

## Field Sampling Plan

Solvay Specialty Polymers USA, LLC

10 Leonard Lane

West Deptford, NJ 08086

Program Interest No: 015010

*Prepared by*

**Integral Consulting Inc.**

923 Haddonfield Road

Suite 300

Cherry Hill, NJ 08002

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## ACRONYMS AND ABBREVIATIONS

bgs	below ground surface
COC	chain-of-custody
DGPS	differential global positioning system
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
FSPM	Field Sampling Procedures Manual
GPS	global positioning system
HSP	health and safety plan
Integral	Integral Consulting Inc.
LDPE	low-density polyethylene
NAD83	North American Datum of 1983
NJDEP	New Jersey Department of Environmental Protection
NOAA	National Oceanic and Atmospheric Administration
NWIS	National Water Information System
PDB	passive diffusion bag
PFAS	per- and polyfluoroalkyl substances
PVC	polyvinyl chloride
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
SGS	SGS North America, Inc.
Site	West Deptford, New Jersey, Plant
Solvay	Solvay Specialty Polymers USA, LLC
SOP	standard operating procedure
TOC	total organic carbon
USGS	U.S. Geological Survey
VOC	volatile organic compound
WGS84	World Geodetic System of 1984

# 1 INTRODUCTION

This document presents the field sampling plan (FSP) that has been prepared on behalf of Solvay Specialty Polymers USA, LLC (Solvay) to study per- and polyfluoroalkyl substances (PFAS) at the West Deptford, New Jersey, Plant site (the Site). The procedures for standard sampling techniques generally conform to the New Jersey Department of Environmental Protection (NJDEP) Field Sampling Procedures Manual (August 2005) and U.S. Environmental Protection Agency (EPA) protocols and, if necessary, are modified to avoid cross-contamination from PFAS-containing equipment.

The 234-acre Site is located at 10 Leonard Lane in West Deptford Township, Gloucester County, New Jersey, on the Delaware River across from the Philadelphia Airport (Figure 1). Active plant operations occur on approximately 34 acres of the property, with the remainder either in a natural state or developed as a solar farm. The far northern area of the Site contains dredge spoils placed there by the U.S. Army Corps of Engineers in the 1960s (ERM 2013). The western edge of the property borders Little Mantua Creek, with undeveloped property located to the east, and a rail line to the south.

Prior to 1970, the property was used for agriculture. Fluorocarbon manufacturing began when Pennwalt constructed a facility in 1970. Manufacturing ceased in 1977, and a new facility was constructed from 1983 to 1985; production of vinylidene fluoride monomers and polymers using PFAS in the manufacturing process started in 1985 (ERM 2013). The facility was purchased by Elf Atochem in 1989 and operated until it was sold to Ausimont USA, Inc. in 1990. The Solvay Group acquired the holding of the parent company of Ausimont in May 2002, and changed the name to Solvay Solexis, Inc. on January 1, 2003. The company and facility name were then changed to Solvay Specialty Polymers USA, LLC on October 31, 2012 (ERM 2013).

The primary objective of remedial investigation is to determine if PFAS are present in Site groundwater, soils, and/or surface water and sediments in the Delaware River.

To execute this investigation, Integral Consulting Inc. (Integral) will conduct the fieldwork and data analysis outlined in this FSP and in the quality assurance project plan (QAPP). The names and quality assurance responsibilities of key project personnel for Integral who will be involved in sampling and analysis activities are provided in the QAPP.

## 1.1 OVERVIEW

The sampling design incorporates a number of different components and sampling media. Future investigation activities involving these same environmental media will refer to this document for general procedures and protocols. As such, this document serves as a project-

specific FSP, subject to one or more addenda that will provide additional details regarding sampling goals and numbers and locations of samples to be collected.

## **1.2 DOCUMENT ORGANIZATION**

This FSP describes the field methods that will be used to collect samples to meet the objectives of the remedial investigation. The background, rationale, data quality objectives, and overall study design are described in detail in the QAPP. Section 2 of this FSP describes the field procedures and sample packaging and shipping requirements that will be followed by the technical team during the field study. Section 3 summarizes field documentation and chain-of-custody (COC) procedures. Field data reporting and field custody procedures are discussed in Section 4.

The following documents are provided as attachments to this FSP:

- Standard Operating Procedures (SOPs). The SOPs describe the procedures that will be used to collect groundwater, surface water, and surface and subsurface sediments (Attachment 1).
- Field Forms. This attachment contains examples of various forms that will be used during field sampling, including a corrective action record, a field change request form, and a COC form (Attachment 2).

The Site health and safety plan (HSP) has been updated to address additional field activities. This document describes the specific requirements and procedures that will be implemented to minimize the safety risk to personnel who carry out the field study program for groundwater, surface water, sediment, and soil collection.



## 2 SAMPLING PROCEDURES

The following sections describe the detailed procedures and methods that will be used during field sampling events, including sampling procedures, recordkeeping, sample handling, storage, and field quality control procedures. Sample collection and processing will be conducted in accordance with the SOPs provided in Attachment 1. Depending on field conditions, procedures specified in the referenced SOPs may be modified if necessary. All field activities will be conducted in accordance with the HSP prepared for the project.

### 2.1 FIELD EQUIPMENT AND SUPPLIES

Field equipment and supplies include sampling equipment, decontamination supplies, sample containers, coolers, shipping containers, log books and forms, personal protection equipment, and personal gear. Protective wear (e.g., gloves) is required to minimize the possibility of cross-contamination between locations. Additional information on protective wear required for this project is provided in the HSP prepared for the Site.

Sample jars, preservatives, distilled/deionized water, coolers, and packaging material for the samples will be supplied by the analytical laboratory. Pursuant to the NJDEP sampling guidance (NJDEP 2005) and where practicable, sample containers will not be held for more than 4 days between sample container shipment from the testing laboratory and sample shipment back to the laboratory for analysis. Details on the numbers and type of sample containers are provided in the QAPP. The field lead and field personnel in charge of sample handling in the field will use a sample matrix table created during each scope of work as a quality control check to ensure that all samples have been collected at a given location.

Commercially available, pre-cleaned jars will be used for the samples, and the testing laboratories will maintain a record of certification from the suppliers. The bottle shipment documentation will include batch numbers. With this documentation, jars can be traced to the supplier, and bottle-wash analysis results can be reviewed. The bottle-wash certificate documentation will be archived in Integral's project file.

Sample containers will be clearly labeled at the time of sampling. Labels will include the task name, sample number, sampler's initials, analyses to be performed, and sample date and time. Sample numbering and identification procedures are described in detail in Sections 3.3 and 3.4.

### 2.2 LOCATION/LOCATION POSITIONING

Latitude and longitude coordinates will be obtained at each sample location proposed in the work plan. A differential global positioning system (DGPS) will be used to document the actual

location where samples were collected. The standard projection method to be used during field activities is New Jersey state plane feet (the North American Datum of 1983; NAD83); coordinates will be collected in the field using World Geodetic System of 1984 (WGS84). The positioning objective is to accurately determine and record the positions of all sampling locations to within  $\pm 2$  m.

The DGPS unit consists of a global positioning system (GPS) receiver and a differential receiver located at a horizontal control point. At the control point, the GPS-derived position is compared with the known horizontal location, offsets or biases are calculated, and the correction factors are telemetered to the GPS receiver. Positioning accuracies on the order of  $\pm 1$  to 3 m can be achieved by avoiding the few minutes per day when the satellites are not providing the appropriate quality of signal (SOP AP-06, Attachment 1). The GPS unit provides the operator with a listing of the time intervals during the day when accuracies are decreased.

## 2.3 SAMPLING VESSEL

If necessary, surface water and sediment samples will be collected from a sampling vessel appropriate in size to accommodate the necessary sampling equipment and personnel and to operate safely within the area of the river/creek to be navigated for sample collection.

## 2.4 WEATHER

During sampling activities, weather will be monitored using the following web site:

Weather conditions and forecasts: National Oceanic and Atmospheric Administration (NOAA) site for the Philadelphia area <http://graphical.weather.gov/sectors/phi.php#tabs>.

## 2.5 SPECIAL SAMPLING CONCERNS

Because PFAS are also found in numerous everyday items, the following special precautions will be taken during all sampling activities:

- No Teflon<sup>®</sup>-containing materials (e.g., Teflon<sup>®</sup> tubing, bailers, tape, sample jar lid liners, plumbing paste) will be used.
- No Tyvek<sup>®</sup> clothing will be worn onsite.
- Clothes treated with stain- or rain-resistant coatings will be avoided or go through several washings prior to use onsite.
- No Post-It<sup>®</sup> notes will be brought onsite.
- No fast-food wrappers, disposable cups, or microwave popcorn will be brought onsite.

- After handling any of the above items, field personnel will wash their hands thoroughly with soap and water prior to any sampling activities.
- No use of chemical (blue) ice packs or foil will be allowed.
- Any insect repellent or sunscreen used during PFAS sampling should not contain any chemicals containing “fluor” in the ingredients list. Any insect repellent or sunscreen used during the sampling events will be applied downwind and well away from sample containers and sample collection equipment.

Nitrile gloves will be worn during all sample collection activities.

## **2.6 EQUIPMENT DECONTAMINATION**

Decontamination procedures for groundwater, surface water, sediment, and soil sampling equipment are provided in the following sections.

### **2.6.1 Groundwater**

Procedures are provided below for decontamination of groundwater sampling equipment used during low-flow and passive sampling methodologies.

#### **2.6.1.1 Low-Flow Sampling and Purging**

Prior to pump installation and collection of each groundwater sample, the pump will be decontaminated and fitted with new ¼-in. polyethylene tubing, eliminating the need for field decontamination of the tubing.

Pump decontamination procedures are as follows:

1. Disassemble the pump and dispose of the used bladder.
2. Rinse the parts with tap water.
3. Vigorously scrub all parts with brush and laboratory-grade standard detergent (e.g., Alconox®) and tap water.
4. Generously rinse with tap water or store-purchased distilled/deionized water.
5. Allow to air dry.
6. Install a new bladder in the pump.
7. Reassemble the pump.

Water level measuring tapes and the flow-through cell will be decontaminated following above procedures in Steps 2 through 6.

### **2.6.1.2 Passive Diffusion Bag (PDB) Samplers**

No reusable materials are used as part of Passive Diffusion Bag (PDB) sampling aside from water level measuring tapes. All dedicated and disposable items, including tethers, bags, weights, etc., will be verified PFAS-free prior to installation into the monitoring well and sampling. Water level measuring tapes will be decontaminated following the procedures described in Steps 2 through 6 of Section 2.6.1.1.

When items are removed from the monitoring well for sampling, they will be placed on PFAS-free sheeting to avoid contact with the ground or materials that may contain PFAS.

### **2.6.2 Surface Water**

Dedicated ¼-in. low-density polyethylene (LDPE) tubing and polycarbonate mixing containers that will be used for collection of surface water samples will be supplied by the analytical laboratory. Therefore, there will be no decontamination of surface water sampling equipment in the field, except for the cleaning of the surface of the work area and water sampler polyvinyl chloride (PVC) structures with a soapy solution (e.g., Alconox®) and a rinse with deionized water and water from the sample locations, respectively, prior to sampling.

### **2.6.3 Sediment**

Before surface sediment sampling begins at a location, the grab sampler will be scrubbed with a standard detergent (e.g., Alconox®), rinsed with water (river, tap, or deionized water), air-dried, and rinsed with river water. Equipment used for compositing the sediment samples (i.e., stainless-steel pot, bowls, and spoons) will follow the same basic decontamination sequence, except that the final rinse will be with laboratory-grade distilled/deionized water. After cleaning, the decontaminated stainless-steel spoon will be placed inside the decontaminated stainless-steel pot and will be covered with the stainless-steel lid for the pot to protect it from possible contamination. The sediment sample collection equipment will be decontaminated immediately before it is used. Decontaminated equipment will not be stored prior to use.

Prior to subsurface sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox®), rinsed with laboratory grade distilled/deionized water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination.

All non-dedicated sampling equipment that comes into contact with the sediment samples (e.g., core catchers, grab samplers, core liners, stainless-steel bowls, pots, and spoons) will be decontaminated prior to use and between samples. Non-dedicated sampling equipment will be decontaminated following procedures in SOP SD-01 (Attachment 1), except that no solvent rinse will be used.

## 2.6.4 Soil

Decontamination procedures for soil sampling are generally similar to those used for sediment sampling, except that equipment rinses will be conducted with deionized/distilled water and tap water. Soil samples will be collected from a 6-in. discrete interval from the disposable acetate liner using a decontaminated stainless-steel spoon and placed into laboratory-provided sampling containers.

All stainless steel macrocores will be decontaminated between intervals in sequence with a standard detergent and rinsed with laboratory grade distilled/deionized water.

Sampling spoons used to transfer the core into laboratory-provided containers will be decontaminated before use and between samples. The sampling spoons will be decontaminated by scrubbing with standard detergent and tap water, rinsing with tap water, and rinsing with distilled water. Any other non-dedicated sampling equipment that comes into contact with the soil samples or core liners will be decontaminated prior to use and between samples following procedures in SOP SD-01 (Attachment 1), except that no solvent rinse will be used.

## 2.7 GROUNDWATER SAMPLING

Initial groundwater samples from newly installed monitoring wells will be collected using low-flow purging and sampling methods, and all following groundwater samples will be collected using PDB samplers. Samples collected using both methods will be collected directly into bottles provided by the certified contract laboratory. Anticipated target analytes are included in Table 1. The site-specific QAPP details the specific laboratory method and quality assurance and quality control for sample analysis.

### 2.7.1 Low-Flow Purging and Sampling

Low-flow purging and sampling methods will be employed to collect groundwater samples from newly installed wells. This method was selected based on prior use at the Site to collect volatile organic compound (VOC) samples, reduced production of purge water, and limited disruption to groundwater conditions during sampling. Field procedures are to follow NJDEP and EPA guidance (NJDEP 2005; USEPA 2010). Unlike prior sampling events, no Teflon® disposable bladders or Teflon®-lined tubing will be used, to minimize risk of PFAS contamination from the sampling equipment. Easy to disassemble and clean low-flow bladder pumps with disposable polyethylene bladders will be employed for sample collection (e.g., Solinst 407 Integra). Dedicated or disposable ¼-in. polyethylene tubing will be used to avoid the need for decontamination of tubing.

Pump intakes will be placed at the midpoint of the 10 ft. well screens and pumps will be secured with non-Teflon® coated cord.

As per NJDEP guidance, stabilization parameters will be monitored during low flow purging to determine if well stability has been achieved prior to sampling. Purging will continue until respective measurements fall within the ranges stated below for three consecutive measurements or 4 hours have elapsed, at which time a sample will be collected and the attempts to reach stabilization documented. Measurements will be collected every 5–6 minutes.

Drawdown	<0.3ft
pH	±0.1 unit
Specific conductance	±3%
Temperature	±3%
Dissolved oxygen	±10%
Turbidity	±10% for values >1 NTU
ORP/Eh	±10 millivolts

Care will be taken to minimize the length of tubing between the top of the well casing and the flow-through chamber. A low volume transparent flow-through cell will be used to allow visual observation of water conditions during purging. The flow-through cell will be connected such that water enters the bottom of the cell and exits the top.

Prior to connection of the flow-through cell, tubing will be flushed for up to 10 minutes while initial drawdown measurements are made. Probes used in the flow-through cell will be calibrated in the field prior to the day's sampling event by personnel certified for the collection of stabilization parameters in New Jersey.

Water level measurements will be recorded prior to pump installation. Following pump installation, the water level probe will be suspended in the well at the point representing 0.3-ft drawdown. Water level readings will be recorded every 5 minutes at the time stabilization parameters are measured.

Pump installation will occur following initial water level measurements. Prior to installation, the pump will be properly decontaminated and fitted with appropriate tubing. Once the pump is prepared for installation, the pump will be lowered in a manner that minimizes any disturbance to the well by slowly lowering the pump to the target sampling interval.

The purge rate will be set initially within a range of 100 to 500 mL/min. The initial few minutes of purge water will be discharge to the wastewater container prior to the connection being made to the flow-through cell. Tubing will be connected to the cell with the pump continuing to operate. If drawdown measurements indicate greater than 0.3 ft of drawdown, the pumping rate will be decreased, but not to less than 100 mL/min. Because of the highly permeable nature of the aquifer being sampled, it is not anticipated that drawdown to the well screen will occur.

Following initial adjustments to the pumping rate, stabilization parameters will begin to be recorded.

### **2.7.2 Passive Sampling**

After the initial groundwater samples are collected using low-flow sampling methodologies, groundwater samples thereafter will be collected using PDB samplers. Per the NJDEP Field Sampling Procedures Manual (FSPM), “The use of [PDB samplers] has been approved by the NJDEP at sites within NJ, and generated data may be used for compliance monitoring and/or to demonstrate that clean-up objectives have been achieved for site closure.” In addition, PDB samplers will be used in sentinel wells with vertical profiling conducted during each sample round (one PDB sampler at the midpoint of every five feet of saturated screen).

Dual-membrane PDBs will be used, which are composed of two separate semipermeable membranes to form the sample chamber: an outer membrane with larger openings to allow large or polar molecules into the sampler, and an inner membrane with smaller pores that allow the diffusion of compounds into the sampler but do not allow water molecules to pass through it.

PDB samplers will be placed in the center of the 10 ft. screened interval and will remain in place for a minimum of 2 weeks in accordance with the NJDEP FSPM.

## **2.8 SURFACE WATER SAMPLING**

Surface water samples will be collected prior to sediment sample collection at any given sample location. Surface water samples will be collected outside the main channel and from the air water interface and/or a single mid-depth interval at each sampling location. Prior to sampling, the water column length will be measured with a lead line from the boat deck or with a fathometer mounted on the research vessel’s hull or by hand. Surface water samples will be collected using either a water collection sampler without Teflon® lining or by using a peristaltic pump and dedicated tubing that will be extended to the desired sampling depths (the depth of the channel and conditions of the surface water body at the time of sampling will dictate the sampling method). Anticipated target analytes are included in Table 1. The site-specific QAPP details the specific laboratory method and quality assurance and quality control for sample analysis.

To obtain the representative volume for sampling, the samples will be composited in a mixing container and the appropriate sample bottles will be filled using a peristaltic pump, with the outflow directed into the sample bottle. Laboratory bottles will be pre-cleaned, and any required preservatives will be placed in the specific bottles by the testing laboratory prior to shipping.

Integral's field lead and field personnel in charge of sample handling will use sample matrix tables as a quality control check to ensure that all samples at a given location are collected and that the appropriate sample container is used for each sample.

### 2.8.1 Sample Handling

Gloved hands are required for sample collection and handling, as described above. Field staff will wear appropriate non-contaminating, disposable, powderless nitrile gloves during the entire sampling operation. The field sampling team will change gloves frequently and before each surface water sample is collected.

Gloved hands are required for all operations that involve equipment that comes into contact with the surface water sample, including the following activities:

- Handling the sample bottle
- Handling the discharge end of the water collection bottle or sample tubing.

The surface water samples will be placed in labeled, laboratory-cleaned sample containers. Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Immediately after sample containers are filled, the samples will be stored on wet ice ( $4 \pm 2^\circ\text{C}$ ) or as specified by the method; no chemical (blue) ice packs will be used during the sampling event. The sampling team leader is responsible for maintaining sample integrity throughout the sampling event.

### 2.8.2 Water Quality Measurements

In addition to surface water collection, general water quality parameters (i.e., water temperature, pH, dissolved oxygen, salinity, oxidation-reduction potential, turbidity, and conductivity) will be measured *in situ* at all sampling locations using a multi-probe (e.g., YSI 6).

The multi-probe will be calibrated at the beginning of every day according to manufacturer specifications (see user manual). The name(s) of the person(s) performing the calibration will be recorded in the field logbook that is used during the sampling event. Calibration records must be kept so the results of the water quality monitoring are traceable. Calibration records will be maintained in accordance with Integral's laboratory certification (Lab ID: 04032).

## 2.9 SEDIMENT SAMPLING

Sediment samples will be collected outside the main channel and biased to depositional areas. The following sections describe the sampling equipment, sampling methods, sample handling,



and shipping for sediments. Anticipated target analytes are included in Table 1. The site-specific QAPP details the specific laboratory method and quality assurance and quality control for sample analysis.

### 2.9.1 Surface Sediment Sampling

Surficial sediment samples (0–6 in.; 0–15 cm) will be collected with a stainless-steel van Veen grab sampler, hand auger, dredge (or equivalent type of equipment) depending upon the conditions encountered in the field. Sediment samples will be collected in accordance with standard methods used by USEPA (1997) and in the NJDEP FSPM (2005). Methods for surface sediment sampling are provided in SOPs SD-04 and SD-13 (Attachment 1).

The samples will be analyzed for the target analytes listed in Table B-1. Additional sediment from each location may be archived and stored at the laboratory for possible future analysis, if necessary.

Material collected with the grab sampling device will be evaluated by the Integral field lead for acceptability using the following criteria:

- The sampler is not overfilled.
- Overlying water may be present.
- The overlying water (if present) is not excessively turbid.
- The sediment surface is relatively undisturbed.
- An adequate penetration depth is attained (i.e., to enable sampling of the undisturbed surface sediment).

If a sample fails to meet any of the above criteria, it will be rejected and discarded away from the location. Removal of material from the sample will be documented in the field logbook.

After a sediment sample is judged to be acceptable, any overlying water will be siphoned off and the upper 6 in. (15 cm) of sediment will be collected in accordance with guidelines (USEPA 1997). Decontaminated stainless-steel spoons will be used to collect the sediment from the grab sampler. A stainless-steel ruler will be used to ensure that the sampling criterion for adequate penetration depth has been met and that the correct amount (i.e., 6 in. [15 cm]) of sediment has been removed from the grab sampler.

Surface sediments from the grab samples will be placed into a decontaminated, stainless-steel bowl and homogenized using a stainless-steel spoon or other stainless-steel mixing implement until the sediment attains a visually uniform color and texture. The sediment sample in the bowl will be covered with the lid until a sufficient volume of sediment (approximately 1 L per

sample location) is collected. Sediment subsamples will then be removed for the various kinds of laboratory analyses and for archiving.

The composite surface sediment samples will be placed in labeled, laboratory-cleaned sample containers without Teflon®-lined lids. Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen (i.e., archived samples) will have 0.5–1 in. (1.3–2.6 cm) of headspace above the sediment to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice ( $4 \pm 2^\circ\text{C}$ ) and within method specifications; no chemical (blue) ice packs will be used during the sampling event.

Integral's field lead and field personnel in charge of sample handling will use a sample matrix table as a quality control check to ensure that all samples at a given location are collected and that the appropriate sample container is used for each sample.

## 2.9.2 Subsurface Sediment Sampling

During subsurface sediment sampling, river gauge height and tides will be monitored using the following web sites:

- Real-time information on wind direction, wind speed, and river elevation: U.S. Geological Service National Water Information System (USGS - NWIS)  
<http://waterdata.usgs.gov/nwis/sw>
- Tides: NOAA site at <https://tidesandcurrents.noaa.gov/>.

Subsurface sediment will be collected using a coring device (e.g., Wildco stainless steel hand corer, piston core, or equivalent type of equipment) depending on conditions encountered in the field. Potential subsurface sediment sampling methods that may be used during sample collection are provided in SOPs SD-08, SD-12, and SD-13 (Attachment 1).

The samples will be analyzed for the target analytes listed in Table 1. If there is a sufficient volume of sediment within a core interval, then additional sediment from each interval will be archived for possible future analysis, if necessary.

A minimum diameter of 3 in. (7.6 cm) will be used for all cores. Any separate sediment horizons that are observed in the core will be noted on the field form (Attachment 2). Sediment will be collected from the entire sediment interval and a discrete sample from the composited, homogenized sediment will be collected. Shorter core lengths will be accepted if native materials are encountered, based on visual inspection of the core, or if multiple attempts (i.e., two attempts) at coring a given location do not provide the anticipated core length.

The core's position will be monitored by observing the angle of the winch line while the corer is being lowered in the water column. When the inlet of the corer is approximately 2 m above the sediment, the corer will stop being lowered, the boat location confirmed, and the angle of the hydrowire determined. When the angle of the hydrowire is less than 5 degrees, the corer will be lowered into the sediment at a rate of 30 cm/s or less. If the weather is windy or tidal conditions warrant it, the boat will be anchored before the core is lowered. Cable will be released through the winch until there is slack in the line. If the boat drifts significantly (e.g., because of wind or tidal conditions), slack in the line will be permitted only briefly to prevent pulling the corer out at an angle.

The corer will be retrieved at a controlled rate to minimize agitation of the core. Retrieval will be stopped as soon as the top of the corer reaches the water surface. If a core catcher is not installed at the bottom end of the core, a plug may be inserted in the bottom end of the corer to prevent the core from slipping out when the corer is raised out of the water. The corer will be brought on board the sampling vessel and immediately stabilized to prevent it from tipping or falling. Care will be taken at all times to keep the corer in a vertical position. After the corer is secured onboard the sampling vessel, the Lexan<sup>®</sup> or polyethylene (or similar) liner that contains the sample will be removed from the corer barrel and inspected.

Each core will be evaluated by Integral's field lead for acceptability using the following criteria:

- The sediment surface is relatively undisturbed.
- Any overlying water is not excessively turbid.
- At least 80 percent core recovery relative to penetration is achieved.

If a sediment core fails to meet any of the above criteria, it will be rejected.

If less than 80 percent core recovery versus penetration is achieved, the recovered core will be retained but considered insufficient, and another attempt to recover a sediment core at the same location will be conducted. If the specified penetration depth is not achieved after two attempts, the location may be relocated slightly. If the slight relocation of the location does not improve the penetration depth, the location may be temporarily abandoned and Integral's project manager will be notified.

After the cores have been collected, both ends of the cores designated for chemical analysis will be securely capped; labeled with the location number, core section, and sediment orientation; and fastened in an upright position. The overlying water will be siphoned or drained off.

Processing of the core may occur either on the sampling vessel or at a specified location onshore. The core will be laid out horizontally on a clean work surface and will either be extruded or the core tube will be cut lengthwise and the split core will be opened. All cores will be placed next to a tape measure and a location number and photographed. Each successful

core sampled will be inspected for physical characteristics and each sampled interval described on a core profile form (see Attachment 2).

The sediment from each core section will be homogenized with a decontaminated stainless-steel mixing implement (e.g., spoon) until the sediment attains a visually uniform color and texture. Sediment will be added to a decontaminated stainless-steel bowl until a sufficient volume (approximately 1 L per core interval) is collected. Sediment subsamples will then be removed for the various kinds of laboratory analyses and for archiving.

The subsurface sediment composite samples will be placed in labeled, laboratory-cleaned sample containers with lids that are not lined with Teflon®. Each sample container will be clearly labeled with the task name, sample number, type of analysis to be performed, date and time, and initials of person(s) preparing the sample. Containers that will be frozen (i.e., archived samples) will have 0.5–1 in. (1.3–2.6 cm) of headspace above the sediment to prevent the jars from breaking during storage at the laboratory. Immediately after sample containers are filled, the samples will be stored on ice ( $4 \pm 2^\circ\text{C}$ ); no chemical (blue) ice packs will be used during the sampling event.

Integral's field lead and field personnel in charge of sample handling in the field will use a sample matrix table as a quality control check to ensure that all samples at a given location are collected and that the appropriate sample container is used for each sample.

## 2.10 PORE WATER SAMPLING

At each co-located surface sediment and surface water location, additional sediment may also be collected for pore water analysis. This bulk sediment sample will be sent to SGS North America (SGS) for pore water extraction and analysis following the laboratory's SOP. An aliquot of the pore water volume will be transferred into an appropriate sample container and analyzed for the parameters described in the applicable scope of work. A fraction of the volume may be archived for future analysis. Anticipated target analytes are included in Table 1. The site-specific QAPP details the specific laboratory method and quality assurance and quality control for sample analysis.

*In situ* pore water samples may also be collected using a PushPoint™ or similar sampler (USEPA 2020). These types of samples consist of a stainless steel tube with a screened zone at one end and a sampling port at the other. The pore water is collected by connecting a syringe or peristaltic pump to extract the sample. The syringe and tubing will be polyethylene or other no-PFAS material. The sample will be transferred directly to the sample containers.

## 2.11 SOIL SAMPLING

Soil sampling will be conducted using direct push methods, stainless steel hand augers or scoops depending on the depth of the samples. When generated, soil cores will be laid out horizontally on a clean work surface. All cores will be placed next to a tape measure and a location number and photographed. Each successful core sampled will be inspected for physical characteristics and each sampled interval described on a core profile form (see Attachment 2). Anticipated target analytes are included in Table 1. The site-specific QAPP details the specific laboratory method and quality assurance and quality control for sample analysis.

Six-in. intervals of core will be collected using a stainless-steel spoon or similar sampling equipment and directly transferred into laboratory-provided containers. The interval selected for analysis will be determined based on field observations. Soil samples will be collected in accordance with the NJDEP's *Field Sampling Procedures Manual* (NJDEP 2005). Methods for soil sampling are provided in the SOPs (Attachment 1).

Samples will be sent to SGS for analysis of the analytes listed in Table 1. Container types, method-recommended hold times, and minimum sample sizes are provided in the QAPP.

Integral's field lead and field personnel in charge of sample handling will use a sample matrix table as a quality control check to ensure that all samples at a given location are collected and that the appropriate sample container is used for each sample.

## 2.12 FIELD QUALITY CONTROL

Field quality control samples will be used to assess sample variability and evaluate potential sources of contamination. The types of quality control samples that will be collected for during the proposed sampling events are described in this section. Detailed information on quality assurance and quality control (QA/QC) procedures, limits, and reporting are described in detail in the QAPP. If quality control problems are encountered, they will be brought to the attention of Integral's quality assurance coordinator. Corrective actions, if appropriate, will be implemented to meet the task's data quality indicators.

### 2.12.1 Groundwater

Field quality control samples for the groundwater investigation will include field duplicates, equipment blanks, and field blanks. The procedures and rationale for collecting these samples are as follows:

- **Field duplicate.** Sample will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and

laboratory as well as chemical analysis. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. A blind field duplicate sample will be collected at a rate of 1 duplicate per 20 samples (sample delivery group). Samples will be assigned unique numbers and will not be identified as field duplicates to the laboratory.

- **Equipment blank.** Sample will be collected to help identify possible contamination from the sampling environment or from the decontaminated sampling equipment (e.g., pump). Equipment blanks will be collected for at least 10 percent of the groundwater wells.

The equipment blank will be collected by filling a 1,000-mL decontaminated, graduated cylinder with distilled/deionized water supplied by the laboratory performing the analysis. A decontaminated pump will be placed into the graduated cylinder with sample tubing and plumbing fittings attached. The required field blank samples will then be collected by operating the pump. As water is removed from the cylinder, additional distilled/deionized water will be added as needed until sufficient sample volume has been collected to perform the required analyses. Because of the larger water volumes required, field blank water may not be supplied in the same identical containers as the sample being collected, as provided for by NJDEP guidance for low-flow purging and sampling.

- **Field (or environmental) blank.** Sample will be prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. One field blank will be collected during the sample event (i.e., at the time that groundwater is being collected into the sample bottle) at a frequency of one field/environmental blank per sample event.

The environmental blank will be prepared by opening the laboratory-prepared sample bottle while at a sample collection site during sample collection, filling the sample bottle with distilled/deionized water, and then sealing it. The environmental blank will be assigned a unique sample number, and the labeled bottle will be sent to the laboratory with the field samples.

## 2.12.2 Surface Water or Pore Water

Field quality control samples for the surface water investigation will include the same types of samples as used for groundwater. The procedures and rationale for collecting these samples are as follows:

- **Field duplicate.** Sample will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. A

blind field duplicate sample will be collected at a rate of 1 duplicate per 20 samples (sample delivery group). Samples will be assigned unique numbers and will not be identified as field duplicates to the laboratory.

- **Equipment blank.** Sample will be collected to help identify possible contamination from the sampling environment or from the decontaminated sampling equipment (e.g., pump). Equipment blanks will be collected for at least 10 percent of the surface water sample locations.

The equipment blank will be collected by rinsing the decontaminated sample collection equipment with distilled/deionized water and collecting the rinsate.

- **Field (or environmental) blank.** Sample will be prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. One field blank will be collected during the sample event (i.e., at the time that surface water is being collected into the sample bottle) at a frequency of one field/environmental blank per sample event.

The environmental blank will be prepared by opening the laboratory-prepared sample bottle while at a sample collection site during sample collection, filling the sample bottle with distilled/deionized water, and then sealing it. The environmental blank will be assigned a unique sample number, and the labeled bottle will be sent to the laboratory with the field samples.

### 2.12.3 Sediment

Field quality control samples for sediment samples will include field duplicate samples, equipment blanks, travel blanks, and field blanks. In addition to the blanks prepared in the field, distilled/deionized water blanks and bottle blanks will be analyzed by the laboratory to assess potential contamination from the laboratory-supplied water and sampling containers. The procedures and rationale for collecting these samples are as follows:

- **Field duplicate.** Samples will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Blind duplicates (field split samples) will be collected at a minimum frequency of 1 duplicate per 20 samples (sample delivery group). Samples will be assigned unique numbers and will not be identified as field splits to the laboratory. Field duplicate samples will be collected from both surface and subsurface sediment samples for chemical analysis. A minimum of one field split sample will be collected for each kind of sample collected.
- **Equipment blank.** Samples will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., stainless-steel

grab sampler, coring device, spoons, bowls, and pots). Equipment blanks will be generated at approximately 10 percent of the sediment sampling locations at a minimum. Field equipment blanks will be collected from equipment used to collect both surface and subsurface sediment samples for chemical analysis. All equipment blank samples will be clearly noted in the field log (e.g., sample identifier, equipment type, date and time of collection, analysis, and filter lot number).

The equipment blank will be collected by rinsing the decontaminated sample collection equipment (grab sampler or corer and sample compositing equipment [stainless-steel bowl or pot, and spoon]) with distilled/deionized water and collecting the rinsate.

#### 2.12.4 Soil

Field quality control samples for soil will include field duplicate samples, equipment blanks, travel blanks, and field blanks. In addition to the blanks prepared in the field, distilled/deionized water blanks and bottle blanks will be analyzed by the laboratory to assess potential contamination from the laboratory-supplied water and sampling containers. The procedures and rationale for collecting these samples are as follows:

- **Field duplicate.** Samples will be used to assess the variability in concentrations of co-located samples due to the combined effects of sample processing in the field and laboratory as well as chemical analysis. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Blind duplicates (field split samples) will be collected at a frequency of 1 duplicate per 20 samples (sampler delivery group) of the low-resolution sampling. Samples will be assigned unique numbers and will not be identified as field splits to the laboratory. A minimum of one field split sample will be collected for each kind of sample collected.
- **Equipment blank.** Samples will be collected to help identify possible contamination from the sampling environment or from the sampling equipment (e.g., stainless-steel grab sampler, coring device, spoons, bowls, and pots). Equipment blanks will be generated at approximately 10 percent of the sediment sampling locations (a minimum of four total). Field equipment blanks will be collected from equipment used to collect both surface and subsurface sediment samples for chemical analysis. All equipment blank samples will be clearly noted in the field log (e.g., sample identifier, equipment type, date and time of collection, analysis, and filter lot number).

The equipment blank will be collected by rinsing the decontaminated sample collection equipment with distilled/deionized water and collecting the rinsate.



## 2.13 SAMPLE PACKAGING AND TRANSPORT

As mentioned above, sample coolers and packing materials will be supplied by the analytical laboratories. Individual sample jars will be labeled and placed into plastic bags and sealed. Samples will then be packed in a cooler lined with a large plastic bag. Glass jars will be packed to prevent breakage and separated in the cooler by bubble wrap or other shock-absorbent material. Ice in sealed plastic bags will then be placed in the cooler to maintain a temperature of approximately 4°C ( $\pm 2^\circ\text{C}$ ) or in accordance with the specific method requirements. When the cooler is full, the COC form will be placed into a zip-locked bag and taped to the inside lid of the cooler for shipment. A temperature blank will be added to each cooler. Each cooler will be sealed with two COC seals, one each on the front and side of the cooler.

The shipping containers will be clearly labeled (i.e., name of task, time and date container was sealed, person sealing the cooler, and company name and address) for positive identification. These packaging and shipping procedures are in accordance with U.S. Department of Transportation regulations (49 CFR 173.6 and 49 CFR 173.24). Coolers containing samples for chemical analyses will be transported to the laboratory by courier or overnight shipping service.

After the samples have been received by the laboratory, they will be stored under refrigeration ( $4 \pm 2^\circ\text{C}$ ) or in accordance with the specific method requirements. Archive sediment samples collected from each composite sample for possible future analysis will be stored frozen at  $-20^\circ\text{C}$ .

## 2.14 STUDY DERIVED WASTES

Any excess phosphate-free, detergent-bearing liquid wastes from decontamination will be deposited in the vicinity of the collection area. Purge water from the groundwater sample collection process will be containerized in drums and temporarily stored at a secure location in the plant for treatment and disposal via the groundwater treatment system. Any surface or subsurface sediment present at the end of the sampling event or other non-liquid investigation derived waste will be managed by the Site. This study-derived waste will be containerized (e.g., 55-gal drums) and disposed of in accordance with Federal and State requirements using a NJDEP-licensed waste disposal company.

### 3 FIELD DOCUMENTATION

The integrity of each sample from the time of collection to the point of data reporting must be maintained. Proper record-keeping and COC procedures will allow samples to be traced from collection to final disposition. Representative photographs will be taken of each area where samples are collected. A photograph will be taken of each surface sediment sample and each subsurface sediment interval collected for testing. Site photographs from various angles and close-up views of the overall conditions may also be collected.

#### 3.1 FIELD LOGBOOK

All field activities and observations will be noted in a logbook or on location specific field sampling sheets or logs.

The field logbook will be a bound document and may contain individual field and sample log forms (depending on the sampling activity). Information will include personnel, date, time, well or location number, sampler, types of samples collected, and general observations. Any changes that occur during sampling (e.g., personnel, responsibilities, or deviations from the FSP) and the reasons for these changes will be documented. The logbook will identify onsite visitors (if any) and the number of photographs taken at each location. Integral's field lead is responsible for ensuring that the respective field logbook for each kind of medium sampled and all field data forms are correct. Requirements for logbook entries will include the following:

- Field forms will be scanned to pdf each day of sample collection.
- Logbooks will be bound, with consecutively numbered pages.
- Removal of any pages, even if illegible, will be prohibited.
- Entries will be made legibly with black (or dark) waterproof ink.
- Unbiased, accurate language will be used.
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be recorded, as well as the time of the observation itself).
- Each consecutive day's first entry will be made on a new, blank page.
- The date and time, based on a 24-hour clock (e.g., 0900 for 9:00 a.m. and 2100 for 9:00 p.m.), will appear on each page.

In addition to the preceding requirements, the person recording the information must sign and date each page of the field logbook during each sampling day. If more than one individual

makes entries in the logbook on any given day, then each recorder must initial and date their respective entries.

Logbook corrections will be made by drawing a single line through the original entry, allowing the original entry to be read. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.

The type of information that may be included in the field logbook and/or field data forms includes the following:

### **All Types of Sampling**

- Task name and task number
- Start date and end date
- Weather conditions
- Name of person making entries and other field staff
- Onsite visitors, if any
- Observations made during sample collection, including any complications, and other details associated with the sampling effort.

Specific information on each type of sampling activity that will be recorded in the field logbook is provided below.

### **Groundwater Sampling**

- Description of all sampling/water quality monitoring equipment used including make, model, and material types for equipment contacting groundwater (e.g., tubing, bladder, pump body, seals, and cord)
- Description of passive sampler types used and any associated reusable and/or disposable materials
- Water conditions including approximate air temperature
- For each well sampled the following will be noted:
  - Well number, sample identifier, and sample number
  - Date and time of sample collection
  - Presence and thickness of any nonaqueous-phase liquid encountered
  - Any observations or problems encountered.

### **Surface Water and Sediment Sampling**

- Sampling vessel, if any
- Location number, sample identifier, and sample number for each sample to be submitted for laboratory analysis
- Date and collection time of each sample
- The specific date and time with corresponding location number associated with the coordinates derived from DGPS
- The sample number, date and time of collection, equipment type, and the lot number for the box of filter papers used for field quality control samples
- Sample description (source and appearance, such as color, oil sheens, odor, and other debris, and sediment type, presence of anthropogenic material and presence and type of biological structures)
- Sediment penetration depth (nearest 0.5 cm) based on sediment depth at the center of the excavation
- Any visible debris near any of the sampling locations
- Any surface vegetation or debris that is removed from the sampling location prior to sampling
- Surface water runoff or seeps that are located near any of the sampling locations.

In addition, a sampling location map will be updated during each sampling activity and will be maintained throughout each sampling event. All logbooks must be completed at the time that any observations are made. Copies of all logbooks and forms will be retained by the technical team.

### **3.2 CHAIN-OF-CUSTODY PROCEDURES**

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals (see SOP AP-03, Attachment 1). A COC record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the COC will be included in laboratory and QA/QC reports. Attachment 2 contains an example of the COC form that will be used during the proposed field events.

At a minimum, the form will include the following information:

- Site name or contract number
- Field lead's name and team members responsible for collection of the listed samples
- Collection date and time for each sample

- Sample type (i.e., sample for immediate analysis or archive)
- Requested analyses
- Sample preservation information (if any)
- Name of the carrier relinquishing the samples to the transporter, noting date and time of transfer and the designated sample custodian at the receiving facility.

Integral's field lead (or delegate) will be the designated field sample custodian for each sampling event and will be responsible for all sample tracking and COC procedures for the samples collected in the field. The field sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The field sample custodian will complete COC forms prior to removing samples from the field. Upon transferring samples to the laboratory sample custodian (if a local laboratory is selected) or shipping courier (as appropriate), the field sample custodian will sign, date, and note the time of transfer on the COC form. The original COC form will be transported with the samples to the laboratories. All samples will be shipped to the testing laboratories in coolers sealed with custody seals.

Each laboratory will designate a sample custodian who will be responsible for receiving samples and documenting their progress through the laboratory analytical process. The sample custodian for each laboratory will establish the integrity of the custody seals upon sample arrival at the laboratory. The laboratory sample custodian will also ensure that the COC and sample tracking forms are properly completed, signed, and initialed upon receipt of the samples.

When the laboratory receives the samples, the laboratory sample custodian will conduct an inventory by comparing sample labels to those on the COC document. The custodian will enter the sample number into a laboratory tracking system by task code and sample designation. The custodian will assign a unique laboratory number to each sample and will be responsible for distributing the samples to the appropriate analyst or for storing samples at the correct temperature in an appropriate secure area.

