the shipping cooler or container is tampered with or opened after leaving custody of the samplers.
- Custody seals should never be placed directly onto a coring tool used as a transport device (e.g., 5 g Sampler) or tared, 40 mL closed-system vials. The seals must be placed on the bag for the coring tool used as a transport device, or on the bag used to enclose the vials. Refer to Table 8-1 for details.

Instructions for completing and attaching a custody seal are included in Table 7-2.

**Table 7-2. Completing and Attaching a Custody Seal**

Step	Action	Important Notes
1	Record the CLP Sample Number.	The space for the CLP Sample Number does not need to be completed on custody seals being placed on the opening of a shipping container, only on those being placed on the opening of sample bottles or containers.
2	Record the month, day, and year of sample collection.	
3	Sign the seal in the signature field.	
4	Print your name and title in the "Print Name and Title" field.	
5	Place the custody seal over the edge of the sample bottle or container such that it will break if tampered with.	Custody seals can be placed directly on any sample container except for coring tools used as a transport device (e.g., 5 g Samplers) and tared VOA bottles. If packing coring tools used as a transport device or tared VOA bottles, place them in a clear plastic bag and place the custody seal on the outside of the bag.
6	If possible, cover the custody seal with clear plastic tape to protect it.	Take special care to not place the protective tape over the seal in such a way that it can be removed and then re-attached without signs of tampering.



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## 8.0 CLP SAMPLE TRANSPORTATION AND SHIPPING

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The sampling organization is not only responsible for the transportation and shipping of the Contract Laboratory (CLP) samples to the scheduled recipient laboratory that will be performing the analysis, but it is also responsible for complying with all applicable packaging, labeling, and shipping requirements. Samplers should review the applicable project plans to be aware of all State, Federal, Department of Transportation (DOT), and International Air Transport Association (IATA) regulations governing environmental and hazardous sample packaging.



### 8.1 Providing Shipment Notification

Some Regions may require that samplers notify their Regional Sample Control Coordinator (RSCC) (or designee) when samples are shipped, while other Regions may allow samplers to contact the Sample Management Office (SMO) directly to provide shipping notification. It is recommended that the electronic Chain of Custody (COC) Record be submitted through the Contract Laboratory Program Support System (CLPSS) (aka “the SMO CLPSS Portal”) as soon as possible after shipping. Submitting the COC Record electronically sends a notification to SMO and to the laboratory that the samples have been shipped. It is recommended that samplers contact the RSCC to verify if such notification is necessary. If samplers are shipping samples after 5:00 PM ET, they should notify the RSCC (or designee) and SMO by 8:00 AM ET on the following business day.

It is strongly recommended that samplers provide shipping notification to the RSCC even if they have received approval to directly notify SMO so that the Region is aware of any changes in the final number and timing of samples delivered.

### 8.2 Packing and Shipping Samples

After collecting the samples, it is important that the samplers properly package them for shipment and ensure that the samples are sent to the appropriate laboratory without delay. Prompt and proper packaging of samples will achieve the following:

- Protect the integrity of samples from changes in composition or concentration caused by bacterial growth or degradation from increased temperatures.
- Reduce the chance of leaking or breaking of sample containers that would result in loss of sample volume, loss of sample integrity, and exposure of personnel to toxic substances.
- Help ensure compliance with shipping regulations.

A single CLP sample may be contained in several different bottles and vials. For example, one water sample may consist of all containers needed for three of the analytical analyses available under this service [i.e., Semivolatile Organic Analyte (SVOA) analysis, Pesticide analysis, and Aroclor analysis], even though the analyses are collected in separate containers. Therefore, the analysis to be performed and the matrix type will determine the type of container(s) that will be used, as well as the volume that should be collected for that particular sample analysis.

## 8.2.1 Inventory of Samples and Documentation

Samplers should inventory the contents of shipping containers against the corresponding Traffic Report/Chain of Custody (TR/COC) Records when packing samples for shipment to laboratories. Check for the following:

- Ensure that the correct number of containers has been collected for each analysis of the samples.
- Confirm that the required Performance Evaluation (PE) and Quality Control (QC) samples and temperature blanks are included in the shipment.
- Verify that the correct sample numbers and analyses have been assigned to each sample.

## 8.2.2 Shipping Regulations

Sample shipping personnel are legally responsible for ensuring that sample shipments comply with all applicable shipping regulations. Verify that the following shipping regulations are adhered to if any of the following conditions apply to the samples:

Note: Field Samplers are legally required to know sample characteristics before shipping.

- ❑ **Foreign soil movement** - Follow U.S. Department of Agriculture (USDA) soil quarantine and shipping requirements, providing soil permits or required soil import agreements to the laboratory for completion.
- ❑ **Chlorinated Dibenzo-*p*-Dioxin (CDD) and Chlorinated Dibenzofuran (CDF)** - Refer to the High Resolution Superfund Method Statement of Work (SOW) for specific information on the safety and handling requirements for samples potentially containing CDD/CDF.
- ❑ **Radiological** - Samples suspected to be radioactive must be screened; follow the instructions from the Analytical Services Branch (ASB) Program Manager. Radioactive/Radiological samples are NOT accepted by the CLP.
- ❑ **Dry ice** - If dry ice is used to ship tissue samples, follow DOT and IATA regulations. Refer to the Code of Federal Regulations (49 CFR 173.217) classifying dry ice as Hazard Class 9 *UN 1845* (Hazardous Material) and IATA Dangerous Goods regulations or DOT regulations and U.S. Environmental Protection Agency (EPA) guidelines. Refer to Appendix C for detailed shipping guidelines when using SW-846 Method 5035A to preserve and ship frozen samples.



When shipping from remote locations, dry ice may be used with water ice for the purpose of keeping the water ice from melting. Wrap the dry ice in newspaper and place above any water ice. Never place dry ice in an air-tight container.

Access more transportation and shipping information using the following websites:



### Dangerous goods regulations

#### IATA website

<https://www.iata.org/en/programs/cargo>

#### DOT/Pipeline and Hazardous Materials Safety Administration (PHMSA)

<https://www.phmsa.dot.gov/phmsa-regulations>

The nature of the samples collected determines the type of shipping materials to be used.

Refer to the project plan to determine which type of shipping container should be used for each type of sample collected during the sampling event.

### 8.2.3 Shipping Temperature

Samples must be stored under conditions that maintain sample integrity.

- Samples requiring cold preservation should be placed in shipping containers or other suitable containers with ice to reduce the temperature as soon as possible after collection.
- Ideally, all samples should be shipped the day of collection for overnight delivery to the laboratory.
- If samples requiring cold preservation cannot be shipped on the day of collection, the sample temperature should be maintained at  $\leq 6^{\circ}\text{C}$ , but not frozen, until they are shipped to the laboratory.

### 8.2.4 Pack Shipping Containers

Packing shipping containers correctly will prevent sample containers from breaking and leaking. Pack shipping containers according to the instructions outlined in Table 8-1.

**Table 8-1. Packing Samples for Shipment**

Step	Action	Important Notes
1	Seal all drain holes in the shipping container, both inside and out, to prevent leakage in the event of sample breakage.	
2	Check all lids/caps to make sure the samples are tightly sealed and will not leak.	
3	Wipe loose soil residue from containers.	
4	Seal samples in a clear plastic bag.	Custody seals can be placed directly on any sample container except for coring tools used as a transport device (e.g., 5 g Samplers) and tared Volatile Organic Analyte (VOA) bottles. If packing coring tools used as a transport device or tared VOA bottles, place them in a clear plastic bag and affix the custody seal to the outside of the bag.
5	Fully chill those samples that require chilling to $\leq 6^{\circ}\text{C}$ (but not frozen) prior to placing in the container with suitable packing materials.	
6	It is recommended that samplers line shipping containers with non-combustible, absorbent packing material prior to placing samples in the shipping container.	The CLP strongly discourages the use of vermiculite and cat litter as sources for packing material. These materials interfere with labeling and documentation and are difficult to remove from sample containers and shipping containers.
7	Place samples in CLEAN, sealed, watertight shipping containers (e.g., metal or hard plastic coolers).	
8	Conduct an inventory of the contents of the shipping cooler/container against the corresponding TR/COC Record(s).	
9	Cover samples in double-bagged ice to prevent water damage to packing materials.	Do NOT pour loose ice directly into the sample container. The ice is used to maintain the temperature of the samples in the shipping cooler.
10	It is recommended that a temperature blank be included each shipping container in a location which will allow for easy access by the laboratory upon opening the shipping container.	The temperature blank is generally a 40 mL vial filled with water and labeled "temperature blank" but does not have a CLP Sample Number.
11	Ensure that the site name or other site-identifying information does not appear on any documentation being sent to the laboratory.	

**Table 8-1. (Continued) Packing Samples for Shipment**

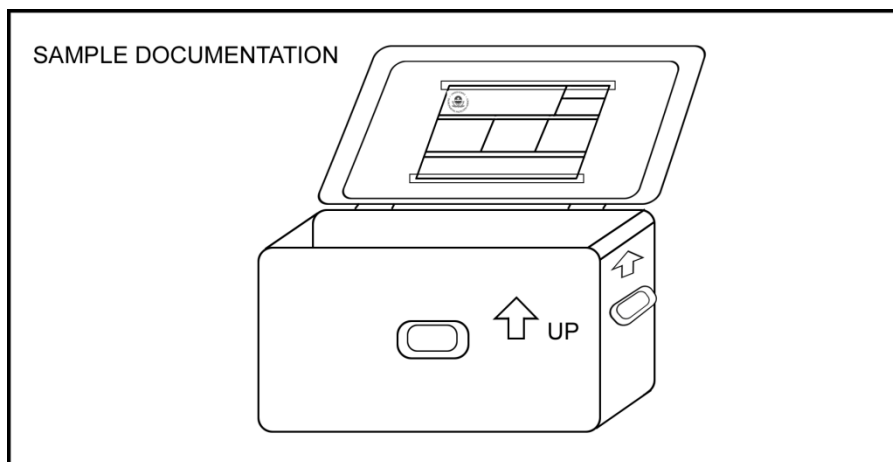
Step	Action	Important Notes
12	Label the outside of the shipping container with any instructions for handling, such as, "This end up," "Do not Tamper With," or "Environmental Laboratory Samples."	
13	If shipping samples containing methanol as a preservative (e.g., samples to be analyzed by SW-846 Method 5035A), use a label to indicate the presence of methanol, the United Nations (UN) identification number for methanol (UN 1230), and Limited Quantity.	



VOA soil samples must be placed on their side prior to being placed on ice. Vials are placed on their side so that the septum is wet on the inside, thereby preventing vapor leaks around it, in case any bubbles form. Also, in case the samples freeze, the water will expand into the flexible septum rather than breaking the vial.

### 8.2.5 Include Required Paperwork

- Attach the necessary paperwork to the shipping cooler or container. All paperwork must be placed in a plastic bag or pouch and then secured to the underside of the shipping container lid (Figure 8-1).



**Figure 8-1. Sample Shipping Container with Attached TR/COC Record, PE Sample Instructions (if applicable), and Container Return Documentation**

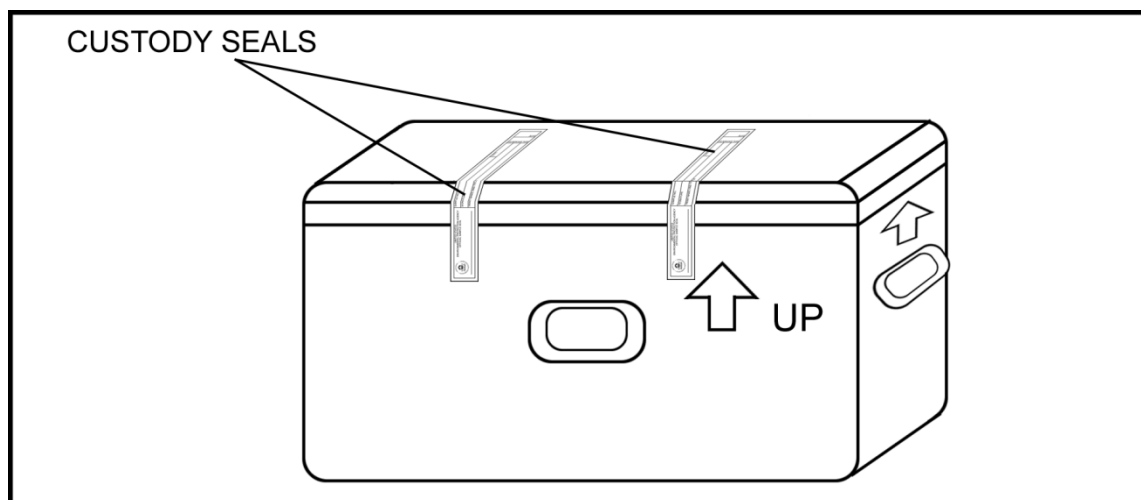
Required paperwork includes:

- TR/COC Records
- Sample weight logs (Figure 4-1), if required for VOA samples
- PE instruction sheets if PE samples are included in the container
- Shipping container return instructions and/or airbills

Contact the RSCC (or designee) for specific paperwork requirements.

## 8.2.6 Label and Seal Sample Shipping Containers

After packing the samples in the shipping containers, samplers must carefully secure the top and bottom of the containers with tape, place return address labels clearly on the outside of the container, and attach the required chain of custody seals (Figure 8-2).