### **3.3 SAMPLE LOCATION NUMBERING**

All wells will be assigned a unique identification code based the location of the monitoring well. Groundwater sample locations will be labeled using the previously assigned well designations. Surface water and sediment location numbers will begin with "SESW" to indicate a collocated surface water and sediment location. Soil sample location numbers will begin with SBXX, where XX indicates the year of soil boring collection (e.g., SB15 is a soil boring collected in 2015).

### 3.4 SAMPLE IDENTIFIERS

Each sample from a given well or location will also have three unique label identifiers. Sample identifiers will be established before field sampling begins and assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., field split samples) to ensure proper data analysis and interpretation (the Sample ID); 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples (the Sample number); and 3) to track individual sample containers to ensure that the laboratory receives all of the material associated with a single sample (the Tag number). To accomplish these purposes, each container is assigned a sample number and a tag number. These codes and their uses are described below:

- A sample ID for each sample will be created as follows: the well or location number (e.g., MW-4X), which is followed by the month, day, and year (MMDDYY) that the sample was collected (e.g., 030115). If possible, a two-letter code for the kind of sample collected at a given location (GW = groundwater, SW= surface water, SL = Soil, SD = sediment) may be added. This is dependent on the character limitations of the laboratory log in process. In addition, subsurface core samples will also have a final series of numbers attached to the sample identifier that will distinguish between the different sample intervals of the core (e.g., 0–6 in. [0–15 cm], 6–12 in. [15–30 cm], 12–24 in. [30–61 cm], and 24–36 in. [61–91 cm]). An example sample identifier for a groundwater sample collected on March 1, 2021 at Well M/H-1D would be M/H-1D-030121-GW. Example sample identifiers for a co-located surface water and surface sediment sample would be SESW001-030115-SW, and SESW001-030115-SD.
- The sample number is an arbitrary number assigned sequentially to each sample collected (e.g., GW0001, GW0002, SW0001, SW0002, SD0001, SD0002). All subsamples of a composited field sample will have the same sample number. Each field split sample will have a different sample number, and the sample numbers of related field quality control samples may not share any content. The sample number will appear on the sample containers, COC forms, and laboratory reports.
- For equipment blanks or field blanks, the samples will be numbered sequentially by date. Equipment blanks will use “EB”, Field blanks “FB” and the sample identification will include the sample date in a MMDDYY format. For example, the first equipment blank of a groundwater sampling event will be labeled EB-1/MMDDYY.

## **4 FIELD DATA MANAGEMENT AND REPORTING PROCEDURES**

During field operations, effective data management is critical to providing consistent, accurate, and defensible data and data products. Daily field records (a combination of field logbooks, field forms, if any, and COC forms) will make up the main documentation for field activities. Upon completion of sampling, field notes, data sheets (if any), and COC forms will be scanned to create an electronic record. Field data will be manually entered into the project database. One hundred percent of the transferred data will be verified based on hard copy records. Electronic quality assurance checks to identify anomalous values will also be conducted following entry.

## 5 REFERENCES

ERM. 2013. Free/residual product and ground water interim remedial investigation submittal. Solvay Specialty Polymers USA, LLC West Deptford, NJ Facility ISRA Case Nos. E89231, E90205, Program Interest No. 015010. Environmental Resources Management, Trenton, NJ.

NJDEP. 2005. Field sampling procedures manual. New Jersey Department of Environmental Protection, Trenton, NJ.

USEPA. 1997. Recommended quality assurance and quality control guidelines for the collection of environmental data in Puget Sound. In: Recommended protocols for measuring selected environmental variables in Puget Sound. U.S. Environmental Protection Agency, Puget Sound Estuary Program, Seattle, WA.

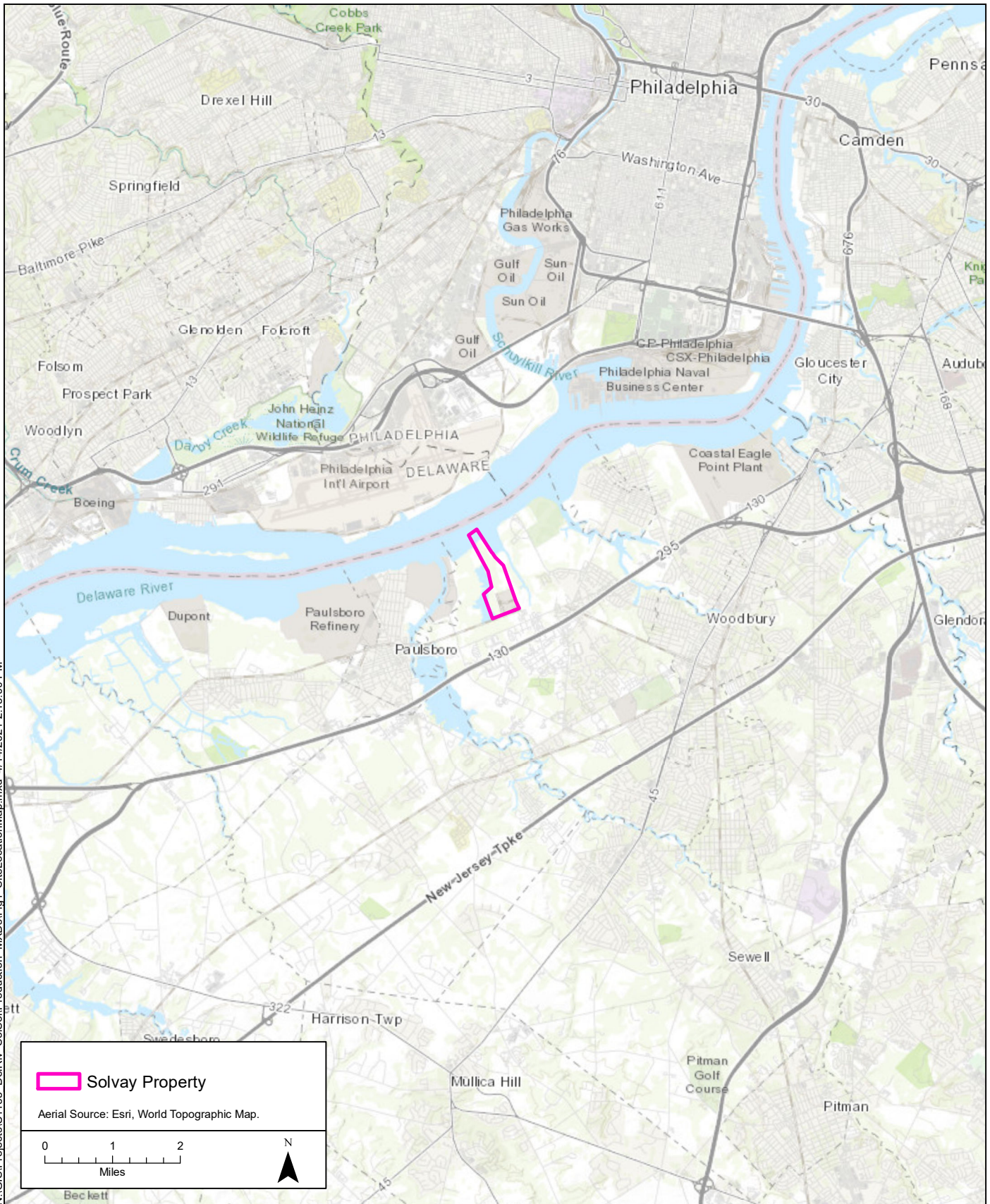
USEPA. 2010. Low stress (low flow) purging and sampling procedures for the collection of groundwater samples from monitoring wells. U. S. Environmental Protection Agency, Washington, DC. Revised January 19, 2010.

USEPA. 2020. Pore Water Sampling Operating Procedure. U.S. Environmental Protection Agency, Athens, GA.



**FIGURE**

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**Figure 1.**  
Location Map

## TABLE

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Table 1. Chemicals of Interest

Analyte	CAS Number	Matrix				
		Groundwater	Pore Water	Surface Water	Sediment	Soil
<b>PFAS</b>						
Perfluorohexanoic Acid (PFHxA, C6)	307-24-4	X	X	X	X	X
Perfluoroheptanoic Acid (PFHpA, C7)	375-85-9	X	X	X	X	X
Perfluorooctanoic acid (PFOA;C8)	335-67-1	X	X	X	X	X
Perfluorononanoic acid (PFNA;C9)	375-95-1	X	X	X	X	X
Perfluorodecanoic acid (PFDA; C10)	335-76-2	X	X	X	X	X
Perfluoroundecanoic acid (PFUnDA; C11)	2058-94-8	X	X	X	X	X
Perfluorododecanoic acid (PFDoDA; C12)	307-55-1	X	X	X	X	X
Perfluorotridecanoic acid (PFTrDA; C13)	72629-94-8	X	X	X	X	X
Perfluorotetradecanoic acid (PFTeDA; C14)	376-06-7	X	X	X	X	X
Perfluorobutanesulfonic Acid (PFBS, C4)	375-73-5	X	X	X	X	X
Perfluorohexanesulfonic acid (PFHxS; C6)	355-46-4	X	X	X	X	X
Perfluorooctanesulfonic acid (PFOS; C8)	1763-23-1	X	X	X	X	X
PFOA (Branched Isomers)	--	X	X	X	X	X
PFOA (Linear Isomer)	--	X	X	X	X	X
PFNA (Branched Isomers)	--	X	X	X	X	X
PFNA (Linear Isomer)	--	X	X	X	X	X
PFHxS (Branched Isomers)	--	X	X	X	X	X
PFHxS (Linear Isomer)	--	X	X	X	X	X
PFOS (Branched Isomers)	--	X	X	X	X	X
PFOS (Linear Isomer)	--	X	X	X	X	X
Monofunctional Surfactants (MFS)	--	X	X	X	X	X
Bifunctional Surfactants (BFS)	--	X	X	X	X	X
<b>Conventional Parameters</b>						
Anions						
Bromide	24959-67-9	X	X	X	--	--
Chloride	16887-00-6	X	X	X	--	--
Fluoride	16984-48-8	X	X	X	--	--
Sulfate	14808-79-8	X	X	X	--	--
Cations						
Calcium	7440-70-2	X	X	X	--	--
Iron	7439-89-6	X	X	X	--	--
Magnesium	7439-95-4	X	X	X	--	--
Manganese	7439-96-5	X	X	X	--	--
Sodium	7440-23-5	X	X	X	--	--
Potassium	7440-09-7	X	X	X	--	--
Total organic carbon	7440-44-0	--	--	X	X	X
Total suspended solids	--	X	--	X	--	--
Total dissolved solids	--	--	--	X	--	--
pH	12408-02-5	X	X	X	--	--
Alkalinity	--	X	--	--	--	--
Grainsize	--	--	--	--	X	X

## Notes:

CAS = Chemical Abstracts Service registry number

PFAS = per- and polyfluoroalkyl substances

X = samples may be collected and analyzed for these parameters

-- = not available

# **ATTACHMENT 1**

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## **INTEGRAL CONSULTING SOPs**

## **STANDARD OPERATING PROCEDURE (SOP) AP-01**

### **SAMPLE PACKAGING AND SHIPPING**

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#### **SCOPE AND APPLICATION**

This SOP describes specific requirements for sample packaging and shipping to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein. This SOP also presents the method to be used when packing samples that will either be hand delivered or shipped by commercial carrier to the laboratory.

#### **EQUIPMENT AND SUPPLIES REQUIRED**

Make sure that you have the equipment and supplies necessary to properly pack and ship environmental samples, including the following:

- Project-specific sampling and analysis plan (SAP)
- Project-specific field logbook
- Sealable airtight bags in assorted sizes (e.g., Ziploc<sup>®</sup>)
- Wet ice in doubled, sealed bags; frozen Blue Ice<sup>®</sup>; or dry ice
- Cooler(s)
- Bubble wrap
- Fiber-reinforced packing tape, clear plastic packing tape, and duct tape
- Scissors or knife
- Chain-of-custody (COC) forms
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick)
- Paper towels
- “Fragile,” “This End Up,” or “Handle With Care” labels
- Mailing labels
- Air bills for overnight shipment

## PROCEDURE

Customize the logistics for sample packaging and shipping to each study. If necessary, transfer samples from the field to a local storage facility where they can be frozen or refrigerated. Depending on the logistics of the operation, field personnel may transport samples to the laboratory or use a commercial courier or shipping service. In the latter case, Integral field personnel must be aware of any potentially limiting factors to timely shipping, such as availability of overnight service and weekend deliveries to specific areas, and shipping regulations regarding “restricted articles” (e.g., dry ice, formalin) prior to shipping the samples.

## SAMPLE PREPARATION

Take the following steps to ensure the proper transfer of samples from the field to the laboratories:

At the sample collection site:

1. Document all samples using the proper logbooks or field forms (see SOP AP-02), required sample container identification (i.e., sample labels with tag numbers), and COC form (example provided in SOP AP-03). Fill out the COC form as described in SOP AP-03, and use the sample labeling techniques provided in SOP AP-04.
2. Make all applicable laboratory quality control sample designations on the COC forms. Clearly identify samples that will be archived for future possible analysis. Label these samples as follows: “Do Not Analyze: Hold and archive for possible future analysis.” Some laboratories interpret “archive” to mean that they should continue holding the residual sample after analysis.
3. Notify the laboratory contact and the Integral project quality assurance/quality control (QA/QC) coordinator that samples will be shipped and the estimated arrival time. Send copies of all COC forms to Integral’s project QA/QC coordinator or project manager, as appropriate.
4. Keep the samples in the possession of the sampling personnel at all times. Lock and secure any temporary onsite sample storage areas to maintain sample integrity and COC requirements.
5. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
6. Complete the COC form as described in SOP AP-03, and retain the back (pink) copy for project records prior to sealing the cooler. Check sample containers against the COC form to ensure all the samples that were collected are in the cooler.

7. Store each sample container in a sealed plastic bag that allows the sample label (example provided in SOP AP-03) to be read. Before sealing the bags, ensure that volatile organic analyte (VOA) vials are encased in a foam sleeve or in bubble wrap.
8. If the samples require storage at a specific temperature, place enough ice in the sample cooler to maintain the temperature (e.g., 4°C) throughout the sampling day.

At the sample processing area (immediately after sample collection) take the following steps:

1. If the samples require a specific storage temperature, then cool the samples and maintain the temperature prior to shipping. For example, place enough ice in each sample cooler to maintain the temperature at 4°C until processing begins at the testing laboratory.
2. Be aware of holding time requirements for project-specific analytes and arrange the sample shipping schedule accordingly.
3. Place samples in secure storage (i.e., locked room or vehicle) or keep them in the possession of Integral sampling personnel before shipment. Lock and secure any sample storage areas to maintain sample integrity and COC requirements.
4. Store samples in the dark (e.g., keep coolers shut).

At the sample processing area (just prior to shipping), do the following:

1. Check sample containers against the COC form to account for all samples intended for shipment.
2. Choose cooler(s) of appropriate size and make sure they are clean of gross contamination inside and out. If the cooler has a drain, close the drain and secure it with duct tape.
3. Line the cooler with bubble wrap and place a large plastic bag (preferably with a thickness of 3 mil), open, inside the cooler.
4. Individually wrap each glass container (which was sealed in a plastic bag at the collection site) in bubble wrap and secure with tape or a rubber band. Place the wrapped samples in the large plastic bag in the cooler, leaving room for ice to keep the samples cold (i.e., 4°C).
5. If temperature blanks have been provided by the testing laboratory, place one temperature blank in each sample cooler.
6. If the samples require a specific storage temperature, add enough wet ice or Blue Ice<sup>®</sup> to maintain that temperature during overnight shipping (i.e., 4°C). Always overestimate the amount of ice that will be required. Keep ice in a sealed plastic bag, which is placed in a second sealed plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it may insulate the samples from the ice. After adding all samples and ice to the cooler, use bubble wrap (or other



available clean packing material) to fill any empty space and prevent the samples from shifting during transport.

7. If possible, consolidate all VOA samples in a single cooler and ship them with (a) trip blank(s) if the project-specific QA project plan calls for them.
8. Sign, date, and include any tracking numbers provided by the shipper on the COC form. Remove the back (pink) copy of the original COC form and retain this copy for the project records.
9. Seal the rest of the signed COC form in a bag and tape the bag to the inside of the cooler lid. Each cooler should contain an individual COC form for the samples contained inside it. If time is short and it becomes necessary to combine all the samples onto a single set of COC forms and ship multiple coolers together, then indicate on the outside of the appropriate cooler, "Chain-of-Custody Inside."
10. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it with fiber-reinforced packing tape. Tape the cooler around the opening, joining the lid to the bottom, and around the circumference of the cooler at both hinges.
11. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid (provided with example field forms). Place one seal on the front right portion of the cooler and one on the back left. Be sure the seals are properly affixed to the cooler to prevent removal during shipment. Additional tape across the seal may be necessary if the outside of the cooler is wet.

## **SAMPLE SHIPPING**

### **Hand Delivery to the Testing Laboratory**

1. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. When hand-delivering environmental samples, make sure the testing laboratory receives them on the same day that they were packed in the coolers.
3. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after it has been faxed. Never leave the original COC form in the custody of non-Integral staff.

## Shipped by Commercial Carrier to the Laboratory

1. Apply a mailing label to the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and "Handle With Care." If the shipment contains multiple coolers, indicate on the mailing label the number of coolers that the testing laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the cooler and to protect it from the weather. This is a secondary label in case the air bill is lost during shipment.
2. Fill out the air bill and fasten it to the handle tags provided by the shipper (or the top of the cooler if handle tags are not available).
3. If samples must be frozen ( $-20^{\circ}\text{C}$ ) during shipping, make sure that dry ice has been placed in the sample cooler. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require.
4. Make sure that benthic infauna samples have been preserved with formalin in the field prior to shipping. Be aware of any additional shipping, handling, and special labeling requirements that the shipper may require for these samples.
5. Notify the laboratory contact and the Integral project QA/QC coordinator that samples will be shipped and the estimated arrival date and time. If environmental samples must be shipped at  $4^{\circ}\text{C}$  or  $-20^{\circ}\text{C}$ , choose overnight shipping for delivery next morning. Fax or scan and e-mail copies of all COC forms to the Integral project QA/QC coordinator. Note: It may be necessary to photocopy the COC form on a slightly darker setting so the form is readable after faxing. Never leave the original COC form in the custody of non-Integral staff.

## **STANDARD OPERATING PROCEDURE (SOP) AP-02**

### **FIELD DOCUMENTATION**

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#### **SCOPE AND APPLICATION**

This SOP describes the Integral procedure for accurate record-keeping in the field for the purposes of ensuring that samples can be traced from collection to final disposition.

Document all information relevant to field operations properly to ensure that activities are accounted for in written records to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. Several types of field documents are used for this purpose and should be consistently used by field personnel. Field documentation should include only a factual description of site-related activities and observations. Field personnel should not include superfluous comments or speculation regarding the field activities or observations.

#### **FIELD LOGBOOKS**

During field sampling events, field logbooks must be used to record all daily activities. The purpose of the field logbook is to document events and record data measured in the field to the extent that someone not present at the site could reconstruct the activity without relying on the memory of the field crew. The project manager (or designee) should issue a field logbook to the appropriate site personnel for the direction of onsite activities (e.g., reconnaissance survey team leader, sampling team leader). It is this designee's responsibility to maintain the site logbook while it is in his or her possession and return it to the project manager or turn it over to another field team.

Make entries in the field logbook as follows:

1. Document all daily field activities in indelible ink in the logbook and make no erasures. Make corrections with a single line-out deletion, followed by the author's initials and the date. The author must initial and date each page of the field logbook. The author must sign and date the last page at the end of each day, and draw a line through any blank space remaining on the page below the last entry.

2. Write the project name, dates of the field work, site name and location (city and state), and Integral job number on the cover of the field logbook. If more than one logbook is used during a single sampling event, then annotate the upper right-hand corner of the logbook (e.g., Volume 1 of 2, 2 of 2) to indicate the number of logbooks used during the field event. Secure all field logbooks when not in use in the field. The following is a list of the types of information that is appropriate for entry in the field notebook:
  - Project start date and end date
  - Date and time of entry (24-hour clock)
  - Time and duration of daily sampling activities
  - Weather conditions at the beginning of the field work and any changes that occur throughout the day, including the approximate time of the change (e.g., wind speed and direction, rain, thunder, wave action, current, tide, vessel traffic, air and water temperature, thickness of ice if present)
  - Name and affiliation of person making entries and other field personnel and their duties, including what times they are present
  - The location and description of the work area, including sketches, map references, and photograph log, if appropriate
  - Level of personal protection being used
  - Onsite visitors (names and affiliations), if any, including what times they are present
  - The name, agency, and telephone number of any field contacts
  - Notation of the coordinate system used to determine the station location
  - The sample identifier and analysis code for each sample to be submitted for laboratory analysis, if not included on separate field data sheets
  - All field measurements made (or reference to specific field data sheets used for this purpose), including the time of collection and the date of calibration, if appropriate
  - The sampling location name, date, gear, water depth (if applicable), and sampling location coordinates, if not included on separate field data sheets
  - For aquatic sampling, the type of vessel used (e.g., size, power, type of engine)
  - Specific information on each type of sampling activity
  - The sample type (e.g., groundwater, soil, surface sediment), sample number, sample tag number, and any preservatives used, if not included on separate field data sheets
  - Sample storage methods

- Cross-references of numbers for duplicate samples
  - A description of the sample (source and appearance, such as soil or sediment type, color, texture, consistency, presence of biota or debris, presence of oily sheen, changes in sample characteristics with depth, presence/location/thickness of the redox potential discontinuity [RPD] layer, and odor) and penetration depth, if not included on separate field data sheets
  - Estimate of length and appearance of recovered cores, if not included on separate field data sheets
  - Photographs (uniquely identified) taken at the sampling location, if any
  - Details of the work performed
  - Variations, if any, from the project-specific sampling and analysis plan (SAP) or standard operating protocols and reasons for deviation
  - Details pertaining to unusual events that might have occurred during sample collection (e.g., possible sources of sample contamination, equipment failure, unusual appearance of sample integrity, control of vertical descent of the sampling equipment)
  - References to other logbooks or field forms used to record information (e.g., field data sheets, health and safety log)
  - Any field results not appearing on the field data sheets (if used), including station identification and location, date, and time of measurement
  - Sample shipment information (e.g., shipping manifests, chain-of-custody (COC) form numbers, carrier, air bill numbers, time addresses)
  - A record of quantity of investigation-derived wastes (if any) and storage and handling procedures.
3. During the field day, as listed above, record in the logbook a summary of all site activities. Provide a date and time for each entry. The information need not duplicate anything recorded in other field logbooks or field forms (e.g., site health and safety officer's logbook, calibration logbook, field data sheets), but should summarize the contents of the other logbooks and refer to the pages in these logbooks for detailed information.
4. If measurements are made at any location, record the measurements and equipment used, or refer to the logbook and page number(s) or field forms on which they are recorded. All maintenance and calibration records for equipment should be traceable through field records to the person using the instrument and to the specific piece of instrumentation itself.

5. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided below.

## **FIELD DATA FORMS**

Occasionally, additional field data forms are generated during a field sampling event (e.g., groundwater monitoring form, sediment core profile form, water quality measurement form) to record the relevant sample information collected. For instructions regarding the proper identification of field data forms, sampling personnel should consult the project-specific SAP.

Upon completion of the field sampling event, the sampling team leader will be responsible for submitting all field data forms to be copied. A discussion of copy distribution is provided below.

## **PHOTOGRAPHS**

In certain cases, photographs (print or digital) of sampling stations may be taken using a camera-lens system with a perspective similar to the naked eye. Ensure that photographs include a measured scale in the image, when practical. If you take photographs of sample characteristics and routine sampling activities, avoid using telephoto or wide-angle shots, because they cannot be used in enforcement proceedings. Record the following items in the field logbook for each photograph taken:

1. The photographer's name or initials, the date, the time of the photograph, and the general direction faced (orientation)
2. A brief description of the subject and the field work shown in the picture
3. For print photographs, the sequential number of the photograph and the roll number on which it is contained
4. For digital photographs, the sequential number of the photograph, the file name, the file location, and back-up disk number (if applicable).

Upon completion of the field sampling event, the sampling team leader is responsible for submitting all photographic materials to be developed (prints) or copied (disks). Place the prints or disks and associated negatives in the project files (at the Integral project manager's location). Make photocopies of photo logs and any supporting documentation from the field logbooks, and place them in the project files with the prints or disks.

## **EQUIPMENT CALIBRATION RECORDS**

Record in the field logbook all equipment calibration records, including instrument type and serial number, calibration supplies used, calibration methods and calibration results, date, time, and personnel performing the calibration. Calibrate all equipment used during the investigation daily, at a minimum, in accordance with the manufacturers' recommendations.

## **DISTRIBUTION OF COPIES**

When the field team has returned from the sampling event, the field team leader is responsible for making sure that the field documentation is 1) scanned and placed into the project file on the portal (in a subfolder named Field under Working\_Files), and 2) a copy of all field logbooks and additional field data forms is made and placed into the project file. Both the scanned copy and the hard copy will be available for general staff use.

The original field logbooks and forms will be placed in a locked file cabinet for safekeeping. One file cabinet at each Integral office will contain the original field documentation for multiple projects. The original field documentation will be filed at the Integral office where the project manager is located.

## **SET-UP OF LOCKING FILE CABINET**

Place each project in its own file folder in a locking file cabinet. On the folder label, include the project name and contract number. Each project folder will include up to six kinds of files:

- Field logbook(s)
- Additional field data forms
- Photographs
- COC forms
- Acknowledgment of Sample Receipt forms
- Archive Record form (to be completed only if samples are archived at an Integral field storage facility or Integral laboratory).

## **STANDARD OPERATING PROCEDURE (SOP) AP-03**

### **SAMPLE CUSTODY**

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#### **SCOPE AND APPLICATION**

This SOP describes Integral procedures for custody management of environmental samples.

A stringent, established program of sample chain of custody will be followed during sample storage and shipping activities to account for each sample. The procedure outlined herein will be used with SOP AP-01, which covers sample packaging and shipping; SOP AP-02, which covers the use of field logbooks and other types of field documentation; and SOP AP-04, which covers sample labeling.

#### **SAMPLE CUSTODY**

A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession
2. The sample is in the person's view after being in his or her possession
3. The sample has been transferred to a designated secure area to prevent tampering after it was in the person's possession.

At no time is it acceptable for samples to be outside of Integral personnel's custody unless the samples have been transferred to a secure area (i.e., locked up). If the samples cannot be placed in a secure area, then an Integral field team member must physically remain with the samples (e.g., at lunch time one team member must remain with the samples).

#### **CHAIN-OF-CUSTODY FORMS**

Chain-of-custody (COC) forms ensure that samples are traceable from the time of collection through processing and analysis until final disposition. The COC form is critical because it documents sample possession from the time of collection through final disposition. The form also provides information to the laboratory regarding what analyses are to be performed on the samples that are shipped.

Complete the COC form after each field collection activity and before shipping the samples to the laboratory. Sampling personnel are responsible for the care and custody of the samples



until they are shipped. The individuals relinquishing and receiving the samples must sign the COC form(s), indicating the time and date of the transfer, when transferring possession of the samples.

Record on the COC form the project-assigned sample number and the unique tag number at the bottom of each sample label. The COC form also identifies the sample collection date and time, type of sample, project name, and sampling personnel. In addition, the COC form provides information on the preservative or other sample pretreatment applied in the field and the analyses to be conducted by referencing a list of specific analyses or the statement of work for the laboratory. The COC form is sent to the laboratory along with the sample(s).

## PROCEDURES

Use the following guidelines to ensure the integrity of the samples:

1. At the end of each sampling day and prior to shipping or storage, enter information for all samples on a COC form. Check the information against the sample container labels and tags and field logbook entries.
2. Do not sign the COC form until the team leader has checked the information for inaccuracies. Make corrections by drawing a single line through any incorrect entry, and then initial and date it.
3. Mark out any blank lines remaining on the COC form, using single lines that are initialed and dated. This procedure will prevent any unauthorized additions.
4. Sign and date each COC form. At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples and the time and date of the transfer. The time the samples were relinquished should match exactly the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.
5. If samples are being sent by a commercial carrier not affiliated with the laboratory, such as FedEx or United Parcel Service (UPS), record the name of the carrier on the COC form. Also enter on the COC form any tracking numbers supplied by the carrier. The time of transfer should be as close to the actual drop-off time as possible. After signing the COC forms and retaining a copy (e.g., the pink copy if the COC form is in triplicate, or an electronic or photocopy if not), seal them inside the transfer container.
6. If errors are found after the shipment has left the custody of sampling personnel, make a corrected version of the forms and send it to all relevant parties. Fix minor errors by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.

Upon completion of the field sampling event, the sampling team leader is responsible for providing copies of all COC forms to the project chemist or laboratory coordinator. A discussion of copy distribution is provided in SOP AP-02.

## **CUSTODY SEAL**

As security against unauthorized handling of the samples during shipping, affix two signed and dated custody seals to each sample cooler. Place the custody seals across the opening of the cooler prior to shipping. Be sure the seals are properly affixed to the cooler so they cannot be removed during shipping. Additional tape across the seal may be prudent.

## **SHIPPING AIR BILLS**

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), the shipper provides an air bill or receipt. Upon completion of the field sampling event, the sampling team leader will be responsible for submitting the sender's copy of all shipping air bills to be copied at an Integral office. A discussion of copy distribution is provided in SOP AP-02. Note the air bill number (or tracking number) on the applicable COC forms or, alternatively, note the applicable COC form number on the air bill to enable the tracking of samples if a cooler becomes lost.

## **ACKNOWLEDGMENT OF SAMPLE RECEIPT FORMS**

In most cases, when samples are sent to a testing laboratory, an Acknowledgment of Sample Receipt form is faxed to the project QA/QC coordinator the day the samples are received by the laboratory. The person receiving this form is responsible for reviewing it, making sure that the laboratory has received all the samples that were sent, and verifying that the correct analyses were requested. If an error is found, call the laboratory immediately, and document any decisions made during the telephone conversation, in writing, on the Acknowledgment of Sample Receipt form. In addition, correct the COC form and fax the corrected version to the laboratory.

Submit the Acknowledgment of Sample Receipt form (and any modified COC forms) to be copied. A discussion of copy distribution is provided in SOP AP-02.

## **ARCHIVE RECORD FORMS**

On the rare occasion that samples are archived at an Integral office, it is the responsibility of the project manager to complete an Archive Record form. This form is to be accompanied by a

copy of the COC form for the samples, and will be placed in a locked file cabinet. The original COC form remains with the samples in a sealed resealable (e.g., Ziploc®) bag.

## STANDARD OPERATING PROCEDURE (SOP) AP-04

### SAMPLE LABELING

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#### SCOPE AND APPLICATION

This SOP describes the general Integral procedures for labeling samples, and the three kinds of labels that can be used on a project (i.e., sample labels, sample tags, and internal sample labels). Consult the project-specific sampling and analysis plan (SAP) to determine the exact sample identifiers and sample labels that are required for a given project. If they are not specified in the SAP, then follow the designations below.

#### SAMPLE IDENTIFIERS

Before field sampling begins, establish sample identifiers to be assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., replicates) to ensure proper data analysis and interpretation, 2) to obscure the relationships between samples so that laboratory analysis will be unbiased by presumptive similarities between samples, and 3) to track individual sample containers to ensure that the laboratory receives all material associated with a single sample. To accomplish these purposes, each container may have three different codes associated with it: the sample identifier, the sample number, and the sample tag number. These codes and their use are described as follows:

- **Sample Identification Code**—The sample identification code (Sample ID) is a unique designation that identifies where and how the sample was collected. The sample identifier is recorded in the field logbook *only* and is not provided on the sample label or chain-of-custody (COC) form. The sample identifier is a multiple-part code. The first component begins with the letter abbreviation; for example, “SWNS” or “SWNB” to designate the surface water sample was collected from the near-surface or near-bottom of the water column. The second part could identify the sampling event; for example, “1” to designate Round 1 sampling. The third part could contain an abbreviation for whether the station is a single point (SP), a transect (TR), a composite (CO), or a vertically integrated station (VI). The station number would be the final component of the sample identifier. Use leading zeros for stations with numbers below 100 for ease of data management and correct data sorting.

If appropriate, add a supplemental component to the sample identifier to code field

duplicate samples and splits. Use a single letter (i.e., a suffix of “A” and “B”) to indicate field duplicates or splits in the final component of the sample identifiers. For equipment decontamination blanks, assign sequential numbers starting at 900 instead of station numbers. Use a sample type code that corresponds to the sample type for which the decontamination blank was collected. Additional codes may be adopted, if necessary, to reflect sampling equipment requirements (see project-specific SAP).

Examples of sample IDs are as follows:

- SWNS-1-SP-002: Surface water sample collected from the near-surface at a single point during Round 1 from Station 2.
- SWNB-1-TR-010-A: Duplicate surface water sample from the near-bottom transect during Round 1 from Station 10.
- **Sample Number**—The sample number is an arbitrary number assigned to each distinct sample or split that is shipped to the laboratory for separate analysis. The sample number appears on the sample containers and the COC forms. Each sample will be assigned a unique sample number. All aliquots of a composited field sample will have the same sample number. In cases where samples consist of multiple bottles from the same location, assign each bottle the same sample number and time. However, assign replicates from the same location different sample numbers and times. Sample numbers of related field replicates will not necessarily have any shared content.

Each field split of a single sample will also have a different sample number and time. The sample number is generally a unique six-digit number that includes a two-digit media code and a four-digit number. The media code may be site-specific, but the Integral default codes are as follows:

- SS—Surface soil
- BH—Subsurface soil or rock (typically from borehole)
- GW—Groundwater
- SW—Surface water
- PW—Pore water
- SD—Sediment
- BT—Biota or biological tissue

The exact sample numbering scheme may vary from project to project. Variances in the sample numbering scheme will be described in the project-specific SAP for the field event. Example sample numbers are PW0001, PW0002, PW0003, etc.

- **Tag Number**—Attach a different tag number to each sample container. If the amount of material (i.e., everything associated with a single sample number) is too large for a single container, assign each container the same sample number and a different sample tag. A sample will also be split between containers if a different preservation technique is used for each container (i.e., because different analyses will be conducted).

The sample tag number is a unique five- or six-digit number assigned to each sample label (or “tag”) for multiple bottles per sample. Integral sample labels come with a preprinted sample tag number. The tag number provides a unique tracking number to a specific sample bottle. This allows for greater flexibility in tracking sample bottles and assists in field quality control when filling out documentation and shipping. Sample tags are not used by many other consultants, and there may be resistance from such firms during teaming situations. However, experience has shown that tags can be very valuable, both in the field and while processing data from field efforts.

Record tag numbers on the COC form. Laboratories use tag numbers only to confirm that they have received all of the containers that were filled and shipped. Data are reported by sample number.

Assign sample numbers sequentially in the field; sample labels are preprinted with sequential tag numbers.

## **SAMPLE LABELS**

Integral sample labels are designed to uniquely identify each individual sample container that is collected during a sampling event. Field sampling teams are provided with preprinted sample labels, which must be affixed to each sample container used. Fill out the labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- A unique number (commonly referred to as the “Tag Number”) that is preprinted on the label consisting of five or six digits; used to identify individual containers.

## **SAMPLE TAGS**

Integral sample tags are designed to be affixed to each container that is used for a sample. Sample tags are required only for environmental samples collected in certain U.S.

Environmental Protection Agency (EPA) regions (e.g., EPA Region 5). Field crews are provided with preprinted sample tags. Attach sample tags to each individual sample container with a rubber band or wire through a reinforced hole in the tag. Mark all sample tag entries with indelible ink. Fill out the tags at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservatives used, if any
- Type of analysis.

A space for the laboratory sample number (provided by the laboratory at log-in) will also be provided on the sample tag.

## **INTERNAL SAMPLE LABELS**

For benthic infaunal samples, wash away the sediment from the sample and collect the remaining benthic infauna into a sample container. Affix sample label (as discussed above) to the outside of the sample container. In addition, place an internal sample label inside the sample container. This internal sample label is made of waterproof paper; be sure to make all internal sample label entries with pencil. Fill out the internal sample labels at the time the samples are collected, documenting the following information:

- Sample number
- Site name or project number
- Date and time sample is collected
- Initials of the samplers
- Preservative used (e.g., formalin).

## **STANDARD OPERATING PROCEDURE (SOP) AP-05**

### **INVESTIGATION-DERIVED WASTE HANDLING**

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#### **SCOPE AND APPLICATION**

This SOP presents the method to be used for handling wastes generated during field sampling activities that could be hazardous. These wastes are referred to as investigation-derived waste and are subject to specific regulations.

All disposable materials used for sample collection and processing, such as paper towels and gloves, are not considered investigation-derived wastes and will be placed in heavyweight garbage bags or other appropriate containers. Disposable supplies will be removed from the site by sampling personnel and placed in a normal refuse container for disposal at a solid waste landfill.

#### **EQUIPMENT AND REAGENTS REQUIRED**

- 55-gallon drums (or appropriately sized waste container)
- Paint markers
- Tools (to open and close drum)
- Ziploc® bags
- Drum labels.

#### **PROCEDURES**

1. Place solid wastes that need to be containerized in properly labeled, DOT- approved, 55-gallon drums.
2. Properly close, seal, label, and stage all filled or partially filled drums before demobilization. Properly profile full drums and have them shipped off site to a RCRA Subtitle C facility.



3. Sampling activities generate personal protective equipment and miscellaneous debris that require disposal. Remove gross contamination from these items, and place the items in plastic bags. It is acceptable to store these items in plastic bags as an interim measure. At the end of each day, dispose of the bags at an appropriate solid waste facility dumpster.

## STANDARD OPERATING PROCEDURE (SOP) AP-06

### NAVIGATION AND STATION POSITIONING

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#### SCOPE AND APPLICATION

This SOP describes procedures for accurate navigation and station positioning required to ensure quality and consistency in collecting samples. Station positioning must be both absolutely accurate, in that it correctly defines a position by latitude and longitude, and relatively accurate, in that the position must be repeatable, allowing field crews to reoccupy a station location in the future (e.g., for long-term monitoring programs).

This SOP is structured as follows:

- Procedures
- Equipment capabilities
- Basic data collection, navigation, and file transfer.

#### PROCEDURES

A global positioning system (GPS) is used to obtain latitude and longitude coordinates for locations where samples are to be collected and to verify the accuracy of coordinates through use of control points and post-processing differential correction to industry standards.

For most sampling events, the GPS unit is used to direct the sampling team to the proposed sampling location, having loaded target locations onto the device prior to field deployment. For some sampling events, the GPS unit is used to record positions on the fly, in the field.

A typical positioning objective is to accurately determine and record the positions of all sampling locations to within 2 m. Positioning accuracies on the order of 1 to 5 m can be achieved<sup>1</sup> but may be diminished during times when the geometry of the satellites above the GPS antenna does not provide the optimum signal. The time intervals during the day when accuracies are decreased are available on Trimble Navigation Limited's (Trimble's) web site: <http://www.trimble.com/gnssplanningonline/#/Settings>.

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<sup>1</sup> GPS accuracy depends on the unit (Table 1).

## Use of Control Points

GPS accuracy should be verified at the beginning and end of each sampling day through use of one (or more) known horizontal control point(s) in the study area. The GPS position reading at any given station can then be compared to the known control point. All GPS signal propagation is controlled by the U.S. government (the U.S. Department of Defense for satellite signals and the U.S. Coast Guard for differential corrections).

## Daily GPS Activities

A consistent routine should be established for each day's positioning activities. After successful reception of differential signals is confirmed, the GPS can be powered up and the software booted. As stated above, accuracy of the system should be verified through use of a horizontal control point.

The sampling team will proceed to the vicinity of a target station location selected by the team leader. That station location is then selected from a number of preloaded station locations that have been entered into the integrated navigation system database. Once the station has been selected, the positioning data are displayed on the computer screen or hand-held unit to assist in proceeding to the station and in maintaining the station position during sampling. A confirmed position is recorded electronically each time a sample collection is attempted (i.e., during sediment grab sampling and coring, the locations of both accepted and rejected grab samples or cores are recorded). Upon recovery of the sampling device, the station position latitude and longitude coordinates from the archived GPS file are read and recorded in the field logbook or on log sheets as a backup to the GPS record. The sampling time and water depth are also recorded, if applicable. Ancillary information recorded in the field logbook may include personnel operating the GPS, tidal phase, type of sampling activity, and the time when coordinates were collected.

## On-Water Sampling Events

For on-water GPS navigation, an assessment should be made of the type of vessel that will be used to do the work and from what type of structure (e.g., side davit, A-frame, moon pool) the sampling equipment be deployed. A GPS antenna must be installed immediately above the location where the sampling equipment will be deployed.

**Note:** On-water GPS navigation can be affected by overhead structures. If sampling from a boat is conducted underneath a bridge or adjacent to tall buildings, a laser range finder such as the Trimble TruPulse 200 Rangefinder may be needed. If sampling is performed in deep water from a boat (e.g., collecting sediments with a remotely operated vehicle), it may be necessary to install an ultrashort baseline (USBL) underwater acoustic positioning system on the sampling equipment. The USBL

system is set up differently from a common GPS unit. **This SOP does not address USBL or laser range-finder navigation.**

When a GPS antenna is mounted on a movable A-frame, the antenna should face up when the A-frame is extended out. The antenna may be mounted on an angle when the A-frame is retracted and not in use. This will optimize satellite signal reception during sampling.

If an antenna cannot be mounted exactly above the point of sampling, an offset should be incorporated into the navigation software so that each time a sample is taken, the correct location of its deployment will be accurately recorded/placed on the map (Figure 1).



Figure 1. GPS Antenna Mounted with an Offset from the Winch Location

GPS antennae can be connected through a cable or via wireless Bluetooth® connection. Bluetooth® connections are typically limited by distances less than 10 m. If the GPS antenna is to be mounted at distances beyond 10 m, a cable connection may be needed.

The GPS antenna should be mounted vertically, with the dome facing toward the sky, at the time of deployment. The GPS antenna can be mounted on top of a davit or A-frame, or offset

over a cabin roof or other boat structure. The GPS antenna may be coupled with a receiver such as the Trimble Pro XH model and connected to a Trimble Yuma tablet or field laptop. The Yuma tablet is waterproof and therefore does not need to be situated inside a cabin (consult user manual for its operation). However, if a standard laptop is used for navigation, there must be enough cable available to connect the laptop to the antenna from inside a cabin or protected area, unless a Bluetooth® connection is available.

1. Mount the GPS antenna for receiving differential corrections on a convenient fixture outside the cabin.
2. Locate the differential corrections receiver and the computer in the cabin. Orient the video screen of the computer to allow the vessel operator to observe on-screen positioning data from the helm. A second monitor may be necessary if the distance between navigator and boat operator makes this setup impractical.
3. Alternatively, manually place a GPS antenna as close as possible to where the sampling will occur (e.g., the moon pool on a barge), and direct the vessel operator to the sampling station location.
4. Once the sampling vessel is anchored or is maintaining its position at the sampling station location, record the horizontal coordinates of the station on the GPS unit and in the field logbook. In some instances, coordinates should be recorded once the sampling device (e.g., core or grab sampler) has contacted the bottom of the water body, or if collecting surface water samples, when the sampling device is in the water at a specific sampling depth.

All target GPS coordinates should be loaded into the GPS unit before field sampling activities begin. The navigator should make sure that the sampling coordinate system is set up according to field sampling plan specifications (e.g., World Geodetic System 1984 [WGS84] or a site-specific state plane, if required). To facilitate navigation, additional background files containing georeferenced aerial photos or polygons of river edges, facility structures, etc. may be preloaded as well.

After sample collection, actual sample location positioning will be checked for precision against the target sampling location to ensure that samples were collected at the target location within the project's navigational error specifications (e.g., within  $\pm 2$  to 10 m from the target, depending on project data quality objectives).

## **EQUIPMENT CAPABILITIES**

### **GPS Units**

Integral maintains up-to-date navigation equipment and some units may not be listed in this SOP. However, the basic principles of GPS navigation, field setup, and data collection are, for

the most part, similar to the ones described herein. Integral owns several types of Trimble GPS units, such as the GeoXH, Yuma with a ProXH receiver, and Juno 3B.

The GeoXH GeoExplorer 2008 series (Figure 2) runs the Windows Mobile operating system, and the newer Juno 3B (Figure 3) runs Windows Handheld Professional. The Trimble Yuma rugged tablet computer (Figure 4) runs the Windows 7 Professional operating system. All units utilize Trimble TerraSync software for GPS data collection. The GeoXH and Yuma are capable of offering submeter accuracy (the Yuma has an internal GPS antenna capable of 2 to 5 m accuracy, but requires an external ProXH antenna for subfoot accuracy). The Juno is capable of 1 to 3 m post-processed accuracy. Table 1 presents an accuracy comparison between the different units. Integral also owns a Trimble TruPulse 200 laser range finder (described in the “Sources of Error” section, below).



Figure 2. GeoXH GeoExplorer 2008 Series Unit



Figure 3. Trimble Juno



Figure 4. Trimble Yuma Rugged Tablet Computer with Pro XH Receiver Mounted on a Waist Belt

Table 1. GPS Unit Comparison

GPS Unit	Horizontal Accuracy <sup>a,b</sup>	Vertical Accuracy <sup>a,b</sup>
GeoXH handheld 2008	≥15 cm–1 m	≥ 2x horizontal error
Yuma with ProXH antenna	≥15 cm–1 m	≥ 2x horizontal error
Yuma without ProXH antenna	2–5 m	≥ 2x horizontal error
Juno 3B	2–5 m	≥ 2x horizontal error

Notes:

<sup>a</sup> The stated accuracy assumes post-processing differential correction.

<sup>b</sup> The vertical and horizontal precisions are provided for each GPS point to a specified confidence level.

## Sources of Error

GPS error is temporal- and location-specific depending on satellite locations and atmospheric conditions. Obtaining high-accuracy GPS data requires rigorous data collection techniques, and data collection can be compromised by inconsistent antenna height, obstructed view of the sky (e.g., tree canopy, docks, bridges), available satellites in view, station occupation time, atmospheric conditions, and distance from the base station. A laser range finder can be used with the unit if the target location is obstructed by tree canopy or structures.

Consistently achieving 15 to 30 cm horizontal accuracy for large field-collection efforts requires preplanning and optimal conditions. Users should confirm GPS accuracy by collocating GPS coordinate collection with a surveyed monument (i.e., base station) prior to high-accuracy fieldwork. Users must set the positional dilution of precision (PDOP) value to 6 as the standard setup for accuracy. However, if field conditions preclude receiving a good satellite signal, the PDOP can be set to “Productivity” in the TerraSync software during fieldwork. This will allow the unit to accept available satellite signals to navigate to a target location; an example of the means for this adjustment is shown in Figure 5 (not applicable for the Juno). Setting the PDOP to Productivity will, however, decrease the level of accuracy in the field.

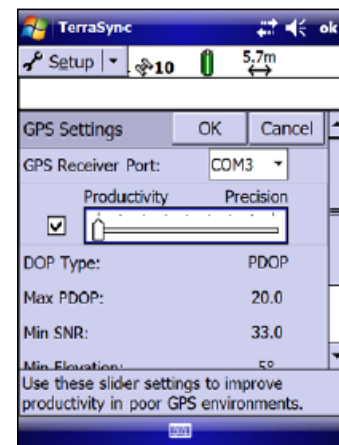


Figure 5. Recommended TerraSync GPS Settings

## Differential Correction

To achieve optimal accuracy, GPS data must be post-processed using GPS Pathfinder Office. Differential correction reduces errors and provides a report that states the estimated horizontal accuracies in error ranges. With the GeoHX and Yuma (with ProXH antenna), the average horizontal error of most GPS field efforts is typically within 0.5 m, although individual station location errors may range from <15 cm to >1 m (Figure 6). With the Juno, the average horizontal error in the field is typically 2 to 5 m. Vertical error is at a minimum 2 to 3 times that of horizontal error, but vertical error is not estimated with the differential correction report. The corrected GPS data include horizontal and vertical precision calculated to a specified confidence level.

Estimated accuracies for 18012 corrected positions are as follows:	
Range	Percentage
0-15cm	27.3%
15-30cm	43.1%
30-50cm	15.4%
0.5-1m	11.1%
1-2m	2.7%
2-5m	0.4%
>5m	0.0%

Figure 6. Error Ranges from Differential Correction Report

Integral's geographic information system (GIS) staff can assist with loading station coordinates and base maps onto the GPS units prior to fieldwork mobilization.

Following field collection, Integral GIS staff can assist with transferring, correcting, archiving, and preparing source files for integration into Integral's data management processes. If a project requires greater accuracy and less uncertainty, a licensed surveyor can provide subcentimeter horizontal and vertical location accuracy using a survey-grade GPS unit or total station instrument.

## BASIC DATA COLLECTION, NAVIGATION, AND FILE TRANSFER

This section outlines basic data collection, navigation, and file transfer using Trimble's TerraSync software. Questions regarding GPS use for fieldwork should be directed to Integral's GIS team. GPS settings related to data accuracy (PDOP, signal-to-noise ratio [SNR], etc.) should not be changed.

If new to using Trimble software, it is strongly recommended that a mock data collection event be conducted *before* actual data collection begins in the field. Any area outside of an office building, in a nearby parking lot, or anywhere that is relatively free of obstructions such as tall buildings or large trees will suffice.



## Basic Data Collection

### Create a New File

1. In the upper left corner, select Data from the Section button.
2. Directly below in the Subsection button, select New and then New File.
3. In the New File screen, set File Type to Rover, Location to Default, type in a File Name, and set Dictionary Name to Generic (unless a specific data dictionary has been created).
4. Confirm antenna height dialogue appears. Enter the correct height if collecting vertical. Select OK.



### Create (Log) GPS Features

1. Tap Create, and the Collect Features screen appears. If the generic data dictionary is chosen (typical), there are three feature options: Point\_generic, Line\_generic, and Area\_generic.
2. To record a point feature, select Point\_generic and tap Create. An attribute entry screen will appear, and the GPS unit will start logging positions. All logging positions will be averaged to compute a final GPS position. The running number of logging positions appears next to the pencil icon at the top of the screen.
3. While the unit is logging positions, remain stationary and fill out the Comment field. The Comment field is a text field that can have any combination of letters, numbers, or symbols (up to 30 characters). Typically, by the time the Comment field is completed, the unit has logged enough positions. Approximately 20 to 30 positions are sufficient; however, a minimum of 40 to 60 logged positions is required for a greater level of positioning accuracy. In theory, a greater number of positions results in a more accurate final position, although additional factors also contribute to accuracy (satellite distribution, canopy cover, etc.); with a very large number of positions, there comes a point of diminishing returns.
4. To stop logging positions and to record the feature, press OK. This returns you to the Collect Features screen.

Line and area features are collected in much the same manner, except that the user walks along the alignment or outline of the feature instead of remaining in place. The pace of the walk should be rather slow, to allow the GPS unit to log enough positions along the way. A line feature will simply create a line that follows the walked path. An area feature will always be a closed polygon, so if the end is not at the point of beginning, the GPS will automatically

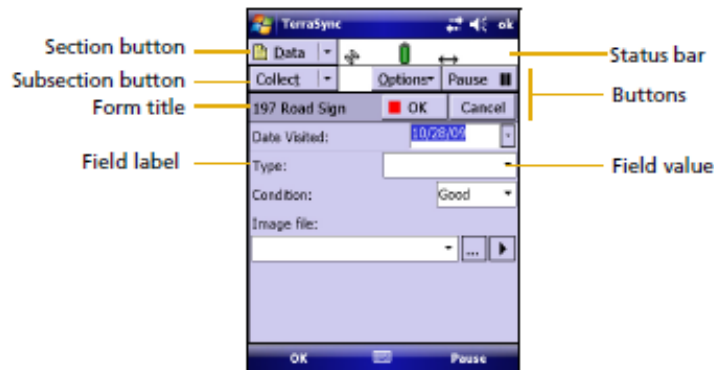
close the loop by connecting the first and last position, regardless of how far apart the two might be. During collection of lines and features, position logging can be paused if there is a need to deviate from the line. Operations are resumed by tapping Resume.

The map can be viewed at any time while features are being collected:

1. Tap the Section button and select Map.
2. To go back to data collection, tap the Section button again, and select Data.
3. To end data collection, tap the Collect button and select Close.

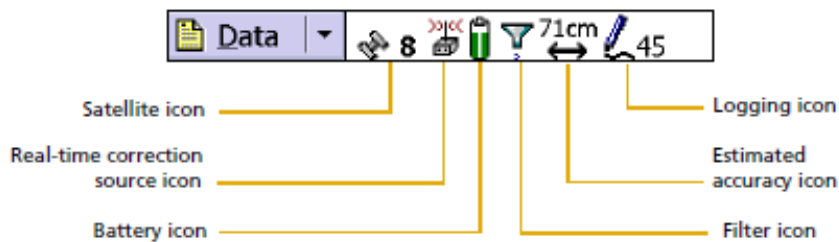
The TerraSync interface and icons are shown below:

The screen below shows elements that are common to all screens in the TerraSync software:



### Status bar

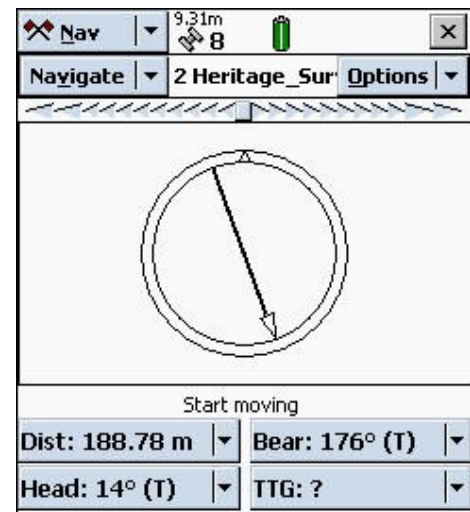
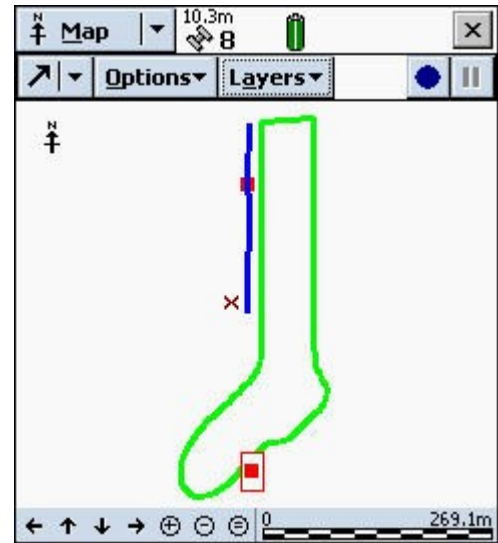
The status bar appears at the top of the TerraSync software screen and provides basic status information about the connected GPS receiver. For information about how to connect to a GPS receiver, see [Connecting to a GPS receiver, page 48](#).



## Navigation

The Navigation section of the program permits users to navigate from their current position (small red **x**) to a selected target or feature.

1. To open the Navigation section, tap the Section List button and select Navigation.
2. To navigate to a point, select the desired point feature in Map view. The selected feature will be displayed as the boxed point feature symbol (at right).
3. Tap Options—Set Nav Target in Map view. The navigation target will now be displayed as a blue crossed-flags navigation target symbol.
4. Select Navigation from the section list, and note the following items (depicted in the example to the right):
  - Target's identification and type (2 Heritage\_Survey\_pt)
  - Distance to target (188.78 m)
  - Bearing to target (176°); the arrow pointer indicates the bearing graphically
  - User's current heading (14°); the pointer on top of the dial represents the user's heading.



TerraSync's "compass" depends on a series of GPS positions to detect the direction of travel, so users must keep moving for the compass to stay in an active state. If they stop, the compass will wander and drift.

Users follow the arrow pointer until the target feature is reached. As the target is approached, an alert tone will sound, and the view changes to a zoomed-in representation of the target feature and the current GPS position.

## File Transfer

Data files are transferred to and from the GPS unit using the Data Transfer utility. This utility is part of the GPS Pathfinder Office software but can also be used as a stand-alone program (free to download onto any computer).

1. Before using the software, connect the GPS unit to your computer via the universal serial bus (USB) cable. Microsoft Mobile Device Center (Windows 7) should successfully connect to the GPS unit.
2. Once that connection is successful, open GPS Pathfinder Office; select Utilities and then Data Transfer (if you are using the stand-alone version, simply open the program).
3. In the device box, select GIS Datalogger on Windows. It should show the GPS as successfully connected. There are two options—Send and Receive.
4. To download your data, select the Receive tab, and hit Add and then Data File. The files that appear are the files in the GPS unit. Any files that have not been downloaded (or modified since the last download) will be selected in bold.
5. Click Open; the Files to Receive dialog box appears. A list of all files that will be downloaded appears, and you can remove any from the list as needed.
6. Click Transfer All; a message box showing summary information about the transfer appears.

Transferring data on the Yuma tablet is done somewhat differently. With the Yuma, the GPS and the computer are both on the same device. The difference is that the files still need to be transferred to and from the computer portion of the device. The easiest method is to use a thumb drive.

1. To load data onto the unit (Send, in the Data Transfer utility), point the path to the thumb drive containing the files to upload.
2. To download data, follow the instructions above, and take note as to where the files are being transferred in the Yuma computer, in the Destination field.
3. After transferring files, navigate to the files in Windows Explorer and copy them to the thumb drive.

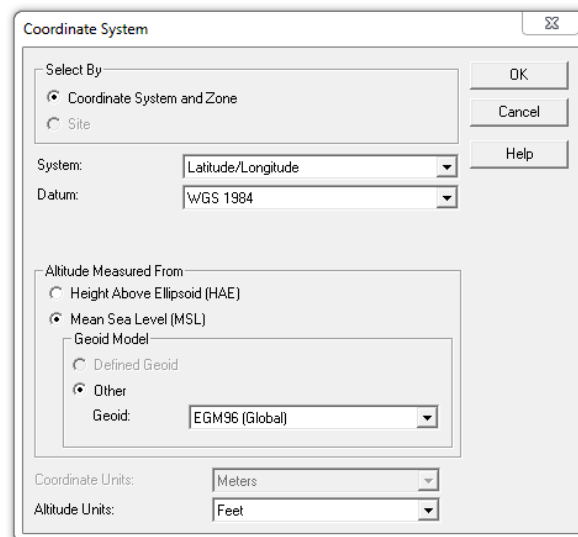
## Loading Background Files

### File Types

Background layers supported by TerraSync include vector data (.shp) and raster data (.bmp, .jpg, .sid, and .tif). The raster data must be uploaded with a world file (.wld, .jgw, .tfw, .sdw), which tells TerraSync the coordinate system in which the data is projected. All data should be projected into WGS84 before it is uploaded to the GPS unit.

### Uploading to the GPS Unit

1. To transfer background data to the GPS unit, open the file in Pathfinder.
2. Set the coordinate system of the Pathfinder Office display to WGS 1984 by going to Options > Coordinate System.
3. To open the background file, go to File > Background and navigate to the file by clicking Add.
4. To check that it is displaying correctly, click on View > Map. Once it is displayed in Pathfinder, it can be transferred to the GPS unit.
5. Connect the GPS unit to the computer and click Utilities > Data Transfer.
6. On the Send tab, click Add > Background and add the file.
7. Click Transfer All for the file to be uploaded to the unit.



### Displaying on the GPS Unit

1. Open TerraSync on the GPS unit and click on the drop-down menu next to Setup.
2. Go to Map, click Layers > Background Files, and choose the background file.
3. Click OK and the file will be added to the map.

## STANDARD OPERATING PROCEDURE (SOP) AP-14

### SAMPLING FOR PFAS COMPOUNDS

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#### SCOPE AND APPLICATION

This SOP provides information on sampling equipment and techniques for various media and is used during field investigations to collect samples to be analyzed for per- and polyfluoroalkyl substances (PFAS). It is based on our current knowledge of PFAS materials and their behavior in the environment and is subject to change as more information regarding PFAS becomes available. This SOP is a supplement to, but does not replace, other Integral SOPs for field collection.

It is possible to cross-contaminate samples from other sources. For this reason, the persons overseeing the sampling event or collecting samples should have a solid understanding of materials known to contain PFAS, and where PFAS-containing materials are suspected. Materials containing PFAS should be avoided during sampling and those suspected of containing PFAS should be approved by the project manager before sampling occurs.

The equipment and procedures described below are designed to help prevent sample alteration or cross-contamination through the use of specific sampling and handling techniques.

#### EQUIPMENT AND SUPPLY CONSIDERATIONS TO PREVENT CROSS CONTAMINATION

Items or materials known to contain PFAS should not be used during sampling. Prohibited items are those containing the fluoropolymers identified in Table 1:

Table 1. Fluoropolymers and Their Trade Names

Fluoropolymer	Abbreviation	Trade Name
Polytetrafluoroethylene	PTFE	Teflon <sup>®</sup> , Hostaflon <sup>®</sup>
Polyvinylidene fluoride	PVDF	Kynar <sup>®</sup>
Polychlorotrifluoroethylene	PCTFE	Neoflon <sup>®</sup>
Ethylene tetrafluoroethylene	ETFE	Tefzel <sup>®</sup>
Fluorinated ethylene propylene	FEP	Teflon <sup>®</sup> FEP and Hostaflon <sup>®</sup> FEP

Many products, equipment, and field supplies commonly used during fieldwork are known to contain PFAS. The following items, and other similar items, should be substituted with an alternative that is known to be PFAS-free or screened to verify that they are PFAS-free:

- Clothing and personal protective equipment (PPE) that is designed to be waterproof, water-repellent, stain-resistant, or UV-protective (e.g., uncoated Tyvek products)
- Clothing that is new or recently washed with fabric softener
- Personal body products such as shampoos, moisturizers, and cosmetics (unless applied on a portion of the body that will be covered by PFAS-free clothing)
- Sunblock and insect repellent
- Food packaging
- Waterproof field books and felt-tip markers
- Adhesive products/materials, such as sticky notes (e.g., Post-It®) and preprinted labels
- Aluminum foil, particularly non-stick foil
- Chemical ice packs
- Canopy tent.

However, *personal safety equipment always take priority* and if a PFAS-free PPE alternative is not available, the PPE used must be approved by the project manager, documented, and discussed in the field sampling report.

The following requirements further apply to fieldwork involving the collection of samples to be analyzed for PFAS.

- Food and food packaging within the sampling zone are not permitted at any time during sampling for PFAS.
- Documentation of field activities should be on loose paper on an aluminum clipboard or in a waterproof field book that has not been treated with PFAS.
- Field notes should be taken with a ball point pen, fine or ultra-fine point Sharpie® marker.
- Chemical ice packs should not be used unless it is verified that they are PFAS-free. Ice used for sample preservation and storage should be from a PFAS-free water source. Samples for PFAS analysis should be placed on water ice immediately and should ideally be received by the laboratory at a temperature less than 6 °C.
- Disposable, powderless nitrile gloves must be worn during PFAS sampling and handling activities and should be changed frequently during and between sampling activities.

- If sampling takes place in inclement weather and a canopy tent is required, disposable nitrile gloves must be worn when assembling and/or moving the tent.

## **GENERAL SAMPLING PROCEDURES**

1. Conduct sampling in accordance with best practices and regulatory guidance, if applicable.
2. Ensure sample containers are made of laboratory-provided polypropylene or high-density polyethylene (HDPE). If liners are present in the sample caps, verify that they are not made of PVDF, PTFE, or Teflon® and do not contain PFAS. Avoid the use of glass bottles as they may result in low-biased sample results.
3. If non-dedicated non-disposable equipment is used for sampling, decontaminate the equipment properly. Check decontamination reagents to ensure that they do not contain PFAS before use. Similarly, check water used for decontamination (i.e., field equipment blanks) to verify that it does not contain PFAS.
4. Use laboratory-certified PFAS-free water should for decontamination purposes. Test water from other sources used for decontamination purposes for the absence of PFAS prior to and throughout a sampling event at frequent intervals (specified in the project Quality Assurance Project Plan).
5. Be aware that some decontamination reagents may not be suitable for the removal of PFAS during decontamination (i.e., they may not be appropriate for unbinding PFAS from the surface of field equipment prior to rinsing). Collect equipment blanks to ensure that the reagents used for decontamination successfully remove PFAS from field equipment.

## **SOIL SAMPLING PROCEDURES**

1. Collect soil samples with stainless steel, acetate, or polypropylene constructed equipment. Do not use liners that contain PFAS for soil sampling. Acetate or single-use PVC liners may be acceptable.
2. Verify that bags to be used to store soil or sediment samples are PFAS-free prior to usage.
3. Exercise care not to cross contaminate PFAS samples if samples for other analyses are being collected. A separate set of sampling equipment and laboratory sample containers is recommended for PFAS sampling, with decontamination, as necessary, and glove changes in between sampling for non-PFAS contaminants of concern and PFAS.



## **GROUNDWATER SAMPLING PROCEDURES**

1. If volumetric purge (submersible pump) or low flow purge are being used, verify that all equipment and components are PFAS-free (manufacturing certification and testing). If confirmation of PFAS-free components cannot be verified prior to sampling, collect an equipment blank for all components to determine the potential PFAS contribution. Use laboratory provided PFAS-free deionized water to collect the equipment blank.
2. Use silicone and/or LDPE/HDPE tubing with the appropriate pump. If a monitoring well contains dedicated tubing, remove the tubing and replace it with recommended tubing. In this situation, collect samples after at least one well volume purge prior to sampling for PFAS.
3. Note that recommendations are variable for length of time that dedicated tubing is in use before it is replaced, and for the amount of purging to be conducted before sampling begins. If it is anticipated that dedicated tubing may be a source of PFAS cross contamination, take extra precautions, such as removing the tubing 14 days prior to sampling or consider purging three well volumes.
4. If a bailer is used to collect groundwater grab samples, ensure it is constructed of non-PFAS containing materials, including its retrieval string. Dispose of bailers and retrieval string after a single use.
5. Exercise care not to cross contaminate PFAS samples if also collecting samples for non-PFAS analyses. For example, if collecting samples to be analyzed for volatile organic compounds (VOCs) and PFAS, collect the VOC samples using a pump with HDPE and silicone tubing, and then collect a second set of samples, the PFAS samples, after changing gloves and switching sample container sets.

## **SURFACE WATER SAMPLING PROCEDURES**

1. If transfer bottles are necessary for surface water sample collection, ensure they are PFAS-free and made of the same material as the laboratory-provided sample containers.
2. If waders are necessary, check to ensure the wader material does not contain PFAS (i.e., has not been coated with waterproof material).

## **WATER SUPPLY SAMPLING PROCEDURES**

1. If a treatment unit is in use, collect both a pre- and post-treatment sample. Carbon filtration, reverse osmosis, and other filter media may bias laboratory results for PFAS.

2. Allow water to run freely until water quality parameter stabilization has occurred, typically between 3 and 5 minutes.
3. Reduce water flow rate for minimal aeration.
4. Do not filter samples for PFAS analysis.

## **QUALITY ASSURANCE AND QUALITY CONTROL**

- Equipment blanks are recommended to ensure that proper decontamination of sampling equipment is performed. It may also be necessary to collect samples of water being used for decontamination prior to use to ensure that water being used for decontamination does not contain PFAS.
- Blank water should be certified as PFAS-free.
- Field blanks are recommended to evaluate the potential for introduction of site-specific contaminants into samples by ambient air.
- Trip blanks are recommended to evaluate the potential for introduction of contaminants during transport of the sample containers from the laboratory to the site, or from the site to the laboratory.
- Field duplicates are recommended to verify laboratory accuracy.
- Matrix spike and matrix spike duplicate samples are recommended to assess interferences caused by the sample matrix.

## **LABORATORY ANALYTICAL METHODS**

The laboratory analytical methods for PFAS include the following:

- Drinking water (not groundwater or surface water): Method 537 (LC/MS/MS)
- Drinking, surface, and groundwater: Method 537.1M (SPE - LC/MS/MS)
- Surface, ground, wastewater: Method 8321 (LC/MS/MS)
- Surface, ground, and wastewater: Method 8327 (method is still in draft form)
- Surface and sludge water: ASTM D7979-17
- Soil: ASTM D7968-17a
- Soil and other solids: Method 8321 (LC/MS/MS).

## NOTES ON LABORATORY METHODS

Some laboratories report PFAS in ground and surface water by Method 537 modified, and U.S. Environmental Protection Agency researchers are currently working on validating methods to measure PFAS in groundwater, surface water, wastewater, and solids. The laboratory methodologies for analysis of PFAS are evolving. Laboratories should be contacted prior to sample submittal to verify that they are certified, as appropriate, to conduct PFAS analyses for the respective media being sampled and analyzed using the most updated sample methodologies. Laboratory reports should be undergo quality assurance validation to ensure that the method selected for analysis is suitable.

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## **STANDARD OPERATING PROCEDURE (SOP) GW-01**

### **DECONTAMINATION OF GROUNDWATER SAMPLING EQUIPMENT**

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#### **SCOPE AND APPLICATION**

This SOP describes procedures for decontaminating sampling equipment used for groundwater sampling that could come in contact with contaminated media. To prevent potential cross contamination of samples, all reusable groundwater sampling and processing equipment will be decontaminated before each use. At the sample collection site, a decontamination area will be established in a clean location, upwind of actual sampling locations, if possible. This decontamination area is where all groundwater sampling and processing equipment will be cleaned. Decontaminated equipment will be stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel will follow all relevant procedures and will wear protective equipment as stipulated in the site-specific health and safety plan.

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either inorganic or organic materials. General procedures were adopted from the Standard Practice for Decontamination of Field Equipment Used at Waste Sites (ASTM 2002).

#### **EQUIPMENT AND REAGENTS REQUIRED**

- Plastic sheeting
- 55-gal, U.S. Department of Transportation-approved drums (if required)
- Alconox<sup>®</sup> or Liquinox<sup>®</sup> detergent
- Acid rinses (for inorganic constituent sampling); either reagent-grade diluted nitric or hydrochloric acid (if required)
- Solvent rinses (for organic constituent sampling); either pesticide-grade hexane, isopropanol, or acetone (if required)
- Deionized/distilled water (generally provided by laboratory) and potable water
- 5-gal buckets or other appropriate containers

- 4-ft length of 2-in. polyvinyl chloride (PVC) tubing with an end cap (if required)
- Scrub brushes
- Personal protective equipment, including appropriate gloves and goggles.

## PROCEDURES

The following sections detail the procedures for decontaminating sampling equipment that has been, or could be, contaminated with inorganic or organic chemicals and for decontaminating the submersible pump.

### ***Inorganic Chemicals—Decontamination of Sampling Equipment***

1. Wipe equipment free of gross solids.
2. Wash equipment with an Alconox® or Liquinox® solution, scrubbing off any residue.
3. Rinse generously with potable water.
4. Rinse equipment with acid (0.1 N nitric or hydrochloric) if specified in the site sampling and analysis plan (SAP).
5. Rinse with deionized water.
6. Allow to air dry, if practical.
7. Wrap equipment in new aluminum foil if it will not be used promptly.
8. Place all sampling equipment, gloves, and other disposable materials in garbage bags after decontaminating. The wash and rinse must be placed in containers for proper disposal.

### ***Organic Chemicals—Decontamination of Sampling Equipment***

1. Wipe equipment free of gross solids.
2. Wash equipment with an Alconox® or Liquinox® solution, scrubbing off any residues.
3. Rinse generously with tap water.
4. Rinse equipment with solvent (pesticide-grade hexane, isopropanol, or acetone) if specified in the site SAP.
5. Rinse with deionized water.
6. Allow to air dry, if practical.
7. Wrap equipment in new aluminum foil if it will not be used promptly.

8. Place all sampling equipment, gloves, and other disposable materials in garbage bags after decontaminating. Place wash and rinse fluids in containers for proper disposal.

### **Decontamination of Submersible Pump**

1. Place the pump in a 5-gal bucket containing potable water and a small amount of Alconox® or Liquinox® detergent. Place discharge hose into same bucket.
2. Turn on the system and pump water through the sampling system. Add more potable water as needed and pump for 2 minutes.
3. Place the pump into a second 5-gal bucket containing tap water leaving the discharge hose in the first bucket. Turn on the system and pump until the soapy water is purged from the pump and tubing. Place the discharge hose into the second 5-gal bucket of water and pump for 1 minute.
4. Turn off system and place the pump into the 4-ft section of 2-in. inside diameter PVC tubing fitted with an end cap. Pour organic-free deionized water into the decontamination tube. Stand by with additional deionized water.
5. Turn on the pump and pull deionized water through the system. Add more water until at least 3 L of deionized water is pumped through the system.
6. Remove the pump from the decontamination tube.
7. Place all sampling equipment, gloves, and other disposable materials in garbage bags after decontaminating. Place wash and rinse fluids in containers for proper disposal.

### **REFERENCE**

ASTM. 2002. Standard practice for decontamination of field equipment used at waste sites. D5088-02. American Society for Testing and Materials, West Conshohocken, PA.

## **STANDARD OPERATING PROCEDURE (SOP) GW-02**

### **MEASUREMENT OF DEPTH TO WATER**

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#### **SCOPE AND APPLICATION**

This SOP describes the required equipment and the procedures used for the collection of water level data. Alternate equipment may be used if necessary, as long as the general procedures described below are followed. Typically water levels are collected from all the site wells as expeditiously as possible so that the water level data can be used to create potentiometric surface maps that are representative of a “single” point in time. This SOP does not address interpretation of water level data and the special care and hydraulic expertise that should be used to interpret water level data sets in unique environments (i.e., tidally influenced wells).

Depth to groundwater surface is measured using an electric water level meter. A light on the water level meter illuminates and an alarm sounds when the weighted probe tip contacts the water surface in the well and completes an electronic circuit. The measured depth to water is determined to within 0.01 ft by noting the point on the probe cable that corresponds to the measuring point at the top of the well/piezometer casing at the initial point of contact. The measuring point should be notched at the lip of the casing, typically either on the high side or on the north side.

#### **EQUIPMENT AND REAGENTS REQUIRED**

- Electronic water level indicator (Solinst® or equivalent)
- Potable and distilled/deionized water
- Alconox® or Liquinox® detergent
- Tape measure with stainless steel weights
- Disposable bailer (if light, nonaqueous-phase liquid [LNAPL] conditions are unknown)

## PROCEDURES

### Water Level Measurements

1. Check the operation of the meter by turning on the indicator switch and pressing the test button.
2. Open well cap to allow equilibration with ambient atmospheric pressure.
3. Monitor air quality at the well head if volatile contaminants are or may be present, or as specified by the project-specific health and safety plan.
4. Check for possible presence of LNAPL using a new 3-ft long disposable bailer affixed to nylon rope if conditions are unknown. Gradually lower the bailer until the bottom of the bailer is approximately 2 ft below the top of the water surface. Slowly raise the bailer to the surface and measure the product thickness using a tape measure. Record the measurement in the field logbook. Properly dispose of the bailer.
5. Decontaminate the probe and graduated cable with an Alconox® or Liquinox® solution followed by a distilled or deionized water rinse.
6. Hold the water level indicator and cable reel above the well casing and lower indicator probe and cable gradually into well until a tone (e.g., buzzer) and/or the indicator light illuminates, denoting that the indicator probe has made contact with the water surface. Stop lowering the cable.
7. Note the point on the graduated cable that corresponds to the measuring point at the top of the casing when the electronic circuit is first completed. If necessary, grasp tape with thumb and index finger exactly at the measuring point marked at the top of the well casing. Pull tape out of well slowly and read the measurement.
8. Draw the cable about 1 ft above the surface of the water, then lower it and repeat Steps 6 through 8. If the two readings differ by more than 0.01 ft, repeat until the measured readings stabilize. Water level records should always use the measurement taken as the indicator is lowered into the well, not as it is raised.
9. Remove the cable from the well or piezometer.
10. Record the stabilized depth-to-water measurement in the field logbook.
11. Decontaminate the probe and graduated cable with Alconox® and tap-water wash and distilled or deionized water, as appropriate.



12. Lower a weighted steel measuring tape slowly from center of well or piezometer if the total depth of the well needs to be measured. Alternately, the water level meter can be used to measure the total depth of the well. However, when measuring the total depth, the depth from the measuring point of the probe to the bottom of the probe must be **added** to the measurement because the graduated cable is referenced to the point of the probe where the electronic circuit is completed. Sounding the bottom of the well prior to sampling of the well is **NOT** recommended because of the potential for resuspension of settled formation solids in the well.
13. Draw tape up very slowly until it is taut again when the weight hits the bottom or until the tape slackens noticeably.
14. Note the tape reading at level of casing top. Record this as well depth in the field logbook to the nearest 0.01 ft.

## STANDARD OPERATING PROCEDURE (SOP) GW-03

### LOW-FLOW GROUNDWATER SAMPLING

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#### SCOPE AND APPLICATION

This SOP presents the methods to be used for monitoring well purging and groundwater sampling using low-flow (minimal drawdown) sampling methods. The procedures outlined in this SOP are in accordance with groundwater sampling methods recommended by USEPA (1992, 1996). Details on site-specific sampling activities, equipment selection (i.e., pumps), site-specific field parameters, field quality control and quality assurance (QA/QC) samples, and laboratory analyses are presented in the work plan, field sampling plan (FSP), or quality assurance program plan (QAPP). Where possible, sampling should first be conducted in areas least affected by chemicals of interest, followed by increasingly affected areas (i.e., clean to dirty).

#### EQUIPMENT REQUIRED

- Electronic water level meter
- Groundwater parameter meter capable of measuring field parameters required by the FSP or the QAPP
- Flow-through cell
- Sampling equipment (one from list):
  - Submersible pump (bladder or Grundfos®): pump, control box, power source (typically a portable generator or 12V battery)
  - Peristaltic pump: pump with pump head, silicone tubing, tubing connectors, power source (typically 12 V battery)
- Decontamination equipment and supplies (buckets, scrub brushes, deionized or distilled water, potable water, and Liquinox® or Alconox® detergent)
- Groundwater sampling forms and logbook
- Sample tubing (type and length are project- and site-dependent)
- Sample tags/labels and appropriate documentation (e.g., chain-of-custody forms, logbook, and groundwater sample collection forms)

- Insulated cooler(s), chain-of-custody seals, Ziploc<sup>®</sup> bags
- Sample containers with preservative (if required), coolers, and ice.

## PROCEDURES

The following sections provide guidelines for preparation for purging, well purging, and groundwater sampling.

### Preparation for Purging

Preparation for purging includes inspecting the condition of the well, monitoring health and safety conditions, and calibrating and decontaminating sampling equipment. General procedures are presented below:

1. Ensure that the area around well head is clean and free of debris. If necessary, place a plastic drop cloth around well head to prevent sampling equipment from coming into contact with the ground surface.
2. Inspect condition of well (e.g., well in locked position, tightness of cap, measuring point well marked, disturbance of surface casing, straightness of well casing, condition of concrete pad). Indicate condition of well on the sampling form.
3. Remove well cap. If the site health and safety plan (HSP) identifies organic compounds as potential contaminants of concern, screen well headspace and breathing-zone headspace (if specified in the HSP) for organic vapors using the appropriate field monitoring instrument (e.g., photoionization detector).
4. Decontaminate all equipment (as specified in the FSP, QAPP, or in accordance with SOP GW-01) before use in each well. Wear nitrile gloves and/or other protective equipment as specified in the site-specific HSP during possible water-contact or equipment-contact activities. At a minimum, change gloves between each well or when it is possible for potential contaminants to be introduced into the well.
5. Measure water level using a decontaminated electronic water level meter as described in SOP GW-02 when the water level in the well has equilibrated.
6. Obtain a sample from the well using a bailer and observe the contents for evidence of free floating product (SOP GW-02), if suspected (see FSP or QAPP). Alternatively, measure free product thickness using an oil-water interface probe.
7. Calculate the well casing volume as follows:

$$\text{well casing volume (gal)} = \pi(r^2)(h)(7.48 \text{ gal/ft}^3)$$

Where:

- $h$  = height of water in the well casing (i.e., depth to bottom of the well minus depth to water) in feet
- $r$  = radius of the inside of the well casing in feet.

8. Calibrate water quality meters for measuring field parameters as appropriate. At a minimum, collect temperature, pH, and specific conductance measurements during purging and prior to sampling. Other field parameters, including dissolved oxygen, redox potential, and turbidity (recommended for inorganics) may be required as specified in the work plan or FSP. Record equipment calibration and maintenance in the field logbook. Decontaminate meters between wells by rinsing with distilled or deionized water. Manage rinsate water used for these measurements in the same manner as purge water, as defined in the work plan or FSP.

## Well Purging

Monitoring wells are purged before groundwater samples are collected for analyses. The purpose of well purging is to remove stagnant groundwater from the well. Field parameters (i.e., pH, temperature, specific conductance, redox potential, dissolved oxygen, and turbidity) are measured during the purging process to verify that stagnant water has been removed and that groundwater conditions are stable prior to sampling to ensure a representative groundwater sample is collected. A variety of pumps can be used to purge and sample the monitoring well (refer to the FSP or QAPP for the specified pump type). Refer to the manufacturer's instructions for operation of the specified pump. General procedures for purging are as follows:

1. Remove well cap.
2. Connect pump.

### **Submersible Pump** (bladder or Grundfos):

- a. Remove the pump from the pump holder and rinse with distilled water.
- b. Connect appropriate length of tubing to pump.
- c. Connect the pump to control box.
- d. Connect the control box to the power supply.

### **Peristaltic Pump:**

- a. Connect new or pre-cleaned tubing to peristaltic pump.
- b. Connect the pump to the power supply.

- c. Lower the pump intake or intake tubing (as applicable) into the water column. The pump intake should be placed at the middle or slightly above the middle of the screened interval in confined aquifers (USEPA 1996) or in unconfined aquifers not screened across the water table. Place the pump intake near the top of the water column for unconfined aquifers screened across the water table (USEPA 1996).
3. Insert multimeter into flow-through cell. Connect the discharge hose from the pump to the flow-through cell. Direct discharge from flow-through cell to an appropriately sized container to manage purge water. **DO NOT** immerse water quality meter probes into purge water containing free product because this may damage the probes.
4. Turn on the pump. Conduct purging at a rate that will minimize drawdown in the well (i.e., purge at a rate less than or equal to recharge, if possible). Recommended purge rates are generally less than 0.13 gal/min (0.5 L/min) (USEPA 1996), or a rate that results in minimal (i.e., less than 0.3 ft) of drawdown in the well. Actual purge rates will vary based on aquifer material and well construction.
5. Record field parameters on the groundwater sampling form or logbook every 3 to 5 minutes. Purging should continue at a constant rate until the water quality parameters have stabilized for three successive measurements according to the stabilization criteria provided in the table below (USEPA 1996). In the event that even very low purge rates result in evacuation of the well, collect groundwater samples for laboratory analyses as soon as sufficient groundwater accumulates in the well, regardless of the stabilization of field parameters.

Field Parameter	Stabilization Criteria
Temperature	± 1°C
pH	± 0.1 standard units
Specific Conductance	± 3 percent
Dissolved Oxygen	± 10 percent
Redox Potential	± 10 mV
Turbidity (nephelometric turbidity units)	± 10 percent

## Groundwater Sample Collection

Groundwater sampling is conducted following proper purging of the well. Where possible, groundwater samples for analyses should be collected directly from the pump discharge at the lowest rate possible to minimize cross contamination, suspension of solids, and aeration of the sample.

Sample groundwater after the water quality parameters have stabilized. The general procedures for groundwater sample collection are as follows:

1. Turn down flow rate on the control box so that water flow is stopped or minimal while maintaining sufficient pressure in the system to prevent water in the tubing or flow-through cell from flowing back into the well. If a peristaltic pump is used, turn off the pump. Take care not to release the pump head because the loss of suction will cause the water in the tubing to drain back into the well.
2. Disconnect the pump discharge hose from flow-through cell or cut the tubing just before the connection to the flow-through cell.
3. Introduce groundwater samples directly from the pump discharge tube into the proper sample container and fill it to capacity. Place a bucket beneath the sampling tube to catch any unsampled water. Target analytes, container types, and preservatives are specified in the FSP or QAPP.
4. Collect groundwater samples for multiple compounds in the recommended following order (USEPA 1992):
  - Volatile organic compounds (VOCs)
  - Dissolved gases and total organic carbon (TOC)
  - Semivolatile organic compounds (SVOCs)
  - Metals and cyanide
  - Major water quality cations and anions
  - Radionuclides.
5. Increase pump flow rate slightly so that the flow rate is approximately the same as was used for purging and fill necessary sample bottles. If sampling for VOCs, flow rate should be just enough to create a trickle of water. If sampling for other analytes, flow rate may be increased. When collecting samples for VOCs, direct the flow from the pump discharge down the side of the sample container to minimize aeration. Hold caps in hand to minimize contamination of sample. Fill all VOC sample containers to the top. A positive meniscus at the top of the container will help ensure that no air is trapped inside when cap is screwed down on the container. No air bubbles should be trapped in the sample when the container is sealed. VOC sample bottles must be checked after filling to ensure no air bubbles are present. Invert the bottle and lightly tap it to release any bubbles beneath the cap. If an air bubble is present, the VOC sample must be retaken using a fresh bottle.