**Figure 8-2. Shipping Container with Custody Seals**

Use the following guidelines to label shipping containers:

- ❑ When shipping more than one container to a laboratory, samplers should mark each container as “1 of 2,” “2 of 2,” etc.
- ❑ An airbill, addressed to the Sample Custodian of the receiving laboratory, must be completed for each container shipped. Samplers should receive the correct name, address, and telephone number of the laboratory to which they must ship samples from the RSCC or the SMO CLPSS Portal.
- ❑ To avoid delays in analytical testing, it is critical that for samples associated with multiple types of analyses the correct containers with the correct types of samples are sent to the correct laboratory. For example, inorganic samples may be shipped to one laboratory for analysis, while organic samples may need to be shipped to another laboratory.
- ❑ Confirm the shipping company’s hours of operation, shipping schedule, and pick-up/drop-off requirements.

## 8.2.7 Overnight Delivery

It is imperative that samples be sent via overnight delivery. Delays due to longer shipment times may result in missed technical holding times or temperatures rising above the preservation limit, which may destroy sample integrity or require the re-collection of samples for analysis.

## 8.2.8 Saturday Delivery

Samplers should notify the RSCC (or designee) and SMO as soon as possible when shipping samples for Saturday delivery, so that the delivery information is received by 3:00 PM ET on the Friday prior to delivery.

## 8.2.9 Shipment Notification

Samplers should upload shipment information to the SMO CLPSS Portal and report all sample shipments to the RSCC (or designee) on the day shipping is completed. **Under no circumstances**

**should the sampler contact the laboratory directly.** If samplers are shipping samples after 5:00 PM ET, they should notify the RSCC (or designee) or SMO by 8:00 AM ET on the following business day. Samplers should receive the name, phone number, and email address of the appropriate SMO coordinator to notify from the Region/RSCC. Samplers should be aware if the Region requires them to notify the RSCC (or designee) and SMO of sample shipment.

Samplers should provide the following information to the RSCC (or designee):

- Name, phone number, and email address at which they can be reached (preferably closest on-site phone number if still in the field)
- SMO CLPSS Portal-generated Case Number
- Number, matrix and analysis types of samples shipped, and MA number (if required)
- Name of laboratory (or laboratories) to which the samples were shipped
- Airbill number(s)
- Date of shipment
- Case status (i.e., whether or not the Case is complete)
- Problems encountered, special comments, or any unanticipated issues
- Information for the next anticipated shipment



For Saturday delivery, samplers should notify the RSCC (or designee) and SMO as soon as possible so that SMO will receive the delivery information (including shipping airbill information) by 3:00 PM ET on the Friday prior to delivery. Uploading the electronic COC to the SMO CLPSS Portal sends a notification to SMO and to the laboratory that the samples have been shipped.

## 8.2.10 Uploading the Electronic COC

The electronic COC Record must be uploaded to the SMO CLPSS Portal as soon as possible after sample shipment. The following is an overview of the steps used to upload the electronic COC Record:

- Using **Scribe**:
  - Under **Sample Management**, click on the **Chain of Custody** link.
  - Click the **Export** button on the top of the menu bar.
  - Select the **COC XML File (.xml)** option.
  - Select the **COC(s)** to be exported.
  - Make sure that the **CLP Region Copy COC XML Template** is checked.
  - Click **OK**.
  - Provide a **filename** for the exported XML file. Per CLP guidance, the XML file name must reference the Region #, Case Number, and the date.
  - Click the **Save** button.
- Using the **SMO CLPSS Portal**:
  - Select the **Submit Chain of Custody** task.
  - **Select** the COC file to upload.
  - Enter any **comments** associated with the COC file.
  - **Submit** the file; CLPSS provides a confirmation page.
  - **Print or download** a copy of the submission summary to keep as a record of the submission.



For a detailed description of how to create and upload electronic COC Record files using Scribe and the SMO CLPSS Portal, refer to each system's user documentation.

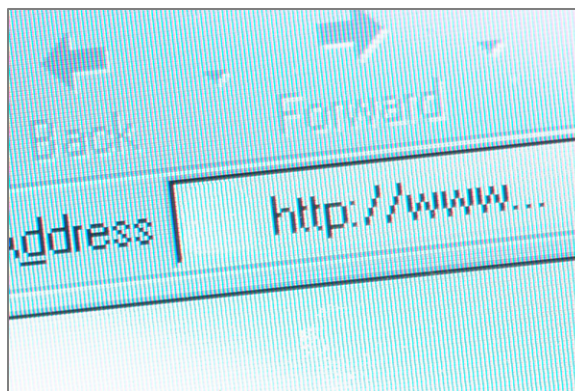
### 8.2.11 Return of Sample Shipping Containers

CLP laboratories must routinely return sample shipping containers within 14 calendar days following shipment receipt. Therefore, the sampler should also include container return instructions with each shipment. Note that the samplers (not the CLP laboratory) are responsible for the costs of return or disposal of these containers and should include shipping airbills bearing the sampler's account number, as well as a return address. **Samplers should use the least expensive return shipping option possible.**

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## 9.0 SAMPLER RESOURCES

This Guidance document provides a summary of the many resources used to define and manage the sampling process. Samplers may need to refer to the original source documents or websites for further information or clarification. The resources cited herein are listed in this section.



### 9.1 List of Resources

Table 9-1 provides a list of the resources available to samplers and referenced throughout this Guidance document.

**Table 9-1. Resources for Samplers**

Resource	Location
<b>U. S. Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) Statements of Work (SOWs)</b>	<a href="https://www.epa.gov/clp/superfund-clp-analytical-statements-work-sows">https://www.epa.gov/clp/superfund-clp-analytical-statements-work-sows</a>
<b>Scribe Software Support</b>	<a href="https://ertsupport.org/scribe">https://ertsupport.org/scribe</a>
<b>Contract Laboratory Program Support System (CLPSS)</b>	<a href="https://smoclpss.com/uaa/login">https://smoclpss.com/uaa/login</a>
<b>EPA Environmental Response Team (ERT) User Manual for Scribe Contract Laboratory Program (CLP) Sampling</b>	<a href="http://www.epaosc.org/sites/ScribeGIS/files/Scribe%20CLP%20User%20Guide.pdf">http://www.epaosc.org/sites/ScribeGIS/files/Scribe%20CLP%20User%20Guide.pdf</a>
<b>CLP Guidance Documents</b>	<a href="https://www.epa.gov/clp/superfund-clp-analytical-services-guidance-documents-documents">https://www.epa.gov/clp/superfund-clp-analytical-services-guidance-documents-documents</a>
<b>Department of Transportation (DOT) Pipeline and Hazardous Materials Safety Administration (PHMSA) regulations</b>	<a href="https://www.phmsa.dot.gov/phmsa-regulations">https://www.phmsa.dot.gov/phmsa-regulations</a>
<b>Use of Dry Ice - Federal Regulations (49CFR 173.217) classified dry ice as Hazard Class 9 UN 1845 (Hazardous Material).</b>	<a href="https://www.gpo.gov/fdsys/pkg/CFR-2004-title49-vol2/xml/CFR-2004-title49-vol2-sec173-217.xml">https://www.gpo.gov/fdsys/pkg/CFR-2004-title49-vol2/xml/CFR-2004-title49-vol2-sec173-217.xml</a>
<b>International Air Transport Association (IATA) transportation regulations</b>	<a href="https://www.iata.org">https://www.iata.org</a>
<b>United States Department of Agriculture (USDA) Regulated Organisms and Soil Permits</b>	<a href="https://www.aphis.usda.gov/plant_health/permits/organism/soil/">https://www.aphis.usda.gov/plant_health/permits/organism/soil/</a>
<b>Common Ground Alliance - marking for underground utilities</b>	<a href="https://call811.com">https://call811.com</a>

Table 9-1. (Continued) Resources for Samplers

Resource	Location
<b>Water Sampling Requirements of Dissolved Metals determinations</b>	<p><b><i>EPA Method SW-846 3005A – Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Section 2.2:</i></b></p> <p>Dissolved Metals - The sample is filtered through a 0.45-µm filter at the time of collection and the liquid phase is then acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested as long as the acid concentrations have been adjusted to the same concentration as in the standards.</p> <p><a href="https://www.epa.gov/sites/production/files/2015-12/documents/3005a.pdf">https://www.epa.gov/sites/production/files/2015-12/documents/3005a.pdf</a></p>
	<p><b><i>Clean Water Act (CWA), §136.3 Identification of test procedures. Table II – Required Containers, Preservation Techniques, and Holding Times</i></b></p> <p>Footnote 7: For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler (e.g., using a 24-hour composite sampler; see 40 CFR 122.21(g)(7)(i) or 40 CFR Part 403, Appendix E), filter the sample within 15 minutes after completion of collection and before adding preservatives. If it is known or suspected that dissolved sample integrity will be compromised during collection of a composite sample collected automatically over time (e.g., by interchange of a metal between dissolved and suspended forms), collect and filter grab samples to be composited (footnote 2) in place of a composite sample collected automatically.</p> <p><a href="https://www.federalregister.gov/documents/2017/08/28/2017-17271/clean-water-act-methods-update-rule-for-the-analysis-of-effluent">https://www.federalregister.gov/documents/2017/08/28/2017-17271/clean-water-act-methods-update-rule-for-the-analysis-of-effluent</a></p>
<b>EPA Method SW-846 5035A - Closed-System Purge-and-Trap Extraction for Volatile Organics in Soil and Waste Samples</b>	<p><a href="https://www.epa.gov/sites/production/files/2015-12/documents/5035a_r1.pdf">https://www.epa.gov/sites/production/files/2015-12/documents/5035a_r1.pdf</a>.</p>
<b><i>NIOSH/OSHA/USCG/EPA Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities</i></b>	<p><a href="https://www.osha.gov/Publications/complinks/OSHG-HazWaste/4agency.html">https://www.osha.gov/Publications/complinks/OSHG-HazWaste/4agency.html</a></p>
<b><i>The Roles of Project Managers and Laboratories in Maintaining the Representativeness of Incremental and Composite Soil Samples, EPA/OSWER 9200.1-117FS</i></b>	<p><a href="https://www.clu-in.org/download/char/RolesofPMsandLabsinSubsampling.pdf">https://www.clu-in.org/download/char/RolesofPMsandLabsinSubsampling.pdf</a></p>

## 9.2 For More Information

For additional information regarding the CLP or this Guidance document, refer to the Superfund Analytical Services/Contract Laboratory Program website at: <https://www.epa.gov/clp>

## APPENDIX A FUNCTIONS WITHIN A SAMPLING PROJECT

The following table describes Quality Assurance Project Plan (QAPP) sampling requirements based on *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5).

**Table A-1. QAPP Requirements**

Functions Within a Sampling Project	Elements of that Function
<b><i>Project Management</i></b>	
Project/Task Organization	Identifies the individuals or organizations participating in the project and defines their specific roles and responsibilities.
Problem Definition/Background	States the specific problem to be solved or decision to be made and includes sufficient background information to provide a historical and scientific perspective for each particular project.
Project/Task Description	Describes the activities to be performed and the schedule for implementation to include: <ul style="list-style-type: none"> <li>• Measurements to be made during the course of the project.</li> <li>• Applicable technical, regulatory, or program-specific quality standards, criteria, or objectives.</li> <li>• Any special personnel and equipment requirements; assessment tools needed.</li> <li>• A work schedule and any required project and quality records, including types of reports needed.</li> </ul>
Quality Objectives and Criteria	Describes the project quality objectives and measurement performance criteria.
Special Training/Certification	Ensures that any specialized training required for modified field sampling techniques, field analyses, laboratory analyses, or data validation should be specified.
Documents and Records	<ul style="list-style-type: none"> <li>• Itemizes the information and records that must be included in the data report package and specifies the desired reporting format for hardcopy and electronic forms, when used.</li> <li>• Identifies any other records and/or documents applicable to the project such as audit reports, interim progress reports, and final reports that will be produced.</li> <li>• Specifies or references all applicable requirements for the final disposition of records and documents, including location and length of retention period.</li> </ul>
<b><i>Data Generation and Acquisition</i></b>	
Sampling Process Design (Experimental Design)	<ul style="list-style-type: none"> <li>• Describes the experimental design or data collection design for the project.</li> <li>• Classifies all measurements as critical or non-critical.</li> </ul>
Sampling Methods	<ul style="list-style-type: none"> <li>• Describes the procedures for collecting samples and identifies sampling methods and equipment. Includes any implementation requirements, support facilities, sample preservation requirements, and materials needed.</li> <li>• Describes the process for preparing and decontaminating sampling equipment to include the disposal of decontamination by-products, selection and preparation of sample containers, sample volumes, preservation methods, and maximum holding times for sampling, preparation, and/or analysis.</li> <li>• Describes specific performance requirements for the method.</li> <li>• Addresses the actions to be taken when a failure in sampling occurs, which party is responsible for corrective action, and how the effectiveness of the corrective action shall be determined and documented.</li> </ul>