6. Conduct field filtration, if required by the FSP or QAPP (recommended for inorganic analytes). If applicable, attach a new, disposable filter cartridge (typically 0.45  $\mu\text{m}$ ) to the discharge line. Collect filtered samples last and pre-rinse them by running a minimum of 0.25 gal of groundwater through them prior to collecting the sample (USEPA 1996). Introduce filtered water directly into the appropriate sample container. Note that alternate field filtration methods may be specified in the FSP or QAPP.
7. Collect QA/QC samples (i.e., duplicate, equipment rinsate, trip blank, laboratory matrix spike, and laboratory matrix spike duplicate, as applicable) at the same time by filling all bottles from the same flow. The number and types of QA/QC samples are specified in the FSP or QAPP.
8. Label sample bottles with date, sample number, time, sampler's name, and type of preservative, as described in the project-specific QAPP and in accordance with SOP AP-04. Place sample bottles in a cooler or on ice to keep samples cool (4°C). Samples must be cooled continuously from the time of collection to the time of receipt at the laboratory, as described in SOP AP-01.
9. Reconnect the discharge tubing to the flow-through cell with the multimeter. Continue pumping for 1 to 2 minutes and collect a set of post-sampling field parameters. Record the parameters on the groundwater sampling form or in the logbook.
10. Remove pump and/or tubing from the well. Close and lock the well. Decontaminate the sampling equipment in accordance with SOP GW-01. Purge, wash, and rinse water should be managed as specified in the FSP or QAPP.
11. Complete chain-of-custody form, package samples for shipment, and ship samples or arrange for courier to laboratory.
12. Document all field observations made and data generated in conjunction with the sample collection on the groundwater field sampling form.

## REFERENCES

USEPA. 1992. RCRA ground-water monitoring: draft technical guidance. U.S. Environmental Protection Agency, Office of Solid Waste, Washington, DC.

USEPA. 1996. Low-flow (minimal drawdown) ground-water sampling procedures. EPA/540/S-95/504. U.S. Environmental Protection Agency, Office of Research and Development, Office of Solid Waste and Emergency Response, Washington, DC.

## **STANDARD OPERATING PROCEDURE (SOP) GW-04**

### **PASSIVE GROUNDWATER SAMPLING**

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#### **SCOPE AND APPLICATION**

This SOP describes the methods to be used for collection of groundwater samples from monitoring wells using passive sampling techniques. The procedures outlined in this SOP are in accordance with groundwater sampling methods described by the New Jersey Field Sampling Procedures Manual (FSPM). Details on site-specific sampling activities, material selection (ie. Type of passive sampler), site-specific field parameters, field quality control and quality assurance (QA/QC) samples, and laboratory analyses are presented in the work plan, field sampling plan (FSP), or quality assurance project plan (QAPP). Where possible, sampling should first be conducted in areas least affected by chemicals of interest, followed by increasingly affected areas (i.e., clean to dirty).

#### **EQUIPMENT REQUIRED**

- Electronic water level meter
- Groundwater parameter meter capable of measuring field parameters required by the FSP or QAPP
- Groundwater logbook and project-specific forms as applicable
- Monitoring well-specific passive diffusion bag (PDB) deployment materials including well caps, suspension tethers, weights, identification tags, and connecting rings
- Passive sampling bag (one from list):
  - Pre-filled equilibrator passive diffusion samplers
  - Dual membrane passive diffusion samplers with certified deionized water for filling of the sampler
  - Hydrasleeve grab sampler with certified deionized water for filling of the sampler



- Disposable sampling materials including zip-ties and sampling straws
- Sample tags/labels and appropriate documentation (e.g., chain-of-custody forms, logbook, and sample collection forms as applicable)
- Insulated cooler(s), chain-of-custody seals, Ziploc® bags

## **PROCEDURES**

The following sections provide guidelines for deployment and collection of passive samplers as well as sample collection.

### **Preparation for Passive Sampler Deployment and/or Collection**

Preparation for purging includes inspecting the condition of the well, monitoring health and safety conditions, and calibrating and decontaminating sampling equipment. General procedures are presented below:

1. Ensure that the area around well head is clean and free of debris. If necessary, place a plastic drop cloth around well head to prevent sampling equipment from coming into contact with the ground surface.
2. Inspect condition of well (e.g., well in locked position, tightness of cap, measuring point well marked, disturbance of surface casing, straightness of well casing, condition of concrete pad). Indicate condition of well in sampling logbook.
3. Remove well cap. If the site health and safety plan (HSP) identifies organic compounds as potential contaminants of concern, screen well headspace and breathing-zone headspace (if specified in the HSP) for organic vapors using the appropriate field monitoring instrument (e.g., photoionization detector).
4. Decontaminate all equipment (as specified in the FSP, QAPP, or in accordance with SOP GW-01) before use in each well. Wear nitrile gloves and/or other protective equipment as specified in the site-specific HSP during possible water-contact or equipment-contact activities. At a minimum, change gloves between each well or when it is possible for potential contaminants to be introduced into the well.
5. Measure water level using a decontaminated electronic water level meter as described in SOP GW-02 when the water level in the well has equilibrated.
6. Calibrate water quality meters for measuring field parameters as appropriate. Record equipment calibration and maintenance on calibration forms. Decontaminate meters between wells by rinsing with distilled or deionized water. Manage rinsate water used for these measurements in the same manner as purge water, as defined in the work plan or FSP.

7. If collecting water quality field parameters, do so as described in the work plan or FSP. Methods used may include retrieval of groundwater using a disposable bailer, deployment of a thoroughly decontaminated meter into the monitoring well to measure groundwater directly, etc.

## Passive Sampler Deployment

This section describes initial deployment of all materials and consumables including reusable features such as well caps, suspension tethers, weights, identification tags, and connecting rings. When completing deployment of a new sampler in a well that already contains the appropriate reusable features, field personnel should start at step 3.

1. Identify spool containing weighted tether measured for the monitoring well you are sampling. Tethers should arrive from the supplier cut to the appropriate length with weights and attachment rings connected at the correct depth.

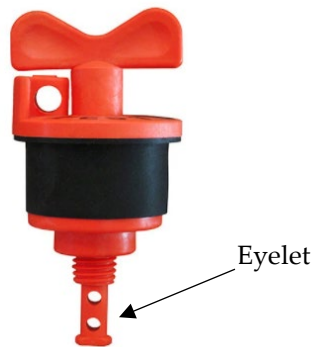


Photo from Envirodesign Products

2. Confirm that monitoring well plug is the correct type: plugs should have an eyelet at the base where the tether can be connected while still allowing the plug to completely close and lock (See photo). If needed, replace monitoring well plug.
3. Remove passive sampler from packaging. If required, fill passive sampler with deionized water using a funnel that has been decontaminated in accordance with the work plan or FSP. Always wear nitrile gloves when handling the passive sampler and/or anything that has had contact with groundwater.
4. Connect passive sampler to the metal rings using zip-ties in accordance with manufacturer's instructions. Confirm that sampler is securely attached to the tether and that tether is properly secured to the monitoring well plug.
5. Gently lower passive sampler and tether into the monitoring well. Secure well plug and close monitoring well cover.

## Passive Sample Collection

This section details collection of groundwater sample from passive sampler. This is completed after the initial deployment following a the time interval described in the work plan, FSP, or applicable state and/or federal regulations and also in accordance with the manufacturer's instructions.

1. While wearing nitrile gloves, secure a clean plastic drop cloth around the monitoring well if it has not been completed already to prevent sampling equipment from coming into contact with the ground surface.
2. Label all sample bottles with the information required by the laboratory and in accordance with the QAPP, work plan, and/or laboratory requirements.
3. Lift passive sampler out of the well by gently pulling the suspension tether. If using a spool or other tool to reel up the tether, confirm that the spool has been decontaminated prior to use as specified in the FSP, QAPP, or in accordance with SOP GW-01. Keep the passive sampler upright during removal to avoid loss of water contained within the sampler.
4. While wearing nitrile gloves, hold sampler and remove from suspension tether by cutting the zip ties. A clean straw provided by the manufacturer should be used to pierce the membrane of the sampler. Be careful to point the straw away from all personnel during this process to avoid contact with potentially contaminated water.
5. Use the straw to fill sample bottles. If there is water remaining in the sampler after all bottles are filled, containerize and dispose of the water in accordance with the project work plan.
6. Once emptied, dispose of sampler, gloves, and other disposable materials in accordance with the project work plan.

## **STANDARD OPERATING PROCEDURE (SOP) SD-01**

### **DECONTAMINATION OF SEDIMENT SAMPLING EQUIPMENT**

#### **SCOPE AND APPLICATION**

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either inorganic or organic materials. To prevent potential cross contamination of samples, all reusable sediment sampling and processing equipment is decontaminated before each use. At the sample collection site, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All sediment sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the site-specific health and safety plan (HSP).

Sampling equipment (e.g., van Veen, Ekman, Ponar, core tubes) may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment used for both analyte groups should follow the order of a detergent wash, site water rinse, organic solvent rinses, and final site water rinse. Sample processing equipment (e.g., bowls, spoons) has a final rinse with distilled/deionized water rinse instead of site water. If the surface of stainless steel equipment appears to be rusting (possibly due to prolonged contact with organic-rich sediment), it should undergo an acid rinse and a site-water rinse at the end of each sampling day to minimize corrosion.

#### **EQUIPMENT AND REAGENTS REQUIRED**

Equipment required for decontamination includes the following:

- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or site water
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial detergent
- Pesticide-grade acetone and hexane (consult the project-specific field sampling plan [FSP], as the solvents may vary by EPA region or state)
- 10 percent (v/v) nitric acid (reagent grade) for inorganic contaminants
- Baking soda
- Long-handled, hard-bristle brushes
- Extension arm for cleaning core liners
- Plastic sheeting, garbage bags, and aluminum foil
- Core liner caps or plastic wrap and rubber bands
- Personal protective equipment as specified in the health and safety plan.

## PROCEDURES

### Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., < 1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not an analyte in the samples. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is sometimes slightly more effective than other solvents, its use is discouraged due to potential toxicity to sampling personnel.

The specific procedures for decontaminating sediment sampling equipment and sediment compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove visible sediment. Perform this step onsite for all equipment, including core liners that will not be used again until the next day of sampling. After removing visible solids, set aside sampling equipment that does not need to be used again that day; this equipment should be thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double rinse the equipment with tap or site water and set right-side-up on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate<sup>1</sup> the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse equipment with tap or site water and set right-side-up on a stable surface to drain thoroughly.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). Hold core liners over the waste container and turn them slowly so the stream of solvent contacts the entire surface. Turn the sample apparatus (e.g., grab sampler) on its side and open it to wash it most effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.

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<sup>1</sup> Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.

7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container (which may need to be equipped with a funnel). If necessary, widen the opening of the squirt bottle to allow enough solvent to run through the core liners without evaporating. (Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the equipment was not thoroughly rinsed with acetone or that the acetone that was purchased was not free of water.) When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the acetone and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.
8. Do a final rinse with site water for the sampling equipment (i.e., van Veen, Ekman, Ponar, core tubes) and with distilled/deionized water for processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

If the sample collection or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.

10. Rinse or wipe with a wetted paper towel all stainless-steel equipment at the end of each sampling day with 10 percent (v/v) normal nitric acid solution. Follow with a freshwater rinse (site water is okay as long as it is not brackish or salt water).
11. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles and disposed of at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

## **Decontamination Procedures for Metals and Conventional Parameters Only**

The specific procedures for decontaminating sediment sampling equipment and sediment processing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove the visible sediment. Perform this step onsite for all equipment, including core liners that will not be used again until the next day of sampling. Set aside pieces that do not need to be used again that day; these pieces should be and thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1–2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. For the polycarbonate core liners, use a round brush attached to an extension arm to reach the entire inside of the liners, scrubbing with a back-and-forth motion. Be sure to clean the outside of core liners, bowls, and other pieces that may be covered with sediment.
4. Double-rinse the equipment with tap or site water and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If the surface of stainless steel equipment appears to be rusting (this will occur during prolonged use in anoxic marine sediments), passivate<sup>2</sup> the surface as follows (if no rust is present, skip to next step). Rinse with a 10 percent (v/v) nitric acid solution using a squirt bottle, or wipe all surfaces using a saturated paper towel. Areas showing rust may require some rubbing with the paper towel. If using a squirt bottle, let the excess acid drain into the waste container (which may need to be equipped with a funnel). Double-rinse sampling equipment with tap or site water and set right-side-up on a stable surface to drain. Double-rinse processing equipment with distilled/deionized water and allow to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area). Seal the polycarbonate core liners at both ends with either core caps or cellophane plastic and rubber bands. Close the jaws of the Ekman and Ponar grab samplers and wrap in aluminum foil.

If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.

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<sup>2</sup> Passivation is the process of making a material less reactive relative to another material. For example, before sediment is placed in a stainless-steel container, the container can be passivated by rinsing it with a dilute solution of nitric acid and deionized water.



7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

## **STANDARD OPERATING PROCEDURE (SOP) SD-02**

### **PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SEDIMENTS**

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#### **SCOPE AND APPLICATION**

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates, equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material; for the purposes herein, all types of reference materials are referred to as standard reference material, or SRM) for sediment sampling efforts. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples are an equipment rinsate blank, a field duplicate, and trip blanks if samples are to be analyzed for volatile organic compounds (VOCs). Definitions of all potential quality control samples are described below.

As part of the quality assurance/quality control (QA/QC) program, all field quality control samples will be sent to the laboratories "blind." To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers with preservatives that are required to complete the field quality control sample for the applicable analyte list shall be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should be recorded only in the field logbook. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent such an occurrence, regular samples should be selected and marked on the chain-of-custody/sampling analysis request (COC/SAR) form or the laboratory should be instructed to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Field quality control samples will be prepared at least once per sampling event, and certain types will be prepared more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of

field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality control sample per 20 is indicated and 28 samples are collected, 2 quality control samples will be prepared. Field quality control samples for sediment sampling activities should be prepared consistent with the requirements discussed below and at the frequency indicated unless different frequency requirements are listed in the FSP and QAPP.

The following table lists the quality control sample types and suggested frequencies for sediment sampling programs. Because sediment quality control sampling may require assessment of site cross-contamination, additional blanks may be required. A detailed explanation of each quality control sample type with the required preparation follows.

Table 1. Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		
		Location	Method	Frequency <sup>a</sup>
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples.
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter wipe	FW	Sampling site	Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be wiped over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1 per 20 thereafter.
Filter paper blank	FB	Sampling site	Clean, unused Whatman filter papers (organic analysis) and Ghost Wipes (metals/mercury analysis) will be sent to the analytical laboratory	Minimum of one for each lot number of filter papers used.
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type.
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment.

Quality Control Sample Name	Abbreviation	Preparation		
		Location	Method	Frequency <sup>a</sup>
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler.
Environmental blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples.
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode.

<sup>a</sup> Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

## FIELD DUPLICATE SAMPLES

Field duplicate (or split) samples are collected to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Field duplicates will be prepared by collecting two aliquots for the sample and submitting them for analysis as separate samples. Field duplicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of field duplicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

## FIELD REPLICATE SAMPLES

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Field replicates will be prepared by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Field replicates will be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The actual number of field replicate samples collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

## **MATRIX SPIKE/MATRIX SPIKE DUPLICATES**

The matrix spike/matrix spike duplicate (MS/MSD) analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated sediment stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The actual number of extra bottles collected during a sampling event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

## **EQUIPMENT RINSATE BLANKS**

Equipment rinsate blanks will be used to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Equipment rinsate blanks will be prepared by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, and then transferring the water to the appropriate sample containers and adding any necessary preservatives. Equipment rinsate blanks will be prepared for all inorganic, organic, and conventional analytes at least once per sampling event per the type of sampling equipment used. The actual number of equipment rinsate blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

## **FILTER WIPES**

Filter wipe samples will be used to help identify possible contamination from the sampling environment or from the decontaminated sediment sampling equipment (e.g., sediment grab sampler, stainless-steel bowls and spoons, shovel, trowel).

Filter wipe samples will be prepared by grasping a piece of clean, ashless filter wipe/paper with decontaminated tongs and/or tweezers and wiping down all surfaces of dry, decontaminated equipment that comes into contact with the sediment sample (e.g., stainless-steel spoon, inside of sediment grab sampler). Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter wipes/papers will be from the same lot used to prepare the filter paper blanks (see below), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be used for each equipment type, solid matrix type, and analysis type. For example, if two pieces of equipment were used for sediment sampling (trowel and stainless-steel spoon) and both metals and

organic compounds were being analyzed, then a total of four filter wipes/papers would be sent to the analytical laboratory.

Tongs and/or tweezers will be used to handle the filter wipe/paper, and all sediment sample-exposed surfaces will be thoroughly wiped down using one piece of filter wipe/paper (per equipment type and for each analysis). The filter wipe sample will then be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory. The filter wipe/paper box will be stored in a clean glass container and must NOT be stored in a plastic bag. In moist environments, the filters should be wrapped thoroughly in aluminum foil to protect them from moisture.

Filter wipe samples will be prepared for all inorganic and organic analytes at least once per sampling event per the type of sampling equipment used. The actual number of filter wipe samples prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter wipe sample collection may vary by EPA region or state).

## **FILTER PAPER BLANKS**

Whenever a filter wipe sample is prepared in the field, a filter paper blank will also be prepared in the field to evaluate potential background concentrations present in the filter paper used for the equipment filter wipe sample.

Filter paper blanks will be prepared by using tongs and/or tweezers to remove the clean ashless filter paper from its box. Whatman filter papers will be used for organic analysis and Ghost Wipes will be used for metals/mercury analysis. The filter papers will be from the same lot used to prepare the filter wipe samples (see above), and the filter lot number will be clearly noted in the field logbook. One filter wipe/paper will be sent to the analytical laboratory for each type of analysis to be performed (i.e., inorganic or organic analytes). The filter paper blank will be placed into a labeled certified pre-cleaned sample jar provided by the analytical laboratory.

Filter paper blanks will be collected at a minimum frequency of one for each filter lot number. The actual number of filter paper blanks prepared during an event will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of filter paper blank collection may vary by EPA region or state).

## **BOTTLE BLANKS**

The bottle blank is an unopened sample bottle. Bottle blanks are submitted along with sediment samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, one bottle

blank per lot of prepared bottles will be submitted for analysis. If more than one type of bottle will be used in the sampling (e.g., high-density polyethylene or glass), then a bottle blank should be submitted for each type of bottle and preservative. The actual number of bottle blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC/SAR form), and send the empty bottle to the laboratory with the field samples.

## **TRIP BLANKS**

Trip blanks will be used to help identify whether contaminants may have been introduced during the shipment of the sediment samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two 40-mL VOC vials and tightly closing the lids. Each vial will be inverted and tapped lightly to ensure no air bubbles exist.

The trip blanks will be transported unopened to and from the field in the cooler with the VOC samples. A trip blank is labeled and placed inside the cooler that contains newly collected VOC samples and it remains in the cooler at all times. A trip blank must accompany samples at all times in the field. One trip blank (consisting of a pair of VOC vials) will be sent with each cooler of samples shipped to the testing laboratory for VOC analysis.

## **TEMPERATURE BLANKS**

Temperature blanks will be used by the laboratory to verify the temperature of the samples upon receipt at the testing laboratory. Temperature blanks will be prepared at the testing laboratory by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank shall be included with each sample cooler shipped to the testing laboratory.

## **ENVIRONMENTAL BLANKS**

The environmental (field) blank is prepared in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If unpreserved bottles are to be used, then the appropriate preservative (i.e., for metals samples use a 10 percent nitric acid solution to bring sample pH

to 2 or less) must be added, as may be required. Environmental blanks should be collected at a minimum frequency of 1 in 20 samples. The actual number of environmental blanks analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water, and then seal it. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

## **REFERENCE MATERIALS**

SRMs are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. The SRMs have undergone multi-laboratory analyses using a standard method that provides certified concentrations. When available for a specific analyte, SRMs provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several SRMs may be required to cover all analytical parameters. For all analytes where available, one SRM will be analyzed at a frequency of one per 50 samples. The actual number of SRMs analyzed during a project will be determined on a case-by-case basis by the project QA/QC coordinator (consult the project-specific FSP and QAPP, as the requirements on frequency of SRM analysis may vary by EPA region or state).



## **STANDARD OPERATING PROCEDURE (SOP) SD-04**

### **SURFACE SEDIMENT SAMPLING**

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#### **SCOPE AND APPLICATION**

This SOP defines and standardizes the methods for collecting surface sediment samples from freshwater or marine environments. Surface sediments are defined as those from 0 to at most 10 cm below the sediment-water interface. The actual definition of surface sediments is typically program-specific and depends on the purpose of the study and the regulatory criteria (if any) to which the data will be compared.

This SOP utilizes and augments the procedures outlined in USEPA (1997) and ASTM (2003) guidelines. A goal of this SOP is to ensure that the highest quality, most representative data are collected, and that these data are comparable to data collected by different programs that follow the USEPA (1997) guidelines.

#### **SUMMARY OF METHOD**

Sediment samples for chemical and toxicity analysis are collected using a surface sediment sampling device (e.g., grab sampler) or hand implements (i.e., spoons, scoops, shovels, or trowels). If a sample meets acceptability guidelines, overlying water is carefully siphoned off the surface in a grab sampler, and the sediment is described in the field logbook. Depending upon the type of analysis to be performed, sediment samples for chemical analysis may be collected directly from an undisturbed surface (e.g., volatile organic compounds and sulfides), or may be homogenized using decontaminated, stainless-steel containers and utensils prior to being placed in sample jars. Sediment from several sampler casts or exposed sediment locations may also be composited and homogenized prior to being placed in sample jars.

#### **SUPPLIES AND EQUIPMENT**

A generalized supply and equipment list is provided below. Additional equipment may be required depending on project requirements.

- Sampling device
  - Grab sampler or box corer (see examples below in procedures for “Sediment Sample Collection”)

- Stainless-steel spoon, scoop, shovel, or trowel
- Field equipment
  - Siphoning hose
  - Stainless-steel bowls or containers
  - Stainless-steel spoons, spatulas, and/or mixer
  - Stainless-steel ruler
  - Project-specific decontamination supplies (e.g., Alconox™ detergent, 0.1 N nitric acid, methanol, hexane, distilled/deionized water)
  - Personal protective equipment for field team (e.g., rain gear, safety goggles, hard hats, nitrile gloves)
  - First aid kit
  - Cell phone
  - Camera
  - Sample containers
  - Ziploc® bags
  - Bubble wrap
  - Sample jar labels
  - Clear tape
  - Permanent markers
  - Indelible black-ink pens
  - Pencils
  - Coolers
  - Ice
- Documentation
  - Waterproof field logbook
  - Field sampling plan
  - Health and safety plan
  - Correction forms
  - Request for change forms
  - Waterproof sample description forms.

## PROCEDURES

### Sediment Sample Collection with a Grab Sampler

Use a sampler that obtains a quantifiable volume of sediment with minimal disturbance of the surrounding sediments to collect sediment for chemical and biological analyses. The sampler should be composed of a material such as stainless steel or aluminum, or have a noncontaminating coating such as Teflon™. Samplers capable of providing high-quality sediment samples include grab-type samplers (e.g., van Veen, Ekman, Smith-McIntyre, Young grab, Power Grab and modified-ponar grab) and box cores (Soutar, mini-Soutar, Gray-O'Hara, spade core). Some programs require a sampler that collects from a specific area (e.g., 0.1 m<sup>2</sup>). Most sampling devices are typically a standard size; however, some non-standard sizes are available to meet the requirements of specific programs. Grab samplers, especially van Veen grab and Ekman grab, are the most commonly used samplers to collect surface sediment. Power Grab samplers are often used for programs requiring collection of sediment deeper than 10 cm (4 in.) or in areas with debris.

Depending on grab weight and water depth, use a hydraulic winch system to deploy the heavier samplers at a rate not exceeding 1 m/second. As the grab nears the bottom, decrease the descent speed to about 0.3 m/second to minimize the bow wake and disturbance of the surface sediment associated with sampler descent. Once the sampler hits the bottom, close the jaws slowly and bring the sampler to the deck of the vessel at a rate not exceeding 1 m/second to minimize any washing and disturbance of the sediment within the sampler. At the moment the sampler hits the bottom, record the time, water depth, and location of sample acquisition in the field logbook.

Retrieve and secure the sampler, and carefully siphon off any overlying water. Inspect the sample to determine acceptability using the criteria detailed in USEPA (1997), except when noted in the project-specific field sampling plan. These criteria include but are not limited to the following:

- There is minimal or no excessive water leakage from the jaws of the sampler
- There is no excessive turbidity in the water overlying the sample
- The sampler is not over-penetrated
- The sediment surface appears to be intact with minimal disturbance
- There is no anthropogenic (i.e., man-made) debris in the sampler
- The program-specified penetration depths are attained.

If the sample meets acceptability criteria, record the sample collection location using a global positioning system (GPS) and enter observations onto a sample collection form or the field logbook. Depending on programmatic goals, remove the sampling interval specified in the field sampling plan. Use a decontaminated stainless-steel ruler to measure the sample

collection depth (0 to 10 cm) within the sampler. To prevent possible cross-contamination, do not use sediments touching the margins of the sampler.

Take a photograph of the sediment in the grab sampler and in the stainless-steel bowl in the field. Verify that the station number or sample ID, time, and date are shown in the photograph.

Typically, sediment from a minimum of three separate casts of the sampler is composited at each station (see project-specific field sampling plan). Once the sample has been characterized, subsample the sediment for chemical and biological analyses using a decontaminated stainless-steel spoon.

## **Sediment Sample Collection with Hand Implements**

Obtain a quantifiable volume of sediment with minimal disturbance of the surrounding sediments to collect sediment for chemical and biological analyses. Hand implements (e.g., spoons, scoops, shovels, or trowels) must be composed of stainless steel.

Use GPS to locate the sampling site and approach the location carefully to avoid disturbing the area of sediment to be sampled. Prior to sample collection, describe and characterize the undisturbed surface sediment in the field logbook. If necessary, expose the sediment surface by clearing an approximately 1-ft<sup>2</sup> area at the sampling site of any rocks greater than approximately 5 in. Remove any anthropogenic (i.e., man-made) debris and organic material on the sediment surface. Note any material removed from the sampling site in the field logbook.

Using a decontaminated, stainless-steel hand implement (i.e., spoon, scoop, shovel, or trowel), excavate the sediment to 10 cm. Place the sediment in a decontaminated stainless-steel bowl and use a decontaminated stainless-steel ruler to confirm that the correct sampling interval has been collected. If the full sample collection interval (i.e., 10 cm) has not been reached, collect additional sediment, place it in the stainless-steel bowl, and reconfirm the sampling interval. Continue this process until the full sample collection interval (0 to 10 cm) has been reached.

Take a photograph of the excavated hole from where the sediment sample was removed. Verify that the station number or sample ID, time, and date are shown in the photograph.

## **Sample Processing**

Complete all sample collection forms, labels, custody seals, and chain-of-custody forms, and record sample information in the field logbook.

Collect samples for volatile compounds (either organics or sulfides) using a decontaminated stainless-steel spoon while sediment is still in the grab sampler or, if the sample is collected using a hand implement, in the stainless-steel bowl. Sediments for volatile analysis are not homogenized. Tightly pack the volatile organics sample jar with sediment (to eliminate obvious air pockets) and fill it so that no headspace remains in the jar. Alternatively, if there is

adequate water in the sediment, fill the container to overflowing so that a convex meniscus forms at the top, and then carefully place the cap on the jar. Once sealed, the jar should contain no air bubbles.

Place the remaining sediment in the grab sampler in a precleaned, stainless-steel bowl; sediment collected using hand implements are already in a stainless-steel bowl. Once a sufficient amount of sediment has been collected, mix the sediment using a decontaminated stainless-steel spoon until it is of uniform color and texture throughout.

If required for analysis, collect samples for grain-size tests before any large rocks are removed from the homogenized sediment. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized sediment volume, note it on the sediment field collection form or in the field logbook, and then discard the rocks.

Dispense the sediment into precleaned sample jars for the various chemical or biological analyses. For toxicity testing, fill sample jars to the top with sediment to minimize available headspace. This procedure will minimize any oxidation reactions within the sediment. For chemical analysis, sample containers may be frozen for storage. Leave enough headspace to allow for sediment expansion.

After dispensing the sediment, place the containers into coolers with ice and either ship them directly to the analytical laboratories or transport them to a storage facility.

## REFERENCES

ASTM. 2003. *Standard Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler*. ASTM Standards on Disc, Volume 11.05.

USEPA. 1997. Recommended protocols for sampling marine sediment, water column, and tissue in Puget Sound. Prepared for Puget Sound Estuary Program, U.S. Environmental Protection Agency, Seattle, WA, and Puget Sound Water Quality Action Team, Olympia, WA. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

## **STANDARD OPERATING PROCEDURE (SOP) SD-06**

### **HOLLOW-STEM AUGER DRILLING/SEDIMENT SAMPLING**

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#### **SCOPE AND APPLICATION**

Soil/sediment cores are collected to evaluate sediment at depths that greatly exceed those achieved by grab or other surface samplers. The purpose of this standard operating procedure (SOP) is to define and standardize procedures for core collection using split-spoon and Shelby tube samplers advanced through hollow-stem auger borings, following American Society for Testing and Materials (ASTM) Method D1586 and Method D1587, respectively. The use of Shelby tube samplers or split-spoon samplers is specified in the Slip 4 Pre-Design Sampling and Analysis Plan Addendum (Integral 2006). Shelby tubes will be used to recover relatively undisturbed soil samples suitable for laboratory tests of engineering properties such as strength, compressibility, permeability, and density.

#### **REQUIRED EQUIPMENT**

- Sampling and Analysis Plan (SAP).
- Health and Safety Plan (HSP).
- Site logbook and boring log.
- Indelible black-ink pens and markers.
- Camera.
- Hollow-stem auger drill rig.
- Driller and helper.
- Split-spoon samplers (typically 2-in. diameter; a larger 3-in. diameter, 2-ft-length split-spoon may be used to obtain more material from each depth interval).
- Shelby tube samplers conforming to thin-walled tube specifications outlined in ASTM D1587 with a 2- to 5-in. O.D and 5 to 10 times the diameter in length. Wax and end caps will also be provided for proper field sealing.
- Photoionization detector (PID).
- Plastic sheeting.
- 55-gallon drums (if required).
- Insulated cooler(s), chain-of-custody seals, Ziploc® bags.
- Sample labels and appropriate documentation.

- Assorted geology supplies (e.g., hand lens, grain-size card, scales, etc.).
- Decontamination equipment (SOP-10).

## Typical Procedures

1. Ensure underground utilities in vicinity of each boring location have been marked prior to mobilizing drill rig to site.
2. Conduct daily site activity/health and safety briefing.
3. Calibrate field instrumentation, if applicable.
4. Record necessary data in field logbook.
5. Obtain photograph(s) of site before drilling.
6. Place plastic sheeting and/or drums at drilling location to collect cuttings (if necessary).
7. Move equipment and supplies to drilling location.
8. Set up decontamination and sampling stations.

## Split-Spoon Sampling

1. Obtain surface soil samples, if required.
2. Drill to first sampling depth, as described in the SAP.
3. Place decontaminated split-spoon sampler on center rods.
4. Drive split-spoon sampler, as described in ASTM Method D-1586. Drive sampler to 18 inch or to refusal (no progress for 50 blows). Record blow counts on boring log form. Retrieve sampler.
5. Screen sampler with PID (if required).
6. Describe soil in accordance with ASTM D2488 on the boring log form.
7. Composite soil sample, as necessary. If volatile organic compound (VOC) samples are to be collected, collect VOC sample prior to describing soil.
8. Continue drilling at next sample location. Collect samples as outlined above.
9. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples.
10. Decontaminate sampling equipment in accordance with the site-specific SAP.
11. Document activities in site logbook.
12. Backfill or grout borehole, as required.

13. Move to next location.

### **Shelby Tube Sampling**

1. Obtain surface soil samples, if required.
2. Drill to first sampling depth, as described in the SAP.
3. Place decontaminated Shelby tube sampler on center rods.
4. Drive Shelby tube sampler, as described in ASTM Method D1587. Retrieve the sampling tube and remove the disturbed material from the top of the tube. In addition, remove 1 inch of soil from the base of the tube. Place an impervious disk at both ends of the tube seal with a wax plug prior to shipment to the laboratory.
5. If Shelby tubes are to be extruded in the field for composite sampling, the driller will use a hydraulic extruder to obtain the sample. The core is then described in accordance with ASTM Method D2488 on the boring log form. Samples will then be composited, as necessary, for analysis.
6. Screen sampler with PID (if required).
7. Label and manage sample containers in accordance with the site-specific SAP section for shipping and handling of samples. The sample tube should be packed in Styrofoam™ plugs or other cushioning material to prevent disturbance of the sample.
8. Continue drilling to next sample location. Collect samples as outlined above.
9. Decontaminate sampling equipment in accordance with the site-specific SAP section.
10. Document activities in site logbook.
11. Backfill or grout borehole, as required.
12. Move to next location.

### **Reference**

Integral. 2006. Lower Duwamish Waterway Slip 4 Early Action Area: Sampling and Analysis Plan for Boundary Definition Addendum: Pre-Design Investigation Sampling. Prepared for Seattle City Light and King County, Seattle, WA. Integral Consulting Inc., Mercer Island, WA.



## **STANDARD OPERATING PROCEDURE (SOP) SD-08**

### **SEDIMENT CORE COLLECTION USING A VIBRACORER**

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#### **SCOPE AND APPLICATION**

This SOP describes the procedure for collecting and processing sediment core samples using a vibracore system, which collects continuous and relatively undisturbed sediment cores. This method of sediment coring is performed from a boat and uses high-frequency low-amplitude vibration to break down the frictional resistance of the sediment and allow the core tube to penetrate into the sediment with minimal distortion. It is best used for sampling coarse, consolidated sediment and very cohesive sediment, where static weight (e.g., piston-type or conventional gravity corers) will not produce adequate penetration into the sediment. In addition, the vibracorer offers a high rate of production, superior retention of shallow samples, and a greater sample volume compared to conventional drilling equipment.

Vibracorers generally consist of a metal corer barrel (usually a 4-in.-outside-diameter, aluminum core barrel) with a location-dedicated polycarbonate or Lexan<sup>®</sup>-lined core tube, and a vibrator mechanism attached to the top of the barrel. The vibration is created either by an electric motor, a hydraulic system, or a pneumatic piston attached to the top of the barrel. Therefore, a generator or air compressor is needed on board to power the corer. The pneumatic piston does not have the same function as a piston in a piston corer. Because vibracorers generally do not have a piston in the corer, some compaction and/or bypass will occur, and recovery will be less than 100 percent.

A continuous sediment sample is retained within the tubing with the aid of a stainless-steel core cutter/catcher or nosecone attached to the bottom of each aluminum tube.

It is always best to keep the core in a vertical position to prevent the top layers of sediment (i.e., the top 5 to 15 cm) from slumping. However, in many cases, it is not feasible to process the core in a vertical position because the tripod needs to be at least twice the height of corer, and sectioning and logging the sample would have to be performed from a ladder. For studies that specify sectioning the sample into coarse intervals (>20 cm), processing the core in a horizontal position will generally not significantly disrupt the stratigraphy. For studies that specify shorter intervals (<5–10 cm), processing the core in a horizontal position is likely to disrupt stratigraphy. In this case, the top layers of sediment that have high water content should be sectioned while the core is in a vertical position, and when the sediment becomes thicker, the corer can be laid horizontally.

## PROCEDURES

### Decontamination

To prevent potential cross-contamination of samples, all reusable sediment sampling equipment must be decontaminated prior to use at each station and between field replicates.

Before each station is sampled, decontaminate the inner surfaces of the corer or core tube liner and all stainless-steel sample compositing equipment. Prior to sampling, all core liners will be washed in sequence with a standard detergent (e.g., Alconox®), rinsed with site water, and then air-dried. During storage and transport, decontaminated core liners will be capped at both ends to prevent contamination. Details on correct decontamination procedures can be found in SOP SD-01, *Decontamination of Equipment—Sediment*. The project-specific field sampling plan (FSP) should also be consulted to determine any project-specific decontamination procedures. The personnel performing the decontamination procedures will wear protective clothing as specified in the site-specific health and safety plan.

All solvent rinsates (if used) will be collected into a bucket or tub and allowed to evaporate over the course of the day. Any rinsate that has not evaporated by the end of the sampling event will be containerized and disposed of in accordance with applicable regulations.

### Vibracorer Deployment and Retrieval

The following procedures are based on using the vibracorer aboard a boat equipped with a tripod or A-frame of sufficient height to allow recovery of the core (see project-specific FSP for information on target coring depth), and a power winch. On pontoon boats, the tripod is centered over a hole in the floor, whereas on other boats, the corer may be lowered over the side or stern. To obtain cores of high quality, the boat must be anchored with at least three anchors so the boat will not drift during the coring process.

1. Maneuver the sampling vessel to the targeted sampling location using the positioning procedures and minimum water depth restrictions.
2. Deploy 3- or 4-point anchor system to maintain position; record and monitor position throughout core acquisition.
3. Once on location, measure the water depth (depth to top of sediment) using the onboard depth sounder (fathometer) or lead line and record measured depth in the field logbook. If the water level is affected by tides, obtain tide level measurements and calculate tidal height in feet above mean low water. The date, time, weather, and water conditions (e.g., high wave activity, strong currents, turbidity, tidal flux) should also be recorded in the field logbook.

4. Assemble the decontaminated core tube, liner, core catcher, and cutter heads (or nose cone depending on the model of vibracorer used), using care to not contact decontaminated surfaces. Attach assembled vibracorer to winch cable. Note that several decontaminated catchers and cutterheads will be on hand, in case of loss. Core catchers and cutter heads can be decontaminated and reused for subsequent core collection.
5. Attach a tape measure to the vibracorer or mark the winch cable in 1/2-ft increments to measure penetration depth.
6. Inspect connections of winch cable and electrical or pneumatic lines to confirm they are secure.
7. Signal the winch operator to slowly raise the vibracorer into a vertical position and guide the vibracorer (with core liner, valve, core catcher, and cutterhead in place) overboard until it is clear of the vessel.
8. Using the winch, slowly lower the vibracorer through the water column at a speed of about 1 ft/s to avoid creating a bow wake or overturning of the vibracorer. Stop lowering the corer a few feet above the sediment and confirm that the boat has not drifted.
9. Continue lowering the vibracorer until the tip of the core is resting on the sediment or to the depth recorded by the fathometer, depending on the consistency of the sediment. Record the vibracorer depth as derived from the attached tape measure or marked winch cable. Measurements will serve as a basis for determining penetration depth.
10. Resume lowering the corer at about 1 ft/s. When the nosecone or core catcher contacts the sediment, turn on the vibracorer motor. The vibracorer is then allowed to slowly penetrate the sediments. Initially, light tension should be maintained on the cable to keep the corer from tipping over.
11. Lower the vibracorer to the target penetration depth as measured by the attached tape measure or marked winch cable. If the targeted penetration depth is met, proceed to the next step; if refusal is met, retrieve the vibracorer, perform gross decontamination (i.e., rinse with river water and brush off visible sediment from the outside of the core barrel) and re-attempt at new location offset at least 3–5 ft from original location.
12. When the target penetration depth is reached, or refusal occurs, turn off the vibracorer and record the time, penetration depth, angle of the cable relative to the boat, and any other observations.
13. Slowly withdraw the core from the bottom sediments at a constant rate, to keep it upright and not dislodge any sediment from within the core barrel, and raise it to the surface.

14. With the corer hanging in a vertical position, clean the vibracorer assembly by hosing down the equipment with site water prior to bringing the core onboard the sampling vessel. If the corer is not plugged, care should be taken not to direct water into the open end of the core barrel.
15. Slowly guide the core onboard the vessel; use care to avoid jostling that might disturb the integrity of the core. Care must be taken to keep the top end of core elevated to prevent sediment from “pouring” out. Use a sawhorse or equivalent to elevate the top of the core. As soon as the nosecone clears the water surface, the bottom of the corer may be plugged with a rubber stopper to prevent loss of sediment.
16. Before removing the core tube from the vibracorer, visually inspect the nosecone or core cutter/catcher to ensure that proper penetration has been attained and that there is no obvious loss of sediment from the tube. Record any presence of noticeable odors, the core penetration depth, and physical characteristics (e.g., color, texture, odor) of the sediment sample as observed at the ends of the tube in the field logbook or on the Field Sediment Core form. In addition, note any sheen in the water in the field logbook.
17. If the core will be processed horizontally, slowly lay the corer down. Unscrew the cutter head (or nosecone) and carefully remove the core catcher, while retaining as much sediment as possible.
18. While removing the core catcher (or nosecone), be ready to immediately seal the end of the core liner by placing clean aluminum foil and a plastic cap over the open end.
19. Carefully remove the core liner that contains the sample by lifting the lower end from the deck as needed to provide clearance. Affix core cap, wrap with tape, label core liner and end of core, remove valve from top of core liner, stand core upright, and place in a processing rack or tray to allow the sediment at the top of the core to settle. Avoid sudden movements to the core that would disrupt the sediment interface.
20. While waiting for sediment to settle, prepare the Field Sediment Core form. Identify any debris and note its depth in the core and what the debris is, if possible.
21. Once resuspended sediment has settled, measure the length of the recovered core, calculate percent recovery ( $100 \times \text{recovered length}/\text{penetration depth}$ ), and record in the logbook or on the Field Sediment Core form.
22. Check the core for acceptability. The following acceptability criteria should be satisfied:
  - The core tube is not overfilled with sample so that the sediment surface presses against the bottom of the vibracorer head.
  - Overlying water is present (indicates minimal leakage).
  - The overlying water is not excessively turbid (indicates minimal disturbance).

- The desired penetration depth (see project-specific FSP for required penetration depth) or refusal has been reached.

Depending on requirements of the project-specific FSP, a core may be rejected based on percent recovery. Commonly, a core is deemed unacceptable if recovery is less than 80 percent. If recovery is less than 80 percent, the core sample will be retained for possible processing, while additional sampling attempts are made to collect a core with greater than 80 percent recovery. If subsequent attempts result in recoveries of less than 80 percent, then the sample with the highest percent recovery may be used for analysis. The number of attempts to collect an acceptable sample will be specified in the project-specific FSP. If recovery is less than 80 percent, the core may be acceptable if the penetration depth is deeper than the target core length. In this case, the recovered length should be equal to the target length.

23. Once sufficient time has been allowed for the sediment to settle (i.e., no sediment is suspended in the overlying water), use a decontaminated saw to cut a drain-slit or a decontaminated drill bit to drill in the side of the core liner approximately 1 to 2 in. above the sediment–water interface; allow excess water to drain. Cut excess polycarbonate liner with decontaminated blade and use a siphon to decant off the overlying water. Ensure that the saw blade, drill bit, or siphon does not contact the sediments and that fine-grained suspended sediment is not removed.
24. Cut cores into manageable sections (3–4 ft) aboard the vessel immediately after their retrieval. Cap each section with aluminum foil and plastic caps, and seal with duct tape. Mark the core with permanent marker using a unique number or alphanumeric code identifying sampling location, core number, core section, and segment orientation (i.e., which end is up). Following sectioning, store the cores in an upright position onboard the vessel in a core box and have them transported periodically throughout each field day by small boat to a field processing area where they are to be stored upright under custody on ice or refrigerated at 4°C to await processing.
25. In preparation for next core, thoroughly rinse the interior of the core barrel until all loose sediment has been washed off. Repeat process at next sampling location. Continue coring until requirements are met.

In situations where there is significant surface water depth and/or water current that could cause the vibracorer setup to lean at an unacceptable angle, a buoyant frame or rigid frame configuration should be used.

With the buoyant frame, the vibracorer is maintained in proper vertical position by two guidelines held taut between a float package and a weight stand. The larger weight stand is provided with ballast boxes so that easy-to-find ballasting material such as lead bags or scrap metal can be used in the field. For deployment, the vibracorer is lowered with the weight

stand hanging on its guidelines from the vibrahead. The float package is hooked up to the guidelines when the vibrahead reaches the deck level.

After coring and pull-up, the system is retrieved in the reverse manner. In case of limited deck space or overhead clearance, or to further accelerate the procedure on the water, the weight stand can be left in as overboard cradle.

## **Sample Handling, Storage, and Processing**

Cores should be processed concurrently with core collection, and every effort should be made to ensure cores are processed within 24 hours of collection. Cores awaiting processing will be sealed tightly at both ends and stored upright in ice or in a refrigerator. If core collection outpaces processing such that significant delays in core processing appear likely, core collection will be suspended to allow the core processing to catch up.

As mentioned above, once coring has been completed at a given location, the cores will be transported in an upright position on ice to a designated field processing area, where they will be logged and processed. The field processing area will be equipped with a core-cutting table, core-processing tables, a decontamination area, and a storage area with appropriate refrigeration. Appropriate lighting will be installed in the field processing area so that consistent, high quality photographs can be taken of the opened cores. Care should be taken to create a field processing area that minimizes the potential for outside contamination.

Sample processing includes removing the sample from the liner, recording observations of sample characteristics, mixing subsamples, and distributing the sample to containers for shipping to the testing laboratory. Vibracore processing most often consists of the following steps:

1. Cut each core tube along the long axis using decontaminated hook blade. Rotate the tube 180° and cut again.
2. After each core is cut, move the entire core tube to an aluminum foil-covered table and open it so that it can be systematically logged, described, and photographed.

However, depending on the project-specific FSP, the core may be extruded from the liner and cut into the specified intervals as it emerges or the core liner may be cut into sections, sealed, and shipped intact to the testing laboratory.

### **Core Observations**

1. Verify that the length of the core, water depth, and all required position data have been recorded in the field logbook together with all pertinent observations and communications with the field team leader.
2. After each core is cut open, describe the sediment on a Field Sediment Core form in the field processing area notebook. When recording the information for each core, follow the guidelines below:

- Physical sediment description (i.e., sediment type [e.g., silt, sand], density/consistency, color) (see SOP SL-04, Field Classification of Soil, based on ASTM D 2488-00 [ASTM 2000])
  - Odor (e.g., hydrogen sulfide, petroleum, creosote)
  - Visual stratification, laminations, and lenses
  - Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
  - Moisture content
  - Vegetation
  - Approximate percentage of vegetation
  - Debris
  - Approximate percentage of debris
  - Presence of biological structures (e.g., detritus, shells, tubes, bioturbation, live or dead organisms, chironomids)
  - Approximate percentage of biological structures
  - Presence of a sheen
  - Other distinguishing characteristics or features.
3. Use other observations (e.g., obvious anthropogenic material, dramatic color changes) to define or help define sample intervals (check project-specific FSP for sample interval definition; depending upon the project-specific requirements the sample interval could be based on lithology or it could be set to a specific interval [e.g., 1 ft]).
  4. Determine the boundaries of lithologic units primarily by changes in the top two dominant grain sizes estimated visually (e.g., a change from a silty sand to a gravelly sand or to a sandy silt).
  5. Photograph the cores after they have been described and before any sediment is removed for processing. It is important for each core section to be photographed with adequate lighting from a standard measured distance from the core. Digital photographs may be used later in the production of digital core logs.

## Mixing and Sample Preparation

1. After the sample is characterized and the core observation logged on the Field Sediment Core form, remove the specified sample interval using a stainless-steel spatula or spoon (see project-specific FSP for correct sampling interval). Exercise care to not include sediment that is in direct contact with the core tube. With the approval of the field team leader, and using a decontaminated stainless-steel instrument, carefully remove unrepresentative material (e.g., large shells, stones). Exercise care not to touch the sediment during this process. Note any unrepresentative material removed from the sample in the field processing area notebook.
2. Remove subsamples for analysis of unstable constituents (e.g., volatile organic compounds, acid-volatile sulfides), and place them directly into sample containers without homogenization. Completely fill the sample container so that there is no headspace or entrapped bubbles.
3. Transfer the remainder of the sample interval to a decontaminated stainless-steel bowl for homogenization. If additional sediment volume is required to fill all sample bottles (see project-specific FSP) and multiple cores need to be collected at a given station, cover the compositing bowl covered with aluminum foil (dull side down) to prevent sample contamination (e.g., from precipitation, engine exhaust, splashing water) and place in a cool dark place until the next core from that location is processed.
4. After all the sediment is transferred to the compositing bowl, homogenize the contents of the bowl using stainless-steel spoons until the texture and color of the sediment appears to be uniform.
5. Distribute subsamples to the various containers specified in the project-specific FSP and preserve the samples as specified in the project-specific FSP. Briefly stir the sediment in the compositing bowl between each spoon transfer to the sample containers.
6. Subsequent intervals should be processed in the same way.

## Field Quality Control Samples

If additional volumes of sediment are required to perform all analyses including quality control analyses, an additional core may need to be collected from the same location and subsampled and homogenized accordingly. Details on collection of field quality control samples (e.g., field duplicates) will be specified in the project-specific FSP. Details on collection of field quality control samples and preparation of the certified reference materials can be found in SOP SD-02, *Preparation of Field Quality Control Samples—Sediment*, and SOP SD-03, *Preparation of Reference Materials—Sediment*. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality



control samples will be described in the project-specific FSP and quality assurance project plan.

## Field Measurements

A water depth measurement must be collected at every sampling location. Depending on the specific project objectives, it may be necessary to perform field measurements of the *in situ* environment. Possible field measurements include temperature and pH of the sediment at the sediment-water interface and concentration of dissolved oxygen, salinity, or conductivity in the overlying water. Details on collection of field measurements can be found in SOP SD-11, *Field Analyses for Sediment*. The specific field measurements, if any, will be specified in the project-specific FSP.

## Station Location Coordinates

Station locations for all field sampling will be determined using a differential global positioning system (DGPS) or by surveying. The accuracy to which the latitude and longitude of a station location is determined will be specified in the FSP. At a minimum, a DGPS capable of providing latitude and longitude coordinates with an accuracy of approximately 3 m is recommended. The DGPS consists of two satellite receivers linked to each other by a VHF telemetry radio system. The receiver will be on the sampling vessel. Details on collection of very accurate station coordinates can be found in SOP AP-06, *Navigation*.

## Sample Custody and Shipping

Sample custody will be maintained in accordance with procedures outlined in SOP AP-03, *Sample Custody*. All samples will be packaged and shipped with other samples in accordance with procedures outlined in SOP AP-01, *Sample Packaging and Shipping*.

## Troubleshooting

### Insufficient Sample

The corer may not collect enough sediment because of 1) inadequate penetration, 2) adequate penetration but poor recovery due to compaction, 3) adequate penetration but poor recovery as a result of bypass, or 4) adequate penetration but loss of sample during retrieval. Compaction and bypass are two different artifacts that are difficult to distinguish and quantify. Following is an approach to identifying the causes and remedies of insufficient sample length. Keep in mind that a combination of these causes may occur:

- **Inadequate Penetration**—Allow more vibration time at the refusal depth, or increase the vibrator frequency.

- **Poor Recovery Due to Compaction**—Compaction is the process of rearranging the sediment particles, so that less volume is occupied by pore water, which results in a shorter column of sediment in the corer than *in situ*. Compaction occurs only in clean coarse silt, sand, and gravel sediments that have a high hydraulic conductivity and are not terminally compacted *in situ*. Fine-grained cohesive sediment (i.e., low hydraulic conductivity) does not compact. The key feature of compaction is that all of the solids ahead of the nosecone are collected as the corer penetrates. So, although the calculated recovery is less than 10 percent, 100 percent of the sediment solids were recovered. Therefore, if the sample has poor recovery, is composed of clean coarse-grained materials, and there is no evidence of sediment falling out the bottom, then the sample is likely to have been compacted. Depending on the project-specific FSP, the specified sample intervals may be shortened proportional to recovery. Because compaction of the solids displaces pore water, minimal compaction is needed for cores that are intended for porewater studies, or cores that will be analyzed for substances that have low  $K_d$  values. Vibration in vibracorer is known to rearrange particles, which leads to compaction, so another type of corer may be appropriate if compaction is a problem.
- **Poor Recovery as a Result of Bypass**—Bypass is the process of pushing sediment out of the path of the nosecone/corer as it penetrates the sediment. This is caused by the friction of sediment inside the core liner making it difficult for more sediment to enter the tube. This is most pronounced in fine-grained sediments that have low hydraulic conductivity, or layers of hard and soft sediment, or long cores. The low hydraulic conductivity prevents porewater from being displaced, so compaction cannot occur. Fine-grained sediments in this context are those in which particles cannot be felt between the thumb and forefinger of an ungloved hand. These are generally “sticky” or cohesive sediments. Therefore, if a sample has poor recovery, is fine grained and cohesive, and there is no evidence of sediment falling out the bottom, then some of the sediment column has likely been bypassed.
- **Poor Recovery Because of Loss of Sample during Retrieval**—This is often diagnosed by observing some of the core falling out the bottom as the corer approaches the water surface during retrieval, or a core liner that is empty near the bottom. Sample slipping out the bottom of the corer can be caused by a loss of suction or noncohesive sediment that does not stick to the liner wall. Depending on the specific design of the vibracorer, there are several places at which suction can be lost. These may include the valve seat, the valve assembly, the nose piece, and couplings between the barrel and extensions. To prevent loss of suction, Teflon® plumber’s tape should be used on all the threaded connections, and the valve assembly should be clean. For coarse-grained sediment (e.g., clean coarse sand and gravel, and shells) that is non-cohesive and falls out the bottom of the corer, it is sometimes possible to penetrate to a lower layer that is finer grained and will effectively plug the bottom of the core. As mentioned above, core catchers may be used to retain sediment in a vibracorer, although they should not be

used if the surface sediments have high water contents and are to be sectioned at less than about 2 inch intervals.

Because recovery can be an important indicator of corer performance, sediment characteristics, and sample quality, some simple tests can be performed as a diagnostic tool. Penetration of the corer can be measured by putting Velcro® tape on the outside of the corer. Velcro® tape can also be used on the inside of the liner during testing to see how far up inside the liner the sediment interface moves, how much sample slips out the bottom, and how much compaction or bypass occurs.

### Notes

1. For long cores that require more than one piece of liner, squarely cut the ends of both pieces with a plastic pipe cutter, butt the ends of the two pieces of liner squarely together and tape them securely so no leaks occur. Do not use too many layers of tape or the liner will not fit into the barrel. Do not use duct tape for this process. Use a high quality tape (i.e., 3M 3750) and dry the tubes before applying.
2. Sometimes tripods are not tall enough to lift the corer so that the barrel will clear the top edge of the liner when removing the liner. To remove the liner in this case, upon unscrewing the cutter head (or nose piece), lower the cutter head (or nose piece) and liner into a pail that has a rope securely tied to the handle. While the corer is raised by the winch, lower the pail through the hole in the deck and into the water (if necessary) until the top edge of the liner clears the bottom edge of the barrel. Then lift it back onto the deck.
3. If the vibrocorer does not penetrate significantly or if the cable is let out too quickly, the vibrocorer will contact the bottom, tip over, and fall sideways. When this happens, the line will initially go slack, then quickly snap to the side and take up the slack. In this case, reject the core and begin again.
4. A good measure of whether the vibrocorer collected the sediment-water interface is to inspect the interface for a thin layer (about 1 mm) of olive green benthic or detrital algae. Also, if the core liner is rotated back and forth gently, the top centimeter will appear to have a gelatinous response.
5. It is sometimes impossible to collect an intact interface because gas bubbles are commonly released from sediment when the corer contacts the sediment. The released gas bubbles entrain surface sediment and cause the overlying water to become turbid. If this is the case, gas bubbles in the sediment can likely be observed through the liner wall.

## REFERENCES

ASTM. 2000. Standard practice for description and identification of soils (visual-manual procedure). ASTM Standard Method No. D 2488-00. In: ASTM Book of Standards, Volume 04.08. American Society for Testing and Materials, West Conshohocken, PA.

## **STANDARD OPERATING PROCEDURE (SOP) SD-12**

### **LOGGING OF SEDIMENT CORES**

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#### **SCOPE AND APPLICATION**

The following procedures for completing the Field Sediment Core Form establish the minimum information that must be recorded in the field to adequately document sediment coring activities. The field sediment core form must be filled out completely. Depending upon project specific requirements, some of the items listed below can be recorded in the observing scientist's field logbook and/or on the Station Core Log. All field forms must be filled out completely.

All of the information addressed in this standard operating procedure (SOP) should be included in the observing scientist's field documentation. Additionally, standards presented may need to be supplemented with additional technical descriptions or field test results (see project specific field sampling plan [FSP]).

#### **ACTIVITIES OF THE OBSERVING SCIENTIST DURING CORING**

1. Record the name of the coring contractor and personnel performing the coring (lead person and any support staff)
2. Record the type and make of the coring equipment being used
3. Note the weather or any special external conditions that influence the coring
4. Be certain that the coring contractor is informed about the nature of the daily records that the contractor will keep
5. Check the coring contractor's daily records to verify their accuracy
6. Note date and time of all activities associated with the coring
7. Make certain that the coring contractor follows all required procedures
8. The observing scientist's daily record shall include, but may not be limited to, the following items:
  - Date and depth of core
  - Depth of start and finish of each sampled interval
  - Depth and size of any casing or core tubing used
  - Time required to advance the core
  - Loss of water, mud, or air during sample retrieval

- Depth of overlying water
- Simplified description of strata
- Total sample recovery (in inches or centimeters)
- Details of delays and breakdowns.

The observing scientist should also record the coring start and finish dates and times. For consecutive sheets, provide, at a minimum, the project number, the station number, and the sheet number. This list excludes any special items that may be required for contractual record purposes or for special tests (see project-specific FSP).

### **Data on Field Sediment Core Form**

**Core Type/Method:** Provide the sampler type (e.g., GC = gravity corer, PC = piston corer, DRCV = drive rod check valve corer, VC = vibracorer, BC = box corer).

**Sample Number/Tag Number:** Provide the sample number. The sample numbering scheme should be established before sampling begins. Consult the project-specific FSP for the sample numbering scheme. The depth of the sample is the depth to the top of the recovered sample to the nearest centimeter. Samples should be obtained from the entire recovered core (depending upon the sampling intervals specified in the project-specific FSP). The tag number(s) and respective sample number(s) of the sample container(s) should also be recorded in the field logbook.

**Photograph Number:** Provide the number of the film roll and the photograph number.

**Odor:** Provide information on presence of any odor associated with the sediment. Document each interval in the core at which an odor is present. Describe the odor in the *Sediment Description* section of the field sediment core form.

**Sheen:** Provide information on presence of any sheen associated with the sediment. Document each interval in the core at which sheen is present. Also note if sheen is present on the water surface during coring activities.

**Blank Columns:** Two blank columns are provided on the field sediment core form. These columns can be used for site-specific information, usually related to the contaminants of concern (e.g., sheen, air quality measurements).

**Water Breaks:** Record the location of any observed breaks in the sediment core.

**Depth Scale:** Enter the depth of the core below sediment surface. Match the sediment descriptions with the depth scale.

**Unified Symbol:** If a geologist is providing the sediment descriptions of the core, then the unified symbol code (USC) for different sediment types (e.g., silt, clay, sand) should be placed in this column. The USC name should be identical to the ASTM D-2488-84 Group Name with the appropriate modifiers.

Table SD-12(1) presents the USC classification system. The USC system is an engineering properties system that uses grain size to classify soils, it can however also be used by a geologist to characterize the sediment in a core.

**Table SD-12(1). USC Classification System**

Major Divisions			Group Symbol	Group Name	
<b>Coarse-grained soils</b>  More than 50 percent retained by No. 200 sieve	<b>Gravel</b> More than 50 percent of coarse fraction retained on No. 4 sieve	Clean Gravel	GW	Well-graded gravel, fine to coarse gravel	
		Gravel with fines	GP	Poorly graded gravel	
		<b>Sand</b> More than 50 percent of coarse fraction passes No. 4 sieve	Clean Sand	GM	Silty gravel
			Sand with fines	GC	Clayey gravel
	<b>Silt and clay</b> Liquid limit < 50	Inorganic	Clean Sand	SW	Well-graded sand, fine to coarse sand
			Sand with fines	SP	Poorly graded sand
		Organic	Sand with fines	SM	Silty sand
			Sand with fines	SC	Clayey sand
<b>Fine-grained soils</b>  More than 50 percent passes No. 200 sieve	<b>Silt and clay</b> Liquid limit < 50	Inorganic	ML	Silt	
			CL	Clay	
	<b>Silt and clay</b> Liquid limit ≥ 50	Inorganic	OL	Organic silt, organic clay	
			MH	Silt of high plasticity, elastic silt	
		Organic	CH	Clay of high plasticity, fat clay	
			OH	Organic clay, organic silt	
Highly organic soils			PT	Peat	

**Note:** Field classification is based on visual examination of soil in general accordance with ASTM D-2488-84.

Soil classification using laboratory tests is based on ASTM D-2487-83.

Descriptions of soil density or consistency are based on interpretation of blow count data, visual appearance of soils, and/or test data.

Liquid limit is the water content of soil-water where the consistency changed from plastic to liquid.

**Sediment Description:** The sediment description should follow the format described in SOP SD-13, *Field Classification of Sediment*. Information on sediment should include sediment type, percent moisture with depth through the core, color, and presence or absence of vegetation or biota. The surface conditions within the core (i.e., overlying water is present, undisturbed sediment/water interface, presence of any vegetation or biota) should also be described. The project-specific FSP should be consulted for any special descriptive items that may be required.

**Comments:** Include all pertinent observations. Coring observations might include coring chatter, core-bounce (hard object hit by corer during penetration), sudden differences in

coring speed, damaged coring equipment, and malfunctioning equipment. Information provided by the coring contractor should be attributed to the coring contractor.

## **Data on Station Core Log**

**Cast Number:** Record the number of coring attempts at each station.

**Start/End Time:** The time should be recorded during coring to determine coring speed. Time should be recorded in 24- hour mode (e.g., 3:00 p.m. = 1500 hours).

**Water Depth:** Record the overlying water depth at the station. Note: The overlying water depth can change between coring attempts and therefore must be measured prior to each attempt.

**Core Penetration Depth:** Record the depth that the core was pushed into the sediment. Note: If this information is not readily apparent, it can be obtained from the coring contractor.

**Retrieved Core Length:** While the sediment core is vertical, record the length of the retrieved core.

**Overlying Water:** Record whether or not there is water on top of the sediment core once the core has been retrieved. This is necessary to determine measurable sediment/water interface.

**Coordinates:** Record the latitude and longitude (or geographic) of the station location. The datum used to collect the station location coordinates (e.g., WGS84) must also be recorded in the field notes.



## STANDARD OPERATING PROCEDURE (SOP) SD-13

### FIELD CLASSIFICATION OF SEDIMENT

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#### SCOPE AND APPLICATION

This SOP presents the field classification of sediments to be used by Integral field staff. Sediment descriptions should be precise and comprehensive without being verbose. Assumptions and personal comments should not be included in the sediment descriptions. These descriptions will be used to interpret environmental conditions and other potential properties, rather than the exact mineralogy or tectonic environment.

Sediment descriptions should be recorded in either the observing scientist's field logbook, or if subsurface sediment is collected, then the sediment description column of the Field Sediment Core Form should be completed for each core collected. If no difference between consecutive sediment samples exists, subsequent descriptions can be noted as "same as above," or minor changes such as "increasing sand" or "becomes dark brown" can be added.

After the overlying water is removed, characterize the sediment. Sediment characteristics that are often recorded in the field logbook or the Field Sediment Core Form if subsurface sediment is collected, include:

- Sediment type (e.g., silt, sand)
- Texture (e.g., fine grain, coarse, poorly sorted sand)
- Color
- Presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia)
- Approximate percentage of moisture
- Presence of biological structures (e.g., chironomids, tubes, macrophytes) and the approximate percentage of these structures
- Presence of organic debris (e.g., twigs, leaves) and the approximate percentage of debris
- Presence of shells and the approximate percentage of shells
- Stratification, if any
- Presence of a sheen
- Odor (e.g., hydrogen sulfide, oil, creosote).

In addition, the project-specific field sampling plan should be reviewed to determine if there are any project-specific reporting requirements.

In general, the similarities of consecutive sediment samples should be noted. Examples of surface sediment descriptions are provided in Table SD-13(1). The minimum elements of the sediment descriptions are discussed below. The format of sediment descriptions for each sample should be consistent throughout the logbook.

**Table SD-13(1). Example of Surface Sediment Descriptions**

Station No.	Grab No.	Example Descriptions
TC01	1	SILT, mottled dark gray (10YR 4/1) with thin layer < 1 cm of very pale brown (10YR 7/4) on surface. Occasional roots, some twigs, and leaves on surface. Slight reducing odor. Sheen on overlying water in grab.
TC02	1	Sandy SILT, fine sand, dark gray (10YR 4/1) throughout grab, with 10 percent medium to coarse sand, trace woody debris. Chironomid on surface.
TC02	2	Same description as first grab at Station TC02.
TC02	3	Same description as first grab at Station TC02, but no sand (SILT only) and color is very dark gray (10YR 3/1) with no chironomid present.

## Definition of Sediment Types

Fine-grained sediments are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-84. If these tests are used, the results should be included in the sediment description. Sediments with high plasticity can be emphasized by describing them as “silty CLAY with high plasticity.” Plasticity is an important descriptor because a sediment can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.

Coarse-grained sediments are classified as predominantly sand. The gradation of a coarse grained sediment is included in the specific sediment name (i.e., fine to medium SAND with silt). Estimating the percentage of size ranges following the group name is encouraged for mixtures of silty sand and sand. If applicable, use the modifiers “poorly graded” or “well graded” when describing the sand component of the sediment.

## Color

The basic color of a sediment, such as brown or gray, must be provided in the description. The color term can be modified by adjectives such as light, dark, or very dark. Especially

note streaking or mottling. The color chart designations provided in either the *Globe Soil Color Book* or the Munsell color guide can be used.

## **Moisture Content**

The degree of moisture present in the sediment should be defined as moist, wet, or very wet. The percent moisture content should be estimated.

## **Other Components**

Other components, such as organic debris and shell fragments, should be preceded by the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). The word “occasional” can be applied to random particles of a larger size than the general sediment matrix (i.e., occasional stone, large piece of wood).

## **Additional Descriptions**

Features such as sloped surface in the grab, root holes, odor, and sheen should be noted if they are observed. Anything unusual should be noted. Additional sediment descriptions may be made at the discretion of the project manager or as the field conditions warrant.

## **STANDARD OPERATING PROCEDURE (SOP) SD-20**

### **SEDIMENT POREWATER SAMPLING**

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The sampling and analysis of pore water obtained from sediment can provide valuable information on chemical changes occurring in the sediment, including equilibrium reactions between minerals and water, and on the transport of toxic chemicals in the sediment–water interface and overlying water. These processes influence the availability of nutrients and toxic chemicals to biota (Azcue and Rosa 1995).

This SOP describes Integral procedures for collecting porewater samples from sediment cores using centrifugation. Sediment cores are collected using a stainless steel box corer equipped with an acrylic liner (SOP SD-17). The box corer can contain a sediment block as large as 50 cm x 50 cm x 75 cm. After cores are retrieved and liners with sediment are removed from the corer, the cores are transported to a field processing area and stored under anoxic conditions in a nitrogen glove bag, according to SOP AP-10, prior to the sectioning of cores and the extraction of pore water. These procedures are not repeated here.

### **SCOPE AND APPLICATION**

This SOP addresses the sectioning of cores and the subsequent extraction and processing of sediment pore water from the cores under anoxic conditions (e.g., high-purity nitrogen atmosphere) to prevent the oxidation of samples during sample processing.

### **SUMMARY OF METHOD**

This procedure requires adequate space for a “wet lab” to be set up. The wet lab must be equipped with a glove bag, benchtops, air conditioning and/or heater, an exhaust hood system to extract sulfide fumes and nitrogen gas, electrical outlets, and space to install a gimbaled table (needed only if operating on a boat), refrigerated centrifuge, and a small refrigerator.

Before sediment core processing begins, the sediment core is visually inspected and photographed. It is then placed in the glove bag, and layers of sediment are sliced according to project-specific depth requirements. The sediment slices are placed in marked bowls, composited, if required, and homogenized. The homogenized sediment is then placed into appropriate jars and prepared for centrifugation. After centrifugation, porewater is extracted and filtered for chemical analysis or *in situ* measurements.

Porewater chemistry may include *in situ* measurements of sulfide, ammonia, pH, salinity, conductivity, temperature, and oxidation-reduction potential (ORP). Temperature, pH, salinity, conductivity, and ORP are measured while in the field.

Sediment pore water sample sizes for sulfide analysis can be minimized from 25 mL to 1 mL and preserved into 9 mL of sodium hydroxide and zinc acetate (i.e., sample is diluted by 1/10). This approach significantly reduces the total porewater volume requirements and reduces the amount of time performing the analysis in the field by sending the samples to be processed at the analytical laboratory.

Short sample holding times for sulfide (i.e., 7 days) should be taken into consideration when planning sampling logistics. Remaining sediment in the jars is reserved for standard chemical analysis at a laboratory.

## SUPPLIES AND EQUIPMENT

- Large glove bag with clamps; inflated work space (37 in. x 37 in. x 25 in.; Cole Parmer Cat. No. EW-04408-38)
- Ultrahigh purity nitrogen gas (99.999%) tank (cylinder size 300) (Airgas)
- Two-stage regulator for nitrogen tank (Airgas part Y12-N415D[CGA])
- Cylinder brackets and clamps (Airgas) depending on number of cylinders and location of use:
  - Y99-241500 bracket to secure a single cylinder directly to a wall
  - Y99-242200 bracket to secure a single cylinder to a workbench or lab table
  - Y99-G200 two-cylinder wall-mount bracket, made of heavy-gauge steel and finished in epoxy
  - Y99-G275 two-cylinder wall/floor stand(If several tanks are needed (i.e., more than six), Airgas can supply a sturdy rack which can be craned to the vessel.)
- Adjustable wrench for installing/removing regulator from cylinder
- High pressure hose to be used between the regulator and a control valve if gas tank is placed outside the lab
- Flow control valve
- 3/8-in. inside diameter Tygon™ tubing for purging nitrogen gas into glove bag
- 3/8-in. adaptor/reducer for attaching tubing to two-stage regulator
- Electric tape

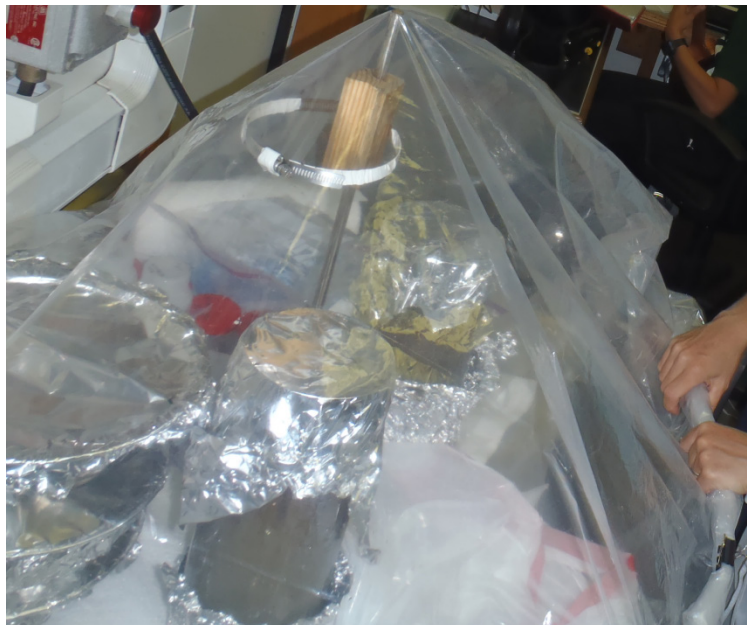
- Kimwipe™ paper towels
- Stainless-steel spatulas
- Stainless-steel bowls
- Cotton gloves
- Nitrile disposable gloves
- Sample jars
- Labels
- Waterproof ink pens (Sharpies™)
- 60 cc Luer-Lok™ tip syringe
- Tygon™ tubing for removing pore water
- Trash bags
- Plastic ruler
- Core rings of the same sediment core diameter and material for each project specific sampling depths (e.g., 0–1 cm, 1–3 cm, 3–5 cm, 5–10 cm)
- Plunger for extruding sediment cores (plunger should fit snugly inside core)
- Metal stand with chain clamp to secure sediment core upright inside bag
- Deionized water squirt bottle for cleaning spatulas
- Data sheet
- Cooler with ice or small refrigerator
- Hach DR/890 colorimeter
- Ammonia nitrogen reagent set for 10-mL samples (Hach 26680-00)
- Sulfide reagent set for 25-mL samples (Hach 22445-00)
- Hach MP-6p combination pH, conductivity, temperature, salinity and ORP meter
- Pipette, 1.0–10 mL
- Pipette tips, 1.0–10.0 mL, non-sterile
- Pipette, 0.1–1.0 mL
- Pipette tips, 0.1–1.0 mL
- Centrifuge and centrifugation materials
  - Eppendorf refrigerated multi-purpose centrifuge; 4 x 250 mL capacity, 115 VAC (Cole Parmer Cat. No. EW-02570-20)

- Swing bucket rotor with rectangular buckets 4 x 250 mL
- 180–250 mL tube adapter (Eppendorf Cat. No. NC-00222SP)
- Gimbaled table (Continental Shelf Associates, custom made; for construction plans, refer to Young and Cline [1983]) (needed only if used on a boat).
- Digital camera
- Low-cost, cast-iron tripod-base supports > 0.43-in. diameter x 24-in. length (Fisher Scientific Cat. No. 14-675CQ)
- Syringe filters, PVDF; Pore size: 0.45  $\mu\text{m}$  (Fisherbrand Cat. No. 09-719-003).

## PROCEDURES

### Staging and Sediment Core Processing

1. Prepare a clean benchtop area large enough to accommodate a glove bag and supplies.
2. Place the glove bag on the benchtop and connect it to the nitrogen tank tubing.
3. Place all materials and supplies needed inside the glove bag for sediment core processing.



4. Remove core from core rack, wash the outside from any excess mud and take a photograph of the entire core. If unusual features, take close-up photos and log photos.

5. Carefully remove the bottom plug of the core and insert plunger while holding the bottom sediment with gloved hand. Use the plunger to slowly push entire contents of corer towards the top. Transport sediment cores inside the glove bag (SOP AP-10) and flush the glove bag with nitrogen three times before the start of processing.
6. Carefully remove top cap of corer.
7. If there is fluid on top of the sediment, decant it and dispose of it with a turkey baster or tubing.



8. Place a pre-cut core ring of the same diameter and thickness at the top of the core and extrude the sediment from the core by sliding the core liner downward.
9. Insert a stainless-steel plate between the core and the ring to section the core (example below is shown outside of the glove bag for clarity).





10. Section the cores as per project specifications (e.g., 0–1 cm, 1–3 cm, 3–5 cm), targeting obvious color or texture changes for divisions.
11. If easily seen through the glove bag, record any general observations of the core intervals, including such information as the vertical changes in sediment characteristics (e.g., texture, density, and moisture) and distribution of visible contamination, color of the sediments, sediment texture, presence of debris (wood chips, wood fibers, human artifacts), presence of oily sheen, and visible fauna or biological structures.

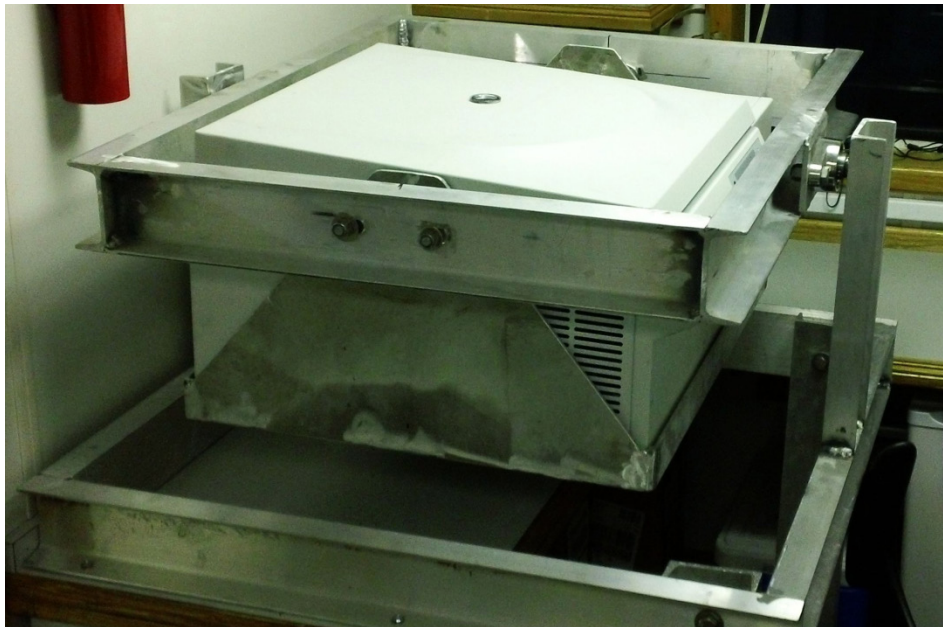
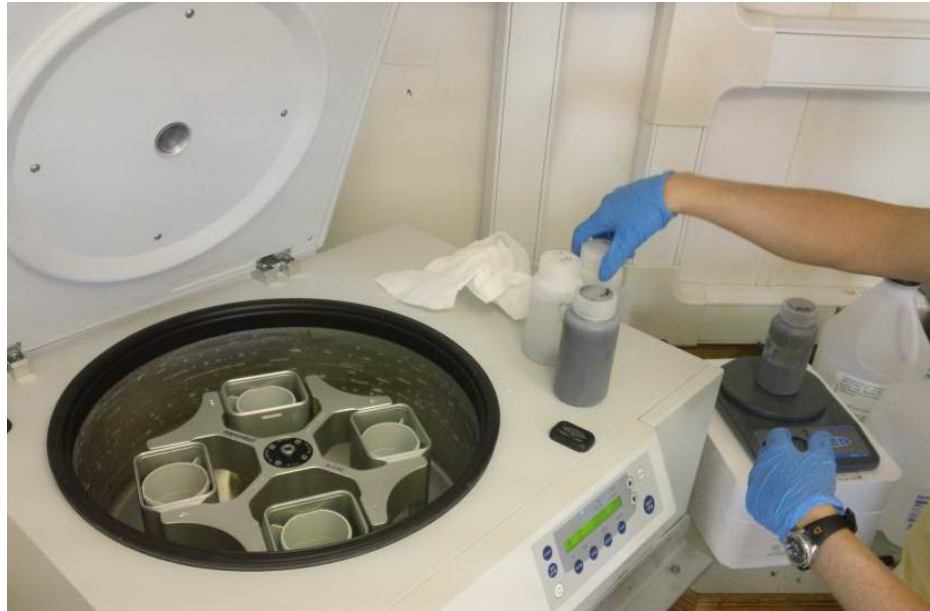
## Sediment Processing

1. Scoop sediment contents from inside the ring, avoiding any smeared upper-layer sediment in contact with the inner walls of the ring. The first top section of the core does not have the smearing problem and can be sampled in its entirety.
2. If the compositing of several cores is necessary, place each sediment section from each core in a marked bowl covered with aluminum foil and homogenize each depth prior to filling centrifuge jars.
3. Place sediment into wide-mouth HDPE jars **rated for centrifugation** and seal them under nitrogen.

## Porewater Extraction

The following procedure is for porewater extraction using commercially available centrifuges. If a centrifuge is not available for extracting pore water from sediment samples, alternate methods of pore water extraction using fritted stones (e.g., the Rhizon system) can be used following the procedures described in Dickens et al. (2007) and Seeberg-Elverfeldt et al. (2005).

1. Weigh the sample jars to determine weight needed for counter balanced jars for the centrifuge. Fill counter balanced jars with similar site sediments and make final adjustments by adding or removing water from the jar.
2. Place each sample jar opposite from its counter balanced jar into a refrigerated centrifuge. If on a boat, the centrifuge must be mounted on a gimbaled table to compensate for boat movement.
3. Ensure that the temperature of the centrifuge chamber is a constant 4°C during operation, unless otherwise specified. Note any deviations of the temperature on the data sheet.



4. Follow the standard operating instructions for use of the centrifuge supplied by the manufacturer. Prepare for a sample run by lowering the rotor assembly into the centrifuge chamber. Set the temperature as per user manual instructions.
5. Centrifuge at 3,000 rpm for 30 minutes.
6. After the sediments are centrifuged, place the jars back into the nitrogen glove bag.

## Porewater Sample Preparation

1. Working inside the glove bag, remove the overlying pore water with a small syringe equipped with tubing at the tip and filter the pore water through a 0.45- $\mu\text{m}$  PVDF Fisherbrand syringe filter back into a sample jar for chemical analysis or *in situ* measurements on board the vessel. A 20 mL volume of pore water is generally required to complete all analyses. However, volume requirements may vary depending on type of analyses needed.
2. Label sample jar and record sample on data forms.
3. Store sediment jars and pore water jars in a cooler with ice or in a refrigerator at 4°C.

## In Situ Pore Water Measurements

1. Measure sulfide and ammonia in the field using a Hach DR/890 colorimeter.
2. Follow the salicylate method in Hach (2009) for measuring total ammonia concentrations. Un-ionized ammonia nitrogen (UAN), the form considered the most toxic to shallow aquatic fauna (USEPA 1989), may be calculated based on the total ammonia concentration and the corresponding salinity, pH, and temperature of the sample (Bowers and Bidwell 1978).
3. Follow the methylene blue method in Hach (2009) for measuring sulfide adapted from APHA Standard Method 4500-S<sup>2-</sup> (APHA 1989). Un-ionized H<sub>2</sub>S, the form considered the most toxic to shallow aquatic fauna, may be calculated based on the sulfide (S<sup>2-</sup>) concentration, pH of the sample, and pK' provided in Standard Method 4500-S<sup>2-</sup> (APHA 1989).

Sediment porewater sample sizes for sulfide analysis can be minimized from 25 mL to 1 mL and preserved into 9 mL of sodium hydroxide and zinc acetate (i.e., sample is diluted by 1/10). This approach significantly reduces the total porewater volume requirements and reduces the amount of time performing the analysis in the field by sending the samples to be processed at the analytical laboratory.

4. Measure temperature, pH, conductivity, salinity, and ORP while in the field using a combination pH and ORP portable meter (e.g., Hach MP-6p). ORP is measured in millivolts and requires the addition of an offset voltage of 210 mV to convert the reading to Eh.

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## **STANDARD OPERATING PROCEDURE (SOP) SL-01**

### **DECONTAMINATION OF SOIL SAMPLING EQUIPMENT**

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#### **SCOPE AND APPLICATION**

This SOP describes procedures for decontaminating sampling and processing equipment contaminated by either organic or inorganic materials. To prevent potential cross contamination of samples, all reusable soil sampling and processing equipment is decontaminated before each use. At the sample collection site, a decontamination area is established in a clean location that is upwind of actual sampling locations, if possible. All soil sampling and processing equipment is cleaned in this location. Decontaminated equipment is stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel must follow all relevant procedures and wear protective clothing as stipulated in the site-specific health and safety plan (HSP).

Sampling equipment may be used to collect samples that will 1) undergo a full-suite analysis (organics, metals, and conventional parameters) or 2) be analyzed for metals and conventional parameters only. Decontamination of sampling equipment (e.g., hand auger, split-spoon sampler) used for both analyte groups should follow the order of a detergent wash, site water rinse, organic solvent rinses, and final site water rinse. Sample processing equipment (e.g., bowls, spoons) is rinsed with distilled/deionized water instead of with site water.

#### **EQUIPMENT AND REAGENTS REQUIRED**

Equipment required for decontamination includes the following:

- Steam cleaner and collection basin (if required)
- 55-gal, Department of Transportation (DOT)-approved drums (if required)
- Polyethylene or polypropylene tub (to collect solvent rinsate)
- Plastic bucket(s) (e.g., 5-gal bucket)
- Tap water or site water (i.e., potable water)
- Carboy, distilled/deionized water (analyte-free; received from testing laboratory or other reliable source)
- Properly labeled squirt bottles

- Funnels
- Alconox®, Liquinox®, or equivalent industrial nonphosphate detergent
- Pesticide-grade ethanol and hexane (consult project-specific field sampling plan [FSP], as the solvents may vary by U.S. Environmental Protection Agency [EPA] region or state)
- 10 percent diluted nitric acid or hydrochloric acid (reagent grade) for inorganic contaminants (if required; see project-specific FSP)
- Baking soda (if required)
- Long handled, hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Personal protective equipment as specified in the HSP.

## PROCEDURES

### Decontamination Procedures for Full Suite Analysis (Organic, Metal, or Conventional Parameters)

Two organic solvents are used in this procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. This allows the second solvent to fully contact the surface of the sampler. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). If ethanol is used, make sure that the denaturing agent in the alcohol is not one of the sample analytes. The second organic solvent is hydrophobic (e.g., hexane) and is intended to dissolve any organic chemicals that are on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific FSP). Integral uses ethanol and hexane as preferred solvents for equipment decontamination. If specified in the project-specific FSP, isopropanol or acetone can be substituted for ethanol, and methanol can be substituted for hexane in the decontamination sequence. The choice of solvents is also dependent on the kind of material from which the equipment is made (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol is slightly more effective than other solvents, its use is discouraged because of its potential toxicity to sampling personnel. Always follow the procedures listed in the site-specific HSP when decontaminating sampling equipment (e.g., always stand upwind when using volatile solvents, wear appropriate gloves and safety glasses or goggles). Containerize all decontamination fluids for proper disposal, following procedures listed in the FSP.