Table A-1. (Continued) QAPP Requirements

Functions Within a Sampling Project	Elements of that Function
Sample Handling and Custody	<ul style="list-style-type: none"> <li>• Describes the requirements and provisions for sample handling and custody in the field, transport, and laboratory, taking into account the nature of the samples, the maximum allowable sample holding times before extraction and analysis, and the available shipping options and schedules.</li> <li>• Includes examples of sample labels, custody forms, and sample custody logs.</li> </ul>
Analytical Methods	<ul style="list-style-type: none"> <li>• Identifies the analytical methods and equipment required, including sub-sampling or extraction methods, waste disposal requirements (if any), and specific method performance requirements.</li> <li>• Identifies analytical methods by number, date, and regulatory citation (as appropriate). If a method allows the user to select from various options, the method citations should state exactly which options are being selected.</li> <li>• Addresses the actions to be taken when a failure in the analytical system occurs, who is responsible for corrective action, and how the effectiveness of the corrective action shall be determined and documented.</li> <li>• Specifies the laboratory turnaround time needed, if important to the project schedule.</li> <li>• Specifies whether a field sampling and/or laboratory analysis Case Narrative is required to provide a complete description of any difficulties encountered during sampling or analysis.</li> </ul>
Quality Control (QC)	<ul style="list-style-type: none"> <li>• Identifies required measurement QC checks for both the field and laboratory.</li> <li>• States the frequency of analysis for each type of QC check, and the spike analyte sources and levels.</li> <li>• States or references the required control limits for each QC check and the corrective action(s) required when control limits are exceeded and how the effectiveness of the corrective action shall be determined and documented.</li> <li>• Describes or references the procedures to be used to calculate each of the QC statistics.</li> </ul>
Instrument/Equipment Testing, Inspection, and Maintenance	<ul style="list-style-type: none"> <li>• Describes how inspections and acceptance testing of environmental sampling and measurement systems and their components will be performed and documented. Identifies and discusses the procedure by which final acceptance will be performed by independent personnel.</li> <li>• Describes how deficiencies are to be resolved and when re-inspection will be performed.</li> <li>• Describes or references how periodic preventative and corrective maintenance of measurement or test equipment shall be performed.</li> <li>• Identifies the equipment and/or system requiring periodic maintenance.</li> <li>• Discusses how the availability of spare parts identified in the operating guidance and/or design specifications of the systems will be assured and maintained.</li> </ul>
Instrument/Equipment Calibration and Frequency	<ul style="list-style-type: none"> <li>• Identifies all tools, gauges, instruments, and other sampling, measuring, and test equipment used for data collection activities affecting quality that must be controlled, and at specific times, calibrated to maintain performance within specified limits.</li> <li>• Identifies the certified equipment and/or standards used for calibration.</li> <li>• Describes or references the calibration procedures to be conducted using certified equipment and/or standards with known valid relationships to nationally recognized performance standards. If no such standards exist, documents the basis for calibration.</li> <li>• Indicates how records of calibration shall be maintained and traced to the instrument.</li> </ul>
Inspection/Acceptance of Supplies and Consumables	<ul style="list-style-type: none"> <li>• Describes how and by whom supplies and consumables shall be inspected and accepted for use in the project.</li> <li>• States acceptance criteria for such supplies and consumables.</li> </ul>

**Table A-1. (Continued) QAPP Requirements**

Functions Within a Sampling Project	Elements of that Function
Non-direct Measurements	<ul style="list-style-type: none"> <li>• Identifies all the types of data needed for project implementation or decision-making that are obtained from non-measurement sources (e.g., computer databases, programs, literature files, historical databases).</li> <li>• Describes the intended use of data.</li> <li>• Defines the acceptance criteria for the use of such data in the project.</li> <li>• Specifies any limitations on the use of the data.</li> </ul>
Data Management	<ul style="list-style-type: none"> <li>• Describes the project data management scheme, tracing the data path from generation in the field or laboratory to their final use or storage.</li> <li>• Describes or references the standard record-keeping procedures, document control system, and the approach used for data storage and retrieval on electronic media.</li> </ul>

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## APPENDIX B GENERAL CLP SAMPLE COLLECTION GUIDELINES FOR AQUEOUS VOA SAMPLES



Regional guidance and/or specific Project Plan requirements will supersede the guidelines listed below.

### Collect the following:

- At least four 40 mL glass containers with polytetrafluoroethylene (PTFE)-lined septa and open top screw-caps that are filled to capacity with no air bubbles, preserved to a pH of 2 with HCl, and cooled to  $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. **DO NOT FREEZE THE SAMPLES.**

**Regular Samples** 4 vials (40 mL filled to capacity with no headspace or air bubbles)

**Regular Samples Requiring Quality Control (QC) Analysis** 4 vials for Sample (40 mL filled to capacity with no headspace or air bubbles)  
2 vials for Matrix Spike (MS) (40 mL filled to capacity with no headspace or air bubbles)  
2 vials for Matrix Spike Duplicate (MSD) (40 mL filled to capacity with no headspace or air bubbles)

- If Selected Ion Monitoring (SIM) analysis is requested, at least two additional 40 mL glass containers with PTFE-lined septa and open top screw-caps that are filled to capacity with no air bubbles, preserved to a pH of 2 with HCl, and cooled to  $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.

**Regular Samples With SIM Analysis** 4 vials for Sample (40 mL filled to capacity with no headspace or air bubbles)  
2 vials (40 mL filled to capacity with no headspace or air bubbles)

### Test for Carbonates, Residual Chlorine, Oxidants, and Sulfides:

- It is important that samplers obtain Regional guidance when testing and ameliorating for:
  - Carbonates;
  - Residual chlorine (e.g., municipal waters or industrial waste waters that are treated with chlorine prior to use or discharge); or
  - Oxidants.
- Volatile Organic Analyte (VOA) samples containing carbonates react with the acid preservative causing effervescence (due to formation of carbon dioxide), which can cause loss of volatile analytes.
- Residual chlorine present in VOA samples can continue to react with dissolved organic matter. This continuous reaction may lead to inaccurate quantitation of certain analytes present in the sample at the time of collection.
- Residual chlorine and oxidants present in VOA samples can cause degradation of certain volatile analytes (e.g., styrene).

### Perform the following for *Pre-Preserved Vials*:

- Pour the sample slowly down the edge of the sample vial to avoid excess aeration or agitation of the sample during filling.
- Fill the vial completely so that a reverse (convex) meniscus is present and ensure that there are no air bubbles present (either in the body or especially at the top of the vial).
- Place the septum on the vial so that the PTFE side is in contact with the sample, and then firmly tighten the cap.

## CLP Sampler's Guide

4. Gently flip the vial a few times to ensure that the sample is mixed with the acid preservative.
5. While holding the vial inverted, gently tap the sample to check for air bubbles (either in the body or especially at the top of the inverted vial).
6. If air bubbles are present, discard the sample and select a new vial in which to collect a new sample. Repeat Steps 1 - 5 above.
7. Do NOT mix or composite samples for VOAs.
8. Cool sample to a temperature of  $\leq 6^{\circ}\text{C}$ , but not frozen. Samplers should begin the cooling process in the field as samples are being collected. Double-bagged ice should be used. DO NOT FREEZE WATER SAMPLES.
9. Immediately transfer the vial to the sample shuttle (device that contains a “set” of VOA vials) once it has been collected. Do NOT allow ice to touch the vials.

**Perform the Following for *Empty* Vials:**

1. Rinse the vial with sample water prior to actual sample collection and preservation.



Regions vary in their approach to pre-rinsing and/or re-using sample vials (e.g., some Regions do not recommend pre-rinsing and/or re-use of pre-cleaned containers using sample water). Be sure to follow Regional guidance.

2. Add 1-2 mL of acid preservative to the vial. Check to ensure that the sample requires a preservative (follow Regional guidance).
3. Pour the sample slowly down the edge of the sample vial to avoid excess aeration and agitation of the sample.
4. Fill the vial completely so that a reverse (convex) meniscus is present and ensure that there are no air bubbles present (either in the body or especially at the top of the vial).
5. Place the septum on the vial so that the PTFE side is in contact with the sample, and then firmly tighten the cap.
6. Gently flip the vial a few times to ensure that the sample is mixed with the acid preservative.
7. While holding the vial inverted, gently tap the vial to check for air bubbles (either in the body or especially at the top of the vial).
8. If an air bubble is present that is larger than ~6 mm (pea size), discard the sample and collect a new sample using the same sample vial. Repeat Steps 1 - 7 above.
9. Check the re-collected sample for air bubbles. If there are air bubbles after three consecutive attempts to eliminate air bubbles by the addition of sample water, the entire sample and sample vial should be discarded and a new sample collected without preservative. Note the absence of preservative due to the impact on holding time.
10. Do NOT mix or composite samples for VOAs.
11. Cool sample to a temperature of  $\leq 6^{\circ}\text{C}$ , but not frozen. Samplers should begin the cooling process in the field as samples are being collected. Double-bagged ice should be used. DO NOT FREEZE WATER SAMPLES.
12. Immediately transfer the vial to the sample shuttle (device which contains a “set” of VOA vials) once it has been collected. Do NOT allow ice to touch the vials.

**Things to Remember:**

- Samples should be shipped as soon as possible, preferably on the same day as sample collection to avoid exceeding sample holding times. If overnight transit is not possible, samples should be maintained at  $\leq 6^{\circ}\text{C}$ , but not frozen, until they are shipped to the laboratory.
- If samples are not preserved (a requirement for certain analyses), the technical holding time is shortened to 7 days.

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## APPENDIX C CLP SAMPLE COLLECTION GUIDELINES FOR SOIL VOA SAMPLES BY SW-846 METHOD 5035A, TCLP EXTRACTION BY EPA METHOD SW-846 METHOD 1311, AND SPLP EXTRACTION BY EPA SW-846 METHOD 1312

- A. Preferred Options for the Contract Laboratory Program (CLP) are Options 1, 2, 3, and 4:

This analytical method employs sample vials that are filled and weighed in the field and never opened during the analytical process. For this reason, sampling personnel should be equipped with a portable balance capable of weighing to 0.01g.



Soil samples must be placed on their side prior to being frozen or placed on ice. Vials are placed on their side so that the septum is wet on the inside, thereby preventing vapor leaks around it, in case any bubbles form. Also, in case the samples freeze, the water will expand into the flexible septum rather than breaking the vial. Dry ice or field freezers are the only options.

### Option 1.

#### Closed-system Vials:

**Container - tared or preweighed 40 mL Volatile Organic Analyte (VOA) Analysis vial containing a magnetic stir bar.**

Collect 5 g of soil per vial (iced or frozen in the field). Check the pre-tared weight of the (dry) Volatile Organic Analyte (VOA) vials prior to departure for the sampling event under controlled conditions. Weigh vials and soil samples to the nearest 0.01 g. This check is to ensure that the original weight was properly recorded.

<b>Regular Samples</b>	3 Vials - Dry (5 g soil per vial)
	<u>1 Vial - Dry (filled with soil, no headspace)</u>
	4 Total Vials

<b>Regular Samples</b>	11 Vials - Dry (5 g soil per vial)
<b>Requiring Quality Control (QC) Analysis</b>	1 Vial - Dry (filled with soil, no headspace)
	<u>12 Total Vials</u>

### Option 2.

#### Closed-system Vials Containing Water:

**Container - tared or pre-weighed 40 mL VOA vial containing a magnetic stir bar and 5 mL water.**

Collect 5 g of soil per vial (iced or frozen in the field). Weigh vials and soil samples to the nearest 0.01 g.

<b>Regular Samples</b>	3 Vials with water added (5 g soil and 5 mL water per vial)
	<u>1 Vial - Dry (filled with soil, no headspace)</u>
	4 Total Vials (3 with water and 1 dry)

<b>Regular Samples</b>	11 Vials with water added (5 g soil and 5 mL water per vial)
<b>Requiring QC Analysis</b>	1 Vial - Dry (filled with soil, no headspace)
	<u>12 Total Vials (11 with water and 1 dry)</u>

**Option 3.**

**Container - 5 g Samplers or equivalent and coring tool used as a transport device.**



All samples should be iced or frozen in the field and bagged individually.

<b>Regular Samples</b>	3 Samplers (5 g soil per Sampler) <u>1 Vial - Dry (filled with soil, no headspace)</u> 4 Total (3 Samplers and 1 Vial)
<b>Regular Samples Requiring QC Analysis</b>	11 Samplers (5 g soil per Sampler) <u>1 Vial - Dry (filled with soil, no headspace)</u> 12 Total (11 Samplers and 1 Vial)

**Option 4.**

**Closed-system Vials:**

**Container - tared or preweighed 40 mL VOA vial.**

Collect 25 g of soil per vial (iced or frozen in the field). Check the pre-tared weight of the (dry) VOA vials prior to departure for the sampling event under controlled conditions. Weigh vials and soil samples to the nearest 0.01 g. This check is to ensure that the original weight was properly recorded.

**Regular Samples**                      6 Vials - Dry (25 g soil per vial)

B. Options 5, 6, and 7 are NOT preferred options for the CLP:

**Option 5.**

**Closed-system Vials:**

**Container - tared or preweighed 40 mL VOA vial containing a magnetic stir bar and preservative.**

Collect 5 g of soil per vial and add Sodium bisulfate (NaHSO<sub>4</sub>) preservative (5 mL water + 1 g NaHSO<sub>4</sub>) - iced in the field.

**Caution: This option is NOT a Preferred Option for the CLP because:**

NaHSO<sub>4</sub> preservation creates low pH conditions that will cause the destruction of certain CLP target analytes (e.g., vinyl chloride, trichloroethene, trichlorofluoromethane, cis- and trans-1,3-dichloropropene). Projects requiring the quantitation of these analytes should consider alternative sample preservation methods. NaHSO<sub>4</sub> also cannot be used on carbonaceous soils. Check the soil before using this method of collection! Soils can be checked by placing a test sample in a clean vial, then adding several drops of NaHSO<sub>4</sub> solution. If the soil bubbles, use Option 5b and note this issue on the Traffic Report/Chain of Custody (TR/COC) Record.

**Option 5a.** Samples preserved in the field

<b>Regular Samples</b>	3 Vials with NaHSO <sub>4</sub> preservative added (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace)
	<hr/> 4 Total Vials (3 with NaHSO <sub>4</sub> preservative and 1 without)
<b>Regular Samples Requiring QC Analyses</b>	6 Vials with NaHSO <sub>4</sub> preservative added (5 g soil per vial) 2 Vials - Dry (filled with soil, no headspace)
	<hr/> 8 Total Vials (6 with NaHSO <sub>4</sub> and 2 without)

**Option 5b.** Samples to be preserved by the laboratory (No NaHSO<sub>4</sub> preservative is added to these samples in the field).