The specific procedures for decontaminating soil sampling equipment and soil compositing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove visible soil. This step should be performed onsite for all equipment. After removing visible solids, set aside sampling equipment that does not need to be used again that day and see that it is thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles, using a back-and-forth motion. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double rinse the equipment with tap or site water and set upright on a stable surface to drain. The more completely the equipment drains, the less solvent will be needed in the next step. Do not allow any surface that will come in contact with the sample to touch any contaminated surface. If acid and solvent rinses are not required by the FSP, skip to step 8.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle using a 10 percent acid solution. Double-rinse equipment with tap or site water and set right-side-up on a stable surface to drain. If solvent rinses are not required by the FSP, skip to step 8.
6. Carefully rinse the equipment with ethanol from a squirt bottle, and let the excess solvent drain into a waste container (which may need to be equipped with a funnel). These solvents act primarily as a drying agent by scavenging water from the equipment surface and carrying it away, but they also work as a solvent for some organic contamination. Hand-augers must be held over the waste container and turned slowly so the stream of solvent contacts the entire surface. The sample apparatus may be turned on its side, and if applicable, opened to be washed more effectively. Set the equipment in a clean location and allow it to air dry. Use only enough solvent to scavenge all of the water and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container. Allow equipment to drain as much as possible. Ideally, the equipment will be dry. The more thoroughly it drains, the less solvent will be needed in the next step.
7. Carefully rinse the drained or air-dried equipment with hexane from a squirt bottle, and let the excess solvent drain into the waste container, which may need to be equipped with a funnel. Hexane acts as the primary solvent of organic chemicals. Ethanol is soluble in hexane but water is not. If water beading occurs, it means that the

equipment was not thoroughly rinsed with ethanol or that the ethanol that was purchased was not free of water. When the equipment has been rinsed with hexane, set it in a clean location and allow the hexane to evaporate before using the equipment for sampling. Use only enough solvent to scavenge all of the ethanol and flow off the surface of the equipment (i.e., establish sheet flow) into the waste container.

8. Do a final rinse with site water for the sampling equipment (i.e., hand-auger) and distilled/deionized water for the processing equipment (i.e., stainless-steel bowls and spoons). Equipment does not need to be dried before use.
9. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

If the sample collection or processing equipment is precleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the project-specific FSP lists special handling procedures.

10. After decontaminating all of the sampling equipment, dispose of the disposable gloves and used foil per the procedures listed in the project-specific FSP. When not in use, keep the waste solvent container closed and store in a secure area. The waste should be transferred to empty solvent bottles for disposal at a licensed facility per the procedures listed in the project-specific FSP. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda or containerized and disposed of per the procedures listed in the project-specific FSP.

## **Decontamination Procedures for Metals and Conventional Parameters Only**

The specific procedures for decontaminating soil sampling equipment and soil processing equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove the visible soil. Perform this step onsite for all equipment. Set aside any pieces that do not need to be used again that day see that they are thoroughly cleaned in the field laboratory at the end of the day.
2. Pour a small amount of concentrated laboratory detergent into a bucket (i.e., about 1 to 2 tablespoons per 5-gal bucket) and fill it halfway with tap or site water. If the detergent is in crystal form, make sure all crystals are completely dissolved prior to use.



3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles. Be sure to clean the outside of the compositing bowls and other pieces that may be covered with soil.
4. Double-rinse the equipment with tap or site water (if an acid rinse is required) or with distilled/deionized water (if no acid rinse) and set right-side-up on a stable surface to drain. Do not allow any surface that will come in contact with the sample to touch any contaminated surface.
5. If an acid rinse is required by the FSP, rinse the equipment using a squirt bottle containing a 10 percent acid solution. Double-rinse equipment with distilled/deionized water and set right-side-up on a stable surface to drain.
6. If the decontaminated sampling equipment is not to be used immediately, wrap small stainless-steel items in aluminum foil (dull side facing the cleaned area).

If the sample collecting or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag until ready for use, unless the project-specific FSP lists special handling procedures.

7. After decontaminating all of the sampling equipment, place the disposable gloves and used foil in garbage bags for disposal in a solid waste landfill. When not in use, keep the waste acid container closed and store in a secure area. The acid waste should be neutralized with baking soda and disposed of per the procedures listed in the project-specific FSP.

## **Decontamination Procedures for Drill Rig or Test Pit Sampling Equipment**

1. Decontaminate sampling equipment before use, between samples and stations, and upon completion of sampling operations.
2. Equipment used during drilling/test pit operations should be decontaminated in the Exclusion Zone prior to transport to the Support Zone (refer to site-specific HSP).
3. If the steam-cleaning location is in an area outside of the Exclusion Zone, remove loose soil on the drill rig, augers, drill pipe, and rods, and other large equipment at the drill site, then move the equipment directly to the steam-cleaning decontamination area for more thorough cleaning.
4. To decontaminate a drill rig or backhoe, pressure wash with a steam cleaner using potable water rinse upon mobilization, between drilling locations, and upon demobilization. Cleaning water can generally be allowed to drain directly on the ground near the station (refer to the FSP).

5. To decontaminate auger, drill rods, and other down-hole tools, pressure wash with a steam cleaner and potable water rinse upon mobilization, between drilling locations, and upon demobilization. All decontamination fluids are to be containerized for proper disposal.
6. To decontaminate split-spoon and hand-auger samplers, follow the decontamination procedures listed above (the selected decontamination procedures is dependent upon analyte list provided in the project-specific FSP). To the extent possible, allow to air dry prior to sampling. If the split-spoon is not used immediately, wrap it in aluminum foil. All decontamination fluids are to be containerized for proper disposal.

## **STANDARD OPERATING PROCEDURE (SOP) SL-02**

### **PREPARATION OF FIELD QUALITY CONTROL SAMPLES FOR SOILS**

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#### **SCOPE AND APPLICATION**

This SOP describes the purpose, preparation, and collection frequency of field duplicate samples, field replicate samples, matrix spike/matrix spike duplicates (MS/MSDs), equipment rinsate blanks, bottle blanks, trip blanks, temperature blanks, environmental blanks, and reference materials (i.e., a standard reference material, a certified reference material, or other reference material) for soil samples. Not all of the field quality control samples discussed in this SOP may be required for a given project. The specific field quality control samples will be identified in the project-specific field sampling plan (FSP) and quality assurance project plan (QAPP). For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if volatile organic compounds (VOCs) are to be analyzed. Definitions of all potential quality control samples are described below.

As part of the quality assurance and quality control (QA/QC) program, all field quality control samples will be sent to the laboratories blind. To accomplish this, field quality control samples will be prepared and labeled in the same manner as regular samples, with each quality control sample being assigned a unique sample number that is consistent with the numbering for regular samples. All of the containers that are required to complete the field quality control sample for the applicable analyte list must be labeled with the same sample number. The sample ID for field quality control samples should allow data management and data validation staff to identify them as such and should only be recorded in the field logbook or field sampling forms. Under no circumstances should the laboratory be allowed to use reference materials, rinsate blanks, or trip blanks for laboratory quality control analysis (i.e., duplicates, matrix spike, and matrix spike duplicates). To prevent this from happening, select and mark regular samples on the chain-of-custody/sampling analysis request (COC) form or instruct the laboratory to contact the project QA/QC coordinator to select appropriate samples for each sample group.

Prepare field quality control samples at least once per sampling event, and prepare certain types more often at predetermined frequencies. If the number of samples taken does not equal an integer multiple of the intervals specified in this SOP, the number of field quality control samples is specified by the next higher multiple. For example, if a frequency of 1 quality

control sample per 20 is indicated and 28 samples are collected, prepare 2 quality control samples. The method of preparation and frequency of field quality control samples required for soil sampling activities are described below. These protocols must be followed, unless different frequency requirements are listed in the FSP and QAPP.

For most projects, Integral's recommended field quality control samples include an equipment rinsate blank, a field duplicate, and trip blanks if VOCs are to be analyzed. The following table lists the possible quality control sample types and suggested frequencies for soil sampling programs (not all types of quality control samples will always be collected; see project-specific FSP and QAPP for actual quality control samples that need to be collected for a particular sampling event). A detailed explanation of each type of quality control sample with the required preparation follows.

#### Field Quality Control Sample Requirements

Quality Control Sample Name	Abbreviation	Preparation		Frequency <sup>a</sup>
		Location	Method	
Duplicate	DUP	Sampling site	Additional natural sample	One per 20 samples. May not be applicable if REP is being collected.
Replicate	REP	Sampling site	Additional natural sample	One replicate per 20 samples. May not be applicable if DUP is being collected.
Matrix spike/matrix spike duplicate	MS/MSD	Sampling site	Additional sample bottles filled for laboratory quality control requirements	One per 20 samples
Equipment rinsate blank	ER	Sampling site	Deionized water collected after pouring through and over decontaminated equipment	Minimum of one per sampling event per type of sampling equipment used and then 1:20 thereafter
Bottle blank	BB	Field	Unopened bottle	One per sample episode or one per bottle type
Trip blank	TB	Laboratory	Deionized water with preservative	One pair per each VOC sample cooler shipment
Temperature blank	TMB	Laboratory	Deionized water	One per sample cooler
Environmental (transfer) blank	EB	Field	Bottle filled at sample site with deionized water	One per 20 samples
Standard reference material	SRM	Field laboratory or sampling site	SRM ampules or other containers for each analyte group	One set per 50 samples or one per episode

<sup>a</sup> Frequencies provided here are general recommendations; specific frequencies should be provided in the project-specific FSP or QAPP.

## **FIELD DUPLICATE SAMPLES**

Collect field duplicate (or split) samples to assess the homogeneity of the samples collected in the field and the precision of the sampling process. Prepare field duplicates by collecting two aliquots for the sample and submitting them for analysis as separate samples. Collect field duplicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of field duplicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

## **FIELD REPLICATE SAMPLES**

Field replicate samples are co-located samples collected in an identical manner over a minimum period of time to provide a measure of the field and laboratory variance, including variance resulting from sample heterogeneity. Prepare field replicates by collecting two completely separate samples from the same station and submitting them for analysis as separate samples. Collect field replicates at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. If field duplicate samples are collected, then it is unlikely that field replicate samples will also be collected during a sampling event. The project QA/QC coordinator will determine the actual number of field replicate samples collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of field duplicate collection may vary by EPA region or state).

## **MATRIX SPIKE/MATRIX SPIKE DUPLICATES**

The MS/MSD analyses provide information about the effect of the sample matrix on the design and measurement methodology used by the laboratory. To account for the additional volume that may be needed by the laboratory to perform the analyses, extra sample volumes may be required to be collected from designated soil stations. MS/MSDs may be collected at a minimum frequency of 1 per 20 samples or once per sampling event, whichever is more frequent. The project QA/QC coordinator will determine the actual number of extra bottles collected during a sampling event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements may vary by analyte group).

## **EQUIPMENT RINSATE BLANKS**

Use equipment rinsate blanks to help identify possible contamination from the sampling environment and/or from decontaminated sampling equipment. Prepare equipment rinsate

blanks by pouring laboratory distilled/deionized water through, over, and into the decontaminated sample collection equipment, then transferring the water to the appropriate sample containers and adding any necessary preservatives. Prepare equipment rinsate blanks for all inorganic, organic, and sometimes conventional analytes at least once per sampling event per the type of sampling equipment used. The project QA/QC coordinator will determine the actual number of equipment rinsate blanks prepared during an event on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of equipment rinsate blank collection may vary by EPA region or state).

## **BOTTLE BLANKS**

The bottle blank is an unopened sample bottle. Submit bottle blanks along with soil samples to ensure that contaminants are not originating from the bottles themselves because of improper preparation, handling, or cleaning techniques. If required, submit one bottle blank per lot of prepared bottles for analysis. If more than one type of bottle will be used in the sampling (e.g., HDPE or glass), then submit a bottle blank for each type of bottle and preservative. The project QA/QC coordinator will determine the actual number of bottle blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of bottle blank analysis may vary by EPA region or state).

To prepare a bottle blank in the field, set aside one unopened sample bottle from each bottle lot sent from the testing laboratory. Label the bottle as "Bottle Blank" on the sample label (and in the "Remarks" column on the COC form), and send the empty bottle to the laboratory with the field samples.

## **TRIP BLANKS**

Use trip blanks to help identify whether contaminants may have been introduced during shipment of the soil samples from the field to the laboratory for VOC analyses only. Trip blanks are prepared at the testing laboratory by pouring distilled/deionized water into two 40 mL VOC vials and tightly closing the lids. Invert each vial and tap lightly to determine if air bubbles exist. There should be no air bubbles in the VOC trip blank vials. If air bubbles are present, then note this information in the field logbook.

Transport the trip blanks unopened to and from the field in the cooler with the VOC samples. Label the trip blank and place it inside the cooler that contains newly collected VOC samples; it must remain in the cooler at all times. A trip blank must accompany samples at all times in the field. Send one trip blank (consisting of a pair of VOC vials) with each cooler of samples shipped to the testing laboratory for VOC analysis.

## **TEMPERATURE BLANKS**

The laboratory will use temperature blanks to verify the temperature of the samples upon receipt at the testing laboratory. The testing laboratory will prepare temperature blanks by pouring distilled/deionized water into a vial and tightly closing the lid. The blanks will be transported unopened to and from the field in the cooler with the sample containers. A temperature blank must be included with each sample cooler shipped to the testing laboratory.

## **ENVIRONMENTAL BLANKS**

Prepare the environmental (i.e., transfer) blank in the field to evaluate potential background concentrations present in the air and in the distilled/deionized water used for the final decontamination rinse. If you use unpreserved bottles, then you must add the appropriate preservative (e.g., for metals samples, use a 10 percent nitric acid solution to bring sample pH to 2 or less), if required. Collect environmental blanks at a minimum frequency of 1 in 20 samples. The project QA/QC coordinator will determine the actual number of environmental blanks analyzed during a project on a case-by-case basis (consult the project-specific FSP and QAPP, as the requirements on frequency of environmental blank analysis may vary by EPA region or state).

To prepare an environmental blank in the field, open the laboratory-prepared sample bottle while at a sample collection site, fill the sample bottle with distilled/deionized water and then seal. Note the location from which the environmental blank was collected along with atmospheric conditions at the time of its collection in the field logbook. Assign the environmental blank a unique sample number, label the bottle, and then send the bottle to the laboratory with the field samples.

## **REFERENCE MATERIALS**

Reference materials (i.e., a standard reference material, a certified reference material, or other reference material) are samples containing known analytes at known concentrations that have been prepared by and obtained from EPA-approved sources. Reference materials have undergone multilaboratory analyses using a standard method which provides certified concentrations. When available for a specific analyte, Reference material samples provide a measure of analytical performance and/or analytical method bias (i.e., accuracy) of the laboratory. Several reference materials may be required to cover all analytical parameters. For all analytes where available, one reference material will be analyzed at a frequency of one per 50 samples. The project QA/QC coordinator will determine the actual number of reference materials analyzed during a project on a case-by-case basis (consult the project-specific FSP

and QAPP, as the requirements on frequency of reference material analysis may vary by EPA region or state).



## **STANDARD OPERATING PROCEDURE (SOP) SL-04**

### **FIELD CLASSIFICATION OF SOIL**

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#### **SCOPE AND APPLICATION**

This SOP establishes the minimum information that must be recorded in the field to adequately document surface soil sampling and soil borehole advancement activities performed during field exploration. The surface soil sampling or borehole log form must be filled out completely for each station.

This SOP presents the field classification of soils to be used by Integral field staff. In general, Integral has adopted the procedures provided in American Society for Testing and Materials (ASTM) Method D-2488-00, Standard Practice for Description and Identification of Soils. ASTM D-2488-00 uses the Unified Soil Classification (USC) system for naming soils. Field personnel are encouraged to study these procedures prior to initiation of fieldwork.

Soil descriptions should be precise and comprehensive without being verbose. The overall impression of the soil should not be distorted by excessive emphasis on minor constituents. In general, the similarities of consecutive soil samples should be emphasized and minor differences de-emphasized. These descriptions will be used to interpret potential contaminant transport properties, rather than interpret the exact mineralogy or tectonic environment. We are primarily interested in engineering and geochemical properties of the soil.

Soil descriptions should be provided on the surface soil field collection form or in the soil description column of the Integral's soil boring log for each sample collected. If there is no difference between consecutive soil samples, subsequent descriptions can be noted as "same as above" or minor changes such as "increasing sand" or "becomes dark brown" can be added.

The format and order of soil descriptions should be as follows:

- Group symbol (in the Unified Symbol column)
- USC name (should be identical to the ASTM D-2488-00 Group Name with the appropriate modifiers)
- Minor components
- Color
- Moisture
- Additional descriptions.

## EQUIPMENT AND REAGENTS REQUIRED

- Surface soil field collection form or borehole log form (see SOP SL-06, *Logging of Soil Boreholes*)
- Munsell® soil color chart.

## PROCEDURES

The USC is an engineering properties system that uses grain size to classify soils. The first major distinction is between fine-grained soils (more than 50 percent passing the No. 200 sieve [75 µm/0.0029 in.]) and coarse-grained soils (more than 50 percent retained by the No. 200 sieve). Small No. 200 sieves are necessary to classify soils near the cutoff size.

1. Fine-grained soils are classified as either silts or clays. Field determinations of silts and clays are based on observations of dry strength, dilatancy, toughness, and plasticity. Field procedures for these tests are included in ASTM D-2488-00. If these tests are used, include the results in the soil description. If these materials are encountered, perform at least one complete round of field tests for a site, preferably at the beginning of the field investigation. The modifiers “fat” and “lean” are used by ASTM to describe soils of high and low plasticity. The soil group symbols (e.g., CL, MH) already indicate plasticity characteristics, and these modifiers are not necessary in the description. Soils with high plasticity can be emphasized by describing them as “silty CLAY with high plasticity.” Plasticity, for example, is an important descriptor because it is often used to interpret whether an ML soil is acting as either a leaky or a competent aquitard. For example, an ML soil can be dilatant/nonplastic and serve as a transport pathway, or it can be highly plastic and very impervious.
2. Coarse-grained soils are classified as either predominantly gravel or sand, with the No. 4 sieve (4.75 mm/0.19 in.) being the division. Use modifiers to describe the relative amounts of fine-grained soil, as noted below:

Description	Percent Fines	Group Symbol
Gravel (sand)	<5 percent	GW, GP (SW, SP)
Gravel (sand) with silt (clay)	5–15 percent	Hyphenated names
Silty (clayey) gravel (sand)	>15 percent	GM, GC (SM, SC)

The gradation of a coarse-grained soil is included in the specific soil name (e.g., fine to medium SAND with silt). Estimating the percent of size ranges following the group name is encouraged for mixtures of silt sand and gravel. Use of the modifiers “poorly graded” or “well graded” is not necessary, as they are indicated by the group symbol.

Show a borderline classification with a slash (e.g., GM/SM). Use this symbol when the soil cannot be distinctly placed in either soil group. Also use a borderline symbol when describing interbedded soils of two or more soil group names when the thickness of the beds are approximately equal, such as “interbedded lenses and layers of fine sand and silt.” Do not use a borderline symbol indiscriminately. Make every effort to place the soil into a single group. (One very helpful addition to the soil log form description is the percentage of silt/sand/gravel. Even if the geologist did not have sufficient time to properly define the soil, this percentage breakdown allows classification at a later date).

3. Precede minor components, such as cobbles, roots, and construction debris with the appropriate adjective reflecting relative percentages: trace (0–5 percent), few (5–10 percent), little (15–25 percent), and some (30–45 percent). Use the word “occasional” to describe random particles of a larger size than the general soil matrix (i.e., occasional cobbles, occasional brick fragments). The term “with” indicates definite characteristics regarding the percentage of secondary particle size in the soil name. It is not to be used to describe minor components. If a nonsoil component exceeds 50 percent of an interval, state it in place of the group name.
4. Give the basic color of a soil, such as brown, gray, or red. Modify the color term with adjectives such as light, dark, or mottled, as appropriate. Especially note staining or mottling. This information, for example, may be useful to establish water table fluctuations or contamination in boreholes. The Munsell® soil color chart designation is the Integral color standard. These charts are readily available and offer a high degree of consistency in descriptions between geologists.
5. Define the degree of moisture present in the soil as dry, moist, or wet. Moisture content can be estimated from the criteria listed in Table 3 of ASTM D-2488-00.
6. If observed, note such features as discontinuities, inclusions, joints, fissures, slickensides, bedding, laminations, root holes, and major mineralogical components. Note anything unusual. Additional soil descriptions may be made at the discretion of the project manager or as the field conditions warrant. The surface soil field collection and soil boring log forms list some optional descriptions, as does Table 13 of the ASTM standard. The reader is referred to the ASTM standard for procedures of these descriptions.

The contact between two soil types must be clearly marked on the surface soil field collection or soil boring log forms. If the contact is obvious and sharp, draw it in with a straight line. If

it is gradational, use a slanted line over the interval. In the case where it is unclear, use a dashed line over the most likely interval.

For drilling activities, the field geologist, who has the advantage of watching the drilling rate and cuttings removal and can talk with the driller in real time, has a much better chance of interpreting the interval than someone in the office.

## **REFERENCE**

ASTM D2488 – 00: Standard practice for description and identification of soils (visual-manual procedure). ASTM International.

## **STANDARD OPERATING PROCEDURE (SOP) SL-05**

### **SURFACE SOIL SAMPLING**

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#### **SCOPE AND APPLICATION**

This SOP defines and standardizes the collection of surface soil samples (e.g., 0 to 12 in. below ground surface). Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

The procedures listed below may be modified in the field upon the agreement of the lead site sampler and field personnel, based on field and site conditions, after appropriate annotations have been made in the field logbook. If specialized sampling methods (e.g., ENCORE®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral's SOP on methanol preservation of soil samples. Record all pertinent information on Integral's surface soil sampling field data form or field logbook.

#### **EQUIPMENT AND SUPPLIES REQUIRED**

- Decontaminated sampling tool (stainless-steel shovel, scoop, trowel, or spoon)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Ziploc® bags
- Camera
- Tape measure
- Field logbook, surface soil field collection form, and pens
- Project-specific field sampling plan (FSP) and health and safety plan (HSP)
- Personal protective equipment (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Decontamination equipment.

## PROCEDURES

1. Locate the sample station as directed in the project-specific FSP. Label containers with sample tags prior to filling in accordance with Integral's SOP on sample labeling (SOP-AP04). If analytical testing will be performed for volatile organic compounds (VOCs), collect the VOC sample first (with a minimum of disturbance) by placing the sample into the container with a minimum amount of headspace and sealed tightly.
2. Don a new pair of nitrile gloves and expose the soil surface by clearing an approximately 1 ft<sup>2</sup> area at the sampling site of any rocks or organic material greater than approximately 3 in. in size. Note any material removed from the sampling site in the field logbook.
3. Using a decontaminated stainless-steel sampling tool, excavate soil to the depth specified in the work plan.
4. If required for analysis, first collect VOC samples (prior to any homogenization) from a discrete location, placing the samples in the appropriate containers. Label sample containers before filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
5. Place additional sample material in a decontaminated plastic or stainless-steel mixing bowl.
6. Describe the soil in accordance with ASTM D2488-00 (see Integral's SOP on field classification of soils, SOP SL-04).
7. Thoroughly mix and homogenize the sample using disposable equipment or a decontaminated stainless-steel spoon until the color and texture are consistent throughout.
8. If required for analysis, first collect samples for grain-size tests before any large rocks are removed from the homogenized soil.
9. Identify any rocks that are greater than 0.5 in. in diameter. Determine their percentage contribution to the homogenized soil volume, note it on the surface soil field collection form or in the field logbook, and then discard the rocks.
10. Remove samples of the homogenized soil from the mixing bowl with the decontaminated stainless steel spoon and place in the appropriate size sample container. Do not touch the sample with your gloves. Fill the sample container with soil to just below the container lip, and seal the container tightly. Label sample containers before filling in accordance with Integral's SOP on sample labeling.
11. Mark the sampling site with a wire flag, wooden stake, metal rebar, or flagging, as appropriate.

12. Complete all pertinent field QA/QC documentation, logbooks, sample labels, and field data sheets. Record any deviations from the specified sampling procedures or any obstacles encountered.
13. Photograph sample location and document it in the logbook.
14. Decontaminate all sampling equipment according to Integral's SOP on decontaminating equipment for soil sampling (SOP SL-01) and in accordance with the project-specific FSP.

## STANDARD OPERATING PROCEDURE (SOP) SL-06

### LOGGING OF SOIL BOREHOLES

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#### SCOPE AND APPLICATION

This SOP describes how to complete a Soil Boring Log form, which must be completed for Integral projects where soil boring techniques are performed during field exploration. A correctly completed form contains all of the information that must be recorded in the field to adequately characterize soil boreholes.

These procedures are adapted from ASTM D-2488-00. Field staff are encouraged to examine ASTM D-2488-00 in its entirety. This SOP represents minor modifications to emphasize environmental investigations rather than geotechnical investigations, for which the standards were written. Because each environmental project is unique and because job requirements can vary widely, the minimum standards presented may need to be supplemented with additional technical descriptions or field test results. However, all soil boring field logs, regardless of special project circumstances, must include information addressed in this SOP to achieve the minimum acceptable standards required by Integral.

#### LOG FORM INFORMATION

**Project Number**—Use the standard contract number.

**Client**—Identify the name of the client and the project site location.

**Location**—If stations, coordinates, mileposts, or similar markers are applicable, use them to identify the location of the project. If this information is not available, identify the facility (e.g., 20 ft NE of Retort #1).

**Drilling Method**—Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, cable tool) and the name of the drill rig (e.g., Mobil B 61, CME 55).

**Diameter**—Provide the diameter of the borehole. If the borehole has variable diameters, provide the depth interval for each diameter.

**Sampling Method**—Identify the type of sampler(s) used (e.g., standard split spoon, Dames & Moore sampler, grab).

**Drilling Contractor**—Provide the name of the drilling contractor.



**Integral Staff**—Enter the name(s) of Integral staff members performing logging and sampling activities.

**Water Level Information**—Provide the date, time, depth to static water, and casing depth. Generally, water levels should be taken each day before resuming drilling and at the completion of drilling. If water is not encountered in the boring, this information should be recorded.

**Boring Number**—Provide the boring number. A numbering system should be developed prior to drilling that does not conflict with other site information, such as previous drilling or other sampling activities.

**Sheet**—Number the sheets consecutively for each boring and continue the consecutive depth numbering.

**Drilling Start and Finish**—Provide the drilling start and finish dates and times.

For consecutive sheets, provide (at a minimum) the job number, boring number, and sheet number.

## TECHNICAL DATA

**Sampler Type**—Provide the sampler type (e.g., SS = split spoon, G = grab).

**Depth of Casing**—Enter the depth of the casing below ground surface immediately prior to sampling.

**Driven/Recovery**—Provide the length that the sampler was driven and the length of sample recovered in the sampler. This column would not apply to grab samples.

**Sample Number/Sample Depth**—Provide the sample number. The sample numbering scheme should be established prior to drilling. One method is to use the boring number and consecutive alphabetical letters. For instance, the first sample obtained from boring MW-4 would be identified as 4A, the second would be identified as 4B, and so on. Another method for sample identification is naming the boring number with the depth. For example, the sample from Boring 1 at 10 ft would be labeled B1-10'. The depth of the sample is the depth of the casing plus the length to the middle of the recovered sample to the nearest 0.1 ft. Typically, split spoon samplers are 18 in. long. Samples should be obtained from the middle of the recovered sample. The depth of the sample with the casing at 10 ft would then be 10.7 ft.

**Number of Blows**—For standard split-spoon samplers, record the number of blows for each 6 in. of sampler penetration. A typical blow count of 6, 12, and 14 is recorded as 6/12/14. Refusal is a penetration of less than 6 in. with a blow count of 50. A partial penetration of 50 blows for 4 in. is recorded as 50/4". Total blows will be recorded for nonstandard split spoons (e.g., 5-ft tube used for continuous sampling).

**Blank Columns**—Two blank columns are provided. Use these columns for site-specific information, usually related to the chemicals of concern. Examples for a hydrocarbon site would be sheen and photoionization detector readings of the samples.

**Depth**—Use a depth scale that is appropriate for the complexity of the subsurface conditions. The boxes located to the right of the scale should be used to graphically indicate sample locations as shown in the example.

**Surface Conditions**—Describe the surface conditions (e.g., paved, 4-in. concrete slab, grass, natural vegetation and surface soil, oil-stained gravel).

**Soil Description**—Enter the soil classification and definition of soil contacts using the format described in SOP SL-04, *Field Classification of Soil*.

**Comments**—Include all pertinent observations. Drilling observations might include drilling chatter, rod-bounce (boulder), sudden differences in drilling speed, damaged samplers, and malfunctioning equipment. Information provided by the driller should be attributed to the driller. Information on possible contaminants might include odor, staining, color, and presence or absence of some indicator of contamination. Describe what it is that indicates contamination (e.g., fuel-like odor, oily sheen in drill cuttings, yellow water in drill cuttings).

## REFERENCE

ASTM D 2488 – 00: Standard practice for description and identification of soils (visual-manual procedure). ASTM International.

## **ATTACHMENT 1. SOIL BORING LOG FORM**



319 SW Washington St., Suite 1150  
 Portland, OR 97204  
 (503) 284-5545

STATION NUMBER \_\_\_\_\_  
 PROJECT \_\_\_\_\_  
 LOCATION \_\_\_\_\_  
 PROJECT NUMBER \_\_\_\_\_  
 LOGGED BY \_\_\_\_\_

SAMPLE INFORMATION						STRATA	DESCRIPTION
Sample ID	Depth	Time	Tag No.	% Recov.	Depth (Feet)		
					2--		
					4--		
					6--		
					8--		
					10--		
					12--		
					14--		














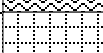

USCS group name, color, grain size range, minor constituents, plasticity, odor, sheen, moisture content, texture, weathering, cementation, geologic interpretation, etc.

DRILLING CONTRACTOR \_\_\_\_\_  
 DRILLING METHOD \_\_\_\_\_  
 SAMPLING EQUIPMENT \_\_\_\_\_  
 DRILLING STARTED \_\_\_\_\_  
 COORDINATES \_\_\_\_\_  
 SURFACE ELEVATION \_\_\_\_\_  
 DATUM \_\_\_\_\_



Location Sketch

**ATTACHMENT 2. FIELD CLASSIFICATION OF SOILS, BASED ON UNIFIED  
SOIL CLASSIFICATION SYSTEM AND ASTM STANDARD D-2488**

## Field Classification of Soils, Based on Unified Soil Classification System and ASTM Standard D-2488

Major Divisions		Symbol and Pattern	General Soil Description	
<b>Coarse-Grained Soils</b> <small>(More than 1/2 of soil &gt;No. 200 sieve size)</small>	<b>Gravels</b>	GW		Well-graded gravels or gravel-sand mixtures, little to no fines
		GP		Poorly-graded gravels or gravel-sand mixtures, little to no fines
		GM		Silty gravels or gravel-sand-silt mixtures
		GC		Clayey gravels or gravel-sand-clay mixtures
	<b>Sands</b>	SW		Well-graded sands or gravel-sand mixtures, little to no fines
		SP		Poorly-graded sands or gravelly sands, little to no fines
		SM		Silty sands, sand-silt mixtures
		SC		Clayey sands, sand-clay mixtures
<b>Fine-Grained Soils</b> <small>(More than 1/2 of soil &lt;No. 200 sieve size)</small>	<b>Silts</b>	ML		Inorganic silts with slight plasticity
		MH		Inorganic elastic silts
		OL		Organic elastic silts
	<b>Clays</b>	CL		Inorganic clays of low to medium plasticity, lean clays
		CH		Inorganic clays of high plasticity, fat clays
		OH		Organic clays of medium to high plasticity
<b>Highly Organic Soils</b>		Pt		Peat, sample composed primarily of vegetable tissue

### Soil Classification Notes

-  Groundwater, First Observed
-  Groundwater, Static

#### Sampling Equipment

- SS Split Spoon
- ST Shelby Tube
- GS Geoprobe® Macrocore Sampler

#### Sheen Types

- NS No Sheen
- LS Light Sheen
- MS Moderate Sheen
- HS Heavy Sheen

#### Sample Moisture

- Dry No moisture, dry to touch
- Moist Damp, but no free water
- Wet Visible free water

#### Sample Plasticity (Fine-Grained Soils)

Non-Plastic - Cannot be rolled at any moisture content.

Low - Can barely be rolled, lump cannot be formed when drier than plastic limit.

Medium - Can easily be rolled, lump crumbles when drier than plastic limit.

High - Can easily be rolled, but takes considerable time to reach the plastic limit. Lump can be formed without crumbling when drier than the plastic limit.

#### Particle Size Range (Coarse-Grained Soils)

Gravel - Fine, Coarse

Sand - Fine, Medium, Coarse

## STANDARD OPERATING PROCEDURE (SOP) SL-07

### SUBSURFACE SOIL SAMPLING

---

#### SCOPE AND APPLICATION

The following procedures are designed to be used to collect subsurface soil samples using a hand auger, direct-push drill rig, and a backhoe. *All underground utilities must be located and cleared prior to drilling or excavating.* Soil samples should be collected from areas having lower levels of constituents of interest first, followed by stations with higher expected levels of constituents of interest.

Based on field and site conditions, the procedures listed below may be modified in the field upon agreement of the field team leader and project management, after appropriate annotations have been made in the project-specific field logbook. If specialized sampling methods (e.g., Encore®) are to be used, refer to the manufacturer's recommended procedures. If methanol preservation is required, refer to Integral SOP SL-08 on methanol preservation of soil samples. Record all pertinent information in the Integral field logbook, subsurface soil field collection form, or boring log (as appropriate).

#### EQUIPMENT AND SUPPLIES REQUIRED

- Subsurface sampling equipment (e.g., hand auger, direct-push drill rig [e.g., Geoprobe®], backhoe, stainless-steel spade) (consult project-specific field sampling plan [FSP] for kind of equipment to be used for a specific field event)
- Large stainless steel mixing bowl and spoon
- Laboratory-supplied sample containers, insulated coolers, and ice
- Chain-of-custody forms, custody seals, sample labels
- Resealable plastic bags (e.g., Ziploc®)
- Camera
- Tape measure
- Logging table
- 6-mil visqueen and duct tape for covering the logging table
- Aluminum foil

- 55-gallon drums for decontamination waters and excess soil (separate drums for liquid and solid wastes) if required by the project-specific FSP
- Field logbook, subsurface soil field collection form, and/or soil boring form, and pens
- Project-specific FSP and health and safety plan (HSP)
- Personal protective equipment (PPE) (safety glasses, steel-toed boots, nitrile gloves, and any other items required by the project-specific HSP)
- Photoionization detector (PID), if required by the project-specific FSP or HSP
- Global positioning system (GPS), if required by the project-specific FSP
- Decontamination equipment.

## HAND AUGER SAMPLER

The following procedures are designed to be used during the general operation of a hand auger sampler. The procedures listed below may be modified in the field upon agreement of the field team leader and drill operators, based on field and site conditions, after appropriate annotations have been made in the field logbook.

1. Locate the sample station as directed in the project-specific FSP. Place sample labels on the sample container prior to filling in accordance with Integral's SOP on sample labeling (SOP AP-04).
2. Place plastic sheeting adjacent to the sampling location.
3. Advance the hand auger into subsurface soil.
4. Empty soil from the first interval (as specified in the project-specific FSP) from the hand auger into a decontaminated stainless steel bowl and cover the bowl with aluminum foil. Continue advancing the hand auger until the next appropriate sample interval has been completed.
5. Screen the soil sample for volatile organic compounds (VOCs) using a PID if required by the project-specific FSP.
6. Photograph each interval with depth and site markers visible in the photograph, if applicable.
7. Log the soils in accordance with SOP SL-04 (*Field Classification of Soils*).
8. If VOC samples are required (see project-specific FSP), collect them prior to homogenizing (i.e., mixing) the sample. Collect the VOC sample (with a minimum of disturbance) by placing the sample into the container with no headspace and sealing it tightly. If an Encore® sampling device is specified in the project-specific FSP, follow the sample collection guidelines provided by the manufacturer.



9. (a) If the soil sample is to be a discrete sample (see project-specific FSP), collect soil from the hand auger using a decontaminated stainless-steel spoon and place the sample into a decontaminated stainless-steel bowl. Homogenize the soil to a consistent color and texture.  
  
(b) If additional sample volume is required to perform the analyses specified in the project-specific FSP, place multiple soil samples collected from nearby locations (it is important to keep the distance between multiple soil borings as close as possible; the maximum distance will be specified in the project-specific FSP) from the same depth interval into a composite sample in a single decontaminated stainless-steel bowl. When a sufficient volume of soil has been obtained, homogenize all of the soil in the bowl to a consistent color and texture using a decontaminated spoon.
10. Discard rocks found in the homogenized soil that are greater than 0.5 in. in diameter after positively identifying them, determining their percentage contribution to the homogenized soil volume, and noting it in the field notebook.
11. Remove samples of the homogenized soil from the compositing bowl and place in the appropriate size sample container. Fill the sample container with soil to just below the container lip, and seal the container tightly.
12. Decontaminate all sampling equipment in accordance with SOP SL-01 and the project-specific FSP.
13. Repeat the process described above for all subsequent sample intervals.
14. Complete the appropriate field books, field data sheets, and quality assurance and quality control (QA/QC) documentation. Record any deviations from the specified sampling procedures or any obstacles encountered.
15. Backfill the borehole with remaining hand auger soil cuttings or place the cuttings in a properly labeled 55-gallon drum, as specified in the project-specific FSP. If soil cuttings are placed in a 55-gallon drum, backfill the borehole with bentonite hole plug pellets and hydrate the pellets with potable water.
16. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate. Collect GPS coordinates of the sample location if specified in the project-specific FSP.

## **DIRECT-PUSH DRILL RIG**

The following procedures are designed to be used during the general operation of direct-push drill rig (e.g., Geoprobe®). The procedures listed below may be modified in the field upon agreement of the field team leader and drill operators, based on field and site conditions, after appropriate annotations have been made in the field logbook. The direct-push drill rig will be operated by a licensed drilling contractor.

The direct-push drilling technique hydraulically pushes tools into the ground to collect soil samples. Direct-push drilling techniques can be used to collect soil samples to depths of 30–100 ft, depending on drilling conditions at the site. In addition to soil sample collection, direct-push techniques can be used to collect soil gas samples, reconnoiter groundwater samples, and install small-diameter monitoring wells.

Soil samples can be collected using two types of Macrocore® samplers, open tip and closed tip. These samplers are typically either 4 ft long by 1.5 in. inside diameter (i.d.) or 5 ft long by 2.5 in. i.d. These samplers have a tubular design and utilize acetate liners to collect the soil samples. The following sections of this SOP describe how to collect soil samples using open-tip and closed-tip Macrocore® samplers.

## **Open-Tip Sampler**

The open-tip sampler is typically used in soils that are cohesive (e.g., stiff silts and clays), where the soil boring is stable and stays open when the sampler and rods are removed from the ground.

1. Ensure all underground utilities are cleared prior to initiating drilling activities.
2. Position the direct-push drill rig over the sample station and remove any surface material that will interfere with sampling. Note in the field logbook any surface material that is removed prior to sampling.
3. Determine the interval to be sampled and install a new clean liner into the open tip Macrocore® sampler.
4. Push the sampler to the bottom of the appropriate sample interval.
5. Retract the rods and Macrocore® sampler.
6. After the Macrocore® sampler has been brought to the surface, remove the liner from the sampler, cap both ends of the liner, and inspect it.
7. After the soil sample is judged to be acceptable, label the sample liner with the station identifier, depth interval, and soil orientation (i.e., arrow pointing toward uppermost soil interval).
8. Place the capped sample liner on a new piece of aluminum foil on the logging table and split the liner open with a hook or utility knife. Process the sample in accordance with the “General Sampling Procedures” listed below.
9. Repeat Steps 2–8 for each subsequent sample interval.

## Closed-Tip Sampler

The closed-tip sampler is typically used to collect soil samples that are noncohesive (e.g., sandy materials), where the soil boring is unstable and collapses when the rods and sampler are removed from the ground.

1. Ensure all underground utilities are cleared prior to initiating drilling activities.
2. Position the direct-push drill rig over the sample station and remove any surface material that will interfere with sampling. Note in the field logbook any surface material removed prior to sampling.
3. Determine the interval to be sampled and install a drive point and a new clean liner into the closed-tip Macrocore® sampler.
4. Push the rods and sampler to the top of the appropriate sample interval.
5. Retract the rods to release the drive point.
6. Push the sampler to the bottom of the appropriate sample interval.
7. Retract the rods and Macrocore® sampler.
8. Once the soil sample has been brought to the surface, remove the liner from the sampler, cap both ends of the liner, and inspect it.
9. After the soil sample is judged to be acceptable, label the sample liner with the station identifier, depth interval, and soil orientation (i.e., arrow pointing toward uppermost soil interval).
10. Place the capped sample liner on a new piece of aluminum foil on the logging table and split the liner open with a hook or utility knife. Process the sample in accordance with the “General Sampling Procedures” listed below.
11. Repeat Steps 2–10 for each additional sample interval.

## General Sampling Procedures

1. After the liner has been split open, screen the soil sample for VOCs using a PID if required by the project-specific FSP.
2. Log the soils in accordance with SOP SL-04 (*Field Classification of Soils*).
3. Photograph each section of the soil boring with appropriate orientation, depth, and site markers visible in the photograph, if specified in the project-specific FSP.

4. If VOC samples are required (see project-specific FSP), collect them prior to sample removal from the liner. Collect the VOC sample (with a minimum of disturbance) by placing the sample into the container with no headspace and seal it tightly. If an Encore® sampling device is specified in the project-specific FSP, follow the sample collection guidelines provided by the manufacturer.
5. Remove the soil from the liner using a decontaminated stainless-steel spoon and place the soil in a decontaminated compositing bowl and thoroughly mix and homogenize the sample using a decontaminated spoon until the color and texture are consistent throughout.
6. (a) If the soil sample is to be a discrete sample (see project-specific FSP), collect soil from the liner using a decontaminated stainless-steel spoon and place the sample into a decontaminated stainless-steel bowl. Homogenize the soil to a consistent color and texture.  
  
(b) If additional sample volume is required to perform the analyses specified in the project-specific FSP, place multiple soil samples collected from nearby locations (it is important to keep the distance between multiple soil borings as close as possible; the maximum distance will be specified in the project-specific FSP) from the same depth interval into a composite sample in a single decontaminated stainless-steel bowl. When a sufficient volume of soil has been obtained, homogenize all of the soil in the bowl to a consistent color and texture using a decontaminated spoon.
7. Discard rocks found in the homogenized soil that are greater than 0.5 in. in diameter after positively identifying them, determining their percentage contribution to the homogenized soil volume, and noting it in the field notebook.
8. Remove samples of the homogenized soil from the compositing bowl and place in the appropriate size sample container. Fill the sample container with soil to just below the container lip, and seal the container tightly.
9. Repeat the process described above for subsequent sample intervals.
10. Complete the appropriate field books, field data sheets, and QA/QC documentation. Record any deviations from the specified sampling procedures or any obstacles encountered.
11. Backfill the borehole with remaining direct-push sampler cuttings or place the cuttings in a properly labeled 55-gallon drum, as specified in the project-specific FSP. If soil cuttings are placed in a 55-gallon drum, backfill the borehole with bentonite grout (mixed to the manufacturer's specifications) or bentonite hole plug pellets and hydrate the pellets with potable water.
12. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate. Collect GPS coordinates of the sample location if specified in the project-specific FSP.

13. Decontaminate all sampling equipment in accordance with SOP SL-01 and the project-specific FSP.

## Test Pit Excavations

The following procedures are to be used during the excavation of pits with construction equipment (i.e., backhoe or track-hoe) prior to soil sampling operations. Adhere to all requirements of the site-specific HSP for this specific activity. The procedures listed below may be modified in the field upon agreement of the field team leader and project management, based on field and site conditions, after appropriate annotations have been made in the field logbook.

1. Locate the sample station as directed in the project-specific FSP. Ensure all underground utilities have been cleared prior to initiating excavation activities. Place sample labels on all sample containers prior to filling in accordance with Integral's SOP for sample labeling (SOP AP-04).
2. Select the appropriate orientation for the excavation, basing it on the judgment of the field team leader, backhoe operator, and onsite conditions. Sampling personnel **MUST** remain in visual contact with the backhoe operator at all times, and out of possible "pinch zones" or areas where heavy equipment may move or swing.
3. Place plastic sheeting from the edge of the proposed excavation leading away for a sufficient distance to the proposed temporary stockpile location so that the excavated soil does not slough back into the pit.
4. Begin pit excavation.
5. Continue excavation of the pit to the required depth. If pit entry is necessary, this depth will not exceed 4 ft from the ground surface. Never enter a trench or pit if conditions are unstable. Excavate the proper pit exit trenches, shoring, and sloping to prevent accidental burial of sampling crew, and to meet or exceed all OSHA Construction Standards (29 CFR § 1926; Attachment 201-2) for entrance by sampling personnel. If pit entry is not necessary for sampling activities, pit depth can exceed 4 ft below ground surface. Instruct the backhoe operator to scrape material evenly along an exposed face to collect (to the extent practicable) a representative sample of the soils across the entire face in the bucket. Collect soil samples from the middle of the backhoe bucket.
6. Screen the soil sample for VOCs using a PID if required by the project-specific FSP.
7. Photograph each interval with depth and site markers visible in the photograph, if applicable.
8. Log the test pit soils in accordance with SOP SL-04 (*Field Classification of Soils*).

9. If VOC samples are required (see project-specific FSP), collect them prior to homogenizing (i.e., mixing) the sample. Collect the VOC sample (with a minimum of disturbance) by placing the sample into the container with no headspace and seal it tightly. If an Encore® sampling device is specified in the project-specific FSP, follow the sample collection guidelines provided by the manufacturer.
10. Collect soil using a decontaminated stainless-steel spoon or disposable sampling tool (depending on project-specific requirements; see FSP), which has been evenly removed from the face of the trench wall or from the bucket, and place the sample into a decontaminated stainless-steel bowl. Homogenize the soil to a consistent color and texture.
11. Discard rocks found in the homogenized soil that are greater than 0.5 in. in diameter after positively identifying them, determining their percentage contribution to the homogenized soil volume, and noting it in the field notebook.
12. Remove samples of the homogenized soil from the compositing bowl and place them in the appropriate size sample container. Fill the sample container with soil to just below the container lip and seal it tightly.
13. Decontaminate all sampling equipment in accordance with SOP SL-01 and the project-specific FSP.
14. Repeat the process described above for all subsequent sample intervals.
15. Complete all pertinent field logbooks, field data sheets, and QA/QC documentation. Record any deviations from the specified sampling procedures or any obstacles encountered.
16. Mark the sampling location with a wire flag, wooden stake, metal rebar, or flagging, as appropriate. Collect GPS coordinates of the sample location if specified in the project-specific FSP. Photograph sample location and document in the logbook.
17. Backfill the test pit with the excavated soils. Depending on historical site data (see project-specific FSP), the plastic sheeting will either be disposed of as garbage or it will be drummed and sent to a hazardous waste landfill.

## STANDARD OPERATING PROCEDURE (SOP) SW-01

# DECONTAMINATION OF SURFACE WATER SAMPLING EQUIPMENT

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### SCOPE AND APPLICATION

This SOP defines and standardizes Integral's methods for decontamination of field sampling equipment for collecting surface water samples to ensure sample integrity and minimize contamination during sample handling.

This SOP utilizes and augments the procedures outlined in the San Francisco Estuary Institute's *Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001), *Interagency Field Manual for the Collection of Water-Quality Data* (USGS various dates), and U.S. Environmental Protection Agency (EPA) Method 1669, *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996). Clean sampling techniques designed for trace metals will be used for the collection of filtered and unfiltered water samples.

Samples may be analyzed for organic compounds, metals, nutrients, and conventional analytes for the surface water sampling events, according to the project-specific sampling and analysis plan (SAP).

To prevent cross-contamination of samples, all reusable surface water sampling equipment will be decontaminated before each use. Decontamination of field sampling equipment can be done in the field or in a commercial laboratory. Depending on the project's complexity and analytical reporting limits (see project-specific SAP), sampling equipment may need to be decontaminated at a qualified laboratory. It is strongly discouraged to decontaminate sampling equipment in the field due to the high risk of contamination. Thorough decontamination procedures should be followed under controlled conditions at the laboratory. However, it is necessary to perform certain decontamination steps in the field.

Set up a decontamination station onsite in a clean location upwind of sampling locations, or perform decontamination in the field office, under a laboratory hood if available. Store decontaminated equipment away from contaminated areas and in a manner that will prevent recontamination prior to use.

When handling decontamination chemicals, follow all relevant procedures outlined in the site-specific health and safety plan.

## EQUIPMENT AND REAGENTS REQUIRED

Equipment required for decontamination includes the following, depending on the target analyte and sampling equipment:

- Plastic brushes with rigid bristles
- Properly labeled squirt bottles
- 5-gal plastic bucket
- Tap water
- Alconox®, Liquinox® detergent, or equivalent
- Pesticide-grade decontamination solvents (e.g., ethanol and methanol, according to the project-specific SAP, as the solvents may vary by EPA region or state)
- Nitric acid (5 percent)
- Hydrochloric acid (10 percent) if nutrients are being analyzed
- Deionized water (analyte-free; received from testing laboratory)
- Sealable waste container equipped with a funnel
- 1 gal sealable plastic bags
- 2.5 L amber glass bottles.

## DECONTAMINATION PROCEDURES

Decontamination methods vary depending on whether the samples collected will be analyzed for conventional analytes, organic chemicals, or trace metals.

### Conventional Analytes and First Use

The following procedure is used when sampling for conventional analytes such as chloride, sulfate, sodium, and calcium. It is also used for new equipment and for equipment that is being used for the first time at a site. Conventional analytes have the simplest decontamination procedure because they tend to be highly soluble in water and detergent solutions, and do not tend to sorb significantly to the surface of the sampling equipment.

For collection of lake water samples at different depths from the same location, equipment needs to be rinsed only with site water three times between stations following an initial decontamination. Similarly, for collection of samples from rivers where stations are close to one another spatially and temporally, only a site-water rinse is necessary. The steps are as follows:

1. Rinse the equipment thoroughly with tap water.



2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (some detergents contain surfactants that are analytes) and set aside to drain.
4. Rinse the equipment three times with site water immediately prior to collecting the sample.

## Organic Chemicals

The following procedure is used for decontaminating equipment (e.g., Kemmerer sampler) used to collect surface water that will be analyzed for organic chemicals. Two organic solvents are used in the procedure. The first is miscible with water (e.g., ethanol) and is intended to scavenge water from the surface of the sampling equipment and allow the equipment to dry quickly. Make sure that the solvent ordered is anhydrous or has a very low water content (i.e., <1 percent). The second organic solvent is hydrophobic (e.g., methanol) and is intended to dissolve any organic chemicals on the surface of the equipment.

The exact solvents used for a given project may vary by EPA region or state (see project-specific SAP). The choice of solvents also depends on the material the equipment is made from (e.g., acetone cannot be used on polycarbonate), and the ambient temperature (e.g., hexane is too volatile in hot climates). In addition, although methanol and hexane are sometimes slightly more effective than other solvents, their use is discouraged because of toxicity to sampling personnel. The decontamination procedure is as follows:

1. Rinse the equipment thoroughly with tap or site water.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap or site water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution.
3. Rinse the equipment with tap or site water and set aside to drain.
4. Rinse the equipment with ethanol dispensed from a squirt bottle and let the excess solvent drain into a waste container equipped with a funnel (ethanol acts primarily as a drying agent, but also works as a solvent for some organic contamination). Rinse the inside of the sampling equipment that comes in contact with sample water. Set the equipment in a clean location and allow it to air dry. In cold temperatures, it may take a long time for equipment to dry. In this case, it is important to remove all water from the surface by thoroughly rinsing with a more volatile solvent such as acetone. In hotter temperatures, use a less volatile water solvent (e.g., isopropanol).

5. Rinse the air-dried equipment with methanol dispensed from a squirt bottle and let the excess solvent drain into the waste container. Methanol acts as the primary solvent, but it is insoluble with water. If water beading occurs, it means that the equipment was not thoroughly rinsed with ethanol or the equipment was not given sufficient time to dry completely. Rinse the inside of the sampling equipment that comes in contact with site water. In hotter climates, use a less-volatile solvent such as methanol. When the equipment has been rinsed thoroughly, set it in a clean location and allow the solvent to evaporate before storing or using it.
6. Close the solvent waste container when not in use and store it in a secure place.
7. Transfer the waste to empty solvent bottles and dispose of it at a licensed facility.

## Trace Metals

In addition to the following decontamination procedures, personnel collecting water samples must be aware of other sources of contamination. Sources commonly encountered in the field include lead batteries used to power pumps, metal objects such as tools, and gasoline cans. To the extent possible, these items should be removed from the sample collection area and the sampling equipment, and anyone collecting the samples should avoid handling these items beforehand. Wear vinyl clean-room gloves (e.g., Oak class 100, powder free) when handling sampling equipment that will be used to collect surface water samples for trace metals analysis. Discard gloves between stations or if they come into contact with any materials known or likely to be contaminated.

The following procedures should be used for decontaminating equipment used to collect surface water samples for trace metals (e.g., Teflon™ tubing, Teflon™ churn splitter, connectors and adapters made of Teflon™ or other similar material, or plastic stands used for holding sample tubing). This procedure is not intended for containers in which samples will be stored and/or shipped to the laboratory for analysis.

1. Rinse the equipment thoroughly with tap or site water.
2. Pour a small amount of Alconox® (or similar product) into a 5 gal bucket and fill it with tap or site water. Using a plastic brush with rigid bristles, scrub each piece in the detergent solution. Fill bottles about halfway with detergent solutions and shake for a few minutes. Pump the detergent solution through any tubing for a few minutes. Small parts can be placed in large-mouth jars that have tight lids and shaken with the detergent solution.
3. Rinse the equipment with tap water to remove all detergent (detergents will neutralize the nitric acid) and set it aside to drain.

4. Clean all equipment surfaces that come into contact with water samples using a 5 percent nitric acid solution for at least 30 minutes. Place small items, such as Teflon™ water intakes, in plastic containers filled with 5 percent nitric acid. Fill sampling containers/bottles with 5 percent nitric acid solution and allow to stand. Cover the containers and keep them away from potential contamination sources.
5. Either pump acid solution through tubing, or leave it static in the tubing for the same duration.
6. Drain all equipment thoroughly and flush with at least three volumes of laboratory deionized water (not deionized water from the grocery store).
7. Drain thoroughly and flush with at least three volumes of site water before collecting a sample.

## **PROCEDURES USED TO DECONTAMINATE SAMPLING DIAPHRAGM PUMPS**

The following procedure is used for samples to be analyzed for trace metals and conventional analytes. Two types of pumps are commonly used for collecting water samples, peristaltic and diaphragm. For peristaltic pumps, only the tubing needs to be cleaned according to the above procedure. It is best to keep precleaned short lengths of tubing for each station when using the peristaltic pump. For diaphragm pumps, the procedure is as follows:

1. Using two short pieces of tube on the pump, place both ends in a 1-gal container with detergent solution and circulate the solution through the system for 2 minutes.
2. Purge the system with about 1 gal of laboratory deionized water, keeping the outflow tubing over a waste bucket. Do not recirculate this solution. Repeat the 1 gal deionized water purge.
3. Connect the two ends of the short tubes with a decontaminated plastic coupler and keep it sealed until sampling time.
4. When ready to sample, remove the short tubing protecting the inlet of the pump, connect the tubing used for sampling to the pump, and purge the system with site water for 2 minutes, or with enough water to rinse the entire system (i.e., pump head and tubing) immediately before collecting the sample.

## **REFERENCES**

David, N., D. Bell, and J. Gold. 2001. Field sampling manual for the regional monitoring program for trace substances. San Francisco Estuarine Institute, San Francisco, CA.

USEPA. 1996. Method 1669 – Sampling ambient water for trace metals at EPA water quality criteria levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC.

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## **STANDARD OPERATING PROCEDURE (SOP) SW-04**

### **SURFACE WATER SAMPLING USING A PERISTALTIC PUMP**

#### **SCOPE AND APPLICATION**

This SOP defines and standardizes the methods for collecting surface water samples from freshwater or marine environments using a peristaltic pump and Teflon™ tubing.

This SOP utilizes and augments the procedures outlined in the *San Francisco Estuary Institute's Field Sampling Manual for the Regional Monitoring Program for Trace Substances* (David et al. 2001), the *Interagency Field Manual for the Collection of Water-Quality Data* (USGS various dates), and *U.S. Environmental Protection Agency (EPA) Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (USEPA 1996). The goal of this SOP is to ensure that the highest quality, most representative data be collected, and that these data are comparable to data collected by different programs that follow EPA guidelines.

By following this SOP, surface water can be collected with a high level of sample integrity and minimal contamination during sample handling. The trace clean sampling method described in this SOP can be used to collect surface water for filtered and unfiltered water analysis, trace metals analysis, analysis of organic compounds, and analysis of conventional analytes, such as total suspended solids, dissolved organic carbon, and total dissolved solids.

#### **STATION ACCESS**

Prior to entering select areas such as private beaches or embayments, or nearing docks, it may be necessary to acquire property access permission from the landowner. Be sure to secure such permission, including any written agreements, in advance of the sampling program.

#### **STATION LOCATION**

When collecting near-bottom surface water samples, take care to avoid resuspending sediments in the water column, which could affect the sample being collected or samples to be collected at other downstream stations. To avoid resuspended sediment interference in the sample being collected, always approach stations from downstream. Avoid sampling near eddies that may circulate water from the sampling location to upstream of the sampling location. To avoid interference from resuspended sediment at other stations, begin collecting samples with the most downstream station and continue upstream.

Collect near-surface water samples at least 6 in. below the surface–air interface or surface water microlayer to avoid collecting non-representative compounds such as transient dust particles and thin oil films, unless otherwise instructed.

Collect water samples from areas that are representative of the surface water body conditions. A station that is located away from immediate point sources (e.g., tributaries and industrial and municipal effluents) is preferred for collecting surface water samples unless sampling is designed to assess these sources. Representative samples can usually be collected in portions of the surface water body that have a uniform cross section and flow rate. Because mixing is influenced by turbulence and water velocity, select a site immediately downstream of a riffle area (e.g., fast flow zone) to ensure good vertical mixing.

Sample tributaries as near the mouth as is feasible. However, consider the impact of the downstream receiving water body on the tributary flow and sediments. The downstream body may decrease water velocity (causing suspended solids to settle) or create eddies (causing mixing of the two waters). The downstream water body may change the water quality (e.g., salinity), temperature, or turbidity in the tributary near its mouth. It is important to determine how far upstream the tributary is influenced by the downstream water body, and then establish a sampling point with a reasonable distance upstream from that boundary.

Pay attention to intakes and outflows within lagoons or settling ponds, which may cause localized concentrations that are not representative of general conditions. Sample locations adjacent to structures (e.g., banks, piers) may also have biased characteristics as a result of flow or release of substances from the structure. Note these kinds of confounding factors in the field logbook. For ponds and lakes that may be vertically stratified, use a multi-parameter water quality meter to collect depth profiles throughout the water body to aid in selecting appropriate sampling points and depths.

## **SUMMARY OF METHOD**

To collect surface water samples for standard chemical and conventional analyses, use a peristaltic pump with an extended sampling tube lowered to the desired depths (see project-specific sampling and analysis plan [SAP]). Two kinds of sampling devices may be used to obtain the water samples, depending upon the project's needs. The near-surface water polyvinyl chloride (PVC) sampling structure (water sampler) has a polyurethane-coated weight suspended from the bottom of the structure to maintain it in an upright position (Figure 1a).

A near-bottom water sampler has a weighted landing base designed to keep the sampling tube at a fixed distance from the bottom (e.g., 30 cm, or 12 in.) and prevent the intake from coming in contact with the sediment (Figure 1b). Both types of water samplers keep the tubing intake pointing into the current with the help of a vane. The vane can be removed if the water is

quiescent. Additional equipment, such as a multi-probe or underwater video camera, may be mounted on the PVC structure.

At each station, when either a near-surface or a near-bottom water sampler is deployed, attach the Teflon™ tubing to the vane with zip ties and place the water intake approximately 10 ft from the bow of the boat with the aid of an A-frame or davit. Keeping the boat facing the current, lower the water sampler unit to the appropriate depth with the help of a hydraulic or electric winch. Using a peristaltic pump, direct the outflow from the sampling tube into either a polycarbonate (for inorganic analyses) or glass or stainless-steel (for organic analyses) composite mixing container (Figure 2). Pump equal volumes of water into each large, pre-cleaned 10- or 20 L mixing container (depending on the project-specific needs) that is equipped with a Teflon™-coated magnetic stirring bar, and place them over a magnetic stir table. Use the containers for mixing and compositing samples for subsequent chemical analysis.

Following sample compositing in the mixing container, fill appropriate sample bottles (see project-specific SAP) using a second peristaltic pump, with the outflow directed into the sample bottle. If enough water volume is available, hold the sample bottle near the pump outlet, rinse the sample container one or two times, discard the rinsate, and then fill the sample bottle. Be aware that laboratory bottles are pre-cleaned and this rinsing option is not mandatory if water volume is an issue. Collect field rinsate blanks to ensure that sampling containers are not a source of contamination. If preservatives are present in the sample bottle, then omit the rinsing step. Cap and label the sample containers, and place them inside a cooler to store at approximately  $4 \pm 2$  °C.

Two types of surface water samples may be collected: unfiltered and filtered. For filtered metals and dissolved organic carbon samples, place the 0.45- $\mu\text{m}$  filter (or project-specific pore size filter; see project-specific SAP) in-line near the tubing outlet to filter samples immediately before the water is discharged into the sample bottle (Figure 2). In general, filter samples for total suspended solids (TSS) and total dissolved solids (TDS) at the laboratory (see project-specific SAP).

Use the same technique described above to collect water for compositing surface water collected at horizontally integrated near-surface and near-bottom stations.

## **EQUIPMENT AND REAGENTS REQUIRED**

This section describes the general types of required equipment and reagents. Attachment 1 provides a detailed supply and equipment list. Additional equipment may be required depending upon project-specific needs.

Use one or two peristaltic pumps at each sampling station (near-surface and near-bottom) for collecting surface water samples. To collect unfiltered and filtered split samples from the mixing containers, use the same pump that is used to fill the mixing containers. Use a sample

processing and preservation chamber (i.e., workbox) made of PVC pipes and 6-mil plastic sheeting to house stir plate(s), a peristaltic pump, sampling bottles, and ancillary equipment. Place a polycarbonate, glass, or stainless-steel mixing container (10 or 20L) on the stir plate. Each mixing container is equipped with a 3-in.-long Teflon™-coated stir bar at the bottom and a lid containing inflow, outflow, and vent Teflon™ spouts (Figure 2). For each sampling station, assemble a filtering kit (laboratory precleaned 0.45-µm filter with C-Flex™ and Teflon™ tubing placed in a double Ziploc™ bag) and attach it to a peristaltic pump and mixing containers. If necessary, attach a precleaned 10-µm prefilter inline to prolong the life of the 0.45-µm filter. You will need the following equipment:

- Peristaltic pump
- Surface water parameter multimeter capable of measuring pH, reduction/oxidation (redox) potential, temperature, specific conductance, turbidity, and dissolved oxygen
- PVC pipes and plastic sheeting
- Polycarbonate (inorganic analyses) and/or glass or stainless-steel (organic analyses) mixing containers (see project-specific SAP for analyte list)
- Sample tubing (type and length are site-dependent)
- Stir plate with Teflon™-coated stir bar
- 0.45-µm filter with C-Flex™ and Teflon™ tubing (if needed; see project-specific SAP to determine if filtered samples are required)
- Water Sampling Log forms (attached)
- Sample tags/labels and appropriate documentation (e.g., chain-of-custody forms)
- Insulated cooler(s), chain-of-custody seals, Ziploc® bags
- Sample containers with preservative, coolers, and blue ice or equivalent.

## PROCEDURES

The sampling team should comprise three people. Two are needed to conduct the sampling and a third must keep track of sample logging and processing. In addition, the third person may be responsible for collecting the surface water quality parameters.

### Equipment Preparation

Bring enough decontaminated sampling tubing and filtering kits to the field to avoid performing decontamination procedures between stations. Each participating laboratory is responsible for preparing its equipment prior to the sampling cruise. Predesignated commercial laboratories will decontaminate sample tubing, mixing containers, and sampling bottles according to their specific SOPs.



Note: Decontamination of large amounts of sampling equipment requires several days, if not weeks, to be ready for sampling. Contract agreements with commercial laboratories and scheduling decontamination work may require several weeks to months. Initiate this critical step as early as possible.

The main components of the peristaltic pump sample collection system are as follows:

- **Processing and Preservation Chamber**—Build a workbox with ¾-in. PVC tubing and cover it with a 6-mil plastic sheet in order to contain the peristaltic pump sampling equipment and conduct the subsampling from the carboys. Leave one side of the workbox open for placing sampling equipment and sample containers. Wash all components with Alconox™ and rinse with tap water. To secure the receiving Teflon™ tubing and filter cartridge, use stands and clamps made of non-metallic components or resin-coated stainless steel, which have been washed with soap, rinsed in tap water, washed in acid, and rinsed with distilled/deionized water.
- **Water Sampler**—The water sampler device for near-surface sampling should be made of PVC tubing with a polyurethane-coated 50-lb weight at the bottom to keep the sampler in the vertical position (Figure 1a) (Note: To reduce potential drag at the water surface, do not include a base on the near-surface sampling device.) The near-bottom sampling device should also be made of PVC tubing and have a polypropylene vane, constructed with a weighted base (Figure 1b). Both sampling devices should be attached to the boat by a Technora™ or Kevlar™ rope. Figure 1b shows the sampling device with a YSI water quality multimeter and underwater camera attached to it. Figure 1b also shows how the Teflon™ tubing is positioned on the vane and the relationship of the inlet to the water sampler. The vane works to keep the water intake into the flow and elevated at a constant height from the bottom. Prior to commencement of sampling activities, wash all components with Alconox™ and rinse with tap water.
- **Water Quality Meter**—Use a YSI 650/6600 multi-probe (newer model or similar) for measuring surface water parameters, such as temperature, pH, dissolved oxygen, conductivity, oxidation-reduction potential (ORP), and turbidity. Attach it to the water samplers as shown in Figures 1a and 1b. The unit will come pre-calibrated from the laboratory and will be checked daily for proper functioning and drift. However, the must be calibrated daily for certain parameters such as pH, conductivity, ORP, and dissolved oxygen. If possible, install a YSI unit on each water sampler (i.e., near-surface and near-bottom) if both are deployed at the same time. A YSI unit installed on the near-bottom water sampler can also take an initial near-surface measurement at the beginning and at the end of the sampling event, therefore avoiding the cost of having to install an additional YSI unit on the near-surface water sampler. The proper handling of the multi-probe is described in detail in SOP SW-06. Except for the probe sensor, wash all components with soap (Alconox™) and rinse with tap water. Because

this equipment will not be in the pathway with the surface water being collected, there is no need for a thorough decontamination.

Take the following steps to set up the surface water collection system:

1. Assemble and secure the water samplers to either the A-frame or a davit.
2. Determine the correct position of the sampling station, have the captain anchor the vessel into the current at the sample site, and switch off the engines. If anchoring is not possible and the engine must be on, make sure the water intake tubing is always facing into the current.
3. Set up a clean area for the workboxes. Set workboxes on a secure table or bench top onboard the sampling vessel to house stir plate(s) and a small peristaltic pump in each workbox. Provide enough space inside the workboxes for a stand to hold the outlet tubing and filter (if necessary; see project-specific SAP) and to collect surface water and processing sample bottles (Figure 3).
4. Place stirring plate(s) inside the workbox and the mixing container(s) on top of the plate. Check each mixing container (a polycarbonate container for inorganic analytes and a glass or stainless-steel container for organics) to ensure:
  - Containers were properly wrapped by the laboratories and are free of rips or holes that may have occurred during shipment to the field.
  - Each container contains a 3-in. stir bar at the bottom.
  - All components such as inflow and outflow tubing have been properly assembled in the laboratory (e.g., one end of the outflow tubing should be touching the bottom inside the container), and that they are intact and securely placed on the cap.
5. Attach the outlet tubing “kits” (i.e., Unit # 3) to the mixing containers (Figure 2). The kits are composed of 10-cm C-Flex™ tubing, 0.5-m Teflon™ tubing, 30-cm C-Flex™ tubing, and 30-cm Teflon™ tubing, placed sequentially.
6. Place the small peristaltic pump inside each of the workboxes.
7. Place a stand inside the workboxes and secure each tubing outlet from both mixing containers with clamps (Figure 3).

8. Attach Teflon™ tubing (collecting end) to 30-cm C-Flex™ tubing and 1-m Teflon™ tubing, sequentially, and then connect these interconnected pieces of tubing to a mixing container (polycarbonate for inorganics and glass or stainless steel for organics). Clamp the C-Flex™ tubing section firmly into place inside the large peristaltic pump head, which is placed outside the workbox. (Note: The length of the Teflon™ tubing will vary depending on project-specific requirements and water depth at a given station. For example, 4 m of Teflon™ tubing could be used for near surface sampling and 25 m could be used for near-bottom sampling)
9. Attach the intake part of the Teflon™ tubing to the vane of the near-surface sampler (Figure 1a) or to the vane of the near-bottom sampler (Figure 1b). Take care not to remove the protective cap from the tip of the sample collection tube until the sampling device is ready for submersion.
10. Secure the pump and pump speed controller, and connect them to the vessel's power source with an extension cord. If vessel power is not available, use the pump's battery power supply.
11. To limit sediment suspension during near-bottom sampling, tether a submersible, underwater video camera to the boat and attach it to the sampling device vane to reveal when the sampling device touches bottom.

## Sample Collection

Take the following steps to collect and process the surface water samples:

1. Remove the protective cap from the sampling tube and lower the sampler gently below the water surface.
2. To sample water near the surface, submerge the sample tubing inlet approximately 1 m (3 ft) below the surface of the water column (consult project-specific SAP for exact sampling water depth).
3. To sample water near the bottom, submerge the sample tubing inlet approximately 3 m (9 ft) above the bottom (consult project-specific SAP for required distance) with the help of the A-frame or davit. If it is necessary to sample surface water at a fixed depth from the bottom, adjust the vane height on the sampler while the sampler rests on the bottom surface. The vane will maintain the sample tubing inlet into the current at a constant depth between 30 cm (12 in.) and 1 m (3 ft) above the sediment–water interface.

4. Begin collecting measurements of water quality parameters at each depth using the water quality meter (e.g., YSI, Hydrolab, Horiba). Set data collection intervals according to data needs. If a vertical water column profile is needed, set the multi-probe to collect data every 1 second for a high-resolution profile. If sampling a vertically integrated water column with several round trips to the bottom, reset the multi-probe to collect data at time intervals relative to the sampling time period after the initial high resolution profile. For example, if sampling a vertically integrated water column during a period of 2 hours, set the multi-probe to record data every 1 second for the first roundtrip to the bottom and then reset to record data every 5 minutes for the subsequent roundtrips until sampling is complete. If collecting surface water at a stationary location for more than 1 hour, and no major changes in water quality are expected, set the multi-probe to collect data every 15 minutes.
5. Note: Failure to adjust the multi-probe for data collection according to sampling periods can result in data loss. That is, if the multi-probe memory bank is quickly filled early in a long sampling period, no additional data will be stored in the memory bank for the remaining sampling time.
6. Record the water quality measurements on the Water Sampling Log forms every 15 minutes during sample collection. If the surface water sample collection is completed within 15 minutes, then collect water quality parameters at least three times: at the beginning, middle, and end of sample collection.
7. Switch the pump on and pump surface water through the sample tubing and into the mixing containers. Once the water reaches one-third of the container's volume, turn on the stir plates.
8. Turn off the pump once the mixing containers have been filled to 1 in. below the inflow spout or when sufficient volume has been collected to fill all of the sample bottles at a given station.
9. Place the C-Flex section of the outflow tubing kit from the first container to be sampled inside the small peristaltic pump head and clamp firmly.
10. Before turning on the small pump, make final adjustments to the stand, holding the outflow spout as close as possible to the sample bottle opening, but without touching the inside of the bottle.
11. Fill container to the "neck" with unfiltered sample water.
12. After collecting the unfiltered samples, attach the 0.45- $\mu\text{m}$  filter cartridge (or appropriate pore size filter) to the sample tubing outlet and secure it to the stand with a clamp (consult project-specific SAP to determine if filtered samples are required). Drain the storage solution inside the filter, and flush the entire sample tubing and filter assembly with sample water. Discard this first "rinse" of sample water.

13. After rinsing the filter and sample tubing, fill the sample bottle to the “neck” with filtered sample water. (Note: If dissolved constituents are being analyzed [per the project-specific SAP], then discard the 0.45- $\mu$ m filtration cartridge after each sampling site.)
14. As soon as a sample container is filled, turn off the peristaltic pump and label the container. Include the date, time, project name or number, sample ID, type of analysis required, and sampler initials on the label (see SOP AP-04).

Once a surface water sample container is properly closed and labeled, place it inside a cooler containing wet or blue ice and store it at approximately 4°C. Store all samples in coolers with ice on board the vessel and transfer them to the field laboratory (if applicable) at the end of the sampling day.

## **Water Quality Measurements**

If specified in the project-specific SAP, measurements of physical and chemical water parameters may need to be collected at surface water stations. Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field if feasible). In addition, measurements of temperature and transparency can be accurately collected only in the field.

It is always best to place the water quality meter directly into the surface water body at the station location at the desired water depth instead of collecting a sample and measuring parameters in a container. However, if this is not possible, use a plastic bucket to collect samples for water quality analyses (e.g., pH, temperature, and conductivity). Rinse a clean bucket twice with the water from the station prior to measuring water quality parameters.

The name(s) of the person(s) making the measurement and the field equipment used to make that measurement must be recorded in the field logbook and on any field forms used during the sampling event. Equipment maintenance and calibration records must be kept in logbooks and field records so that the procedures are traceable.

## **Sample Handling**

Gloved hands are required for sample collection and handling, as described above. Field staff will wear appropriate non-contaminating, disposable, powderless nitrile gloves during the entire sampling operation. Change gloves frequently, usually with each change in task (wearing multiple layers of gloves allows rapid glove changes).

Gloved hands are required for all operations that involve equipment that comes into contact with the sample, including the following activities:

- Handling the sample bottle
- Handling the discharge end of the sample tube or line
- Setting up working space inside the processing and preservation chambers
- Setting up the equipment (i.e., the sample bottles, mixing containers, and the filtration and preservation equipment) inside the chambers
- Working inside the chambers during collection, processing, and preservation
- Handling the filter (if needed)
- Changing the chamber covers as needed.

Ungloved hands take care of all operations that involve contact with potential sources of contamination, including the following activities:

- Working exclusively exterior to the processing and preservation chambers
- Preparing a clean workspace (inside boat)
- Preparing and operating the sampling equipment, including the pumps and discrete samplers, peristaltic pump switch, pump controller, and manifold system
- Handling the generator or other power supply for samplers
- Handling the tools, such as hammers, wrenches, keys, locks, and sample-flow manifolds
- Handling the single or multi-parameter instruments for field measurements
- Setting up and checking the field-measurement instruments
- Measuring and recording the water depths and field measurements.

Store all samples in coolers with ice at approximately 4°C on board the vessel and transfer them to the field laboratory (if applicable) at the end of the sampling day. The sampling team leader is responsible for maintaining sample integrity throughout the sampling event.

If storage freezers or refrigeration units are available at the field laboratory, monitor these units daily to ensure temperature compliance. Each unit will have a separate log form containing date, time, and temperature information.

Avoid contaminating samples by handling the sample containers with clean gloves and transferring the samples into clean refrigerators (or clean coolers) immediately after the samples have been brought back from the field. Always wear disposable, powderless nitrile gloves when handling samples. This includes any and all sample handling that may occur during sample packing and shipping (see SOP AP-01).

## RELATED SOPS

- Pack and ship all surface water samples in accordance with procedures outlined in SOP AP-01.
- Record field activities in accordance with procedures outlined in SOP AP-02.
- Maintain sample custody in accordance with procedures outlined in SOP AP-03.

## REFERENCES

David, N., D. Bell, and J. Gold. 2001. Field sampling manual for the Regional Monitoring Program for Trace Substances. San Francisco Estuarine Institute, San Francisco, CA.

USEPA. 1996. Method 1669 – Sampling ambient water for trace metals at EPA water quality criteria levels. U.S. Environmental Protection Agency, Office of Water Engineering and Analysis Division (4303). Washington, DC.

USGS. [various dates]. National field manual for the collection of water-quality data: U.S. Geological Survey techniques of water-resources investigations, Book 9, Chap. A1-A9. Available online at <http://pubs.water.usgs.gov/twri9A>. U.S. Geological Survey. Accessed February 5, 2008, at <http://water.usgs.gov/owq/FieldManual/index.html#Citation>.

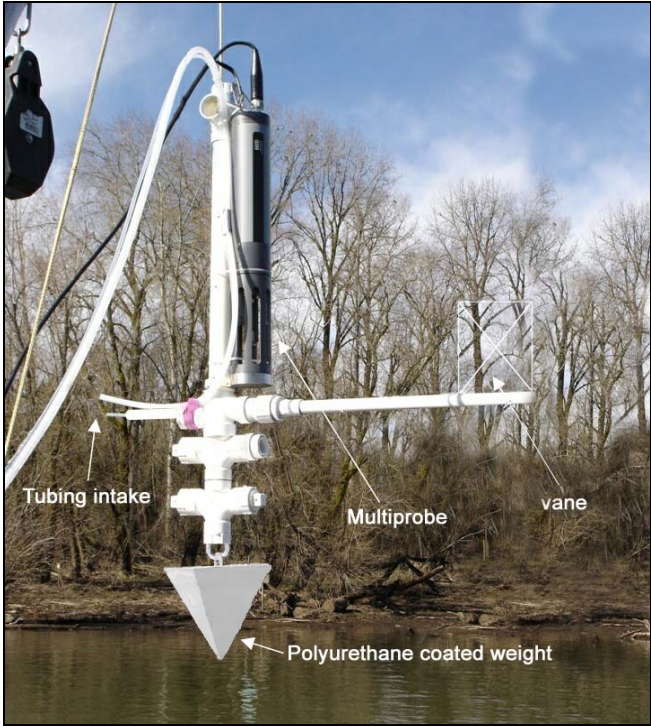


Figure 1a. Near-Surface Water Sampler

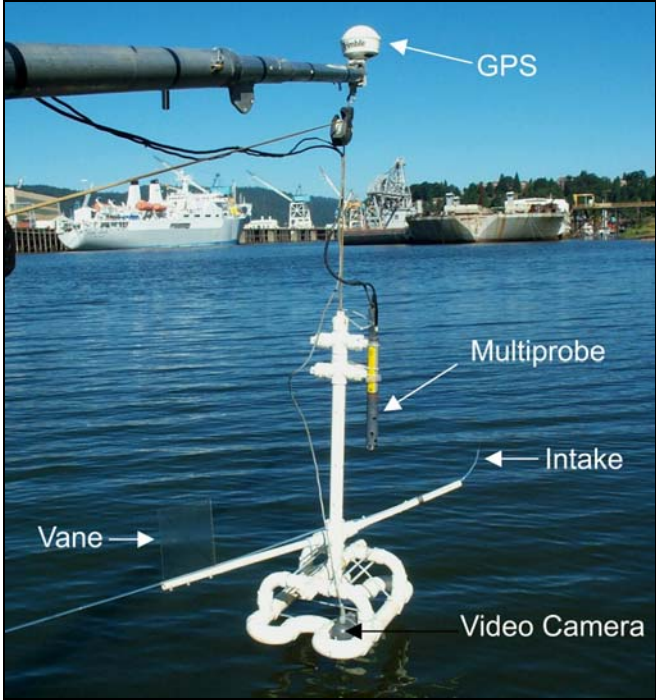


Figure 1b. Near-Bottom Water Sampler



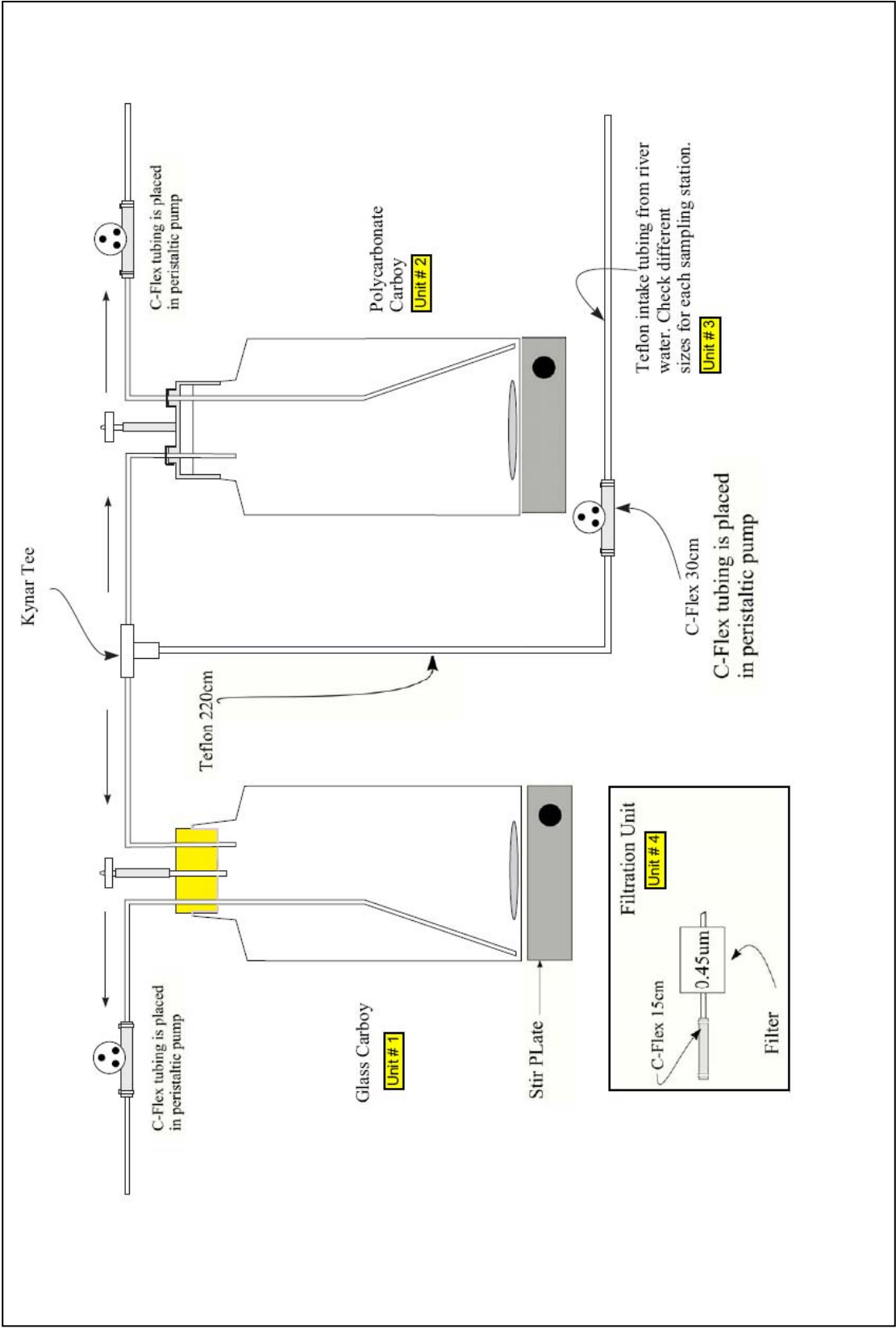
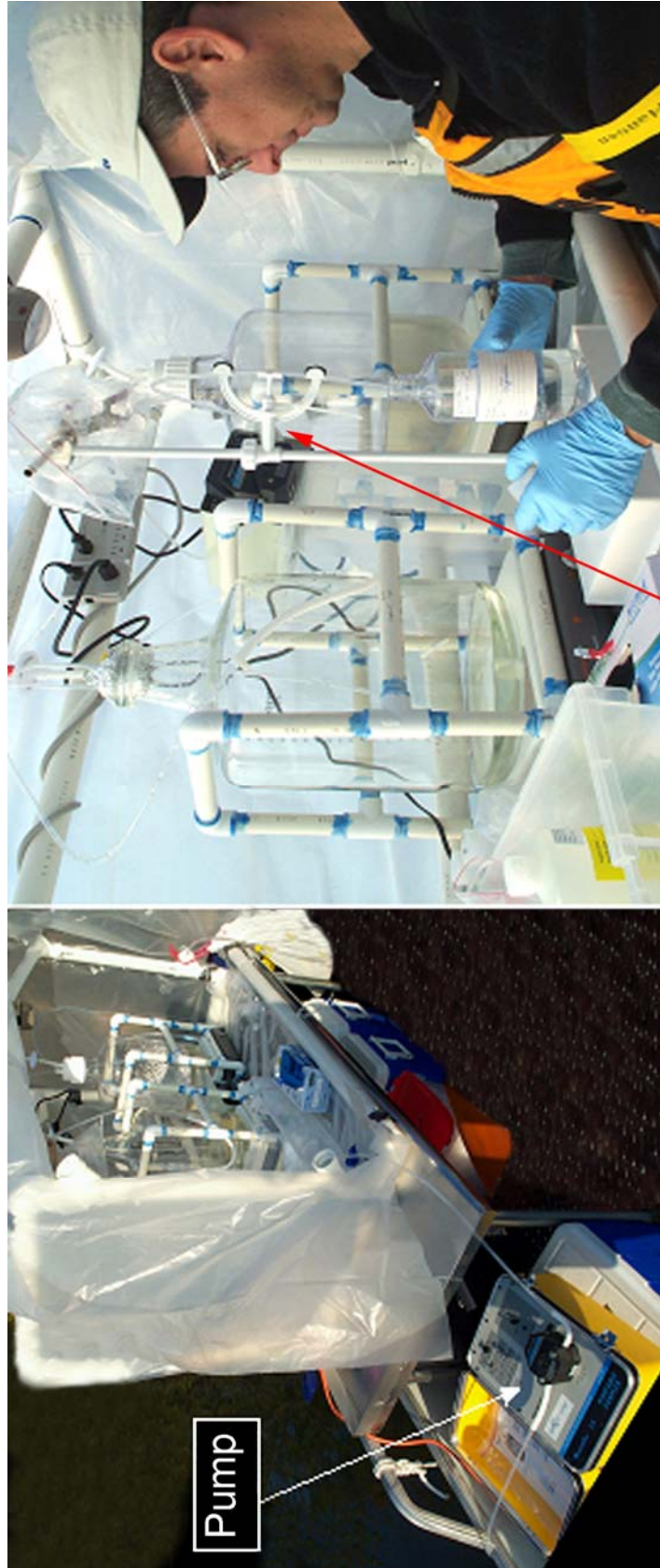


Figure 2. Peristaltic Pump Sampling Apparatus



stand for holding outlet tubing

Figure 3. Peristaltic pump setup in workbook.