<b>Regular Samples</b>	3 Vials - Dry (5 g soil per vial) 1 Vial - Dry (filled with soil, no headspace)
	<hr/> 4 Total Vials
<b>Regular Samples Requiring QC Analyses</b>	6 Vials - Dry (5 g soil per vial) 2 Vials - Dry (filled with soil, no headspace)
	<hr/> 8 Total Vials

**Option 6.****Methanol Preservation (medium-level analysis only):**

**Container - tared or pre-weighed 40 mL VOA vials containing 5 mL methanol.**

Collect 5 g of soil per vial (iced in the field).

**Caution: This is NOT a preferred option for the CLP because:**

Samples preserved with methanol can only be analyzed by the medium-level method. Low-level Contract Required Quantitation Limits (CRQLs) cannot be achieved when samples are preserved in this manner. If this soil option is used, then samples for low-level analysis by one of the other options should also be collected and accompany the medium-level soil.

Additional problems associated with use of methanol as a preservative in the field include:

- Possible contamination of the methanol by sampling-related activities (e.g., absorption of diesel fumes from sampling equipment);
- Leakage of methanol from the sample vials during shipping, resulting in loss of VOAs prior to analysis.

<b>Regular Samples</b>	2 Vials (5 g soil and 5 mL methanol per vial) 1 Vial - Dry (filled with soil, no headspace)
	<hr/> 3 Total Vials (2 with methanol and 1 dry)
<b>Regular Samples Requiring QC Analyses</b>	6 Vials (5 g soil and 5 mL methanol per vial) 1 Vial -Dry (filled with soil, no headspace)
	<hr/> 7 Total Vials (6 with methanol and 1 dry)



If shipping samples that contain methanol as a preservative, a shipping label must be used to indicate the presence of methanol. This label must also contain the United Nations (UN) identification number for methanol (UN 1230), and indicate Limited Quantity. Refer to <http://www.phmsa.dot.gov> for additional information about the safe shipping of methanol.

**Option 7.****Glass Containers filled with sample - No Headspace:****Container - 4 oz. Glass Jars.**

Glass container filled with soil with no headspace and iced.

**Caution: This is NOT a preferred option for the CLP because:**

Samples collected in this manner lose most of their volatile analytes prior to analysis when the sample containers are opened and sub-sampled in the laboratory. This option is only available when required by the Region.

<b>Regular Samples</b>	2 Glass Jars (4 oz.) filled with sample, no headspace 1 Vial - Dry (filled with soil, no headspace)
	<hr/> 3 Total Containers
<b>Regular Samples Requiring QC Analyses</b>	2 Glass Jars (4 oz.) filled with sample, no headspace 1 Vial - Dry (filled with soil, no headspace)
	<hr/> 3 Total Containers

**C. Caution:**

1. Extreme care should be taken to ensure that frozen or iced samples do not break during shipment.
2. Before adding soil to pre-weighed vials containing a stir bar, weigh the vials to confirm the tared weight. If the weight varies by more than 0.1 g, record the new weight on the label and the sample documentation. Do NOT add labels to these vials once the tared weight has been determined or confirmed.

**D. Dry Samples:**

Collect in a dry 40 mL VOA vial (or a 4 oz. wide mouth jar) with no headspace. No water, NaHSO<sub>4</sub>, or methanol is added to this sample. This sample is collected to determine moisture content; therefore, it does not need to be tared or have a stir bar.

**E. Iced or Frozen Samples:**

1. Iced means cooled to  $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.
2. Frozen means cooled to  $< -7^{\circ}\text{C}$  immediately after collection.
3. Dry ice is not a long-term freezing agent and may contaminate samples.

**F. Sample Delivery:**

The CLP strongly recommends that all samples arrive at the laboratory by close of business the next day following sample collection.

**G. Notes:**

1. For Options 2, 5, and 6, check the weight of the pre-tared VOA vials plus liquid in the field due to the possibility that liquid may have leaked out during packing, transit, or deployment in the field just prior to sampling. This check is to ensure that the original weight is properly recorded.
2. For Option 5, samples can be preserved with NaHSO<sub>4</sub> either:
  - In the field; or
  - The samplers must evaluate whether NaHSO<sub>4</sub> would be compatible with the sample prior to requesting the laboratory to add it to the sample.

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3. Regional Quality Assurance Project Plans (QAPPs) may require the use of Option 6. Note that this option is for medium-level analysis ONLY.
4. If water, methanol, or NaHSO<sub>4</sub> preservative is added to the vials in the field, a field blank containing the appropriate preservative used in the vials should be sent to the laboratory for analysis.

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## APPENDIX D CLP SAMPLE COLLECTION REQUIREMENTS BY ANALYSIS TYPE

The following tables (D-1 through D-4) specify the number of containers of each type necessary for each analysis based on the sampling methodology and QC requirements.

**Table D-1. Sample Collection Requirements for CLP Organics [Volatile Organic Analytes (VOAs) Only]**

Analysis	Matrix	Sample Type	Container Type <sup>1</sup>	Closure <sup>2</sup>	Minimum Number of Containers Needed				Minimum Volume/Mass <sup>3</sup>	Important Notes	Preservative <sup>4</sup>	Technical Holding Time <sup>5</sup>	
					with Water	Dry	% Moisture	TOTAL					
VOAs	Water	Samples Only	40 mL amber or clear glass vial, 24 mm neck finish.	Polypropylene or phenolic, open-top screw-cap, 1.5 cm opening, 24-400 size.	-	-	-	4	Fill to capacity	Containers/vials must be filled to capacity with no headspace or air bubbles. Refer to Appendix B for samples requiring QC analyses. If amber containers are not available, the samples should be protected from light.	Preserve to a pH of 2 with HCl and cool to ≤ 6°C, but not frozen, immediately after collection. DO NOT FREEZE water samples.	14 days	
		Samples with SIM						6					
		Samples with Matrix Spike (MS)/MS Duplicate (MSD) <sup>6</sup>						8					
	Soil/ Sediment	Water	Samples Only	<b>OPTION 1</b> Closed-system 40 mL amber or clear glass vial containing magnetic stirrer, 24 mm neck finish.	Polypropylene or phenolic, open-top screw-cap, 1.5 cm opening, 24-400 size.	-	3	1	4	5g	Place samples on side prior to being iced. <sup>7</sup> Refer to Appendix C for samples requiring QC analyses.	Frozen to < -7°C OR iced to ≤ 6°C, but not frozen.	14 days OR 48 hours (unpreserved) <sup>7</sup>
			Samples with MS/MSD <sup>6</sup>						11				
		Soil/ Sediment	Samples Only	<b>OPTION 2</b> Closed-system 40 mL amber or clear glass vial containing magnetic stirrer, 24 mm neck finish and 5 mL water.	Polypropylene or phenolic, open-top screw-cap, 1.5 cm opening, 24-400 size.	3	-	1	4	5 g	Place samples on side prior to being iced. <sup>7</sup> Refer to Appendix C for samples requiring QC analyses.	Frozen to < -7°C OR iced to ≤ 6°C, but not frozen.	14 days OR 48 hours (unpreserved) <sup>7</sup>
			Samples with MS/MSD <sup>6</sup>						11				

Table D-1. (Continued) Sample Collection Requirements for CLP Organics [Volatile Organic Analytes (VOAs) Only]

Analysis	Matrix	Sample Type	Container Type <sup>1</sup>	Closure <sup>2</sup>	Minimum Number of Containers Needed				Minimum Volume/Mass <sup>3</sup>	Important Notes	Preservative <sup>4</sup>	Technical Holding Time <sup>5</sup>
					with Water	Dry	% Moisture	TOTAL				
		Samples Only	<b>OPTION 3</b> Coring tool used as a transport device.		-	3	1	4	5 g	Refer to Appendix C for samples requiring QC analysis. Place samples on side prior to being iced. <sup>7</sup>	Frozen to < -7°C <b>OR</b> iced to ≤ 6°C, but not frozen.	14 days (frozen) <b>OR</b> 48 hours (iced) <sup>7</sup>
		Samples with MS/MSD <sup>6</sup>			-	11	1	12				
		Samples for TCLP/SPLP Only	<b>OPTION 4</b> Closed-system 40 mL amber or clear glass vial 24 mm neck finish.	Polypropylene or phenolic, open-top screw-cap, 1.5 cm opening, 24-400 size.	-	6	-	6	150 g <sup>8</sup>	Place samples on side prior to being iced. <sup>7</sup>	Iced to ≤ 6°C but not frozen.	14 days

**Notes**

<sup>1</sup> Vials for soil analysis are typically pre-labeled and tared. Vials for water analysis are not pre-labeled or tared.

<sup>2</sup> Septums for VOA vials 24 mm disc of Polytetrafluoroethylene (PTFE) bonded to silicone for a total thickness of 0.100 - 0.125 in.

<sup>3</sup> Minimum volume/mass to be collected in order to ensure sample analysis can be performed. Collect additional volume for MS/MSD samples to allow for sufficient volume for these analyses in the event sample volume is lost as a result of sample containers breaking, leaking, or laboratory accidents.

<sup>4</sup> Check Regional guidance regarding use of acid as a preservative of samples that may contain carbonates, residual chlorine, and other oxidants.

<sup>5</sup> Technical holding time is calculated from the time of sample collection to sample extraction, and determined as 14 days for preserved (frozen or iced) samples and 48 hours for non-preserved (iced) samples.

<sup>6</sup> For Gas Chromatograph/Mass Spectrometer (GC/MS) organic analyses, a Region may decide to reference Deuterated Monitoring Compounds (DMCs) rather than MS/MSD for accuracy and precision.

<sup>7</sup> Vials are placed on their side so that the septum is wet on the inside, thereby preventing vapor leaks around it, in case any bubbles form. Also, in case samples freeze, the water will expand into the flexible septum rather than breaking the vial.

<sup>8</sup> Samples with low solids content that are scheduled for Toxicity Characteristic Leaching Procedure (TCLP) or Synthetic Precipitation Leaching Procedure (SPLP) may require up to 1000 mL total volume.

**Table D-2. Sample Collection Requirements for CLP Organics [Semivolatile Organic Analytes (SVOAs), Pesticides, and Aroclors]**

Analysis	Matrix	Sample Type	Container Type	Closure	Minimum Volume/Mass <sup>1</sup>	Important Notes	Preservative/Collection	Technical Holding Time <sup>2</sup>	
SVOAs	Water <sup>3</sup>	Samples Only	1 L amber round glass bottle, 33 mm pour-out neck finish.	Polypropylene or phenolic cap, 33-430 size; 0.015 in. PTFE liner.	2 L (per Test)	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. DO NOT FREEZE water samples.	7 days	
		Samples with SIM			2 L				
		Samples with MS/MSD			5 L				
		Samples with SIM with MS/MSD			5 L				
	Soil/ Sediment/ Waste <sup>4</sup>	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish or two 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner. Or Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	150 g		Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	14 days	
		Samples with SIM	Two 8 oz. short, wide mouth, straight-sided, glass jars, 70 mm neck finish or three 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.		300 g				
		Samples with MS/MSD			Samples for Only TCLP/SPLP				450 g
		Samples with SIM with MS/MSD							

Table D-2. (Continued) Sample Collection Requirements for CLP Organics [Semivolatile Organic Analytes (SVOAs), Pesticides, and Aroclors]

Analysis	Matrix	Sample Type	Container Type	Closure	Minimum Volume/Mass <sup>1</sup>	Important Notes	Preservative/Collection	Technical Holding Time <sup>2</sup>
Pesticides	Water <sup>3,5</sup>	Samples Only	1 L amber round glass bottle, 33 mm pour-out neck finish.	Polypropylene or phenolic cap, 33-430 size; 0.015 in. PTFE liner.	2 L	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. DO NOT FREEZE water samples.	7 days
		Samples with MS/MSD			5 L			
	Soil/ Sediment/ Waste <sup>4</sup>	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish or two 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner. Or Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	150 g		Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	14 days
		Samples with MS/MSD	Two 8 oz. short, wide mouth, straight-sided, glass jars, 70 mm neck finish or three 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.		300 g			
		Samples for Only TCLP/SPLP	Two 8 oz. short, wide mouth, straight-sided, glass jars, 70 mm neck finish or four 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.		450 g			
	Wipes	Samples Only	20 mL glass vial.	Polypropylene or phenolic cap, PTFE liner.	1 wipe			14 days
Aroclors	Water <sup>3,5</sup>	Samples Only	1 L amber round glass bottle, 33 mm pour-out neck finish.	Polypropylene or phenolic cap, 33-430 size; 0.015 in. PTFE liner.	2 L	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. DO NOT FREEZE water samples.	7 days
		Samples with MS/MSD			5 L			
	Soil/ Sediment/ Waste <sup>4</sup>	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish or two 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner. Or Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	150 g		Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	14 days
		Samples with MS/MSD	Two 8 oz. short, wide mouth, straight-sided, glass jars, 70 mm neck finish or three 4 oz. tall, wide mouth, straight-sided, glass jar, 48 mm neck finish.		300 g			
	Wipes	Samples Only	20 mL glass vial.	Polypropylene or phenolic cap, PTFE liner.	1 wipe			14 days

**Table D-2. (Continued) Sample Collection Requirements for CLP Organics [Semivolatile Organic Analytes (SVOAs), Pesticides, and Aroclors]**Notes

- <sup>1</sup> Minimum volume/mass to be collected in order to ensure sample analysis can be performed.
- <sup>2</sup> This technical holding time is calculated from the time of sample collection to sample extraction. Sample extracts are to be analyzed within 40 days of extraction. It is recommended that samplers ship samples to the laboratory on the same day that they are collected, or as soon as possible thereafter.
- <sup>3</sup> An aqueous sample for SVOA analysis would require the field sampler to collect at least 2 L for field samples and at least 3 L for the MS and MSD samples for a total volume of 5 L. If Pesticide or Aroclor MS/MSD analyses are required for the same sample, only two extra 1 L bottles of each sample would be needed, for a total of seven bottles (which includes samples and MS/MSD for both methods). Collect additional volume for MS/MSD samples to allow for sufficient volume for these analyses in the event sample volume is lost as a result of sample containers breaking, leaking, or laboratory accidents.
- <sup>4</sup> If one or two extractable analyses are required for soil/sediment, only a single 8 oz. or two 4 oz. jars are required. If three extractable analyses are required, two 8 oz. or four 4 oz. jars are required. The number of jars should be doubled if MS/MSD is required.
- <sup>5</sup> Samplers must test for chlorine in aqueous samples in the field upon collection. Refer to the Sampling and Analysis Plan (SAP) and Appendix E of this document for guidance.

Table D-3. Sample Collection Requirements for CLP Inorganics

Analysis	Matrix	Sample Type	Container Type	Closure	Minimum Volume/ Mass <sup>1</sup>	Important Notes	Preservative/ Collection <sup>2</sup>	Technical Holding Time <sup>3</sup>
Metals/ICP-AES, Metals/ICP-MS, and/or Mercury/CVAA	Water	Samples Only	0.5 L high density polyethylene, cylinder-round bottle, 28 mm neck finish.	Polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner.	0.5 L	DO NOT FREEZE water samples.	Acidify to pH < 2 with HNO <sub>3</sub> immediately after collection. <sup>4</sup>	6 months for all metals except Mercury (28 days)
		Samples with MS/Duplicate			1 L			
	Soil/ Sediment/ Waste <sup>5</sup>	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner.	Fill to capacity		Cool to ≤ 6°C, but not frozen, immediately after collection.	6 months
		Samples with MS/Duplicate						
		Samples for Only TCLP/SPLP						
	Metals/ICP-AES <sup>6</sup>	Wipe	Samples Only	1 qt. polymer zip-top bag.	Has self-closing mechanism.	N/A		Store at room temperature.
Cyanide/ Spectrophotometric Determination	Water	Samples Only	0.5 L high density polyethylene, cylinder-round bottle, 28 mm neck finish.	Polyethylene cap, ribbed, 28-410 size; F217 polyethylene liner.	0.5 L	DO NOT FREEZE water samples.	To neutralize residual chlorine, add 0.6 g ascorbic acid for each liter of sample collected, immediately upon collection. <sup>7</sup> Add NaOH until pH > 10 and cool to ≤ 6°C, but not frozen, immediately after collection.	14 days
		Sample with MS/Duplicate			1 L			
	Soil/ Sediment/ Waste <sup>5</sup>	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner.	Fill to capacity		Cool to ≤ 6°C, but not frozen, immediately after collection.	14 days
		Samples with MS/Duplicate						
		Samples for SPLP						

Table D-3. (Continued) Sample Collection Requirements for CLP Inorganics

Analysis	Matrix	Sample Type	Container Type	Closure	Minimum Volume/ Mass <sup>1</sup>	Important Notes	Preservative/ Collection <sup>2</sup>	Technical Holding Time <sup>3</sup>
Anions/Ion Chromatography (IC) <sup>8</sup>	Water	Samples Only	125 mL high density polyethylene, cylinder-round bottle, 28 mm neck finish.	Polyethylene or polypropylene cap, ribbed.	125 mL	DO NOT FREEZE water samples.	For the analysis of nitrate, nitrite, orthophosphate, and sulfate cool to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. Do not allow samples for orthophosphate to be held at room temperature more than 12 hours.	48 hours for nitrate, nitrite, and orthophosphate. 28 days for bromide, chloride, fluoride, and sulfate.
		Samples with MS/Duplicate			250 mL			
	Soil	Samples Only	One 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner.	Fill to capacity		Cool to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	48 hours for nitrate, nitrite, and orthophosphate. 28 days for bromide, chloride, fluoride, and sulfate.
		Samples with MS/Duplicate						
Hexavalent Chromium/Ion Chromatography (IC) <sup>8</sup>	Water	Samples Only	125 mL high density polyethylene or polypropylene, cylinder-round bottle, 28 mm neck finish.	Polyethylene or polypropylene cap, ribbed.	125 mL	DO NOT FREEZE water samples.	Cool to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. Field filter. Preserve to pH > 8 using either liquid preservative [ $\text{NH}_4\text{OH}/(\text{NH}_4)_2\text{SO}_4$ 1 mL per 100 mL sample] or solid mix [13.3 mg $\text{Na}_2\text{CO}_3/10.5$ mg $\text{NaHCO}_3/33$ mg $(\text{NH}_4)_2\text{SO}_4$ per sample].	28 days
		Samples with MS/Duplicate			250 mL			
Total Organic Carbon (TOC) <sup>8</sup>	Water	Samples Only	125 mL round glass bottle, 22 mm pour-out neck finish.	Polypropylene or phenolic cap, PTFE liner.	125 mL	DO NOT FREEZE water samples.	Acidify to pH < 2 with $\text{H}_2\text{SO}_4$ or $\text{H}_3\text{PO}_4$ immediately after collection and cool to $\leq 6^{\circ}\text{C}$ , but not frozen.	28 days
		Samples with MS/Duplicate			250 mL			
	Soil	Samples Only	4 oz. (120 mL) tall, wide mouth, straight-sided, amber glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	Fill to capacity		Cool to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	28 days
		Samples with MS/Duplicate						

**Table D-3. (Continued) Sample Collection Requirements for CLP Inorganics****Notes**

- <sup>1</sup> Minimum volume/mass to be collected in order to ensure sample analysis can be performed.
- <sup>2</sup> Check Regional guidance regarding use of acid as a preservative of samples that may contain carbonates, residual chlorine, and other oxidants.
- <sup>3</sup> This technical holding time is calculated from the time of sample collection to sample analysis.
- <sup>4</sup> Water samples collected for total metal and filtered metal analyses from the same sampling location must be assigned separate (unique) CLP Sample Numbers.
- <sup>5</sup> Only one 8 oz. jar is needed for soil/sediment when all metals (including mercury) and cyanide analyses are required for soil/sediment samples. Collect more than one jar when TCLP or SPLP are scheduled.
- <sup>6</sup> Wipe materials have varied from laboratory tissues (e.g., Kimwipes®) to pre-moistened “baby wipes” from the nearest store.
- <sup>7</sup> Samplers must test for sulfide and oxidizing agents (e.g., chlorine) in aqueous samples in the field upon collection. Refer to the SAP for guidance. Sulfides adversely affect the analytical procedure. The following can be done to test for and neutralize sulfides. Place a drop of the sample on lead acetate test paper to detect the presence of sulfides. If sulfides are present, treat 25 mL more of the sample than that required for the cyanide determination with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide or black lead sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complication or occlusion of cyanide on the precipitated material. Sulfide removal should be performed in the field, if practical, prior to pH adjustment with NaOH.
- <sup>8</sup> Analyses available through Modified Analysis only.

**Table D-4. Sample Collection Requirements for CLP Chlorinated Dibenzo-*p*-Dioxins/Chlorinated Dibenzofurans (CDDs/CDFs) and Chlorinated Biphenyl Congeners (CBCs)**

Analysis	Matrix	Container Types	Closure	Minimum Volume/Mass <sup>1</sup>	Important Notes	Preservative	Technical Holding Time <sup>2</sup>
CDD/CDF and CBC	Water <sup>3</sup>	1 L amber round glass bottle, 33 mm pour-out neck finish.	Polypropylene or phenolic cap, 33-430 size; 0.015 in. PTFE liner.	2L	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection. DO NOT FREEZE water samples. If residual chlorine is present, add 80 mg sodium thiosulfate/L of water. If pH is greater than 9, adjust to 7-9 with sulfuric acid.	1 year
	Soil/Sediment/Oil/Sludge Ash/Biosolids	8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish or 4 oz. (120 mL) tall, wide mouth, straight-sided, amber glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner. Or Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	Fill to capacity	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , but not frozen, immediately after collection.	
	Tissue	Heavy duty aluminum foil as transport device. 8 oz. short, wide mouth, straight-sided, glass jar, 70 mm neck finish or 4 oz. (120 mL) tall, wide mouth, straight-sided, amber glass jar, 48 mm neck finish.	Polypropylene or phenolic cap, 70-400 size; 0.015 in. PTFE liner. Or Polypropylene or phenolic cap, 48-400 size; 0.015 in. PTFE liner.	20 g for each analytical method	If amber containers are not available, the samples should be protected from light.	Cool all samples to $\leq 6^{\circ}\text{C}$ , or freeze immediately after collection.	

Notes

<sup>1</sup> Minimum volume/mass to be collected in order to ensure sample analysis can be performed.

<sup>2</sup> This technical holding time is calculated from the time of sample collection to sample extraction. Sample extracts are to be analyzed within 1 year of extraction when sample extracts are stored under the appropriate conditions. It is recommended that samplers ship samples to the laboratory on the same day that they are collected, or as soon as possible thereafter.

<sup>3</sup> Samplers must test for chlorine in aqueous samples in the field upon collection. Refer to the SAP for guidance.

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## APPENDIX E SAMPLING TECHNIQUES AND CONSIDERATIONS

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During a sampling event, samplers are expected to follow prescribed sampling techniques. Samplers should also be aware of any special sampling considerations, contaminant issues, and sample compositing and mixing methods that could affect their sampling efforts.

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Regional guidance will take precedence over any of the techniques and considerations listed below.

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### 1. General Sampling Techniques

Information regarding surface water, groundwater, sediment, soil, and surface wipe sampling can be found in many documents including, but not limited to, the following sources:

- *Compendium of ERT Surface Water and Sediment Sampling Procedures*, EPA/540/P-91/005
- *Compendium of ERT Soil Sampling and Surface Geophysics Procedures*, EPA/540/P-91/006
- *Compendium of ERT Groundwater Sampling Procedures*, EPA/540/P-91/007
- *The Roles of Project Managers and Laboratories in Maintaining the Representativeness of Incremental and Composite Soil Samples*, EPA/OSWER 9200.1-117FS
- *Lead in Surface Wipe Samples*, NIOSH Method 9100, August 15, 1994
- *Elements on Wipes*, NIOSH Method 9102, March 15, 2003
- *Surface Wipe Sampling Procedure*, IH75190, Brookhaven National Laboratory, Industrial Hygiene Group, May 10, 2011

When working with potentially hazardous materials, samplers should follow U.S. Environmental Protection Agency (EPA) and Occupational Health and Safety Administration (OSHA) requirements, specific health and safety procedures, and Department of Transportation (DOT) requirements.

### 2. Special Sampling Considerations

Samplers should refer to the Regional Standard Operating Procedures (SOPs) to obtain specific procedures for proper collection and preservation of samples in the field. For additional guidance regarding sampling for Volatile Organic Analytes (VOAs) Analysis in soil and water, see Appendices B and C. Samplers should obtain Regional guidance when testing and ameliorating for:

- Carbonates in VOA soil and water samples
- Residual chlorine in VOA soil and water samples, or in aqueous cyanide samples
- Oxidants in VOA soil and water samples
- Sulfides and oxidizing agents in aqueous cyanide samples

### 3. Contaminant Sampling

Certain analytes can be detected in the parts-per-billion (ppb) and/or parts-per-trillion (ppt) range. Extreme care MUST be taken to prevent cross-contamination of these samples. The following precautions should be taken when trace contaminants are a concern:

- Disposable gloves should be worn for each different location sampled.
- When collecting both surface water and sediments, surface water samples should be collected first to reduce the chance of sediment dispersal into the surface water, and the resulting loss of surface water sample integrity.

- Sampling should occur progressively, from the least to the most contaminated area, if this information is known to the sampling team.
- Samplers should use equipment constructed of polytetrafluoroethylene (PTFE), stainless steel, or glass that has been properly pre-cleaned for the collection of samples for trace organic and/or inorganic analyses. Equipment constructed of plastic or polyvinyl chloride (PVC) should NOT be used to collect samples for trace organic analyte analyses.
- Equipment constructed of stainless steel should NOT be used to collect samples for trace metals analyses.

#### 4. Sample Compositing

Sample compositing is a site-specific activity that must be conducted according to the Sampling and Analysis Plan (SAP). Compositing is typically used for large sites to improve the precision (i.e., lower the variance) of the estimated average contaminant concentrations. **Samples for VOA analysis should NOT be composited to minimize loss of analytes.**

Composite samples consist of a series of discrete grab samples that are mixed together to characterize the average composition of a given material. The discrete samples are usually of equal volume, but may be weighted to reflect an increased flow or volume. Nevertheless, all discrete samples must be collected in an identical manner and the number of grab samples forming a composite should be consistent. There are several compositing techniques that may be required, such as:

- Flow-proportioned - Collected proportional to the flow rate during the compositing period by either a time-varying/constant volume or a time-constant/varying volume method. This technique is usually associated with wastewater or storm water runoff sampling.
- Time - Composed of a varying number of discrete samples collected at equal time intervals during the compositing period. This technique is typically used to sample wastewater and streams, and in some air sampling applications.
- Areal - Collected from individual grab samples collected in an area or on a cross-sectional basis. Areal composites are comprised of equal volumes of grab samples where all grabs are collected in an identical manner. This technique is typically used for estimating average contaminant concentrations in soils or sediments. It is also useful when contaminants are present in nugget form (i.e., TNT chunks, lead shot, etc.), thus exhibiting large differences in concentration over a small sample area.
- Vertical - Collected from individual grab samples taken from a vertical cross section. Vertical composites are comprised of equal volumes of grab samples where all grab samples are collected in an identical manner. Examples would include vertical profiles of a soil borehole or sediment columns.
- Volume - Collected from discrete samples whose aliquot volumes are proportional to the volume of sampled material. Volume composites are usually associated with hazardous waste bulking operations where the sample represents combined or bulked waste.

When compositing solid or tissue samples (i.e., sediment, soil, or sludge) for analysis of analytes present in trace quantities, use a stainless steel or PTFE bowl and spatula as applicable.

#### 5. Sample Mixing and Homogenizing

Mixing of the sample for the remaining parameters is necessary to create a representative sample media. It is extremely important that solid samples be mixed as thoroughly as possible to ensure that the sample is representative of the sample location. Refer to the project-specific SAP for instructions on removal of any extraneous materials (e.g., leaves, sticks, rocks, etc.). The mixing technique will depend on the physical characteristics (e.g., particle size, moisture content, etc.) of the solid material. For example, grinding and homogenization of tissue is easier when it is partially frozen. The mixing container should be large enough to hold the sample volume and accommodate the procedures without spilling. Both the mixing container (generally a bowl or tray) and the mixing implement should be properly decontaminated before use. Samples should be homogenized

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according to procedures listed in the project-specific SAP. Table E-1 provides a brief procedure for mixing a soil sample with a small particle size (less than 1/4 in.) and filling sample containers in the field.

**Table E-1. Mixing a Sample and Filling Sample Containers**

Step	Action
1	Roll the contents of the compositing container to the middle of the container and mix.
2	Quarter the sample and move to the sides of the container.
3	Mix each quarter individually, then combine and mix OPPOSITE quarters, then roll to the middle of the container.
4	Mix the sample once more, and then quarter the sample again.
5	Mix each quarter individually, then combine and mix ADJACENT corners, then roll to the middle of the container. The goal is to achieve a consistent physical appearance before sample containers are filled.
6	Flatten piled material into an oblong shape.
7	Using a flat-bottomed scoop, collect a strip of soil across the entire width of the short axis and place it into a sample container.
8	Repeat Step 7 at evenly-spaced intervals until the sample containers are filled.
9	Record the approximate quantity of each subsample in the field logbook.

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## APPENDIX F INTERNATIONAL SHIPPING

The following section provides information on shipping Contract Laboratory Program (CLP) samples to laboratories outside the United States.

### F.1 United Parcel Service (UPS) Procedure for Shipping to Canada

#### 1. Introduction

Samplers should refer to the Standard Operating Procedure (SOP) for the responsibilities and procedures for shipping to Canadian laboratories. The following sections detail the export shipping process.

#### 2. Overview

UPS will provide pick-up and delivery service for small package (weighing less than 150 lbs. each) shipments to laboratories in Canada.

#### 3. Contact Information

UPS Strategic Support for Government Accounts	<a href="https://www.ups.com/us/en/services/government.page">https://www.ups.com/us/en/services/government.page</a>
UPS International Customer Service	<a href="http://www.ups.com/international">www.ups.com/international</a>
Preferred Customer Associate Team	<a href="https://www.ups.com/us/en/help-support-center.page?">https://www.ups.com/us/en/help-support-center.page?</a>

#### 4. Customs Broker

The laboratory should be notified of each shipment so they know to contact their customs broker to have the shipment released. Otherwise, shipments may be held in customs. Laboratories may also act as their own broker.

#### 5. Product Profile

**Service Level** – EPA receives government pricing for shipments via UPS *Worldwide Express Saver* service. This service provides guaranteed delivery within 1 – 3 business days, by end of day, to Canada. The maximum weight per package is 150 lbs. (70 kg), maximum length is 108 in. (270 cm) per package and the maximum dimensions are 165.0 in. (419.10 cm) per package, length and girth combined.

#### 6. Commercial Invoice

A commercial invoice form (see Figure F-1) is used for all shipments containing non-documents. It is the primary document used for importation control, valuation, and duty determination. This document identifies the items in the shipments.

##### The form should include:

- Complete name and address information for both shipper and consignee
- Phone numbers for shipper and consignee
- Terms of Sale (Incoterm)
- Reason for export
- A complete description of the item
- Type of item
- Item use
- Harmonized Tariff Codes, if known

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- Country of origin (where manufactured) for each commodity
- Number of units, unit value, and total value (purchase price) of each item
- Number of packages and total weight
- Shipper's signature and date
- A nominal or fair market value must be stated for items of no commercial value

### Invoice

From		Ship To		Sold To			
Tax ID/VAT No.:	Waybill Number:                      Shipment ID:	Tax ID/VAT No.:	Tax ID/VAT Number:	Tax ID/VAT No.:	Tax ID/VAT Number:		
Contact Name:	Date:	Contact Name:	Contact Name:	Contact Name:	Contact Name:		
Company Name:	Invoice Number (Reference 1):	Company Name:	Company Name:	Company Name:	Company Name:		
Address:	Purchase Order Number (Reference 2):	Address:	Address:	Address:	Address:		
City State/Province:	Terms of Sale (Incoterm):	City State/Province:	City State/Province:	City State/Province:	City State/Province:		
Postal Code Country/Territory:	Reason for Export:	Postal Code Country/Territory:	Postal Code Country/Territory:	Postal Code Country/Territory:	Postal Code Country/Territory:		
Phone:		Phone:	Phone:	Phone:	Phone:		
Units	UM	Description of Goods/Part Number	Harmonized Code	C/O	Unit Value	Total Value	Currency
Additional Comments:			Invoice Line total: _____ Discount/Rebate: _____ Invoice Sub-Total: _____ Freight: _____ Insurance: _____ Other: _____ Currency				
Declaration Statement:			Total Invoice amount: _____				
Shipper's Signature / Title			Date			Total Number of Packages: _____ Total Weight: _____	

These commodities, technology, or software were exported from the United States in accordance with the Export Administrator Regulation. Diversion contrary to U.S. law is prohibited.

**Figure F-1. Commercial Invoice Template**

## 7. Packing with Coolants and Refrigerants (Dry Ice)

Coolants and refrigerants are used to keep temperature-sensitive items cold or frozen while in transit. Dry ice (frozen carbon dioxide) and ice are the most common types of coolant/refrigerants used for transportation.

### International Air Shipments Containing Dry Ice

International Air shipments containing dry ice require the shipper to have a UPS International Special Commodities contract. For additional information, please contact the UPS Hazardous Materials Support Center at 1-800-554-9964, or visit our online UPS Guide for Shipping International Dangerous Goods.

<https://www.ups.com/us/en/help-center/packaging-and-supplies/special-care-shipments/international-dangerous-goods/shippers-responsibilities.page>

### International Dry Ice Packages Shipped via Air Service Require the Following Under International Air Transport Association (IATA):

- Process through WorldShip 2019, CampusShip, or compliant software
- An acceptance audit is performed
- Mark the outer carton with:
  - The words “Dry Ice” or “Carbon Dioxide, Solid” and “UN 1845”
  - The amount of dry ice contained in the package in kg
  - Class 9 Diamond label

### Requirements for Preparing Dry Ice Shipments:

- Fill any empty space in the shipping container with appropriate packing material to prevent product movement during transit.
- Wrap temperature-sensitive products in two watertight plastic bags or use absorbent material along with a plastic liner.
- Avoid shipping temperature-sensitive products over the weekend.
- Wrap the refrigerant in paper or another carton to slow the sublimation rate and prevent excess space when using dry ice.
- Do not place the refrigerant at the bottom of the package because cold air will not circulate.
- Do not seal the inner insulated container when using dry ice. Venting is required to allow some carbon dioxide gas to escape the package.

\* UPS CampusShip is a web-based, UPS-hosted shipping solution that helps to increase efficiency and reduce costs.

## 8. Additional Information

Common items that may be hazardous require the shipper to have a UPS International Special Commodities contract. For additional information, contact the UPS Hazardous Materials Support Center at 1-800-554-9964, or visit the online UPS Guide for Shipping International Dangerous Goods.

Descriptions that Indicate Dangerous Goods – watch for any of the following descriptions that could indicate Dangerous Goods or Hazardous Materials:

- Acidic
- Caustic
- Combustible Communicable
- Compressed Gas
- Corrosive
- Explosive
- Flammable
- Infectious
- Inflammable

- Poison
- Radioactive
- Refrigerated
- Toxic
- Volatile

**Note:** The above is only a sampling of terms which should prompt further questions about a shipment.

## 9. Tools and Resources

### UPS Global

Access UPS Global website to get up-to-date information on everything international, including information on how to prepare an international shipment, track your package, and import/export country regulations and international forms.

### Processing International Package using UPS CampusShip

[https://www.ups.com/media/en/CampusShip\\_shipping\\_QuickStart\\_Guide.pdf](https://www.ups.com/media/en/CampusShip_shipping_QuickStart_Guide.pdf)

### Step-by-Step Instructions for International Shipping

#### Step 1. Verify That The Commodity Can Be Shipped

Determine if service restrictions apply to either the United States or Canada.

[Check for Embargoed/Suspended Service](#)

Verify that your commodity can be shipped to or from the United States and Canada.

[Check List of UPS Export Prohibited Articles](#)

**Step 2. Select an International Service** If price and time are your primary considerations in selecting an international service, use the Service Comparison tool on UPS CampusShip to find the guaranteed delivery time and price of every service available to and from the United States and Canada.

#### Step 3. Select an International Billing Option

UPS offers flexible international billing options. Select the applicable option.

[Learn More About International Billing Options](#)

#### Step 4. Create Required Documentation

Determine the export documentation required for the shipment and complete each form.

[Learn How to Create Documentation](#)

#### Step 5. Prepare The Shipment

Use UPS International Shipping Basics to learn more about preparing your shipment.

[Learn What Packing Materials to Use for Your International Shipment](#)

[Identify Specific Weight and Size Limitations Convert the Weight, Length, Area, and Volume of Your Shipment](#)

Decide whether to declare a value for high value items. If the value of the goods exceeds \$100.00 (USD), declare a higher value, up to \$50,000.00 (USD) per package. For packages that exceed the maximum declared value, a waiver must be obtained. Refer to the applicable terms and conditions of service for additional limits and restrictions.

[Learn More About Declared Value](#)

**Step 6. Ship The Package**

Use UPS CampusShip or Internet Shipping on [ups.com](https://www.ups.com) to prepare the international shipping label and the international forms required for certain shipments.

**Step 7. Get The Shipment to UPS**

In instances where automatic pickup has not been scheduled, UPS On-Call Pickup<sup>®</sup> may be requested to pick up all ground, air, and international shipments from any location. UPS On-Call Pickup can be scheduled at [ups.com](https://www.ups.com) or by calling 1-800-PICK-UPS<sup>®</sup> for same day or future day pickup. Packages can also be shipped from UPS locations including The UPS Store<sup>®</sup>.

[Find Locations](#)

**Step 8. Check Shipment Status**

Since each UPS package is assigned a unique tracking number, shipment information is easily accessible. Tracking information is always available at [ups.com](https://www.ups.com), by e-mail, and through optional services.

[Track Your Shipment](#)

[Learn About More Ways to Track](#)

## F.2 Completed Customs Forms Example

Shipping samples to an international laboratory requires the completion of a customs form. The following is an example of a correctly completed customs form for shipment to a laboratory in Canada.



 <b>United States-Mexico-Canada Agreement Certification of Origin Form (USMCA/CUSMA/T-MEC)</b>		
<b>1. Importer, Exporter or Producer Certification of Origin</b> <input type="checkbox"/> Importer <input checked="" type="checkbox"/> Exporter <input type="checkbox"/> Producer		<b>2. Blanket Period</b> <b>From:</b> <u>12/Dec/2020</u> (DD/MM/YYYY) <b>To:</b> <u>13/Dec/2020</u> (DD/MM/YYYY)
<b>3. Certifier</b> (Name, Title, Address, Country, Phone, Email)  US EPA 980 College Station Rd. Athens, GA 30605 United States		<b>4. Exporter – if different from the certifier</b> (Name, Address (Place of Export), Country, Phone, Email)
<b>5. Producer – if different from the certifier or exporter</b> (Name, Address (Place of Production), Country, Phone, Email)  XYZ Laboratory 530 St. Clair Ave W Toronto, ON M6C0A2 Canada		<b>6. Importer</b> (Name, Address (within a USMCA Party's Territory), Country, Phone, Email)  XYZ Laboratory 530 St. Clair Ave W Toronto, ON M6C0A2 Canada
7. Description of Good(s) and HS Tariff Classification Number(s)		8. Origin Criterion
Description	HS # (6-digit level)	
Infiltrax Water Sampler Stainless Steel Columns w 250g XAD resin (adsorbent) Scientific Testing Only	HS Code 9015.90	CA
Wound Glass fiber filter cartridges with samples for Scientific Testing Only. Not for resale, no com value	HS Code 9015.90	CA
<b>9. Certification</b> I certify that the goods described in this document qualify as originating and the information contained in this document is true and accurate. I assume responsibility for proving such representations and agree to maintain and present upon request or to make available during a verification visit, documentation necessary to support this certification.		
_____ <b>Authorized Signature</b>		<div style="border: 1px solid black; padding: 5px;"> <b>Date (DD/MM/YYYY)</b> </div>
_____ <b>Name and Title</b>		
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Figure F-2. International Shipping Form (1 of 3)




**Certification of Origin (USMCA/CUSMA/T-MEC)  
Continuation Sheet (Sections 7 & 8)**

7. (cont'd) Description of Good(s) and HS Tariff Classification Number(s)		8. (cont'd) Origin Criterion
Description	HS # (6-digit level)	

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**Figure F-3. International Shipping Form (2 of 3)**



## Certification of Origin Form (USMCA/CUSMA/T-MEC)

### Instructions

A certification of origin that is the basis for a claim for preferential tariff treatment under the USMCA shall include the below data elements in accordance with the below instructions. UPS provides customers with this information in an effort to help them better understand the USMCA. This information, however, including this USMCA certification of origin form and any other UPS-provided USMCA information, is for informational purposes only. Neither the certification of origin form nor the USMCA information constitutes legal advice. In addition, you, the customer ("you"), agree that it is your responsibility to confirm – and that you have confirmed – that any and all goods for which you claim USMCA preferential treatment and/or that you identify on this USMCA certification of origin form meet all of the applicable USMCA rules of origin for that good(s) (e.g., tariff shift, regional value content, steel and aluminum content, labor value content, etc.) and any other applicable USMCA requirement.

- 1. Importer, Exporter or Producer Certification of Origin**  
Indicate whether the certifier is the exporter, producer or importer.
- 2. Blanket Period**  
Include the period if the certification covers multiple shipments of identical goods for a specified period of up to 12 months.
- 3. Certifier**  
Provide the certifier's name, title, address (including country), telephone number, and email address.
- 4. Exporter**  
Provide the exporter's name, address (including country), e-mail address, and telephone number if different from the certifier. This information is not required if the producer is completing the certification of origin and does not know the identity of the exporter. The address of the exporter shall be the place of export of the good in a Party's territory.
- 5. Producer**  
Provide the producer's name, address (including country), e-mail address, and telephone number, if different from the certifier or exporter or, if there are multiple producers, state "Various" or provide a list of producers. A person that wishes for this information to remain confidential may state "Available upon request by the importing authorities". The address of a producer shall be the place of production of the good in a Party's territory.
- 6. Importer**  
Provide, if known, the importer's name, address, e-mail address, and telephone number. The address of the importer shall be in a Party's territory.
- 7. Description and HS Tariff Classification of the Good**
  - (a) Provide a description of the good and the HS tariff classification of the good to the 6-digit level (except, in the case that the relevant rule of origin for the good requires eight digits, identify to the 8-digit level using the HS tariff classification of the USMCA Party into whose territory the good is imported). The description should be sufficient to relate it to the good covered by the certification; and
  - (b) If the certification of origin covers a single shipment of a good, indicate, if known, the invoice number related to the exportation.
- 8. Origin Criterion**  
General Rules of Origin (RoO) Section 202 of the USMCA Implementation Act specifies the rules of origin used to determine whether a good qualifies as an originating good under the Agreement. The HTSUS GN 11 includes both the general and specific rules of origin, definitions, and other related provisions. In general, under the USMCA, a good is originating based on the following four RoO criterion A-D and the good satisfies all other applicable requirements:
  - **Criterion A:** The good is wholly obtained or produced entirely in the territory of one or more of the USMCA countries, as defined in Article 4.3 of the Agreement;
  - **Criterion B:** The good is produced entirely in the territory of one or more of the USMCA countries using non-originating materials, provided the good satisfies all applicable requirements of product-specific rules of origin;
  - **Criterion C:** The good is produced entirely in the territory of one or more of the USMCA countries exclusively from originating materials; or
  - **Criterion D:** The good is produced entirely in the territory of one or more of the USMCA 6 countries. It is classified with its materials, or satisfies the "unassembled goods" requirement, and meets a regional value content threshold of not less than 60% if the transaction value method is used, or not less than 50% if the net cost method is used (not including RVC for autos); except for goods in Chapter 61-63 of the HTSUS.

<https://www.cbp.gov/sites/default/files/assets/documents/2020-Jun/Updated%20USMCA%20Interim%20Implementing%20Instructions%20-%202020%20Sep%2016.pdf>

- 9. Certification, Authorized Signature and Date**  
The certification must be signed and dated by the certifier and accompanied by the following statement:  
I certify that the goods described in this document qualify as originating and the information contained in this document is true and accurate. I assume responsibility for proving such representations and agree to maintain and present upon request or to make available during a verification visit, documentation necessary to support this certification.

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Figure F-4. International Shipping Form (3 of 3)

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## APPENDIX G SAMPLING CHECKLISTS

**Table G-1. Personnel Preparation Checklist**


(Page 1 of 2)

Personnel Briefing	Yes	No	Comments
1. Did you review sampling team responsibilities and identify individual(s) responsible for corrective actions?			
2. Did you ensure that you have met the appropriate personal safety and protection requirements?			
3. Did you identify sampling locations and receive permission to access them, as appropriate?			
4. Did you contact the appropriate utility companies PRIOR to the start of sampling?   By law, utility companies must be contacted prior to the start of digging/sampling so that any underground utilities (gas lines, water lines, electrical lines, etc.) can be marked. A list of one-call centers for each state may be found at: <a href="https://call811.com">https://call811.com</a>			
5. If sampling on private property, do you have sample receipts to provide to the property owner for all samples collected and removed from the property?			
6. Have you determined the number and type of samples to be collected?			
7. Did you review sample collection methods?			
8. Have you reviewed sample container requirements?			
9. Did you review decontamination requirements, procedures, and locations?			
10. Did you determine holding times and conditions?			
11. Did you determine Performance Evaluation (PE) and Quality Control (QC) sample requirements?			
12. Have you obtained shipping container temperature blanks, if required?			
13. Did you review sample label requirements?			
14. Did you review Traffic Report/Chain of Custody (TR/COC) Record and custody seal requirements?			
15. Have you obtained the laboratory name, shipping addresses, and telephone number?			
16. Did you review shipping container return instructions?			
17. Have you obtained shipping company information (name, telephone number, account number, and pickup schedule)?			
18. Have you obtained shipping schedules?			
19. Did you review shipment reporting requirements and the appropriate contact names and telephone numbers for reporting?			


**Table G-1. (Continued) Personnel Preparation Checklist**  
**(Page 2 of 2)**

Personnel Briefing	Yes	No	Comments
20. Have you included any sampler comments regarding sampling issues (e.g., low volumes, matrix, suspected concentrations based on field measurements)?			




**Table G-2. General Sample Collection Checklist**  
(Page 1 of 1)

General Sample Collection	Yes	No	Comments
1. Did you identify and mark the sampling location with buoys, flags, or stakes according to the sampling plans, maps, and grids?			
2. If the sampling location is inaccessible, did you contact the appropriate field or Regional personnel for instructions?			
3. Did you use the correct sampling equipment?			
4. Did you follow the correct decontamination procedures?			
5. Did you follow the correct collection procedures?			
6. Did you use the correct sample containers for each sample collected?			
7. Did you use certified clean containers for all samples? Are certificates kept on record?			
8. Did you use certified clean water for all field, trip, equipment, and rinsate blanks? Are certificates kept on record?			
9. Did you collect the correct volume for each sample?			
10. Did you collect the correct type of sample, including primary samples and QC samples?			
11. Did you properly preserve each sample collected?			
12. Did you correctly document and label each sample with all necessary information?   Under no circumstances should the site name or address appear on any documentation being sent to the laboratory, unless the laboratory is a Regional U.S. Environmental Protection Agency (EPA) laboratory.			
13. If sampling on private property, did you provide a sample receipt to the owner of the property for all samples collected and removed from the property?			

**Table G-3. Completing Field Logbook Checklist****(Page 1 of 1)**

Completing Field Logbook	Yes	No	Comments
1. Did you use waterproof ink when writing in the field logbook?			
2. Did you document sampling project information such as: <ul style="list-style-type: none"> <li>• Project name, Project ID, and location</li> <li>• Names of samplers</li> <li>• Geological observations, including maps</li> <li>• Atmospheric conditions</li> <li>• Field measurements</li> <li>• Sampling dates, times, and locations?</li> </ul>  Under no circumstances should the site name or address appear on any documentation being sent to the laboratory, unless the laboratory is a Regional EPA laboratory.			
3. Did you record sampling activity information such as: <ul style="list-style-type: none"> <li>• Sampling dates and times</li> <li>• Sample identifications</li> <li>• Sample matrices</li> <li>• Sample descriptions (e.g., odors and/or colors)</li> <li>• Number of samples collected</li> <li>• Sampling methods/equipment</li> <li>• Description of QC samples?</li> </ul>			
4. Did you document any and all deviations from the sampling plan?			
5. Did you document any and all difficulties in sampling and/or any unusual circumstances?			
6. Were all errors corrected by crossing a line through the error, initialing the error, dating the error, and then adding the correct information?			


**Table G-4. Completing Handwritten Sample Labels Checklist**  
(Page 1 of 1)

Completing Handwritten Sample Labels	Yes	No	Comments
1. Did the Regional Sample Control Coordinator (RSCC) provide Contract Laboratory Program (CLP) Sample Numbers and Sample Management Office (SMO) Contract Laboratory Program Support System (CLPSS) Portal-generated CLP Case Numbers?			
2. If additional CLP Sample Numbers were needed, did you contact the appropriate Regional personnel?			
3. Were the CLP Sample Numbers and SMO CLPSS Portal-generated CLP Case Numbers on the labels correct?			
4. Were samples uniquely numbered and designated to only one sample?   Samples collected for total metal and filtered metal analyses must receive separate, unique, CLP Sample Numbers.			
5. Were QC samples numbered accordingly?			
6. Were the specific requirements followed for total and filtered metals analysis, QC and PE samples, and SW-846 Method 5035A?			
7. Were all temperature blanks labeled with "TEMPERATURE BLANK"?			
8. Was a sample label containing the CLP Sample Number, SMO CLPSS Portal-generated CLP Case Number, location, concentration, preservative, and the analysis, attached to each sample bottle or container as the sample was collected?   Under no circumstances should the site name or address appear on any documentation being sent to the laboratory, unless the laboratory is a Regional EPA laboratory.			
9. Was clear tape placed over the sample labels to protect the labels from moisture and to help the labels adhere to the sample bottle?   Use only CLEAR tape over the sample labels and avoid wrinkles in the tape and the sample labels.			
10. Were all errors corrected by drawing a line through the error, initialing and dating the error, and then adding the correct information?			



**Table G-5. Completing Handwritten Custody Seals Checklists****(Page 1 of 1)**

<b>Completing Custody Seals</b>	<b>Yes</b>	<b>No</b>	<b>Comments:</b>
1. If required for the project, did you sign and date the custody seal?			
2. Did you attach a completed custody seal to the sample bottle, container, or plastic bag, placing the seal over the cap or lid of each sample bottle or container or on the bag opening such that it will be broken if the sample bottle, container, or bag is opened or tampered with?			
3. As appropriate, did you attach the completed custody seal to the sample shipping container or cooler, placing the seal such that it will be broken if the container or cooler is opened or tampered with?			
4. Were all errors corrected by crossing a line through the error, initialing and dating the error, and then adding the correct information?			

**Table G-6. Packing Sample Container Checklist****(Page 1 of 1)**

Packing Sample Container	Yes	No	Comments
<p>1. Did you follow all State, Federal, Department of Transportation (DOT), and International Air Transportation Association (IATA) regulations governing the packaging of environmental and hazardous samples?</p>  <p>If samples contain methanol preservation (e.g., samples to be analyzed by SW-846 Method 5035A), refer to the packaging instructions in Appendix C.</p>			
<p>2. Were all CLP Sample Numbers, SMO CLPSS Portal-generated CLP Case Numbers, analyses, labels, and custody seals attached to the correct sample containers?</p>			
<p>3. Is Modified Analysis indicated if requested?</p>			
<p>4. Was an inventory conducted of CLP Sample Numbers, SMO CLPSS Portal-generated CLP Case Numbers, analyses, and containers, and verified against the TR/COC Records?</p>			
<p>5. Were the correct number and type of PE and QC samples collected?</p>			
<p>6. Were all sample containers sealed in clear plastic bags with the sample label and tag (if applicable) visible through the packaging?</p>			
<p>7. Was suitable absorbent packing material placed around the sample bottles or containers?</p>			
<p>8. Were the outsides of metal containers labeled properly with the CLP Sample Number, SMO CLPSS Portal-generated CLP Case Number, and the analysis of the sample inside?</p>			

**Table G-7. Packing Shipping Container Checklist**  
(Page 1 of 2)

Packing Shipping Container	Yes	No	Comments
1. Were shipping samples in a clean waterproof metal or hard plastic ice chest, cooler, or container in good condition?			
2. Were all non-applicable labels from previous shipments removed from the container?			
3. Were all inside and outside drain plugs closed and covered with suitable tape (e.g., duct tape)?			
4. Was the inside of the shipping container lined with plastic (e.g., large heavy-duty garbage bag)?			
5. Was the lined shipping container packed with non-combustible absorbent packing material?			
6. Were sample containers placed in the shipping container in an upright position not touching one another?			
7. Was a shipping container temperature blank included in the cooler?			
8. Did the documentation in the cooler only address the samples in that container?			
9. Was the site name absent from all documentation?   Under no circumstances should the site name or address appear on any documentation being sent to the laboratory, unless the laboratory is a Regional EPA laboratory.			
10. Was there sufficient packing material around and in between the sample bottles to avoid breakage during transport?			
11. If required, was double-bagged ice placed on top and around sample bottles to keep the samples cold at $\leq 6^{\circ}\text{C}$ ?   Do not pack loose ice into the cooler.			
12. Was the top of the plastic liner fastened and secured with tape?			
13. Was a completed custody seal placed around the top of the fastened plastic liner (if required by the Region)?			
14. Were all sample documents enclosed within the shipping container (e.g., TR/COC Record, PE instructions, and container return instructions) in a waterproof plastic bag?			
15. Was the plastic bag, containing the documentation, taped to the underside of the lid?			
16. Were shipping container return instructions and airbills taped to the underside of the cooler lid?			
17. Was the return address of the shipping container written with permanent ink on the underside of the lid?			
18. Was tape placed around the outside of the entire container and over the hinges?			
19. Were the completed custody seals placed over the top edge of the container so the container cannot be opened without breaking the seals?			
20. Was the return address label attached to the top left corner of the container lid?			

**Table G-7. Packing Shipping Container Checklist**  
(Page 2 of 2)

Packing Shipping Container	Yes	No	Comments
21. Were instructional labels attached to the top of the container, as necessary (e.g., "This End Up," "Do Not Tamper With," or "Environmental Laboratory Samples")?			
22. Have all U.S. DOT regulations been met for the shipment when shipping hazardous samples?			
23. If shipping samples containing methanol as a preservative (e.g., samples to be analyzed by SW-846 Method 5035A), was a label used to indicate the presence of methanol, the United Nations (UN) identification number for methanol (UN 1230), and Limited Quantity?			

**Table G-8. Shipping & Reporting CLP Samples Checklist****(Page 1 of 1)**

<b>Shipping CLP Samples</b>	<b>Yes</b>	<b>No</b>	<b>Comments:</b>
1. Did you follow all State, Federal, DOT, and IATA regulations governing the shipment of environmental and hazardous samples?			
2. Was a separate airbill filled out for each container being shipped?			
3. Was the airbill filled out completely, including correct laboratory name, address, and telephone number, identification of recipient as "Sample Custodian," and appropriate delivery option (e.g., overnight or Saturday)?			
4. Was the completed airbill attached to the top of the container with the correct laboratory address?			
5. If more than one container was being shipped to the same laboratory, were they marked as "1 of 2," "2 of 2," etc.?			
6. Were the samples being shipped "overnight" through a qualified commercial carrier?			
7. Did you export the electronic COC XML file from Scribe?			
8. Did you upload the electronic COC XML file using the Submit Chain of Custody task in the SMO CLPSS Portal?			
<b>Reporting CLP Samples</b>	<b>Yes</b>	<b>No</b>	<b>Comments:</b>
1. Did you contact the RSCC (or designee) and the Contract Laboratory Program Sample Management Office (SMO) on the same day samples were shipped?			
2. If the samples were shipped after 5:00 PM Eastern Time (ET), were they reported to the RSCC (or designee) and to SMO by 8:00 AM ET the following business day?			
3. Did you notify the RSCC (or designee) and SMO so that SMO will receive the delivery information (including shipping airbill information) by 3:00 PM ET on Friday for sample shipments that will be delivered to the laboratory on Saturday?			
4. Did you provide the RSCC (or designee) or the SMO CLPSS Portal with: <ul style="list-style-type: none"> <li>• Your name, phone number, email address, and Region number;</li> <li>• Case Number of the project;</li> <li>• Modified Analysis Number, if requested;</li> <li>• Exact number of samples, matrix(ces), and type of analysis;</li> <li>• Laboratory(ies) to which the samples were shipped;</li> <li>• Carrier name and airbill number;</li> <li>• Date of shipment;</li> <li>• Date of next shipment; and</li> <li>• Any other information pertinent to the shipment?</li> </ul>			

## APPENDIX H GLOSSARY

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**Analyte** -- The specific compound, mixture, element, or ion an analysis seeks to determine.

**Analytical Services Branch (ASB)** -- The branch of the Technology Innovation and Field Services Division (TIFSD) of the United States Environmental Protection Agency's (EPA's) Office of Superfund Remediation and Technology Innovation (OSRTI) responsible for the overall management of the Contract Laboratory Program (CLP) under the Office of Land and Emergency Management (OLEM).

**Aroclor** -- Polychlorinated biphenyls (PCBs) or a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl and a general chemical formula of  $C_{12}H_{10-x}Cl_x$ . PCBs, commercially produced as complex mixtures containing multiple isomers at different degrees of chlorination, were marketed in North America under the trade name Aroclor.

**Case** -- A finite, usually predetermined, number of samples collected over a given time period from a particular site. Case Numbers are assigned by the Sample Management Office (SMO). A Case consists of one or more Sample Delivery Groups (SDGs).

**Chlorinated Dibenzo-*p*-Dioxin/Chlorinated Dibenzofuran (CDD/CDF)** -- A group of organic compounds of tetra- through octa-chlorinated dibenzo-*p*-dioxins (CDDs) and dibenzofurans (CDFs).

**Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)** -- First authorized by Congress in December 1980, and amended in 1986, CERCLA provided broad Federal authority to respond directly to the release or possible release of hazardous substances that may endanger human health or the environment. CERCLA also established a Trust Fund to provide for cleanup when no responsible party could be identified; hence, CERCLA is commonly referred to as "Superfund."

**Congener** -- Individual compound belonging to a group or class of compounds with a similar general structure.

**Contract Laboratory Program (CLP)** -- Supports the EPA's Superfund effort by providing a range of state-of-the-art chemical analytical services of known and documented quality. This program is directed by the Analytical Services Branch (ASB) of the Technology Innovation and Field Services Division (TIFSD) Office of the Superfund Remediation and Technology Innovation (OSRTI) of the EPA.

**Custody Seal** -- An adhesive label or tape that is used to seal a sample bottle or container that maintains chain of custody and that will break if the sample bottle or container is opened or tampered with.

**Cyanide (Total)** -- Cyanide ion and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

**Data Quality Objectives (DQO)** -- Qualitative and quantitative statements that clarify technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

**Data Validation** -- Data validation is based on Region-defined criteria and limits, professional judgment of the data validator, and (if available) the Quality Assurance Project Plan (QAPP) and Sampling and Analysis Plan (SAP).

**Deuterated Monitoring Compound (DMC)** -- Compounds added to every Gas Chromatograph/Mass Spectrometer (GC/MS) calibration standard, blank, and sample to evaluate the efficiency of the extraction/purge-and-trap procedures, and the performance of the GC/MS systems. DMCs are isotopically labeled (deuterated) analogs of native target compounds. DMCs are not expected to be naturally detected in the environmental media.

**Duplicate** -- Quality Control (QC) sample required by the laboratory's contract to check the accuracy and precision of inorganic analyses. It is a second aliquot of the same sample to determine the precision of the method.

**Equipment Blank** -- A sample used to check field decontamination procedures. See Field Blank.

**Field Blank** -- Any sample that is submitted from the field and identified as a blank. A field blank is used to check for cross-contamination during sample collection, sample shipment, and in the laboratory. A field blank includes trip blanks, rinsate blanks, bottle blanks, equipment blanks, preservative blanks, decontamination blanks, etc.

**Field Duplicate** -- A duplicate sample generated in the field, not in the laboratory. Checks reproducibility of laboratory and field procedures and indicates non-homogeneity.

**Field QC Sample** -- Any QC samples submitted from the field to the laboratory. Examples include, but are not limited to, field blanks, field duplicates, and field spikes.

**Field Sample** -- Primary sample material collected from the field from which other samples, such as duplicates or split samples are derived. A field sample can be prepared in the field and sent for analysis in one or multiple containers, and is identified by a unique EPA sample number.

**Field Sampling Plan (FSP)** -- Developed to outline the actual steps and requirements pertaining to a particular sampling event, and explains, in detail, each component of the event to all involved samplers.

**Holding Time** -- The elapsed time expressed in hours, days, or months from the date of collection of the sample until the date of its analysis.

**Contractual** -- The maximum length of time that the CLP laboratory can hold samples prior to extraction and/or analysis, as specified in the CLP analytical services Statements of Work (SOWs).

**Technical** -- The maximum length of time that samples may be held from time of collection to time of preparation and/or analysis and still be considered valid.

**Laboratory Blank** -- See Method Blank.

**Laboratory Duplicate** -- A sample required by the laboratory's contract to check the precision of inorganic analyses.

**Laboratory QC Sample** -- An additional volume of an existing sample, as required by the laboratory's contract, used to detect contamination or error in the laboratory's practices.

**Matrix** -- The predominant material of which a sample to be analyzed is composed. Matrix is not synonymous with phase (liquid or solid).

**Matrix Spike (MS)** -- QC sample required by the laboratory's contract to check the accuracy of organic and inorganic analyses. It is an aliquot of a sample (water or soil) that is fortified (spiked) with known quantities of a specific analyte and subjected to the entire analytical procedure to indicate the appropriateness of the method for the matrix by measuring recovery. See Matrix Spike Duplicate.

**Matrix Spike Duplicate (MSD)** -- QC sample required by the laboratory's contract to check the accuracy and precision of organic analyses. It is a second aliquot of the same matrix as the Matrix Spike (MS) that is spiked to determine the precision of the method. See Matrix Spike.

**Method Blank** -- An analytical control consisting of all reagents, internal standards and surrogate standards (or DMCs for GC/MS organic analysis), that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination, also referred to as laboratory blank when defining the level of laboratory contamination.

**Modified Analysis (MA)** -- A contractual document with modified Statement of Work (SOW) requirements that append to the SOW. All contract and analytical SOW requirements remain in effect unless explicitly modified.

**Performance Evaluation (PE) Sample** -- A sample of known composition to the EPA; however, it is unknown to the Contractor and is provided to evaluate Contractor performance.

**Pesticides** -- Substances intended to repel, kill, or control any species designated a “pest,” including weeds, insects, rodents, fungi, bacteria, and other organisms. Under the CLP, only organochlorine pesticides are analyzed (e.g., DDT, Dieldrin, Endrin, etc.).

**Polychlorinated Biphenyls (PCBs)** -- A group of toxic, persistent chemicals used in electrical transformers and capacitors for insulating purposes, and in gas pipeline systems as a lubricant. The sale and new use of PCBs were banned by law in 1979.

**Quality Assurance (QA)** -- An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

**Quality Assurance Project Plan (QAPP)** -- Document written to meet requirements outlined in the document *EPA Guidance for Quality Assurance Project Plans* (EPA QA/R-5). Prepared in advance of field activities and used by samplers to develop any subsequent plans such as the Sampling and Analysis Plan (SAP) or the Field Sampling Plan (FSP).

**Quality Control (QC)** -- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality.

**Regional Sample Control Coordinator (RSCC)** -- In most Regions, coordinates sampling efforts and serves as the central point of contact for sampling questions and problems. Also assists in coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services.

**Regional Site Manager** -- Coordinates the development of data quality objectives and oversees project-specific remedial or removal contractors, State officials, or private parties conducting site sampling efforts.

**Rinsate Blank** -- A sample used to check decontamination procedures. Also see Field Blank.

**Sample** -- A discrete portion of material to be analyzed that is contained in single or multiple containers, and identified by a unique sample number.

**Sample Delivery Group (SDG)** -- A unit within a sample Case that is used to identify a group of samples for delivery. An SDG is defined by the following, whichever is most frequent:

- Each Case of field samples received; or
- Each 20 field samples (excluding PE samples) within a Case; or
- Each 7 calendar day period (3 calendar day period for 7-day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).

In addition, all samples and/or sample analyses assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG. Sample may be assigned to SDGs by matrix (e.g., all soil samples in one SDG, all water samples in another) at the discretion of the laboratory.

**Sample Label** -- An identification label attached to a sample bottle or container to identify the sample.

**Sample Management Office (SMO)** -- A Contractor-operated facility operated under the SMO contract, awarded and administered by the EPA.

**Sample Number** -- A unique number used to identify and track a sample. This number can be recorded on a sample label or written on the sample bottle or container using indelible ink.

**Sampling and Analysis Plan (SAP)** -- A document that explains how samples are to be collected and analyzed for a particular sampling event.

**Scribe** -- A stand-alone Windows-based desktop application that samplers can use to automatically create and generate sample documentation prior to and during sampling events.

**Semivolatile Organic Analyte (SVOA)** -- A compound amenable to analysis by extraction of the sample using an organic solvent.

**Standard Operating Procedure (SOP)** -- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps, and that is officially approved as the methods for performing certain routine or repetitive tasks.

**Statement of Work (SOW)** -- A document that specifies how laboratories analyze samples under a particular Contract Laboratory Program (CLP) analytical program.

**Superfund** -- The program operated under the legislative authority of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and Superfund Amendments and Reauthorization Act (SARA), that funds and carries out EPA removal and remedial activities at hazardous waste sites. These activities include establishing the National Priorities List (NPL), investigating sites for inclusion on the list, determining their priority, and conducting and/or supervising cleanup and other remedial actions.

**Superfund Amendments and Reauthorization Act (SARA)** -- The 1986 amendment to the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

**Traffic Report/Chain of Custody (TR/COC) Record** -- A record that is functionally similar to a packing slip that accompanies a shipment of goods. Used as physical evidence of sample custody and functions as a permanent record for each sample collected.

**Trip Blank** -- A sample used to check for contamination during sample handling and shipment from field to laboratory. Also see Field Blank.

**Volatile Organic Analyte (VOA)** -- A compound amenable to analysis by the purge-and-trap technique. Used synonymously with the term purgeable compound.



Copies of all reference materials are available upon request through LDCE