## ATTACHMENT 1. CHECKLIST OF SUPPLIES FOR SURFACE WATER SAMPLING WITH PERISTALTIC PUMP

All sampling equipment described here will be sent to Battelle Marine Laboratories at Sequim, Washington, or other approved laboratory for decontamination and assembly prior to sampling. Each unit below shall be wrapped in plastic bags and clearly labeled on the outside in large letters.

### UNIT #1

*For polycarbonate carboys*

#### Teflon

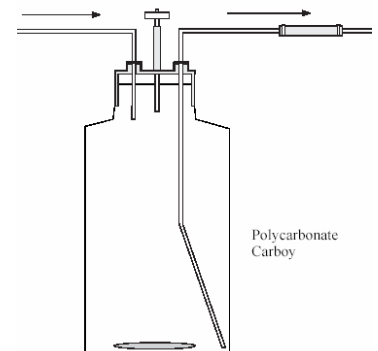
- 50 cm for inflow from Kynar tee into carboy
- 140 cm for outflow from carboy to small peristaltic pump
- 60 cm for outflow from small peristaltic pump to sample bottle

#### C-Flex

- 6 cm for connecting air filter on carboy (additional internal tubing is not needed)
- 30 cm for connecting outflow tubing from carboy to tubing for filling sample bottles

#### Other

- 3-in. stir bar
- Vacu-guard filter
- 2 small plastic zip-ties for C-Flex tubing



The total number of bags labeled UNIT #1 will depend on the number of sampling stations per specific sampling event.

## UNIT #2

*From sample intake tubing to large peristaltic pump to carboys*

### Teflon

220 cm for inflow from large peristaltic pump to Kynar tee

### C-Flex

30 cm for connecting Kynar tee and inflow tubing to variable lengths of sampling intake tubing

### Other

Kynar tee for connecting carboys to intake tubing  
Variable lengths of sampling intake tubing (station dependent) to large peristaltic pump

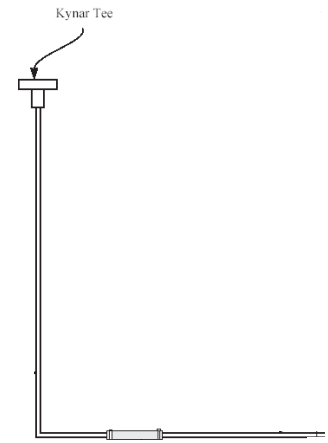
### Teflon

40 to 100 m for near-bottom sampling at transect stations

8 m for near-surface water sampling at any station

15 m for near-bottom sampling at shallow stations

The total number of bags labeled UNIT #2 will depend on the number of sampling stations per specific sampling event.



## UNIT #3

*Set of one filter in line*

### C-Flex

15 cm for connecting the filter to the outflow from small peristaltic pump to sample bottle

### Filter

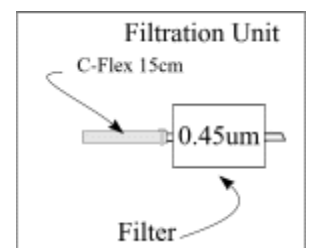
0.45  $\mu\text{m}$  Whatman POLYCAP 36 TF

### Other

Small plastic zip-tie for C-Flex tubing

Loose small plastic zip-tie (extra zip-tie to be placed in bag to connect to carboy outflow)

The total number of bags labeled UNIT #3 will depend on the number of sampling stations per specific sampling event.





## **STANDARD OPERATING PROCEDURE (SOP) SW-06**

### **MEASUREMENT OF SURFACE WATER FIELD PARAMETERS**

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This SOP is based on the procedures outlined in *Field Measurements: U.S. Geological Survey Techniques of Water-Resources Investigations* (Wilde various dates).

#### **SCOPE AND APPLICATION**

Information and general instructions for field measurement of water quality parameters (pH, Eh [oxidation-reduction potential, ORP], specific conductance, dissolved oxygen, and temperature) are presented below. Because the types and complexity of water quality meters available vary widely, calibration and measurement procedures should be conducted in accordance with manufacturer's recommendations for the specific meters used. The following information describes general procedures for the measurement of water quality parameters. Where possible, sampling should be conducted first in areas expected to be least affected by constituents of interest, followed by increasingly affected areas.

#### **EQUIPMENT AND REAGENTS REQUIRED**

- Water quality parameter multimeter or meters specific to parameters of interest (i.e., temperature, dissolved oxygen, pH, transparency, turbidity, salinity, specific conductance, and ORP)
- Calibration solutions and deionized distilled water.

#### **PROCEDURES**

Before any calibration takes place, allow the probe and all calibration solutions to acclimate to the ambient field temperature along for at least 1 hour.

Calibrate the meter(s) in the field at the beginning of each day of field or laboratory work when water quality parameters will be measured. If feasible, meters must be checked for drift with calibration standards after every 4 hours of continuous use. Otherwise, a final check must be done at the end of the sampling event. If drift is evident, recalibrate.

1. Calibrate meter(s) in accordance with manufacturer's instructions using fresh (unused) calibration buffers and standards for each sensor.
2. Check slope reading with specifications (in operating manual) to verify slope is within the manufacturer's specified range.
3. Thoroughly rinse a 500-mL beaker or 8-oz jar with sample water. Discard sample water.
4. Rinse electrodes with sample water to acclimate them.
5. Fill beaker with fresh sample water.
6. Immerse electrodes in sample while swirling the sample, if needed, to provide thorough mixing. Turn on meter(s). If a flow-through cell is used, install probes and connect sample water to bottom port of flow-through cell, directing sample water up through the cell, exiting through the top port. Direct effluent tubing back in the water or into an appropriate container for storage and handling.
7. When the readings have stabilized, record the measurements displayed on the meter. It is important to determine that the correct units and unit scale are displayed on the meter and recorded for each parameter measured. Record and correct any problems encountered during measurement.
8. If available, field measurement results should be compared to previous measurements for quality control.

Several physical and chemical water parameters are best measured in the field because of the unstable nature of the parameter or because the information is needed to direct further sampling. It is frequently preferable to perform these analyses in the field, especially if the samples will not be immediately transported to the analytical laboratory (pH, in particular, should be measured in the field, if feasible). In addition, measurements of temperature and transparency can be collected accurately only in the field. Eight parameter measurements for water are described in the following sections of this SOP.

## Temperature

Measure water temperature with either an alcohol or digital thermometer. It is recommended that mercury thermometers not be used to avoid possible breakage and introduction of mercury into the environment and to remove a source of possible contamination to samples collected for the analysis of mercury. Measure temperature as soon as the sample is collected to obtain a measurement that is an accurate representation of the *in situ* sample temperature. All instruments used to measure temperature should be traceable to a National Institute of Standards and Technology temperature reference. In the case of digital thermometers, follow the calibration procedure recommended by the manufacturer, if provided. Multiprobes in

general contain a temperature probe; check these probes against a calibrated thermometer before use. For more detailed procedures, see discussion in Wilde (2006).

## Dissolved Oxygen

Dissolved oxygen may be measured in the field by either a dissolved oxygen polarographic-membrane type sensor or a luminescent type sensor. Dissolved oxygen can also be measured by a field-portable Winkler titration kit.

It is recommended that calibration be done at temperatures that are at least within 10°C of the ambient water temperature. The smaller the temperature difference is between the environmental water and the calibration chamber, the more accurate the calibration will be.

When using static samples (i.e., water sample collected in a container), protect samples from absorbing oxygen from the atmosphere by using a low or zero-headspace container. If using a meter and probe, calibrate the system according to the manufacturer's procedure prior to use with a zero oxygen standard and a second standard of known oxygen content. Check the second standard by performing a Winkler titration. Other probes are calibrated by percent oxygen saturation in an enclosed container with a small amount of water. When measuring dissolved oxygen with certain polarographic-membrane probe in water samples held inside zero-headspace containers, swirl or stir samples constantly until the reading stabilizes and the measurement is recorded. Stirring the sample is not necessary if a luminescent-sensor is used. For other probes, immerse the probe in the water column and monitor a constant measurement (dynamic measurement) until the readings are stabilized. Once the readings stabilize, record the oxygen concentration readings manually or digitally. For more detailed procedures, see discussion in Lewis (2006).

## pH

The pH of a water column sample can be measured in the field using a pH meter. Calibrate the meter according to manufacturer's specifications with at least two standards of known pH. The pH of these standards should bracket the expected pH at the sampling site. For example, if the pH at the sampling site is expected to be basic (pH 7 to 14), standards of pH 7.00 and 10.00 should be used to calibrate the meter. The pH of the buffer solution is temperature dependent. That is, pH 10 buffers change more per unit change in temperature than do pH 4 buffers. The temperature of buffer solutions must be measured, and temperature-correction factors must be applied before calibration adjustments are made. Calibration and operating procedures differ with instrument systems—check the manufacturer's instructions. If pH measurements at the sampling site do not fall within the initial calibration range, the meter should be recalibrated with appropriate standards and sample pH remeasured for those samples that fell outside the calibration range. For more detailed procedures, see discussion in Wilde et al. (2006).



## Transparency

Water column transparency is measured with a Secchi disk, which is a weighted, black-and-white or all-white disk that is lowered into the water body on a calibrated rope or line.

Always perform these measurements from the side of the boat that faces away from the sun. Lower the disk slowly until it is no longer visible and then raise it until it is visible again. Record the depth, measured from the water surface, in feet or meters. The all-white disk may be preferable when the water transparency is high. However, either disk is acceptable to use.

## Turbidity

Turbidity can be measured in the field on static water samples contained in jars with a field-portable nephelometer (turbidity meter) or *in situ* with a turbidity probe mounted in a multiprobe device. Calibrate the meter prior to use with at least two standards of different but known turbidity (in nephelometric turbidity units, or NTUs). The two standards should bracket the range of turbidity measurements expected at the sampling site.

Perform field analysis for turbidity on static water samples as soon as possible after collection. If immediate analysis is not possible, agitate the sample prior to analysis to resuspend any settled solid material. If the sample temperature increases, air bubbles may form and cause erroneous values.

When performing field analysis for turbidity *in situ*, monitor the turbidity probe constantly with a remote display and record data manually or digitally.

For more detailed procedures, see Anderson (2005).

## Conductivity or Salinity

Salinity can be measured in the field with a salinometer, and conductivity with a conductivity meter. There are two types of conductivity sensors as described below.

- **Contacting-type sensors with electrodes**—Electrodes contained in a dip cell can be suspended in the sample. The cell constant is the distance between electrodes (in centimeters) divided by the effective cross-sectional area of the conducting path (in square centimeters). A cell constant is chosen on the basis of the expected conductivity. The greater the cell constant, the greater the conductivity that can be measured.
- **Electrodeless-type sensors**—Conductivity is measured by inducing an alternating current in a closed loop of solution, and measuring the magnitude of the current. Measuring errors in this type of electrode are minimized because sensors do not have issues with electrode polarization or electrode fouling.

Calibrate the conductivity meter prior to use in accordance with the manufacturer's directions using a standard of known conductivity. The conductivity of the standard should be close to the expected value at the sampling site. When measuring a sample for conductivity, swirl or stir the sample until the meter is stabilized and a measurement is recorded. For more detailed procedures, see Radtke et al. (2005).

Salinity can be automatically calculated from conductivity, temperature, and barometric pressure readings in the same multiprobe and displayed on the meter of most models. Salinity may also be calculated from the measured conductivity and temperature of a sample according to Standard Method 2520B (APHA 1998). Gross salinity measurements may also be taken with a field-portable refractometer. This instrument provides salinity measurements with an accuracy of 1 to 2 parts per thousand. For more detailed procedures, see APHA (1998).

## **ORP or Eh**

ORP or Eh may be measured in the field with an inert metal electrode and read relative to a reference electrode that is immersed in the same medium. For most multiprobe units, the inert metal electrode is a button or ring made of platinum and the Ag/AgCl reference electrode is the same one connected to the pH probe. The readout of the sensor is a voltage (relative to the reference electrode), with positive values (e.g., +300 mV) indicating an oxidizing environment (ability to accept electrons) and negative values (e.g., -300 mV) indicating a reducing environment (ability to donate electrons) (YSI 2005).

ORP and Eh are the same parameters in that both measure the potential of the medium to transfer electrons. However, the ORP reference electrode is made of different material than the Eh standard hydrogen electrode; therefore, a voltage offset needs to be taken into account when converting ORP measurements to Eh values.

More detailed explanation on the theoretical concept, voltage offset conversions, method limitations and interferences can be found in the attached YSI Tech Note (YSI Environmental 2005) and in Nordstrom and Wilde (2005).

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## **ATTACHMENT 1. MEASURING ORP ON YSI 6-SERIES SONDES: TIPS, CAUTIONS AND LIMITATIONS**



## Measuring ORP on YSI 6-Series Sondes: Tips, Cautions and Limitations

### Introduction and Basic Theory

As described in *Standard Methods for the Examination of Water and Wastewater* (Section 2580 B.), ORP is a potentiometric measurement in which the potential (or tendency) of the medium for electron transfer is sensed by an inert metal electrode and read relative to a reference electrode that is immersed in the same medium. This determination can also be referred to as a “redox” measurement (combination of REDuction and OXidation). For most multiparameter monitoring systems, the inert metal electrode is a button or ring made of platinum and the reference electrode is the same one associated with the pH sensor, usually Ag/AgCl. The readout of the sensor is a voltage (relative to the reference electrode), with positive values (e.g., +300 mV vs. Ag/AgCl) indicating an oxidizing environment (ability to accept electrons) and negative values (e.g. -300 mV) indicating a reducing environment (ability to furnish electrons).

The determination of ORP is particularly worthwhile in water which contains a relatively high concentration of a redox-active species, e.g., the salts of many metals ( $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ) and strong oxidizing (chlorine) and reducing (sulfite ion) agents. Thus, ORP can sometimes be utilized to track the metallic pollution in ground or surface water or to determine the chlorine content of wastewater effluent. However, ORP is a nonspecific measurement, i.e., the measured potential is reflective of a combination of the effects of all the dissolved species in the medium. Because of this factor, the measurement of ORP in relatively clean environmental water (ground, surface, estuarine, and marine) has only limited utility unless a predominant redox-active species is known to be present. Users should thus be careful not to “over-interpret” ORP data unless specific information about the site is known.

### ORP vs. Eh: Calibration of ORP Sensors

Many users of YSI 6-series sondes that make field or laboratory redox measurements have questions about the difference between ORP and Eh. In essence, the two parameters are the same in that both quantify the potential of the medium to transfer electrons -- the difference is the reference electrode (and thus the voltage offset) against which the potential of the platinum sensor is reported. Eh is defined as a voltage reading vs. the Standard Hydrogen Electrode (SHE), while ORP is a much less specific term in which the measurement can be made relative to any

practical or theoretical reference electrode, such as Ag/AgCl, calomel, or SHE. Generally, it is not easy to use the SHE in laboratory or field measurements and thus redox readings are made using either the Ag/AgCl or calomel reference electrodes, with Ag/AgCl being more popular in multiparameter water quality instrumentation. Thus, Eh is usually not determined directly. However, the voltages obtained as ORP readings vs. non-SHE electrodes can easily be converted into Eh values by two mechanisms:

- Adding (or subtracting) an offset voltage to the ORP readings obtained vs. a practical reference electrode to account for the fixed difference between the SHE and the other reference system. The offset voltage can easily be obtained for several practical reference electrodes in Table 2580: II of the section of *Standard Methods* on ORP. For example, the potential of Zobell solution vs. the Ag/AgCl reference electrode using 4 M KCl is +228 mV while the same solution read vs. the SHE is +428 mV. Therefore to convert ORP readings taken under these conditions to Eh, simply add 200 mV to the ORP voltage. For example, ORP readings of +150 and -172 mV translate to Eh values of +350 and +28 mV, respectively.
- Using the instrument software to automatically add the offset voltage to the ORP readings as they are displayed or logged. This method is implemented during calibration of the ORP sensor in YSI 6-series sondes. For example, when calibrating an instrument with Zobell solution that has an ORP reading of 228 versus the YSI reference electrode, enter 428 mV at the calibration prompt instead of 228. After the calibration is confirmed, 200 mV (the difference between Ag/AgCl and SHE reference electrodes) will automatically be added to all displayed and logged ORP values, effectively converting them to Eh with no further correction needed. The software in YSI 6-series sondes is likely to interpret the entry of the higher voltage value as an “out of range” calibration error and provide a warning to this effect. As long as the user is knowledgeable about the procedure, the error can be “overridden” with no ill effects.

### Effect of Temperature

The temperature of the water for which ORP is being determined will affect the voltage output of the sensor. This factor definitely needs to be taken into account for calibration and should be considered when reporting field ORP values.

(continued)

For calibration, the following table can be used when using Zobell solution, the YSI-recommended standard. Thus, if the Zobell calibration standard is at 15° C instead of 25° C, enter 241 mV at the calibration prompt instead of 228 mV (the 25° C value which is commonly quoted).

Temperature, C	Zobell Solution Value, mV vs. Ag/AgCl (4 M KCl)
-5	267.0
0	260.5
5	254.0
10	247.5
15	241.0
20	234.5
25	228.0
30	221.5
35	215.0
40	208.5
45	202.0
50	195.5

The user may be able to locate similar temperature-dependence data in the literature for other ORP standards such as Light's Solution and quinhydrone standards in pH buffers.

Temperature will also clearly have an effect on field readings, but, in this case, the variation is usually not definable since the temperature effect depends on the dissolved species responsible for the ORP reading, and these species are usually not known exactly for environmental water. For this reason, ORP readings on YSI 6-series sondes are **not** temperature compensated in any manner. The user must remember that ORP variation in field water could be due to temperature changes rather than analyte compensation. Usually, however, gross changes in ORP (>100 mV) are not due to the effect of temperature.

### Confirming ORP Response

Unlike pH, YSI 6-series sondes only allow a single point calibration for ORP, i.e., an offset adjustment as described above. This is almost always adequate if the ORP sensor has been maintained properly. However, some users like to confirm that their ORP system tracks changes in ORP correctly, in the same way that a pH sensor responds to immersion in pH 7 and pH 10 buffers.

To check the "slope" and response characteristics of the ORP sensor, YSI recommends that the user purchase item number B125 (ORP Calibration Kit) from the manufacturer of one of our ORP sensors:

Sensorex  
Tel. +1 714 895 4344  
Fax. +1 714 894 4839  
Email. info@sensorex.com  
Web. www.sensorex.com

The kit contains solid quinhydrone which, when added to the supplied buffers, yields two solutions with well-defined, but different, ORP values.

### Problems with ORP Sensors

Although based on relatively simple theory, ORP is, unfortunately, also a measurement that can show more problems than other water quality sensors with regard to consistency between different instruments and overall accuracy. In addition, these issues are further complicated in that their extent is likely to depend on both the condition of the sensor and the makeup of the water being tested. The most common problem reported with regard to ORP determination in environmental water is that readings from various instruments (sometimes with exactly the same sensor type and electronics) differ by a significant margin (50-100 mV) even though the sensors are in the same container of water. To make the problem more perplexing, all of the sensors show identical readings in an ORP standard such as Zobell solution. The exact explanation for this paradox is sometimes elusive, but there are at least three possible reasons for its occurrence.

- First, ORP sensors can show a slow response in environmental water if the platinum button of the probe has been contaminated with extraneous material. Common contaminants include hard water deposits, oil/grease, or other organic matter. If the platinum electrodes in the above example are variably contaminated, then some of them (the more contaminated) will be likely to approach potentiometric equilibrium slower. Under this scenario, if left long enough all the sensors would read the same. However, it might take days for the contaminated sensors to reach their final value, and, therefore, they appear in the time frame of a sampling experiment (< 1 hour), to be different. Naturally, if the electrode contaminant is redox-active, either in itself or because it contains redox-active impurities, the reading from that sensor will exhibit erroneous readings that may never change unless the contaminant is removed.

(continued)

- Second, in clean environmental water, there may be very few redox-active species present, and those that are present may be in very low concentration. In many cases, the concentration can be so low that the redox influence of the species is effectively below the detection limit of the method. Under these conditions, the readings will have questionable meaning and could show this type of variation described above. Note that the ORP reading variance associated by this scenario is likely to be exacerbated if any of the electrodes is also contaminated as described above.
- Third, the makeup of the surface composition of the electrode may not be ideal for the measurement in the medium under investigation. While “platinum” ORP electrodes are primarily composed of the metal itself (in a neutral state), it is well known that the surface of the electrode (where the redox action takes place) is coated to varying extents with a molecular layer of platinum oxide (PtO). The Pt/PtO ratio can change over time, depending on the medium in which the probe is stored, and thus the surface of the electrode actually possesses its own potential that can be variable. If this surface potential is similar to the ORP potential of the medium, then electrode response can be sluggish. The cleaning procedure recommended later in this document will result in a surface characterized by a low Pt/PtO ratio and one that possesses a very positive potential. This should be suitable for most environmental measurements.

The fact that similar or identical ORP sensors read differently in environmental water yet the same in Zobell solution is due to the fact that the concentration of redox-active species (ferricyanide/ferrocyanide for Zobell) is much greater in the standards. This higher concentration usually “swamps out” the inconsistencies related to detection limit problems (caused by low amounts of redox-active species) and response time issues (caused by electrode contamination), thus all sensors respond rapidly and read within the YSI specification of +/- 20 mV when in standards.

If you observe inconsistency between different sensors or experience ORP readings which seem unusual for the water being tested with your YSI 6-series multiparameter instrument, YSI recommends the following steps to identify and/or correct the problem:

First, make certain that the pH sensor is functioning properly. The reference electrode of the sonde is common to both pH and ORP sensors and, therefore, if both pH and ORP sensors are malfunctioning this is likely to be the source of the problem. Reference electrode problems usually appear as either total failure or as a slow response in both pH and ORP readings. If

a reference electrode problem is suspected, test the ORP sensor in a standard and make certain that it is within 20-30 mV of the predicted value. If reference electrode performance is indicated, clean the sensor according to the instructions shown below and then retest.

Second, if the sensor performs well in the ORP standard, remove the probe from the sonde and carry out the sequential cleaning process documented in the next section.

### ORP Electrode Cleaning

The following procedure will result in removal of many common contaminants from the platinum ORP electrode. Fouling of the electrode can, however, be deployment-specific, and some contaminants from polluted water may not be dissolved by this method. The use of other solvents and reagents may be possible, but they must be selected carefully so as not to damage the reference electrode or pH glass of the combination sensors nor to leach or dissolve the CPVC body of the probe itself. Consult YSI Customer Service before using cleaning methods other than those documented below.

YSI recommends that the user perform the cleaning/reconditioning operation in the order indicated. Performance can be rechecked at the conclusion of each major section (A, B, and C) and the cleaning discontinued if, at that point, the performance problem has been corrected.

#### Procedure A

1. Soak the probe for 10-15 minutes in clean water containing a few drops of commercial dishwashing liquid.
2. Wipe the platinum button or ring by rubbing with a cotton swab soaked in the cleaning solution. CAUTION: For 6565 probes, be certain not to damage the glass bulb of the combination sensor during this process.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, and then re-rinse with clean water.

#### Procedure B

1. Soak the probe for 20-30 minutes in one molar (1 M) hydrochloric acid (HCl). This reagent can be purchased from most laboratory supply dealers. Be sure to follow the safety instructions supplied with the reagent.
2. Wipe the platinum button by rubbing with a cotton swab soaked in the acid. CAUTION: For 6565 probes, be certain not to damage the glass bulb of the combination sensor during this process.
3. Rinse the probe in clean water, wipe with a cotton swab saturated with clean water, then rerinse with clean water.

*(continued)*

**Procedure C**

1. Soak the probe for approximately 1-2 hours in a 1 to 1 dilution of commercially available chlorine bleach.
2. Rinse the probe with clean water and then soak for at least 1 hour in clean water to remove residual bleach from the reference junction. **CAUTION:** If removal of the chlorine bleach is incomplete, this cleaning reagent can seep into either your calibration standards or measurement media and cause erroneous ORP readings until it is dissipated. Always err on the side of caution in the chlorine bleach removal. Soaking the probe in clean water for periods of time longer than 1 hour can do no harm, however, lesser soaking times can cause problems.
3. Dry the sonde port and probe connector with compressed air and apply a very thin coat of O-ring lubricant to all O-rings before re-installation of the probe. After the probe is reinstalled, place the sensors in Zobell solution and make certain that observed ORP readings stabilize within a few minutes and remain stable for 15-20 minutes.

**Typical ORP Data in Standards and Freshwater**

Probe #	Zobell Reading		
	Initial	1 hour	After Testing
1	228	228	233
2	227	226	227
3	227	227	228
4	224	224	228
5	227	227	228

Table 1. ORP sensor performance in Zobell Solution.

Experiments have been performed at YSI to demonstrate the typical performance of YSI ORP sensors in both standards and in freshwater. Five (5) new 6565 sensors were taken from stock and placed directly into Zobell solution at 22° C. As shown in Table 1 below, all sensors read within 4 mV of each other. The sensors were left in the Zobell solution for 1 hour and the values recorded again. Finally, the sensors were retested in Zobell solution after the entire regimen of testing described below was completed. The values were basically unchanged, demonstrating the stability of the sensor in redox buffer.

The sensors were then rinsed and soaked in DI water and then transferred to tap water that had been diluted with deionized water to a specific conductance of 290 uS/cm and saturated with air. The ORP readings were recorded 1 minute after transfer and then again after 2.5 hours in the low-to-medium conductivity water. Note that all readings are fairly close at 1

minute, with probe 5 showing a somewhat more positive reading. Note also that the discrepancy between probe 5 and the others increased slightly after longer-term exposure to the water sample. Cleaning the ORP platinum sensor of probe 5 with clean water and a cotton swab resulted in a decrease of the reading to 207

Probe #	ORP Reading	
	1 minute	2.5 hours
1	138	178
2	143	161
3	132	177
4	135	169
5	166	221

Table 2. ORP sensor performance in 290 uS/cm natural water.

mV -- significantly closer to the other sensors. Finally, note that all readings increased by an average of about 40 mV after longer-term exposure to the natural water. This stabilization pattern, along with some variation in probe readings, is likely to occur with all ORP sensors when used in environmental water samples. The difference in behavior between Zobell solution and the water sample is striking and demonstrates that a lower accuracy specification must be tolerated in natural water samples than in buffers. (Note that the YSI accuracy specification of +/- 20 mV refers to readings taken in redox standards.) See Table 2 for the data described in this experiment.

The sensors were then cleaned using the 1 M HCl treatment described above, soaked in DI water to remove all acid traces, and then placed back into the 290 uS/cm natural water sample. ORP readings were taken 5 minutes after placing the probes in the water. The calibration of the sensors was then checked in Zobell solution the probes returned to the natural water sample, and the readings recorded after 5 minutes. Results are shown in Table 3.

Probe #	Water Sample 5 minutes post cleaning	Zobell Solution Calibration check	Water Sample 5 minutes post cal check
1	195	233	186
2	188	227	183
3	214	228	184
4	197	228	184
5	280	228	230

Table 3. ORP sensor performance in 290 uS/cm natural water after cleaning sensors with 1 M HCl.

(continued)



Note the following from the data in Table 3:

- The results in the natural water sample are about the same after the cleaning as before -- probe #5 is still significantly higher than the other 4 that are fairly tightly bunched.
- Even after multiple exposures to standards, the natural water sample, and 1 M HCl, the probes (including probe #5) all read effectively the same in Zobell solution.
- Although the effect is relatively minor, the water sample readings are somewhat dependent on the previous reagent to which the probes were exposed. Note that the results are more consistent and slightly lower overall after the probes had been in Zobell solution (column 3) than after they had been in 1 M HCl (column 1).

Most users would consider the performance of probes 1-4 in natural water acceptable in terms of their consistency with one another, but might wonder why probe 5 always seems to read somewhat more positive than the other sensors except in Zobell solution where it has the same reading. Although difficult to prove, the difference is most likely due to a different Pt/PtO ratio on the surface of probe 5. Consistent with this hypothesis, the final experiments indicate that probe 5 responds to ORP changes and that its ORP reading in natural water becomes closer to those of the other four sensors after longer-term exposure to this medium.

In the final testing, the probes were placed in a sodium sulfite solution, a reducing environment that should produce a decrease in the ORP readings. As shown in Table 4, this effect was indeed observed. The probes were then carefully cleaned and returned to the natural water sample for 18 hours and then the ORP values recorded to conclude the test protocol. These final values are found in Table 4.

Probe #	Sodium Sulfite Solution, after 5 minute exposure	Natural Water, after 18 hour exposure
1	135	196
2	125	174
3	140	207
4	120	195
5	95	218

Table 4. ORP sensor performance in sodium sulfite solution and after long-term exposure to natural water.

YSI would consider Probe 5 as an acceptable sensor for use with our 6-series sondes for the following reasons even though it reads an average of 50 mV different from the other sensors tested:

- The sensor responds quickly and shows the proper reading in Zobell solution;
- The sensor's reading in natural water is not radically different (>100 mV) from the other sensors and becomes closer after extended exposure to this medium;
- The sensor tracks changes in ORP properly.

**Summary**

The determination of ORP in environmental water can provide valuable insight into the sample as long as there is a significant concentration of a redox-active species present. However, in the absence of these species, ORP can be a significantly less exact measurement than for most other sensors found in YSI 6-series sondes. The inexactness is usually due to contamination of the electrode surface (either physically or chemically), but can also be due to the lack or low concentration of redox active agent in the environmental water.

The quoted accuracy specification for the YSI ORP sensor (+/- 20 mV) refers to redox- standards, such as Zobell solution, and not to environmental water of variable, and usually unknown, content. In many cases, the +/- 20 mV specification will be met in natural water, but it cannot be guaranteed.

Periodic maintenance of your YSI ORP sensor (6032 or 6565) will increase your field consistency and accuracy, but may not overcome all problems.

The value of ORP in determining the content of environmental water is greatly enhanced if the user has some knowledge or history of the site.

*For additional information please contact*

**YSI Environmental**

Tel. +1 937 767 7241

US 800 897 4151

Fax +1 937 767 1058

Email. [environmental@ysi.com](mailto:environmental@ysi.com)

Web. [www.ysi.com](http://www.ysi.com)

## **ATTACHMENT 2**

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### **FIELD FORMS**



923 Haddonfield Road, Suite 300  
 Cherry Hill, NJ 08002  
 (856) 399-7720

**Log of Boring:**

Project Name: \_\_\_\_\_  
 Project Number: \_\_\_\_\_  
 Logged by: \_\_\_\_\_  
 Date: \_\_\_\_\_

Page \_\_\_\_\_ of \_\_\_\_\_

**SAMPLE INFORMATION**

Sample ID	Time	Sample Type	% Recovery	PID (ppm)	Sheen	Depth (Feet)	Symbol	Soil Description (USCS group name, minor components, color, moisture, additional descriptions)
						--1.0		
						--2.0		
						--3.0		
						--4.0		
						--5.0		
						--6.0		
						--7.0		
						--8.0		
						--9.0		
						--10		
						--12		

**Notes**

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

Drilling Contractor: Walker-Hill Environmental

Equipment: Geoprobe

Sampling Equipment: SS bowls and spoons

Start/End Time: \_\_\_\_\_

Latitude: \_\_\_\_\_

Longitude: \_\_\_\_\_

Location Sketch

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

**Integral Consulting Inc. - Groundwater Sampling Field  
Calibration Log: Laboratory Certification #04032**



Site / Project Number: _____	
Personnel: _____	Instrument (Make/Model): _____
Date: _____	Serial Number: _____
Start Time: _____	End Time: _____

NIST Temperature Calibration <i>(From Rental Company)</i>	Specific Conductance <i>1.413 or 1.000 Solution</i>	Ox-Redux Potential (ORP) <i>(Optional)</i>
Standard: _____ °C	Standard: _____ ms/cm <sup>c</sup>	Standard: _____ mV
Reading: _____ °C	Temp: _____ °C	Temp: _____ °C
Deviation: _____ %	Initial Read: _____ ms/cm <sup>c</sup>	Initial Read: _____ mV
	Cal. Read: _____ ms/cm <sup>c</sup>	Cal. Read: _____ mV
	Perc. Recovery: _____ %	Perc. Recovery: _____ %
Correction Factor: _____ °C	<b>Pass if ± 1%</b>	

Three Point pH Calibration		
Standard: <u>4.</u> S.U.	Standard: <u>7.</u> S.U.	Standard: <u>10.</u> S.U.
Temp: _____ °C	Temp: _____ °C	Temp: _____ °C
Initial Read: _____ S.U.	Initial Read: _____ S.U.	Initial Read: _____ S.U.
Cal. Read: _____ S.U.	Cal. Read: _____ S.U.	Cal. Read: _____ S.U.
Difference: _____ S.U.	Difference: _____ S.U.	Difference: _____ S.U.
<b>Pass if ± 0.05 S.U.</b>	<b>Pass if ± 0.05 S.U.</b>	<b>Pass if ± 0.05 S.U.</b>

pH Initial Check	Turbidity Two Point Calibration	
Standard: <u>7.</u> S.U.	Standard: _____ NTU	Standard: _____ NTU
Temp: _____ °C	Probe Temp: _____ °C	Probe Temp: _____ °C
Reading: _____ S.U.	Initial Read: _____ NTU	Initial Read: _____ NTU
Difference: _____ S.U.	Cal. Read: _____ NTU	Cal. Read: _____ NTU
<b>Pass if ± 0.1 S.U.</b>	Difference: _____ NTU	Difference: _____ NTU
	<b>Pass if ± 1/10</b>	<b>Pass if ± 1/10</b>

Dissolved Oxygen <i>(Use either membrane or optical boxes as applicable)</i>		
Water-Sat. Air Calibration	Zero Check ( <b>Membrane</b> )	Air-Sat. Water Check ( <b>Optical</b> )
Standard: _____ %	Standard: _____ mg/L	Temp: _____ °C
Probe Temp: _____ °C	Probe Temp: _____ °C	True DO (chart): _____ mg/L
Baro. Pressure: _____ in Hg	Reading: _____ mg/L	Reading: _____ mg/L
Initial Read: _____ %	Difference: _____ mg/L	Perc. Recovery: _____ %
Cal. Read: _____ %	<b>Pass if ± 0.3 mg/L</b>	<b>Pass if between 95.8 - 104.8%</b>
Perc. Recovery: _____ %		
Winkler Val: _____ mg/L	Analyst Signature	
Cal. Read: _____ mg/L		
<b>Pass if ± 0.3 mg/L</b>		

# Integral Consulting Inc. - Groundwater Sampling Field Calibration Log: Laboratory Certification #04032



Drift Checks: (every 3 hours)	1ST CHECK		2ND CHECK		3RD CHECK		Pass Criteria
	Reading / Temp	Pass	Reading / Temp	Pass	Reading / Temp	Pass	
Time		--		--		--	-----
pH (7.0), S.U.							± 0.2
Conductivity (span), ms/cm <sup>c</sup>		--		--		--	-----
Turbidity (span), NTU		--		--		--	-----
Dissolved Oxygen (% Sat.)		--		--		--	-----

## Quick Reference Guide & Notes - Refer to Standard Operating Procedures for full details

### RETAIN ALL CALIBRATION DOCUMENTS PROVIDED WITH THE INSTRUMENT FOR 5 YEARS

For grab sampling: duplicate samples shall be collected every 20 samples (at least daily). Integral will record and monitor relative percent difference (RPD).

Winkler calibration conducted by equipment rental company. Additional Winkler calibration details provided in rental documentation.

### Methods

pH - N.J.A.C. 7:18 - 3.3 (a) 3 and/or SM 4500-H B-11

Turbidity - SM2130 B-11

Specific Conductance - N.J.A.C. 7:18-3.3 (a) 6 and/or SM2510 B-2011

Membrane Dissolved Oxygen - N.J.A.C. 7:18-5.2 and 5.5 as appropriate and SM4500 OG-2011

Optical Dissolved Oxygen - HACH 10360-11

Temperature - N.J.A.C. 7:18 - 3.3 (a) 5 and/or SM2550 B-201

### Drift Check Notes

pH check 5 sample/3 hour check required of buffer 7. Record to significant to confirm range.

Optional QA check for DO (% Saturation), Turbidity, and Specific Conductance, but not required.

### Equations

$$Spec. Cond. = \frac{Cond. Reading}{1 + C_{temp} \times (T - 25)}$$

$$\% Recovery = \frac{Read Value}{True Value} * 100\%$$

Cond. Reading = non-specific conductivity reading

C<sub>temp</sub> = 0.0191

T = Temperature at time of reading

### pH Standards Temperature Quick Chart - See SOP for full tables:

Temp °C	0	10	15	20	25	30	35
pH	4.01	4.00	4.00	4.00	4.00	4.01	4.01
pH	7.12	7.06	7.04	7.02	7.00	6.99	6.98
pH	10.20	10.12	10.08	10.04	10.00	9.96	9.92

### Saturated Dissolved Oxygen Quick Chart - See SOP for full tables:

Temp °C	DO mg/L	Temp °C	DO mg/L	Temp °C	DO mg/L	Temp °C	DO mg/L	Temp °C	DO mg/L
0	14.16	17	9.37	22	8.53	27	7.87	32	7.32
5	12.37	18	9.18	23	8.39	28	7.75	33	7.22
10	10.92	19	9.01	24	8.25	29	7.64	34	7.13
15	9.76	20	8.84	25	8.11	30	7.53	35	7.04
16	9.56	21	8.68	26	7.99	31	7.42	40	6.59



<b>Site:</b>	<b>Well Location ID</b>	
<b>Project Number:</b>	<b>Well Permit ID:</b>	<b>DTW (Before Pump Placement):</b>
<b>Date:</b>	<b>Well Depth (ft):</b>	<b>DTW (After Pump Placement):</b>
<b>Sampler:</b>	<b>Well Diameter:</b>	<b>Column Height:</b>
<b>Weather:</b>	<b>Screen Interval:</b>	<b>Start Purge:</b>
	<b>Pump Intake Depth (ft):</b>	<b>Purge Method:</b>
<b>PID Readings (Background):</b>	<b>Pump Type:</b>	<b>Sample Method:</b>
<b>Below Cap:</b>	<b>Tubing Type:</b>	

Time	pH (S.U.)	Specific Conductivity (mS/cm)	Turbidity (NTU)	Dissolved Oxygen (mg/L)	Temp (°C)	ORP (mV)	Purge Rate (mL/min)	Depth to Water (ft bgs)	Notes
	+/- 0.1 S.U.	+/- 3%	+/-10% OR < 5 NTU	+/-10%	+/-3%	+/-10mV	<500 mL/min	+/- 0.3 ft	

Rental Equipment Details (Serial/Vendor):

Comments:





# SURFACE SEDIMENT COLLECTION FORM

Project Name: \_\_\_\_\_ Project #: \_\_\_\_\_ pg \_\_\_ of \_\_\_

Date: \_\_\_\_\_ Weather: \_\_\_\_\_

Crew: \_\_\_\_\_

Sampling Method: \_\_\_\_\_

## Station Positioning Tolerance:

Station: \_\_\_\_\_ Time: \_\_\_\_\_ Attempt #: \_\_\_\_\_ Acceptable Grab:  Yes  No

"On Bottom" Coordinates: \_\_\_\_\_

Estimated Distance from Target Station: \_\_\_\_\_ Acceptable Distance:  Yes  No

Bottom Depth: \_\_\_\_\_ Penetration Depth: \_\_\_\_\_ Photo ID: \_\_\_\_\_

Sample ID: \_\_\_\_\_  discrete  composite

Type:  cobble  gravel  sand [coarse / medium / fine]  silt clay  organic matter  wood/shell fragments

Color:  drab olive  gray  black  brown Apparent Redox Discontinuity Depth: \_\_\_\_\_

Odor:  none  slight  moderate  strong  sulfidic  petroleum  other

## Comments/Description:

Station: \_\_\_\_\_ Time: \_\_\_\_\_ Attempt #: \_\_\_\_\_ Acceptable Grab:  Yes  No

"On Bottom" Coordinates: \_\_\_\_\_

Estimated Distance from Target Station: \_\_\_\_\_ Acceptable Distance:  Yes  No

Bottom Depth: \_\_\_\_\_ Penetration Depth: \_\_\_\_\_ Photo ID: \_\_\_\_\_

Sample ID: \_\_\_\_\_  discrete  composite

Type:  cobble  gravel  sand [coarse / medium / fine]  silt clay  organic matter  wood/shell fragments

Color:  drab olive  gray  black  brown Apparent Redox Discontinuity Depth: \_\_\_\_\_

Odor:  none  slight  moderate  strong  sulfidic  petroleum  other

## Comments/Description: