

# **ALASKA DEPARTMENT OF ENVIRONMENTAL CONSERVATION**

## **DIVISION OF SPILL PREVENTION AND RESPONSE CONTAMINATED SITES PROGRAM**



**Field Sampling Guidance  
January 2022**

# FIELD SAMPLING GUIDANCE

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## 1.0 Introduction

The purpose of the Alaska Department of Environmental Conservation (DEC) Contaminated Sites Program's (CSP) *Field Sampling Guidance* is to provide fundamental guidelines, methods, and equipment options for sample collection at contaminated sites. This document builds on the sampling procedures found in the *Underground Storage Tank (UST) Procedures Manual* (DEC, March 2017), adopted by reference in the 18 Alaska Administrative Code (AAC) 78 regulations. Alternatives to the procedures and equipment described in this guidance may be proposed in project work plans on a site-specific basis.

The *Field Sampling Guidance* can be used for developing site characterization work plans under 18 AAC 75.335, cleanup work plans (including sampling and analysis per 18 AAC 75.355) under 18 AAC 75.360, and corrective action plans (including sampling and analysis per 18 AAC 75.355) under 18 AAC 78.250. Additionally, in accordance with 18 AAC 75.370(b), the *Field Sampling Guidance* can be used to develop work plans for construction projects at contaminated sites. All plans must be reviewed and approved by CSP.

The *Field Sampling Guidance* is not a stand-alone manual and includes web links to other resources. This creates a comprehensive system of tools to guide the environmental professional. This guidance is not intended to be used for site characterization of munitions or unexploded ordnance. Munitions and unexploded ordnance guidance can be found at: <https://www.epa.gov/fedfac/military-munitionsunexploded-ordnance#tab-2>.

Additional CSP guidance documents necessary to work plan development and sampling design and procedures are located at <http://dec.alaska.gov/spar/csp/guidance-forms/> and include, but are not limited to, the following:

- *Guidance on Developing Conceptual Site Models* (January 2017)
- *Underground Storage Tank Procedures Manual* (March 2017)
- *Biogenic Interference and Silica Gel Cleanup - Technical Memorandum* (December 2021)
- *Procedures for Calculating Cumulative Risk* (February 2018)
- *Determining the Fraction of Organic Carbon for Methods Three and Four* (March 2017)
- *Arsenic Technical Memorandum* (March 2009)
- *Vapor Intrusion Guidance* (November 2017)
- *Ecoscoping Guidance: A Tool for Developing an Ecological Conceptual Site Model* (March 2014)
- *Monitoring Well Guidance* (September 2013)
- *Risk Assessment Procedures Manual* (February 2018)
- *Site Characterization Work Plan and Reporting Guidance for Investigation of Contaminated Sites* (March 2017)
- *Minimum Quality Assurance Requirements for Sample Handling, Reports and Laboratory Data Technical Memorandum* (October 2019)
- *Guidance for Evaluating Metals at Contaminated Sites* (August 2018)

Send suggestions on how CSP can improve the *Field Sampling Guidance* to Todd Blessing at [todd.blessing@alaska.gov](mailto:todd.blessing@alaska.gov).

The use of trade names is for descriptive purposes only and does not constitute endorsement of these products by the State of Alaska, DEC, or the Division of Spill Prevention and Response.

## **2.0 Qualifications for Environmental Sampling**

DEC established a set of qualification standards for environmental samplers to ensure sampling, interpretation, and reporting is performed or supervised by experienced and knowledgeable persons. Both 18 AAC 75 and 18 AAC 78 require "qualified environmental professional" status for those who have direct responsibility in investigation and cleanup at a contaminated site, including leaks from regulated underground storage tanks. A qualified environmental professional is defined in 18 AAC 75.333 and 18 AAC 78.088:

A qualified environmental professional means a person who is an impartial third party and who is qualified to perform site characterization and cleanup activities including fate and transport analysis, remediation design and other activities associated with contaminated sites; actively practices in the field of environmental science or another related scientific field including engineering, geology, physical science, hydrology, biology, chemistry, or a related field; has not been found to have falsified environmental data or committed other acts of fraud directly related to environmental work; and meets one or more of the following minimum educational qualifications and experience requirements:

- A. has an undergraduate or graduate degree from a nationally or internationally accredited postsecondary institution in environmental science or engineering, geology, hydrology, physical science, biology, chemistry, or a related field and at least one year of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional completed after the degree described in this paragraph was obtained;
- B. has a four year degree in any field or a two year associates degree in environmental science, geology, hydrology, physical science, biology, chemistry, or a related field from a nationally or internationally accredited postsecondary institution, and at least three years of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional;
- C. is certified as an environmental technician under an apprenticeship program that conforms to the requirements of the U.S. Department of Labor, Employment and Training Administration, 29 CFR Part 29, Labor Standards for the Registration of Apprenticeship Programs, Amendment of Regulations, Final Rule, October 29, 2008, and has at least three additional years of professional experience in contaminated site characterization and cleanup activities under the direct supervision of a qualified environmental professional.

Site characterization and cleanup work plans and reports required under 18 AAC 75 and site assessment, release investigation, and corrective action work plans and reports required under 18 AAC 78 must be prepared by a qualified environmental professional.

In some cases, a “qualified sampler” may conduct the field sampling in accordance with 18 AAC 75 and 18 AAC 78. A qualified sampler is defined in 18 AAC 75.333 and 18 AAC 78.088 and refers to an impartial third party with experience and/or a degree that has the following minimum training:

- A. applied field work involving environmental sample collection of soil, groundwater or surface water associated with coursework for a completed degree in environmental science, engineering, geology, hydrology, physical science, or a related scientific field, at a nationally or internationally accredited postsecondary institution; or
- B. an environmental sampling training program recognized by DEC; and
- C. at least three months of experience in environmental sampling under the direct supervision of a qualified environmental professional.

### 3.0 Sampling Work Plan

It is important to have a defined sampling strategy in the work plan prior to conducting field work. CSP recommends the use of systematic planning to generate data that is of sufficient quality and quantity in order to serve the goals of the investigation. The type of systematic planning described below is called the data quality objective (DQO) process, a systematic planning process based on the scientific method. DQOs help clarify study objectives, define the appropriate type, quantity, and quality of data, and specifies tolerable levels of potential decision errors to answer specific environmental questions and support proper decision making. Using the DQO process helps ensure environmental data collection activities result in sufficient information being collected to make decisions that will meet the goals of the study and leads to more efficient use of resources. DEC staff and the qualified environmental professional project team should agree on project DQOs during the development of the work plan before sampling occurs.

Each work plan or quality assurance project plan (QAPP) submitted to CSP should answer the following questions with regard to quality assurance and the DQO process:

1. What is the problem that needs to be addressed?
2. What are the questions this project should answer? Is there a conceptual site model developed that can guide the questions to be answered?
3. What information is needed to answer the questions? What pollutants are to be evaluated? Are there visual or other signs of pollution that could guide what chemicals need to be evaluated? How will decisions be made (i.e., what rules govern how to answer the questions)?
4. What are the boundaries of the study (i.e., area of concern, media of concern, spatial and temporal variability, and constraints to collection of data)?
5. What is the proposed analytical approach (i.e., equipment needed to collect data, discrete samples versus incremental sampling or statistical evaluation on 95% upper confidence

#### **Work Plan**

**Requirements:** CSP will not accept work plans that reference this guidance in lieu of providing detailed sampling and analysis procedures. Each work plan submitted to CSP should include a description of data quality objectives.



- level of the mean results, screening or cleanup levels, laboratory detection and reporting levels, soil or groundwater parameter data collection to support alternative cleanup level calculations or modeling, target receptors, etc.)? What are the consequences and probabilities of making an incorrect decision based on the proposed analytical approach?
6. What decisions will be made with the results (i.e., initial survey or screening for presence/absence determination, delineate nature and extent of contamination, calculation of exposure point concentration with 95% upper confidence level or maximum concentrations, modeling, calculating alternative cleanup levels, or collection of background data to compare to contaminated site data)? What is the performance or acceptance criteria for qualifying or rejecting analytical data (i.e., method or project specific quality control limits/criteria, method quantitation limits, and decision errors from sampling, measurement)?
  7. What are the methods and procedures for obtaining the data (i.e., number of samples, sample types, collection methods, sample locations and depths, sample handling, preservation, packaging, and analytical methods, etc.)? How do these procedures accomplish the objectives of the project and answer questions?

#### **Standards for Validating and Evaluating Data Usability**

Each work plan or QAPP should discuss the standards or criteria for qualifying or rejecting data. To aid in assessing laboratory data usability, the CSP program recommends adhering to the most current version of EPA's Contract Laboratory Program *National Functional Guidelines for Data Review*. You can access these guidance documents here:

<https://www.epa.gov/clp/superfund-clp-national-functional-guidelines-data-review>. In cases where investigation or cleanup of contamination is occurring at a Department of Defense or a Department of Energy facility, then the consolidated Quality Systems Manual (QSM) for Environmental Laboratories (DOD/DOE, September 2019) could be referenced for data validation and usability assessments. You can access the latest QSM document here: <https://denix.osd.mil/edqw/documents/manuals/qsm-version-5-3-final/>

The full DQO process is described in the EPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, February 2006). This document can be accessed at: <https://www.epa.gov/quality/guidance-systematic-planning-using-data-quality-objectives-process-epa-qag-4>.

Sampling procedures should also be described in detail so the study objectives can be met and the work plan adequately evaluated by CSP. The sampling procedures section should describe the proposed number of samples to be collected per location, considering level of effort, logistical limitations, weather conditions, and other issues that may affect sample integrity. The sampling strategy or design may have flexibility to be adjusted based on conditions in the field. Deviations from the *Field Sampling Guidance* may be approved by CSP on a site-specific basis but should be clearly identified and described in the work plan and report. The document *Guidance on Choosing a Sampling Design for Environmental Data Collection* (EPA, December 2002) available at: <https://www.epa.gov/sites/default/files/2015-06/documents/g5s-final.pdf>, provides detailed information on a number of sampling designs that may be applied to a particular site or support the

data use objectives. Different approaches may be applicable to sites with multiple source areas or to account for contaminant type, fate and transport considerations, or other factors.

CSP will review and approve the work plan based on 18 AAC 75 and 18 AAC 78 regulatory criteria and an evaluation of whether the planned work is likely to provide sufficient information to meet the DQOs and support the site decisions expected as a result of the field work and sampling. For work under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the Resource Conservation and Recovery Act (RCRA), or the Toxic Substances Control Act (TSCA), EPA guidance documents will also be used.

During work plan development, the qualified environmental professional should describe how nondetects are to be evaluated before sampling commences. Refer to CSP's *Guidelines for Treating Non-Detect Values, Data Reduction for Multiple-Detections and Comparison of Quantitation Limits to Cleanup Value* (DEC, April 2017) at: <http://dec.alaska.gov/spar/csp/guidance-forms/> as a guide to treatment of nondetects and comparison of quantitation limits to cleanup values.

Refer to CSP's *Site Characterization Work Plan and Reporting Guidance* (DEC, March 2017) at <http://dec.alaska.gov/spar/csp/guidance-forms/> and the specific requirements outlined in 18 AAC 75.335 and 18 AAC 75.360 for further guidance on general and required work plan elements. These may vary on a site-specific basis. Work plan approval by CSP is required prior to conducting any sampling.

The qualified environmental professional should notify the CSP project manager prior to mobilizing for field activities and must obtain approval prior to implementing field modifications not provided for in the approved work plan. Site-specific field modifications that are not approved by CSP may result in the rejection of site data, qualification of the site data as estimated, and/or a requirement that additional supplemental data be collected. All work plan modifications and decision rationale should be documented in the field log and the final report. While in the field, environmental samplers are required to retain a reference copy of the approved work plan and should have a copy of the CSP approval letter.

## **4.0 Notification To Agencies**

Notification to the CSP is required before any site assessment work is performed for UST closure or change-in-service and may be subject to additional notifications per 18 AAC 78.085.

## **5.0 Soil Sampling**

In accordance with 18 AAC 75 and 18 AAC 78, a responsible party shall characterize the nature and extent of contamination at a site. To achieve this goal, the qualified environmental professional must delineate the extent of soil contamination to the default method two most stringent cleanup levels (i.e., lowest cleanup level for the appropriate climate zone) listed in 18 AAC 75.341, Tables B1 and B2. For hazardous substances listed in Table B1 or, if not listed in Table B1, calculated using the DEC Cleanup Levels Calculator, the most stringent of the human health and migration to groundwater exposure pathway cleanup level for the applicable climate zone should be used for delineation. For petroleum hydrocarbon ranges listed in Table B2, the most stringent of the ingestion, inhalation and

migration to groundwater exposure pathway cleanup level for the applicable climate zone should be used for delineation. For guidance on the delineation of soil contamination in the Arctic Zone, please refer to DEC's *Technical Memorandum on Establishing Arctic Zone Cleanup Levels* (DEC, April 2019) which can be downloaded at: <http://dec.alaska.gov/spar/csp/guidance-forms/>.

Use Appendix F of this *Field Sampling Guidance* for determining what site characterization analytes and methods should be employed for each source type. Please also note that site characterization activities must be conducted so cumulative risk can be evaluated as required under 18 AAC 75.325(g) and 18 AAC 78.600(d).

Under Alaska regulation 18 AAC 75.990, surface soil is defined as soil that extends no more than two feet below the surface, subsurface soil is defined as soil that is more than two feet below the surface.

Deviations from guidance provided in this section may be approved by CSP on a site-specific basis but should be clearly identified and described in the work plan and report.

## **5.1 General Guidelines**

The soil sampling methodology should be clearly described in the work plan and should support data quality objectives and intended site decisions. Unless approved by the CSP project manager on a site-specific basis, all laboratory soil samples should be discrete samples and not composited before analysis, except when approved as part of a plan that includes incremental sampling methodology (ISM), or when required by federal regulations (e.g., TSCA for polychlorinated bi-phenyls (PCB) or RCRA waste disposal characterization). Environmental professionals are encouraged to check with the appropriate EPA office for the most recent federal guidance on compliance with applicable federal regulations.

Judgmental sampling involving the collection of discrete analytical samples based on field screening results is the most common sampling approach used at contaminated sites in Alaska. Therefore, field sampling guidelines and procedures relevant to judgmental sampling are emphasized in this guidance.

Alternatives to discrete sampling, such as incremental sampling methodology (ISM), may be approved by the CSP on a site-specific basis. Alternative sampling approaches can also be designed to perform a statistical analysis of the results. The CSP recommends the use of a systematic random sampling design when statistical analysis of results is proposed for cleanup complete decisions. A general guideline for a systematic random sampling design is to collect a minimum of 30 samples from each source area in order to adequately perform statistical analysis, such as a derivation of a 95% upper confidence limit (UCL). The collection of incremental samples should be collected in accordance with the 2020 Interstate Technology & Regulatory Council (ITRC) *Incremental Sampling Methodology Guidance* (ITRC, October 2020).

For a regulated UST system site assessment, the sampling must meet or exceed the minimum requirements in 18 AAC 78.090.

Soil samples should be collected and analyzed for all applicable contaminants of potential concern using the method specifications listed in Appendix D or in accordance with a site-specific, CSP approved work plan.

Potential seasonal groundwater fluctuations should be assessed at sites where groundwater contamination is a concern. If soil contamination has the potential to extend to seasonal high groundwater, in accordance with a CSP site-specific approved work plan, install short-term or long-term monitoring wells to assess potential groundwater contamination (see groundwater section). For light non-aqueous phase liquid (LNAPL) contaminants collect soil samples above, within, and below the zone of seasonal saturation. For dense non-aqueous phase liquid (DNAPL) or other contaminants, additional sampling of other intervals may be required.

The creation of a preferential contaminant migration pathway during site work may impact groundwater. As necessary, implement precautionary measures to assure the groundwater will be protected (i.e., grouting boreholes and compacting soil). If groundwater is encountered, grout soil borings in accordance with CSP's *Monitoring Well Guidance* (DEC, September 2013).

## 5.2 Field Screening

Field screening supports, and is used in conjunction with, a judgmental sampling approach. Field screening is required for site characterization and assessment of underground storage tanks in accordance with 18 AAC 78.090. Field screening is useful to determine where to collect samples for laboratory analysis, segregate excavated soils, identify release points, and estimate the extent of contamination. The chosen field screening method should differentiate degrees of contamination at the site.

The proposed field screening method(s) and frequency should be stated in the work plan and support the data use objectives.

If applicable, describe minimum field screening device detection/quantitation levels and possible interferences in the work plan.

**Note:** The guidelines in Section 5.2 are appropriate for most petroleum contaminated sites, but may not be appropriate for non-petroleum contaminated sites. Field screening at sites contaminated with metals, PCB, solvents, or other contaminants may be subject to additional requirements and should follow appropriate guidance for those contaminants. Prior to developing a sampling plan for non-petroleum contaminated sites environmental professionals should consult the appropriate guidance and consult with CSP to determine an appropriate sampling approach.

The CSP recommends site-specific correlation studies be conducted to compare field screening results with analytical laboratory results when variable field screening results are anticipated, such as at sites with aged fuel releases or sites with contaminants that have very low cleanup levels. The work plan should describe how the correlation study will be conducted and how the results will be applied during the fieldwork.

The tables below are provided as a general guide to some of the available field screening methods. For each field screening method cited in Table 1 and in Appendices B and C, there may be other types

and sources of field screening equipment, methods, or test kits available. For example, there are numerous companies that manufacture and sell petroleum hydrocarbon immunoassay test kits. Each manufacturer uses similar methods but different techniques to detect and measure petroleum hydrocarbons. These differences may be important to you when selecting a field screening technology for your site. Include for approval in the proposed work plan a discussion of field screening method detection limits and accuracy, along with the expected quantification tolerances for any proposed detection levels used for decision making.

Additional guidance on the methods available can be found in Solid Waste (SW) -846 or through ASTM.

Table 1. Field Screening Methods Guide <sup>1,2</sup>

Type	Media	Contaminants of Concern
Direct Reading Devices photoionization detector (PID), flame ionization detector (FID)	Soil	Volatile Organic Compounds (VOC), Gasoline Range Organics (GRO), Diesel Range Organics (DRO)
Qualitative Physical Screening Methods (stick test, jar shake test, sheen test)	Soil/sediment	Hydrocarbons
Field test kits (Hach®, etc.)	Soil /Water	Metals, PCB, Total Petroleum Hydrocarbons (TPH), Organics
Ultra Violet Fluorescence (site LAB®, etc.)	Soil/Water/ Sediment	Polycyclic Aromatic Hydrocarbons (PAH), DRO, GRO, TPH, PCB
Hanby® Diesel Dog®	Soil/Water	Hydrocarbons, Aromatics
Dexsil®-Petroflag®	Soil	Hydrocarbons
Immunoassay (EnSys, EnviroGard <sup>TM</sup> , RaPID Assay, etc.)	Soil/water	PCB, PAH, Benzene, Toluene, Ethylbenzene and Xylenes (BTEX), TPH, Pesticides, Pentachlorophenol (PCP)
Colorimetric Wet/Gas Chemistry	Soil/Water/ Air	Target specific including petroleum hydrocarbons
UV/ROST technology	Soil	TPH, PAH
Membrane Interface Probe	Soil	TPH, VOC
X-Ray Fluorescence (XRF)	Soil/Sediment	Metals
Field gas chromatography	Soil/Water	Hydrocarbons, VOC, Semivolatile Organic Compounds (SVOC)

Type	Media	Contaminants of Concern
Infrared Spectrophotometry Field Analyzer (Wilks)	Soil/Water	TPH
<sup>1</sup> The CSP does not endorse or recommend any specific brand test kit for use. While Table 1 provides methods available at the time this document was written, it's important to note that new and/or improved methods may become available and should be considered in the work plan design phase of the project. <sup>2</sup> Some field screening procedures have an associated EPA approved method.		

## 5.2.1 Primary Field Screening Methods

### 5.2.1.1 Photoionization Detector and Flame Ionization Detector

Two commonly used field instruments for detecting volatile organic vapors at petroleum sites are PID and FID. Because PID and FID are limited to compounds that readily volatilize, they should not be used as the sole method of field screening for weathered fuels, used oil, or other compounds with low or no volatile components.

Heated headspace organic vapor monitoring involves the measurement of volatile organics emitted from soil samples in a sealed container. The container is typically warmed and then tested for volatile organic vapors using photo- or flame-ionization techniques. The results generated by this method are qualitative to semi-quantitative and are limited to compounds that readily volatilize and are able to be ionized and measured via PID or FID.

Conduct headspace analysis in glass jars or re-sealable polyethylene bags. If using re-sealable polyethylene bags, a blank sample should be tested prior to field screening to account for potential interferences caused by the bags themselves. In addition, the presence of moisture may interfere with instrument readings. Results should be presented in the report and documented in the field notes.

To ensure that field instruments will be properly calibrated and remain operable in the field, the following procedures should be followed:

- PID and FID instruments must be calibrated before each testing session to yield "total organic vapors" in parts per million to a benzene equivalent. The PID instrument should be operated with a lamp source that is able to detect the contaminants of concern, operates at a minimum of 10.6 eV, and is capable of ionizing those contaminants of concern.
- All standards used to calibrate field instruments should meet the minimum requirements for source and purity recommended in the instrument's operation manual.
- If the instrument's operation manual recommends specific calibration requirements for other criteria in calibrating the instrument (such as pH, conductivity, temperature, etc.), those criteria should be adhered to.
- Acceptance criteria for calibration should be determined depending on the potential contaminant(s) and should be within the limits set in the manufacturer's operations manual.
- The dates, times, and results of all calibrations and repairs to field instruments should be recorded in the field record.

- All users of the instrument should be trained in the proper calibration and operation of the instrument and should be required to read the operation manual before initial use.
- Operation, maintenance, and calibration should be performed in accordance with the instrument manufacturer's specifications.
- All users of the instrument should be trained in routine maintenance, including battery and lamp replacement, lamp and sensor cleaning, and battery charging.
- Each instrument's operation and maintenance manual should be present at the site.
- Field instruments should be inspected before departure for the site and also while on site.
- Instrument battery charge should be inspected far enough ahead of time to bring the instrument up to full charge before departure for the site.
- At a minimum, a source of extra batteries and lamps (if applicable) should be readily available.

The following heated headspace field screening procedure should be used:

- Partially fill (one-third to one-half) a glass jar or re-sealable polyethylene bag with the sample to be analyzed. Total capacity of the jar or bag may not be less than eight ounces (approximately 250 mL), but the container should not be so large as to allow vapor diffusion and stratification effects to significantly affect the sample.
- If the sample is collected from a split spoon, after collecting analytical sample, transfer it to the jar or re-sealable polyethylene bag for headspace analysis immediately after opening the split spoon.
- Collect the sample from freshly uncovered soil if it's collected from an excavation or soil stockpile.
- If a jar is used, quickly cover the top with clean aluminum foil or a jar lid. Use screw tops, strong rubber bands, or other methods that will tightly seal the jar. If a re-sealable polyethylene bag is used it should be quickly sealed shut.
- From the time of collection, allow headspace vapors to develop in the container for at least 10 minutes but no longer than one hour.
- Shake or agitate containers for 15 seconds at the beginning and end of the headspace development period to assist volatilization. Temperatures of the headspace should be warmed to at least 40°F (approximately 5°C).
- After headspace development, insert the instrument sampling probe to a point about one-half the headspace depth. The container opening should be minimized and care should be taken to avoid uptake of water droplets and soil particulates.
- After probe insertion, record the highest meter reading. This normally will occur between two and five seconds after probe insertion.
- Complete headspace field screening within one hour from the time of sample collection.
- Document all field screening results in the field record or logbook.
- Do NOT reuse soil from the head space sample in subsequent laboratory samples or analyses; separate samples from undisturbed, freshly exposed soil are to be collected and used for laboratory analyses.

#### **5.2.1.2 Immunoassay**

Immunoassay field screening involves the detection and measurement of petroleum hydrocarbons using specific binding characteristics of antibodies and antigens. The antibodies form antibody/antigen compounds with molecules of specific organic compounds present in the petroleum hydrocarbon mixtures such as gasoline, diesel fuel, and motor oils. Most immunoassay test kits use an enzyme-linked immunosorbent assay (ELISA) process. In this process, the samples being tested are combined with a labeled enzyme that competes for binding antibody sites. The process requires incubation prior to separation of bound and unbound antibodies. The bound antibodies are then quantified using secondary processes.

The immunoassay methods generate quantitative and semi-quantitative results. Most of these methods have been designed to measure the presence and concentration of a variety of petroleum hydrocarbon mixtures. Concentration determinations are based upon a relative response to specific types of organic compounds or molecular structures present in all hydrocarbon mixtures. Therefore, it is possible to monitor for gasoline, diesel, and other hydrocarbon mixtures using immunoassay methods.

Immunoassay methods require methanol extraction of a known mass of soil containing petroleum hydrocarbons. The methanol extract is then introduced to the antibody/antigen reaction to focus the testing process on the appropriate target compounds. Once the antibody/antigen reaction has been terminated, colorimetric or turbidimetric processes are used to quantify the petroleum hydrocarbon mixture present in the soil.

#### **5.2.1.3 Infrared Spectrophotometry**

Infrared spectrophotometry (IRS) is typically used to measure the carbon-hydrogen bonds (C-H bonds) present in all petroleum hydrocarbon mixtures. IRS field screening techniques recommend the addition of silica gel to a known mass of petroleum hydrocarbon impacted soil prior to extraction using a suitable solvent. The soil extract is then analyzed directly following calibration of the infrared spectrophotometer adjusted to the appropriate wavelength to measure the C-H bond emissions. The quantitative results are prepared using the extraction solvent and appropriate petroleum hydrocarbon target analyte. The concentrations are determined based on project-specific data quality objectives and require an understanding of basic chemistry for proper preparation.

#### **5.2.1.4 Colorimetric Wet Chemistry**

Colorimetric test methods employ visible monitoring techniques to identify and quantify the presence of petroleum hydrocarbons. The methods require visual observation and quantification using visual comparison or spectrophotometric equipment. These methods usually employ organic wet chemistry techniques for determination of petroleum hydrocarbons on a qualitative, semi-quantitative, or quantitative basis.

Colorimetric wet chemistry methods require mixing soil containing petroleum hydrocarbons with coloring reagents. The presence of petroleum hydrocarbon mixtures is then determined through visible wavelength spectrophotometry or by visual observance of color in the reaction vessel.

#### **5.2.1.5 Qualitative Physical Screening Methods**



Physical screening methods, such as visual and olfactory screening, are qualitative and provide only basic information related to the presence or absence of petroleum hydrocarbons. However, these should be reported and documented in the field notes if observed. Physical screening methods require little or no preparation prior to a direct observation to evaluate the presence of petroleum hydrocarbons. If other field screening methods do not detect contaminants but visual or olfactory evidence suggests that they are present, soils should be treated as contaminated until analytical results are received.

#### **5.2.1.6 Selection Criteria**

Petroleum hydrocarbon field screening methods use different technologies to measure or respond to the presence of petroleum hydrocarbons. These methods can react differently under similar conditions. To select a field screening method that will provide the user with the desired results, several criteria should be considered and evaluated during the selection process. These criteria include:

- Determination of the target analytes (volatile, semi-volatile, or relatively non-volatile petroleum hydrocarbons);
- Estimation of the target analyte concentration ranges (generally comparable to applicable cleanup standards);
- Determination of the data quality objectives, such as the need for quantitative, semi-quantitative or qualitative data;
- Required expertise to perform the screening analysis; and
- An understanding of the capabilities and limitations of the screening methods. These criteria are discussed in greater detail in the following sections.

#### **5.2.1.7 Target Analytes**

Each field screening method is designed to respond to various petroleum hydrocarbon mixtures or classes of organic compounds. Some screening methods are capable of testing only for volatile organics, while others are capable of measuring higher molecular weight petroleum hydrocarbons. To select an appropriate field screening method, first try to identify the petroleum hydrocarbon mixture in the soil being tested. This is typically established using fixed laboratory analyses and/or prior knowledge of the source of contamination.

#### **5.2.1.8 Multiple Petroleum Hydrocarbon Mixtures**

The presence of multiple petroleum hydrocarbon mixtures complicates the use of field screening methods. The field screening methods are based on the detection of a variety of hydrocarbon mixtures or a combination of the petroleum fractions - GRO, DRO, and RRO. Without knowing which petroleum hydrocarbon mixture(s) are present, a range of field screening methods may need to be used to adequately estimate concentrations and guide analytical sample locations.

Field screening methods have limitations concerning the applicable ranges of concentrations they can detect. The concentration ranges are different for each field screening method. The screening method user should identify project-specific data quality objectives and identify the field screening

method that can meet those objectives. Field screening methods alone are not capable of generating results that correspond directly to the analytical methods required for GRO, DRO and RRO (AK 101, 102 and 103). Instead, the field screening methods are capable of detecting multiple ranges or varying portions of these hydrocarbon mixtures. The user should be familiar with information provided by various equipment and test kit manufacturers to ensure the selected screening method will evaluate the desired petroleum hydrocarbon ranges or mixture.

### 5.2.2 Training and Expertise

Operation of the various field screening methods requires different levels of personnel training and expertise. Some of the simpler field screening methods can easily be completed after reviewing general procedures and becoming familiar with the operation of instrumentation and equipment. Other field screening methods require training and/or support from experienced personnel, test kit manufacturers, or trained chemists. It is important that the operator responsible for the direct reading of instrumentation, test kits, and field-adapted laboratory equipment fully understand the principles used to measure and quantify target analytes. This knowledge allows the operator to maximize the reliability and usability of the data being generated.

### 5.2.3 Capabilities and Limitations

It is important to know the specific capabilities and limitations of the various methods when selecting an appropriate field screening method. It may be important to consult with the equipment/method manufacturers to further investigate the capabilities and limitations for application to particular projects. Some factors that affect the applicability of various field screening methods are listed below, along with examples of the limitations or effects.

**Moisture in soil** may interfere with the operation of direct reading instruments, which may result in erroneous data.

**Natural organic matter** in the soil may bias screening results due to the contribution of organic compounds similar to those present in refined petroleum hydrocarbons of concern.

**Soil types** may interfere with testing procedures and results. Examples are: moist, dense, plastic clay that is not easily broken apart may limit the generation of headspace hydrocarbon vapors for monitoring using a direct reading instrument; organic peat lithologies can introduce significant quantities of natural organics causing high bias in immunoassay screening results; gravel and rock lithologies may decrease the accuracy of all screening methods due to limited sample surface areas, increased sample mass, and the limited sample size required by most screening methods.

**Low temperature and high altitudes** may limit or preclude the operation of some direct reading instruments.

**Temperature fluctuations** may alter the response from field screening instrumentation and equipment requiring frequent calibration.

**Electrical power source stability** is required for operation of some field screening method equipment. Continuous power with limited voltage and current fluctuations is typically required when using electrical equipment requiring an alternating current (AC) power supply.

#### **5.2.3.1 Other Technology Selection Considerations**

**Logistical concerns** require attention when shipping United States Department of Transportation (USDOT) hazardous substances such as methanol, hexane, isobutylene, or other chemicals or compressed gases to project sites. Shippers, including environmental consultants, must ensure that they have received the appropriate training and certification to be able to ship hazardous substances. Some immunoassay methods require low temperature preservation during shipment and storage prior to use.

**Timeframe for testing** should be considered. Some of the field screening methods will allow the user to test hundreds of samples per day, while others will be limited to fewer than 40.

**Cost** will play an important role. The number of samples to be tested and the usability of the data will have a direct bearing on cost feasibility. It is suggested that the user perform a cost-benefit analysis prior to selecting field screening methods.

#### **5.2.4 Selecting Appropriate Field Screening Methods**

Appendix B provides general information for each field screening method category described in this guide. It should be used as a preliminary or initial guide to select the field screening technologies or categories that meet the site-specific data quality objectives and approximate concentration ranges for the contaminants of potential concern.

Once the selection criteria in Appendix B are understood, Appendix C can be used to identify the field screening method category or categories most appropriate for a site. Appendix C further elaborates on the technical and logistical criteria important to selecting a field screening method. Factors affecting accuracy and precision are noted for each category. A relative comparison of the training and desirable expertise for the field operator is noted. General causes of interference and the associated effects on the screening results are described for each category. Finally, other logistical considerations such as waste by-products, transportation, storage, and shelf life are briefly compared.

The task of selecting “the best fit” field screening method can be difficult and is dependent upon site-specific technical data.

### **5.3 Soil Laboratory Analytical Sample Collection**

#### **5.3.1 General Guidelines**

Sample holding times must conform to the specifications in the required analytical method (see Appendix D). When sampling frozen soils, the equipment and techniques described in the following sections may or may not apply. Refer to Appendix E for a list of recommended sampling materials.

### **5.3.2 Excavated Soil Characterization Sampling**

In accordance with 18 AAC 75.360(8) and 18 AAC 75.370(a), known and suspected contaminated soil must be managed and contained separate from soil known or suspected to be free of contamination. Field screening samples and laboratory analytical samples must be collected from all stockpiled soils, including those soils suspected of being free of contamination, except as noted below.

If instruments or other field observations indicate contamination, soil should be separated into stockpiles based on apparent degrees of contamination. Segregate all excavated soils into different stockpiles of apparent degrees of contamination based on field screening results and site observations.

To assist in characterizing excavated soil, use Table 2A to determine the minimum number of screening and laboratory analytical samples to collect from any excavated soils (including overburden soils and soils placed into stockpiles, drums, and other containers) at contaminated sites. Excavated overburden soils should be sampled as any other excavated soil unless it has been demonstrated to CSP's satisfaction that the overburden soils meet the applicable site cleanup levels.

For each stockpile, use Table 2A to determine the appropriate number of field screening and laboratory analytical samples. Table 2A was originally developed for petroleum contaminated sites; however, it may also be appropriate for sites with other types of contaminants if there is a known release source.

Field screening and associated laboratory analytical samples should be collected at different depths and locations to adequately represent soil contaminant heterogeneity and be of sufficient quantity to ensure representativeness.

Field screening samples for volatiles should be collected at least 18 inches beneath the exposed surface of the stockpile unless additional field screening samples are needed to represent soil contaminant heterogeneity. Petroleum contamination can be persistent near the bottom of long-term stockpiles, so it is important that some field screening samples be collected near the base.

For non-petroleum contaminants, or sites without a known source, CSP may require a different frequency of screening and analytical samples depending on data use, contaminant type, site management decisions, remediation goals, and other site-specific factors to determine the proper management of the excavated soil. Sample collection procedures, including frequency should be clearly outlined in a site-specific work plan for all contaminated sites, and should be submitted to CSP for review and approval prior to sampling.

Table 2A. Excavated Soil Sample Collection Guide<sup>1</sup>

By Volume (cubic yards)	Number of Screening Samples	Associated Number of Laboratory Samples
0-10	5	1
11-50	5	2
51-100	1 per 10 cy	3
More than 100	1 per 10 cy, or as the CSP determines necessary	3, plus 1 per each additional 200 cubic yards, or portion thereof, or as the CSP determines necessary
<sup>1</sup> The Table is appropriate for characterizing the levels of petroleum contamination in soil prior to requesting approval for, transport to a treatment or disposal facility, as required by 18 AAC 75.325(i). Consult with CSP for determining the appropriate numbers of field screening and laboratory soil samples for characterizing maximum petroleum concentrations in soil for on-site treatment.		

There are several situations where CSP may exempt the excavated soils from the field screening and laboratory sampling requirements as part of a site-specific work plan. Excavated soils that are clearly contaminated, known to be impacted only by petroleum, not in need of characterization, and will be taken directly to a CSP-approved facility for the treatment of petroleum contaminated soils may be excluded from the field screening and laboratory sampling frequency in Table 2A. Pre-treatment laboratory sampling may be required by the treatment facility to establish contaminants are suitable for treatment.

If the nature of the contamination has been sufficiently determined through a site characterization, CSP may approve of direct hauling of excavated contaminated soil without additional sampling and analysis. There may be other sampling and analysis requirements for landfarming or other types of contaminants and disposal facilities (e.g., RCRA, TSCA, or Class III landfills). DEC guidance on landfarming (DEC, March 2020) can be found at <http://dec.alaska.gov/spar/csp/guidance-forms/>.

Class I landfills and industrial waste landfills with liners and leachate collection systems are permitted to accept soil that is contaminated below a level approved by the DEC Solid Waste Program (SWP). Class I and Class II landfills that do not operate with geomembrane liners may accept polluted soils with prior approval from the SWP. “Low level” polluted soil may be accepted by Class III landfills for beneficial use (or disposal if contaminated with petroleum only), with prior approval by the SWP.

If disposal of contaminated soil at a regulated landfill is anticipated, contact that landfill and the SWP during work plan development to ensure the facility-specific sampling requirements and approvals are met. For a complete discussion of SWP requirements for landfill disposal of polluted soils, please see the links under “polluted soil” at the following URL <https://dec.alaska.gov/eh/solid-waste/how-do-i-dispose-of/>. Please note that no landfill is required to accept polluted soil or other hazardous materials.

Excavated soils which are not taken directly to a landfill or CSP-approved treatment facility may be temporarily stockpiled onsite for sampling and analysis to determine disposal options. Stockpiled soil must be placed on a liner and covered in accordance with 18 AAC 75.370. The footprint or area where soil is stockpiled should be sampled before stockpile placement and after the soil is moved

offsite. 18 AAC 78.605(b) lists the required number of confirmation grab samples for post-treated excavated soil subject to the UST regulations (18 AAC 78).

Surface and sub-surface field screening samples should be collected directly from an excavation area, the center of the excavation equipment bucket, or by using direct push or split spoon methods. Field screening samples should be collected in a manner that minimizes the loss of volatile organic compounds (VOC).

If instruments or other field observations indicate contamination, excavated soil should be segregated and contained based on apparent degrees of contamination in accordance with Chapter 5.3. When sampling soil for volatile organic compounds, remove 2 to 6 inches of soil immediately before sample collection. If the excavation has been open for longer than one hour, remove 6 to 12 inches of soil immediately before collection. Do not collect samples from disturbed soil that has fallen into the bottom of the excavation pit.

For non-volatile samples (metals, PCB, DRO, RRO, and PAH) it may not be necessary to expose fresh soil by removing any overburden, prior to collection.

If excavation depth precludes safely collecting samples from the bottom of the excavation, samples can be collected from the center of an excavation bucket by first removing 4 to 6 inches of soil immediately, prior to collection.

Use Table 2B to determine the minimum number of screening and laboratory analytical samples to collect from a petroleum contaminated excavation. The analytical samples should be selected from the areas with the highest screening sample results.

For non-petroleum contaminants, CSP may require a different frequency of screening and analytical samples depending on data use, contaminant type, site management decisions, remediation goals, and other site-specific factors. Sample frequency and collection procedures for all contaminated sites should be proposed in the site-specific work plan submitted to the CSP for review and approval. To receive a cleanup complete determination, a sufficient number of sample results be collected to demonstrate the cleanup levels have been achieved at all sidewalls and at the base of the excavation, unless otherwise approved by CSP.

Table 2B. Surface/ Excavation Base and Excavation Sidewall Soil Sample Collection Guide<sup>1</sup>

Base or Sidewalls	By Surface Area (square feet)	Number of Screening Samples	Associated Number of Laboratory Samples
Base	0-50	5	1
	51-124	5	2
	125-250	1 per 25 sq. ft.	2
	More than 250	10, plus 1 per additional 100 sq. ft., or as the CSP determines necessary	2, plus 1 sample per additional 250 sq. ft., or portion thereof; or as the CSP determines necessary.
Sidewalls	Any	For each sidewall, 1 per 10 sq. ft. (depth and length), or portion thereof, with field screening sample collection focused on soil horizon(s) demonstrated as most likely to be contaminated.**	Minimum 1 per each sidewall, plus 1 additional sample for each sidewall area over 250 total square feet (depth and length), or portion thereof, at the highest field screening reading in all soil horizons, or as the CSP determines necessary. For example, a 12'x 30' sidewall [360 square feet total] would require 2 laboratory sidewall samples.**

<sup>1</sup>This Table may not be appropriate for identifying the necessary number of field screening or laboratory soil samples of a landspread, landfarm, or other soil treatment facility. For guidance on landfarming, please refer to CSP's Technical Memorandum *Landfarming at Sites in Alaska*.

\*\* Field screening samples and laboratory samples are to be collected within a soil horizon at the area most likely to be contaminated, such as on top of confining layers, at the base of more porous layers, at the groundwater interface, or along any other preferential pathways identified in the field. Consult with the DEC project manager for sampling frequency of sidewalls of 2 feet or less in depth.

### 5.3.3 In-Situ Soils Characterization Sampling

In-situ soil sampling is typically part of site characterization under 18 AAC 75.335 or site assessment and release investigation under 18 AAC 78, and may include samples from the surface or subsurface. Subsurface sampling is generally accomplished by installing soil borings with a drill rig in order to recover soil cores so that sampling can occur at specific depths. However, hand auger samples or test pits may be used as well when data quality objectives can be met via these methods. Sampling of surface soils is often conducted using hand tools unless part of a work plan in which subsurface samples are also collected, in which case surface soils may be sampled from a soil core.

The frequency and location (including depth) of field screening and laboratory analytical samples should be proposed in the work plan submitted to CSP for approval.

When sampling from soil borings two or more laboratory samples are collected from each boring or test pit. Collect samples above, within, and below the zone of seasonal groundwater fluctuation commonly called "smear zone" if light non-aqueous phase liquids (LNAPL) are contaminants of concern. Collect sample intervals within each zone based on field screening results of the soil cores. For dense non-aqueous phase liquids (DNAPL) or other contaminants, additional sampling of other intervals may be required when site conditions warrant.

### 5.3.4 Underground Storage Tank Sites

For a regulated UST system site assessment, the field screening and sampling must meet or exceed the minimum requirements in 18 AAC 78.090. For example, at least one analytical soil sample at each dispenser and at least one analytical soil sample along piping in areas most likely to be impacted must be collected. Before sampling is conducted, an observation of the site's surface must be conducted to assist in determining field sampling approaches and locations. Activities that must be completed during this observation include:

- locating the aboveground components of USTs;
- confirmation of the amount of fuel currently in each tank;
- determination of tank size;
- observation for aboveground utilities;
- underground utility locations (contact utility location centers where available);
- visual inspection for surface indications of releases;
- if practical and no safety hazard exists, check for odor of petroleum in nearby structures (basements); and
- check sumps and access manholes for evidence of pump leakage.

Key areas that must be observed for surface indications of a release include:

- vent pipes and fill holes;
- pavement depressions, buckling, cracks, or patches that indicate subsurface problems have historically occurred;
- cracks or stains at base of pumps; and
- evidence of stressed vegetation that may have resulted from a release or spill.

The results of the site observations must be recorded in a field logbook or another appropriate document. Conduct field screening and confirmation sampling at UST locations as follows:

Tank Area:

- Below the tank, as per Table 2B;
- Adjacent to and below all fill and vent pipes;
- Excavation sidewalls, as per Table 2B;
- For an in-place assessment, no more than 5 feet from the tank; and
- Other areas of suspected contamination.

Piping Run (including vent piping) and Dispensers:

- Within two feet below piping joints, elbows, connections, damaged piping components, and every 10 foot length of piping; if these locations are unknown then screening must occur within 2 feet below original level of piping at a minimum frequency of one field screening sample for every 10 foot length of piping;
- Adjacent to and within 2 feet below all dispensers; and



- Other areas of suspected contamination.

Absence of positive field screening results or those field screening results below a threshold cannot be used as justification for not taking the required number of laboratory analytical samples. If groundwater is encountered, soil samples should be collected from the first six inches of groundwater-saturated soil or the zone of seasonal water table fluctuations in accordance with this document.

### 5.3.5 Volatile Soil Sampling Procedure

When collecting soil samples for VOC analysis, in an effort to minimize volatilization, do not use a soil collection device that causes mixing or unnecessary disturbance of the soil. Do not use an air rotary drill rig for VOC sample collection or use a vacuum truck or air knife within four feet (vertically or horizontally) of where a VOC sample is to be collected.

Core type samples are preferred to reduce the loss of volatiles during sampling. Core samplers used for VOC sample collection should be constructed of non-reactive materials that will minimize loss of VOC in the sample and should be of adequate size to obtain the minimum required soil mass. A large coring device or multiple core samples may be required to obtain the necessary soil mass, e.g., 25-50 grams. Certain soil types and/or site conditions are not amenable to core type devices and sampling procedures. In such cases, a spoon or scoop type sampling method may result in less soil disturbance, more immediate soil field preservation and therefore, less volatile loss.

While soil core samplers are the preferred method for collecting VOC soil samples, other sampling tools may also be acceptable with CSP approval on a site-specific basis. Therefore, detailed sampling procedures should be included in the site work plan submitted to the CSP for review and approval.

Collect and preserve GRO and VOC soil samples immediately upon exposing the soils as follows:

- Collect a minimum of 25 grams of soil with minimum disturbance directly into tared 4 oz or larger jar with a Teflon®-lined septum fused to the lid. Interim storage/containers (e.g., re-sealable polyethylene bags) are not allowed.
- Immediately after collection, carefully add 25 mL aliquot of methanol (methanol should include a surrogate for method AK101) until the sample is submerged and then seal the lid on the jar. This step should be completed as quickly as possible, within approximately 10 seconds of placing the soil in the sample jar. If an extended time period between soil collection and preservation is necessary due to site conditions or safety concerns, this should be specified in CSP site-specific approved work plan, recorded in the field notes and documented in the final report.
- For low level VOC analysis, place a five gram soil sample into a 40 mL vial with 10 mL of deionized water. Quickly brush any soil off the vial threads and immediately seal the vial and freeze the sample to less than 0°C. The sample vial should be placed on its side while being frozen and transported to the laboratory. Consult with CSP regarding approval of low level preservation using sodium bisulfate.
- Do not place tape, including evidence tape, on the sample container directly.

- Cool and retain samples at less than 6°C except for frozen low-level VOC samples.
- Collect a sample of the same material from the same location in an unpreserved jar for percent moisture determination.
- Collect appropriate field and laboratory quality control samples (see Table 6).
- Collect sample parameters in the following order:
  1. Volatile Organic Compounds (VOC, GRO, BTEX),
  2. Semi-volatile organic compounds (SVOC); including pesticides, herbicides, DRO, RRO, and PCB,
  3. Total Organic Carbon (TOC), and
  4. Metals.
- Soils that are frozen in-situ ( $< -7^{\circ}\text{C}$ ) may not be required to be preserved immediately for VOC analysis as specified above. In these cases, the soil should be maintained frozen ( $< -7^{\circ}\text{C}$ ) in appropriate containers and sub-sampled and preserved as soon as practical. The soil should not be thawed prior to sub-sampling and preservation. Sub-sampling and preservation should follow the procedure specified above. The collection, maintenance of frozen soil at temperature, and sub-sampling/preservation procedures should be detailed in a site-specific work plan submitted to the CSP for review and approval.
- The EPA Contract Laboratory Program *Sample Collection Guidelines for Volatile Organic Aromatics (VOAs) in Soil* (EPA, January 2011) may also be required for VOC collection at some sites (Appendix B of EPA 540-R-09-03).

### 5.3.6 Incremental Sampling Methodology

DEC recommends adhering to ITRC's *Incremental Sampling Methodology Guidance* (ITRC, October 2020), found at: <http://www.itrcweb.org> for developing and implementing incremental sampling methodology (ISM) approaches. ISM is not an appropriate methodology for delineating the extent of contamination from point source releases such as a release from an underground storage tank, fuel hydrant system, or pipeline. ISM is appropriate for characterizing contaminant concentrations in a landfarm, landspread, stockpile, or an area of known contamination. It is also an appropriate methodology for characterizing non-point sources of pollution. ISM data are not appropriate for comparing to discrete data using statistics since the sampling schemes are different.

#### ISM Decision Units

DEC shall be consulted prior to determining the ISM decision unit size and shape. Decision unit size and shape shall be determined based on the known or estimated extent of contamination. In general, decision units shall not be over  $\frac{1}{2}$  acre in area and the soil density across the decision unit should be similar. ISM must be conducted in accordance with an approved work plan.

The sampling design chosen should include randomization which allows statistical inferences to be made about a sample population. Common sampling design approaches include simple random, stratified random and systematic random approaches (See ITRC's ISM guidance for details about these designs). A minimum of 30 increments should be collected per ISM sample in a random fashion. The ISM final bulk sample mass should be at least 500 grams to reduce the fundamental error associated with the sampling event. For large decision units (DUs) exceeding 10,000 cubic yards, 50 to 100 increments should be collected per ISM sample. Any vegetation or oversized material removed from the ISM sample should be documented in a field log or field form. ISM samples slated

to be analyzed for analytes other than VOC, DRO, SVOC or GRO can be air dried and sieved to less than 2 mm particle size prior to sub-sampling. Particle size reduction such as grinding can facilitate a reduction in fundamental error and can be approved for metals analysis provided the target analytes are not lost or transformed during the particle size reduction step. Splitting of a bulk ISM sample to be analyzed by two different methods is not recommended. Sampling devices should be decontaminated or disposed of between decision units to avoid cross contamination. Sub-sampling of the ISM sample can be conducted by a laboratory if a standard operating procedure has been approved by DEC. For the collection of ISM samples for the analysis of GRO or VOC, methanol is often used for sample preservation. Large quantities of methanol may be difficult to transport. Therefore, in accordance with an approved work plan, sample increments or composited increments can be collected and preserved with methanol in the field and then individual methanol aliquots can be combined by the laboratory to generate the ISM sample for VOC or GRO analysis.

The probability of underestimating the mean concentration in a decision unit from a single ISM sample result is significant if the decision unit data variability is high (i.e., coefficient of variability greater than 1). The data variability of a decision unit is often not known before ISM is implemented. Therefore, triplicate ISM samples should be collected from at least one decision unit at each site; preferably a decision unit which is expected to exhibit the greatest variability in contaminant concentrations. For sites with greater than 10 decision units, in cases where the geology of a contaminated site is highly variable, or when there is high variability expected between decision units in the types and concentrations of contaminants, DEC may request multiple decision units be sampled in triplicate when ISM is proposed. The heterogeneity of the decision unit can be evaluated by calculating the percent relative standard deviation (RSD) of the triplicates. RSD calculations are discussed in Section 12.6. If the RSD of the triplicate samples exceeds thirty percent, then there is an increased chance a small area with elevated contaminant concentrations was sampled in one of the triplicate samples. If elevated heterogeneity is determined, DEC recommends that the DU size be reduced or more increments be collected in future sampling events.

Triplicate ISM results are used to calculate a standard deviation or coefficient of variation to quantify the variability of decision units results within the study area. An investigator can infer that the variability of the decision unit sampled in triplicate is similar to other adjacent decision units not sampled in triplicate if the geology and contaminants of concern are similar across the study area. The standard deviation or coefficient of variation of the decision unit sampled in triplicate should be evaluated to select an appropriate equation to calculate a 95% UCL of the mean. The calculated UCL should then be compared to DEC cleanup levels. The Student's t-test method should not be used to calculate a UCL if the coefficient of variability is greater than 0.23. In cases where the coefficient of variability is greater than 0.23, the Chebyshev equation should be used to calculate the UCL.

### **5.3.7 Total Organic Carbon**

The amount of organic carbon present in the soil affects the amount of contamination that can be held by the soil reducing the potential for it to migrate to the groundwater. CSP allows for the collection and use of Total Organic Carbon (TOC) samples in order to determine a site-specific cleanup level. Refer to CSP's technical memorandum, *Determining the Fraction of Organic Carbon for Methods Three and Four* (DEC, March 2017) for requirements.

### 5.3.8 Sampling Requirements for Naturally Occurring Compounds

Naturally occurring inorganic compounds may be found in concentrations above the regulatory cleanup levels in 18 AAC 75.341(c) Table B1 and 18 AAC 75.345, Table C. The presence of inorganic compounds may be considered naturally occurring if no known or suspected anthropogenic inorganic contaminant sources are present. See CSP's *Guidance for Evaluating Metals at Contaminated Sites* (DEC, August 2018) for additional information.

CSP also recommends the use of EPA's *Guidance for Comparing Background and Chemical Concentrations in Soil for CERCLA Sites* (EPA, September 2002), for sampling and proposing site-specific background concentrations.

Naturally occurring organic material is present in many Alaskan soils. Biogenic interference is the term that is used to describe the naturally occurring organic material that is quantified and reported as DRO and/or RRO in accordance with the AK102 and AK103 methods. For more information see CSP's technical memorandum, *Biogenic Interference and Silica Gel Cleanup Technical Memorandum* (DEC, December 2021).

### 5.3.9 Sampling Requirements for Environmental Molecular Diagnostics

Environmental Molecular Diagnostics (EMD) is a term that describes a variety of techniques to analyze biological and chemical features of environmental processes in soils, sediments, water, and air. These techniques focus on the analysis of stable chemical isotopes and biological molecules such as nucleic acids (e.g., DNA or RNA) or enzymes. Most environmental media (soil, sediments, groundwater, etc.) can be sampled for EMD analyses, and sampling typically requires the same equipment and collection containers as traditional soil or groundwater sampling. However, additional sampling requirements (e.g., maintenance of sterility or use of specialized passive samplers) may also need to be considered. For more information, see ITRC's Technology Overview, at: [itrcweb.org](http://itrcweb.org).

## 5.4 Soil Sampling Equipment

### 5.4.1 Scoop/Trowel/Stainless Steel Spoon

A trowel, scoop (Figure 1), or stainless steel spoon may be used to collect soil samples. They can also be used for homogenizing soil or for collecting a variety of other waste samples. Scoops come in different sizes and makes. Some are coated with chrome paint, which can peel off and get into the sample: these are unacceptable. Stainless steel scoops are preferred; however, scoops made from alternative materials may be applicable in certain instances (e.g., polyethylene for trace element sampling in sediments).

Samples can be put directly into sample containers or be processed through sieves to acquire the desired grain size. Stainless steel trowels and scoops can be purchased from scientific or environmental equipment supply houses.



Figure 1. Stainless Steel Scoop. (Photograph by D.Dibblee)

#### Procedures for Use:

- At specified intervals, take small, equal portions of sample from the surface and immediately below the surface.
- Transfer samples into laboratory cleaned sample bottles and follow procedures for preservation and transport.

#### Advantages:

- Easy to use and clean.

#### Disadvantages:

- Not preferred for volatile organic sample collection due to loss of volatiles.

### 5.4.2 Bucket Auger

The bucket auger (Figure 2) consists of a stainless steel cylindrical body with sharpened spiral blades on the bottom and a framework above allowing for extension rod and T-handle attachments. When the tool is rotated clockwise by its T-handle, it advances downward as it cuts into the soil and moves loosened soil upward where it's captured in the cylindrical body.

Cutting diameters vary. The overall length of an auger is about 12 inches and extensions can extend the sample depth to several feet. There are three general types of augers available: sand, clay/mud, and augers for more typical mixed soils.



Figure 2. Bucket Augers. (Photographs publish with permission by Art's Manufacturing and Supply)

Depending on soil characteristics, choose the auger best suited for your needs. These tools can be purchased from scientific or forestry equipment supply houses. The auger is particularly useful in collecting soil samples at depths greater than 8 cm (3 in.).

This sampler destroys the cohesive structure of soil and clear distinction between soil collected near the surface or toward the bottom may not be readily apparent as a result of the mixing effect. Due to lost volatiles, a bucket auger should not be used when sampling for VOCs unless specifically approved in a CSP site-specific work plan. Bucket augers are acceptable for inorganic analysis.

Additional auger flights can be used to increase the depth obtainable by the unit. The bucket auger is used to bore just above the desired sampling depth. A coring device, smaller in diameter than the auger flight, is then used to obtain the sample from undisturbed soil at the base of the augured hole.

#### Procedures for Use:

- Remove unnecessary rocks, twigs, and other non-soil materials from selected sampling point.
- Attach the bucket and handle to an extension rod.
- Begin turning the auger with a clockwise motion and continue until the desired sampling depth is obtained.
- Transfer the sample into laboratory cleaned sample containers using a decontaminated stainless steel spoon or trowel.
- When collecting samples at depths greater than 12 inches, it's advisable to discard one-half inch of material in the top portion of the auger due to cave-in.
- Follow procedures for transport.

#### Advantages:

- Relatively speedy operation for subsurface samples.

#### Disadvantages:

- Don't use for volatile organic sample collection, unless approved by CSP in a site-specific work plan.

### 5.4.3 Soil Coring Device

A soil-coring device (Figure 3) consists of a stainless steel, machined split-cylinder with threaded ends, cutting shoe and end cap with a slide hammer used for advancement into the soil. The cutting shoe and end caps of the corer are also constructed of stainless steel. Use of a plastic collection tube and soil-retaining basket is optional.



Figure 3. Soil Coring Device (Photograph by J. Schoenleber)

Once the desired depth is reached, the slide hammer can be used to assist in pulling back the device. Caution should be used when back-hammering so as not to loosen soil captured within the barrel if a liner/retaining basket is not used. This device may be used in conjunction with a soil auger if core analysis of depth profiles needs to be performed.

Once opened, collect a soil sample for volatile organic analysis using a soil core device or other appropriate sampler. Subsequently, field screen the remaining core with a PID or FID as needed.



#### Procedures for Use:

- Assemble the split barrel and screw on cutting shoe and end caps. Liner and basket retainers are optional.
- Place the sampler in position with the bit touching the ground.
- Drive with slide hammer until unit is completely advanced. Avoid sample compression.
- After reaching the required depth, use the slide hammer to back out device using caution so as not to lose sample.
- Remove both ends and tap barrel to break open split sections.
- Use a utility hook knife to open plastic liner.
- For volatile organic analysis, use a soil core device or other appropriate sampler to collect the sample prior to preservation.
- Record visual observations and field screening data in boring log.
- Follow procedures for transport.

Soil coring devices should be of stainless steel construction.

#### Advantages:

- Can be used in various substances.
- Core sample remains relatively intact.
- Bit is replaceable.

#### Disadvantages:

- Depth restrictions.
- Not useful in rocky or tightly packed soils.

### **5.4.4 Split Spoon Sampler**

A split spoon sampler (Figure 4) is used to collect representative soil samples at depth. The sampler itself is carbon or stainless steel tubing split longitudinally and equipped with a drive shoe and a drive head. These are available in a variety of lengths and diameters and are typically advanced by blows of a hammer dropped from a drill rig mast. The weight and throw of the hammer varies by drill rig.



*Figure 4. Split Spoon Sampler (Photograph by D. Dibblee)*

#### Procedures for Use:

- Assemble the sampler by aligning both sides of the barrel and then screwing the drive shoe with retainer on the bottom and the heavier headpiece on top.
- Drive the tube utilizing a sledgehammer or well drilling rig if available. Do not drive past the bottom of the headpiece as this will result in compression of the sample.
- Record the length of the tube that penetrated the material being sampled, the weight of the hammer and distance dropped and the number of blows required to obtain this depth.
- Once soil core is acquired, if collecting samples for analyses of volatile compounds, collect and preserve samples in accordance with Appendix D. Conduct field screening readings with PID/FID. Field screening should be conducted in accordance with Section 5.2 of this document.
- Volatile samples should be collected within two minutes of core retrieval.
- Collect additional samples for non-volatile analyses as necessary.

When split tube sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D1586 *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM, 2018).

#### Advantages:

- Easily available.
- Strong.
- Ideal for split sample collection.

#### Disadvantages:

- Requires drilling or tripod for deeper samples.
- Requires split spoon decontamination after each sample.

### **5.4.5 Shelby Tube Sampler**

A Shelby tube is used mainly for obtaining geological information but may be used in obtaining samples for chemical analysis. The Shelby tube consists of a thin walled tube with a tapered cutting head. This allows the sampler to penetrate the soil and aids in retaining the sample in the tube after the tube is advanced (without excessive force) to the desired depth.





Figure 5. Shelby Tube Sampler (photo taken from Diedrich Drill, Inc.)

#### Procedures for Use:

- Place the sampler in a perpendicular position on the material to be sampled.
- Push the tube into the soil by a continuous and rapid motion, without impact or twisting.
- In no instance should the tube be pushed further than the length provided for the soil sample.
- Let sit for a few minutes to allow soils to expand in the tube.
- Before pulling out the tube, rotate the tube at least two revolutions to shear off the sample at the bottom. If the sample is to be shipped for further geologic analysis, the tube should be appropriately prepared for shipment. Generally, this is accomplished by sealing the ends of the tube with wax in order to preserve the moisture content. In such instances, the procedures and preparation for shipment should be in accordance with ASTM D1586 *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils* (ASTM, 2018).

#### Advantages:

- Inexpensive.
- Tube may be used to ship the sample without disturbing the sample.
- Provides core sample.
- Easily cleaned.

#### Disadvantages:

- Sometimes difficult to extract sample.
- Not durable when rocky soils are encountered.

### **5.4.6 Soil Core Samplers (VOC)**

While soil core samplers are the preferred method for collecting VOC samples, other sampling tools may also be acceptable, with CSP approval on a site-specific basis.

There are a number of soil core sampling devices available for VOC sample collection which are approved for EPA method 5035A. The En Core® sampler, or equivalent brand of soil core sampler, is acceptable to collect soil samples for VOC analysis as described in ASTM D6418-09 *Standard*

*Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis* (ASTM, 2009). These devices are used to collect a specific soil sample mass for volatile organic analysis in a manner that minimizes loss of contaminants due to volatilization, biodegradation, or both. In performing the ASTM collection procedure, the integrity of the soil sample structure is maintained during sample collection, preservation, storage, and transfer in the laboratory for analysis. The sample is expelled directly from the coring body/storage chamber into the appropriate container for preservation without disrupting the integrity of the sample and as a result has limited exposure to the atmosphere during the collection, storage, and transfer process. Methanol field preservation is required for all volatiles soil analysis. Alternate low level volatile collection and analysis techniques per EPA SW846 Method 5035A can be approved by the CSP on a site-specific basis.

Below are examples of coring devices for collecting soil to be tested for volatile contaminants. Soil is extruded from sampler after collection and placed in a container and preserved with methanol.



Figure 6. Examples of Soil Coring Devices: Core N' One™ tool Soil Moisture Equipment Corp. 0200 Soil Core Sampler

Procedures for Use (En Core® provided as an example only):

- Open foil package containing 5-gram En Core®.
- Insert 5-gram Teflon® sampler into En Core®.
- DO NOT pull plunger back prior to use.
- Set device aside on a clean surface.

T-handle:

- In a controlled setting, open coring device and expose core for field screening with direct reading instrument.
- Once the sample increment is identified, carefully prepare soil core surface for sub-core sampling by scraping away a small portion of soil with a stainless steel spatula.
- Position En Core® with T-handle squarely over the prepared surface and press into soil to a depth of approximately 5/8" to achieve 5-gram sample.
- Extrude core into sample jar with methanol preservative and **repeat as necessary to obtain the required sample mass.**

Advantages:

- Engineered to maintain integrity of soil sample without loss of volatile organics.

Disadvantages:

- Plunger is designed to open as it is pressed into the soil core. Depending on the cohesive nature of the material being sampled, obtaining a full sample in one movement may be difficult.
- Cores consisting of small rocks, shale, cobble, tight clays, peat/tundra, or similar material cannot be effectively sampled. If soil matrix is not amenable, other sampling methods may be proposed in the work plan for CSP review and approval on a site-specific basis.
- Depending on the size of the core sampling device and the required sample mass, multiple cores may be required, resulting in additional sample handling and possible VOC loss.

### **5.4.7 Power Auger**

The power auger is not a tool for sample collection, in and of itself. Instead, a power auger is used in lieu of a bucket auger to reach the depth of a desired sample interval. The power auger is composed of a length of auger flight, usually three feet; attached to a power source which turns the auger either hydraulically or mechanically. Various sizes and types of power sources are available, from one man to equipment mounted units.

Additional auger flights can be used to increase the depth obtainable by the unit. The power auger is used to bore just above the desired sampling depth. A coring device, smaller in diameter than the auger flight, is then used to obtain the sample from undisturbed soil at the base of the augured hole.

Advantages:

- Reduces sampling time.
- Inexpensive.

Disadvantages:

- Use of gasoline powered engine increases possibility of contamination of sample.
- Not useful in rocky soils.
- Extensive decontamination procedure (high pressure, hot water cleaning of auger flights).

### **5.4.8 Direct Push Technology**

Use of Direct Push (DP) Technology to obtain soil samples is widely accepted. The relative ease to collect minimally disturbed soil cores at the surface or at depth plus the ability to provide a wide array of geotechnical options has made this an attractive system. While various manufacturers make and distribute their own equipment and accessories, the same general principles still apply when collecting soil samples.

Direct push systems use hollow steel rods to advance a probe or sampling tool. The rods are typically 3-5 feet long and have treaded ends. As the DP rods are pushed, hammered and/or vibrated into the ground, new sections are added to reach target depths. There are two types of rod systems, single rod, and dual rod/cased systems. Both systems allow for the collection of soil, soil gas and groundwater samples. Single rod systems use only one string of rods to connect to the probe or sample tool and the rods are removed from the hole each time a sample is collected. Cased systems or dual rod systems advance an outer tube (casing) and a separate inner sampling rod (sometimes referred to as piston samplers) simultaneously. When sample depths are reached the inner sample rod and sample core are removed, and the outer drive casing remains in the ground and keeps the hole open. The inner rod can then be refitted with a new sample sleeve, then re-inserted to collect the next sample interval. For more information related to direct push technology, go to the following web site:

<http://geoprobe.com/>

#### Procedures for Use:

- Hammer sampling barrel to desired sampling interval and remove.
- Use sleeve caps to seal ends of core section sleeves to reduce loss of volatiles while awaiting sample collection.
- Volatile samples should be collected within two minutes of core retrieval.
- Open the sampling barrel, remove the plastic sleeve containing the core and cut open the sleeve.
- If collecting samples for analyses of volatile compounds, use appropriate DP rod system and methods to reduce error as described below. Collect and preserve samples in accordance with Appendix D and below.
- Conduct field screening readings with PID/FID. Field screening should be conducted in accordance with Section 5.2 of this document.
- Collect additional samples for non-volatile analyses as necessary.

#### Sample Collection by Direct Push:

The potential for loss of volatiles resulting in non-representative samples will depend on both the type of soil sampler and the type of DP rod system. A major concern is for non-sealed samplers that have an open bottom which may, when used with single-rod systems, allow them to collect soil that has sloughed from an upper section of the probe hole; therefore, they may collect samples that are not representative of the sampling zone. If the sloughed soil contains contaminants, an incorrect conclusion could be made regarding the presence of contaminants at the target interval. Alternatively, if the overlying soil is less contaminated than the soil in the targeted interval, erroneously low concentrations could be indicated. As a result, non-sealed samplers should not be used with single-rod DP systems where contaminated soils are present. In such cases, piston samplers (Macro Core) are the only appropriate soil samplers.

Care should be taken to keep drilling rates/rod removal rates slow enough that water has a chance to drain through weep holes in the sheath drive head. Ensure that water does not wash back through the core and preferentially remove fines from the sample.

Care should be taken to coordinate drilling and sampling rates so that core sections are not left waiting to be sampled while exposed to air.

Ensure that core recoveries are appropriate. Sample core recovery may be lower in cores from greater depth. Loss of core and incomplete core sections showing slough and/or loss of fines are cause for recollecting the sample interval.

#### Advantages:

- Allows for continuous sampling.
- Profiling and collection of soils over large areas can be accomplished in less time. Efficient access to remote locations due to equipment size and mobility.
- Direct push techniques produce a minimal amount of investigation-derived waste material compared to most other drilling methods.

#### Disadvantages:

- Direct push sampling is limited to soils and unconsolidated materials that can be penetrated with available equipment.
- Decontamination of reusable equipment is required between boreholes.

General guidance on the construction of temporary wells installed via direct push technology can be referenced through ASTM D6001-96 *Direct Push Water Sampling for Geoenvironmental Investigations* (ASTM, 2002), and via the following Internet links:

<http://www2.epa.gov/superfund>

<http://www2.epa.gov/ust>

<http://geoprobe.com/>

#### Additional Considerations:

When collecting soil samples for EMD analyses of the microbial community, sterility of sampling tools and containers needs to be maintained using aseptic techniques; if impractical, contamination from other locations or sampling tools should be minimized. Sampling equipment may need to be decontaminated with disinfectant prior to sampling a new location. Use of sample blanks can provide information on the presence of contaminating microorganisms introduced during sampling.

## **6.0 Groundwater Sampling**

When conducting groundwater sampling, care should be taken to ensure that the sample is not altered or contaminated by the sampling equipment, sampling process, or the sample handling procedure. Sampling should be targeted at the interval(s) within the water column based on the physical characteristics of the contaminant. It is important to have representative samples collected from the same depth interval to evaluate trends between sampling events. Deviations from this section may be approved by CSP on a site-specific basis and should be clearly identified and discussed in the work plan and report.

## 6.1 General Guidelines

In accordance with 18 AAC 75.335, the extent of groundwater contamination that exceeds the groundwater cleanup levels of 18 AAC 75.345 Table C must be determined. Unless otherwise directed by CSP, if groundwater monitoring wells are required for delineation of contamination, the installation of the groundwater monitoring well should be performed in accordance with 18 AAC 78.615(b), and the following procedures should be used:

- If the direction of groundwater flow is known, at least three monitoring wells should be installed and sampled, one upgradient and two downgradient of the potential contamination source;
- If the direction of groundwater flow is unknown, it is recommended that the number of wells installed be sufficient to characterize the groundwater flow using horizontal and vertical control measures; at least three monitoring wells should be installed and sampled;
- Well drilling equipment should be decontaminated as outlined in Section 12.8 before drilling at each new location; and
- Wells should be driven with a hollow stem auger, cable drill, or direct push drill rig.

For monitoring well design, construction, development, maintenance, and decommissioning information, refer to CSP's *Monitoring Well Guidance* (DEC, September 2013). During monitoring well installation, the grout needs be allowed to set for a minimum of 24 hours before the surface pad and protective casing are installed unless approved by CSP on a site-specific basis. The surface pad should be allowed to cure for a minimum of 24 hours before the monitoring well is developed unless approved by CSP on a site-specific basis. CSP recognizes that remote site work may make these installation times impractical.

Except when compressed air is being used for well development, monitoring well sampling can be initiated as soon as the groundwater has re-equilibrated, is free of visible sediment, water quality parameters have stabilized (see below), or 24 hours have passed following development. In the case of using compressed air to develop a monitoring well, wait at least 7 days before sampling. If sampling is conducted prior to the prescribed waiting period, the data for that sampling event may be considered screening level only and subsequent sampling event(s) may be required. Well screen intervals and length of screens should be approved by CSP on a site-specific basis and need to be designed so that samples are representative of the overall groundwater contaminant plume.

The groundwater sampling methodology should be described in the work plan, should support the intended data use to make site decisions, and be approved by CSP. Groundwater samples need to be collected and analyzed for all appropriate contaminants of concern using the method specifications listed in Appendix E and the approved work plan, unless other methods are approved by CSP on a site-specific basis. Well screen intervals and length of screens should be approved and need to be designed so that samples are representative of the intended portion of the aquifer as needed to define or monitor impacted groundwater.

Before sampling, the monitoring well should be developed and the depth from the top of the well casing to groundwater measured after static conditions have returned to the well. Horizontal

groundwater flow direction should be determined from measured well piezometer levels (i.e., groundwater elevation levels) in a minimum of three monitoring wells screened in the same aquifer formation. Water elevation measurements should be measured to the nearest 0.01 foot. The groundwater elevation data should be plotted on a map or cross section and equipotential lines created. Groundwater flow will occur in the direction of the maximum gradient (from high groundwater elevations to low groundwater elevations) which are at right angles to the equipotential lines. Refer to EPA's 2014 guidance on the three-point solution method (*3PE: A Tool for Estimating Groundwater Flow Vectors*) for determining groundwater flow.

The potential for vertical groundwater flow should be evaluated on a site-specific basis. To determine vertical groundwater flow, a minimum of two nested or clustered monitoring wells should be installed parallel to the horizontal groundwater flow direction for the measurement of hydraulic head. The vertical gradient is then determined using Darcy's law. The following link provides calculators for determining horizontal and vertical groundwater flow:

[https://www3.epa.gov/ceampubl/learn2model/part-two/onsite/b0\\_onsite.html](https://www3.epa.gov/ceampubl/learn2model/part-two/onsite/b0_onsite.html).

Groundwater samples should be collected from wells screened through the smear zone and into the permanently saturated zone. However, site conditions (e.g., diving plumes, confined aquifers, etc.) and contaminant types may dictate that representative groundwater samples also be collected from other depth intervals. If further vertical delineation of contaminant concentration(s) is necessary, the interval(s) within the water column where samples are collected should be based on the physical characteristics of the contaminants and the site specific hydrogeology. This is a consideration at sites with chlorinated solvents or other DNAPLs and possibly other sites. The field notes and the report should document the depth that the groundwater samples were collected (e.g., pump intake depth) relative to the ground surface and the static water level in the well.

Groundwater sampling devices should complement the intended data use and site decisions. Select groundwater purging and sampling equipment to minimize increases in sample temperature, water column agitation, and sample agitation. Materials comprising sampling devices and tubing should not adsorb, desorb, or leach contaminants of concern and should be resistant to chemical and biological degradation.

Due to the loss of volatiles with using these methods, inertia pumps (Section 6.4.4), peristaltic pumps (Section 6.4.5), and bailers (Section 6.4.10) should not be used for the collection of volatiles or other air sensitive parameters unless approved by the CSP project manager in a site-specific work plan. Volatile or other air sensitive samples collected by these methods may be considered biased low.

Rather, bladder pumps (Section 6.4.1), positive pressure submersible pumps (Section 6.4.2), gear pumps (Section 6.4.3), passive diffusion bag samplers (Section 6.4.6), or samplers like HydraSleeve (Section 6.4.8) or Snap Samplers (Section 6.4.9) should be used to reduce the loss of volatiles during sampling.

The application of EMD to groundwater may involve collecting groundwater for (off-site) laboratory analysis, or the use of sterile, in-line field filtration devices to facilitate the field collection of biomass. Field filtration increases the likelihood of collecting suspended particles and attached microorganisms, decreases shipping costs, and significantly reduces costly laboratory extraction



procedures. Shipping filtration devices to the laboratory, in lieu of groundwater samples, may also require maintenance of the samples at 4°C during handling and transport.

The creation of a preferential pathway during site work may impact groundwater. As necessary, implement precautionary measures to assure the groundwater will be protected (i.e., grouting boreholes and compacting soil). If groundwater is encountered, grout soil borings and decommission well points/monitoring wells in accordance with CSP's *Monitoring Well Guidance* (DEC, September 2013).

## **6.2 Drinking Water**

DEC's Division of Environmental Health, Drinking Water Program (DWP) regulates public water systems in accordance with 18 AAC 80 and provides guidance on private drinking water systems. For information on how to collect water samples from private or public drinking water systems, please go to the DWP's web page at: <http://dec.alaska.gov/eh/dw.aspx>

## **6.3 Groundwater Laboratory Analytical Sample Collection**

Groundwater samples should be collected from representative locations and depths as needed to define the horizontal and vertical extent of the contaminant plume and should be clearly described in a work plan approved by the CSP. The geologic interval most likely to be contaminated should be sampled. For petroleum contaminated sites, samples are generally targeted near the top of the aquifer (e.g., the top foot of the water column), due to the potential for LNAPL presence. However, petroleum contamination can extend to significant depths in groundwater due to hydrogeological and other conditions, therefore the CSP may require groundwater samples at greater depths when site conditions warrant it.

The use of Teflon® sampling equipment (e.g., tubing) is preferred, except for the analysis of per- or polyfluorinated alkyl substances (PFAS). The use of HDPE equipment should be minimized to the extent practicable. Refer to Appendix F for a list of recommended sampling materials.

Sample holding times should conform to the specifications in the required laboratory method (see Appendix E). In some cases, sterility may need to be maintained (e.g., practice aseptic technique) and dedicated, disposable sampling equipment may be required when collecting biological samples (e.g., microorganisms).

Prior to sampling, determine depth to groundwater to within 0.01 feet. Check the monitoring well for the presence of NAPL that might be floating on top of the water or in a separate layer at the bottom of the casing. If wells contain NAPL then alternate wells that are representative of the affected groundwater should be sampled, if available. Alternatively, water samples should be collected using methods that minimize the potential for NAPL inclusion in samples that will be analyzed to measure dissolved phase concentrations; the field notes and report should describe the presence and observed thickness of NAPL. Sampling and analysis of petroleum analytes is not required for monitoring wells that contain NAPL.



Identify NAPL by an electronic device designed to detect non-aqueous liquids and to measure the thickness of the non-aqueous layer. Do not use bailers; because of the lower density of the NAPL, bailers will measure a smaller NAPL thickness than is actually in the monitoring well or measure no NAPL at all.

When samples are collected for laboratory analysis, any devices used to collect additional information, such as a flow-through cell, should be removed from the sample train before sampling. During laboratory analytical sample collection, do not use a constricting device on the sample tubing to reduce the flow rate because the constrictor will cause a pressure difference in the water column leading to loss of VOC and dissolved gasses.

Water samples typically should not be filtered prior to analysis. If filtering is approved by CSP in a site-specific work plan, both filtered and non-filtered samples should be collected and analyzed so the effects (bias) of the filtering process on the contaminant concentrations can be evaluated.

Unless approved by CSP to conduct no purge sampling, removal of at least one well volume and stabilization of water quality parameters is required before sampling. Sample the wells least likely to be contaminated first.

More information on groundwater sampling techniques and equipment can be found via the following Internet links:

<http://www2.epa.gov/superfund>

<http://www2.epa.gov/ust>

<http://geoprobe.com/>

<https://www.itrcweb.org/Guidance>

## **6.4 Groundwater Sampling Equipment**

### **6.4.1 Bladder Pump**

An example of positive-displacement, the bladder pump (Figure 6) consists of a Teflon® or stainless steel housing that encloses a flexible Teflon® or HDPE membrane. Below the bladder, a screen may be attached to filter any material that may clog check valves located above and below the bladder. The pumping action begins with water entering the membrane through the lower check valve and once filled, compressed gas is injected into the cavity between the housing and bladder. Utilizing positive-displacement, water is forced (squeezed) through the upper check valve and into the sample discharge line. The upper-check valve prevents back flow into the bladder. All movement of gas and sample is managed through a series of regulators housed in a control mechanism at the surface.

The source of gas for the bladder is either bottled (typically nitrogen, carbon dioxide, or ultra-zero air) or via an on-site oil-less air compressor. Flow rates can be reduced to levels much like the variable speed centrifugal submersible pump without fear of motor stall.



Figure 7. Example of a Teflon® constructed bladder pump, complete (top) and exploded version illustrating internal Teflon® bladder (Photograph by J. Schoenleber)

Field cleaning of bladder pumps is acceptable if the bladder pump housing is constructed of stainless steel or Teflon® with an internal disposable bladder.

#### Procedures for Use:

- Check all fittings for tightness.
- Lower decontaminated pump and dedicated tubing into the well below the smear zone.
- Connect compressor to power source ensuring the power source is downwind to prevent fumes from entering sampling area. If compressor is not used, connect to external air source.
- Engage air source (compressor or external) via control box. Full water flow will begin after five to fifteen pumping cycles. After stabilization of well water has been observed and recorded, sampling may begin.
- Adjust the refill and discharge cycles in accordance with manufacturer's instructions to optimize pumping efficiency. Reduce the flow rate, by adjusting the throttle control, to 100-150 milliliter (mL)/minute or less while sampling volatile and semi-volatile organics.
- Collect sample directly from discharge line into laboratory-cleaned sample bottles after well has stabilized and follow procedures for transport.

#### Advantages:

- Positive-displacement.
- Acceptable for well evacuation and sample collection for all parameters.
- Operational variables are easily controlled.
- Minimal disturbance of sample.
- In-line filtration possible.
- Available in a variety of diameters.
- No variances from the Technical Requirements for Site Remediation necessary.
- Sample depth up to 1,000 feet.

#### Disadvantages:

- Large gas volumes may be needed, especially for deep installations.
- Only pumps with disposable bladders may be field cleaned for portable use when approved decontamination methods are employed.

- Sample flow rate of a typical bladder pump is not sufficient for purging some larger diameter wells.
- The check ball design of bladder pumps may make it an unacceptable method for sampling wells that contain excessive amounts of sediment (check balls may not seal properly if dirty).
- At high pumping rates, use of the bladder pump may cause disturbance of the water column.

### 6.4.2 Variable Speed Submersible Centrifugal Pump

Improvements in the design of submersible centrifugal pumps over the last decade have resulted in pumps significantly reduced in overall size with variable speed discharge control. These two key features, coupled with stainless steel and Teflon® construction, have enhanced the desirability of this pump for application of low-flow purging and sample collection. Some examples include:

- Proactive Environmental Products®
- QED®
- Grundfos®

The variable speed feature is one of the key design items, which allows for application of low-flow purging and sample collection. When using variable speed submersible pumps to collect the equipment blank, one should follow the same general rules for all groundwater sampling equipment. This includes the requirement that all sampling equipment that comes in contact with the sample should also come into contact with the equipment blank water.



Figure 8. Proactive SS Monsoon® Pump. Example with disassembled pump (right)



Figure 9. Disassembled Grundfos® Pump being prepared for decontamination (Photograph by J. Schoenleber)

Procedures for Use (depending on pump manufacturer and/or model):

- Decontaminate pump, electrical leader, and all associated fittings.
- For low-flow purging and sampling, attach precut tubing whose length has been predetermined based upon well-specific pump intake depth.
- For volume-average sampling, set the pump either within three feet of the top of water column, or, immediately above the well screen depending on chosen method.
- Install pump slowly through water column wiping down tubing with deionized saturated paper towel.
- If a portable gasoline generator is used, it should be placed downwind. Initiate purge based on procedure selected.
- After purging, collect sample as specified in CSP approved site-specific work plan.

Advantages:

- Positive-pressure.
- Variable speed control at surface allows for fine tuning of flow rate.
- Stainless steel and Teflon® construction.
- Complete disassembly allows for access to all parts for thorough decontamination.
- Acceptable for low-flow purging and sampling.

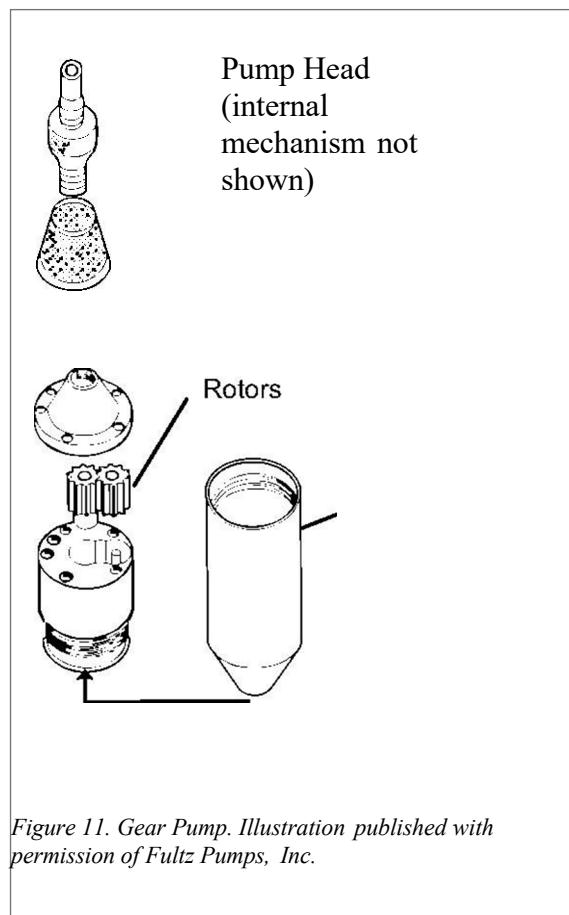
Disadvantages (depending on pump manufacturer and/or model):

- During low-flow purging and sampling, temperature increases may be observed.
- At extremely low-flow rates, motor stall is possible. To re-establish flow, high pumping rate may be needed to restart.

- Should manufacturer's disassembly instructions for decontamination not be followed, cross contamination of well is possible.
- Low yielding wells can also test the limits of variable speed design.
- Decontamination issues.

### 6.4.3 Gear Pump

Positive-displacement pumps, e.g., Fultz Pumps, Inc., also have the capacity for variable speed control (Figure 9). The applications of this pump are similar to the variable speed submersible centrifugal pump. Choose a pump with stainless steel housing and fluorocarbon polymer rotors or gears (Figure 10). Internal parts (gears) may not be readily accessible on-site, therefore careful attention should be made when cleaning. This should be considered when choosing to use this pump for a portable application. Pumps may be designed with the power supply molded into the sample tubing. This makes custom length of tubing based on individual well requirements impractical during a portable application. Single molded power supply and sample tubing is also difficult to decontaminate when using this pump on a portable basis. Instead, pumps whose power supply and pump discharge lines are separate are also available. This pump may be best applied when used in a dedicated system.



#### Procedures for Use:

- Decontaminate pump, electrical leader, and all associated fittings.
- For low-flow purging and sampling, attach precut tubing whose length has been predetermined based upon well-specific targeted zone of influence information. For volume average sampling, set the pump either within three feet of the top of water column, or, immediately above the well screen depending on chosen method.
- Install pump slowly through water column, wiping tubing with DI saturated paper towel.
- Initiate purge based on procedure selected.
- At end of purge, collect sample as specified in approved CSP site-specific work plan.

#### Advantages:

- Positive-displacement.
- Light weight.
- Good variable speed control, especially at low rates.
- Acceptable for low-flow purging and sampling.

#### Disadvantages:

- For portable sampling, many are designed with power supply molded into tubing, which is difficult to decontaminate.
- Turbid purge water wears on fluorocarbon gears and may clog the pump.
- New rotors require wear-in time before the pump can be put back into service.
- Submersible motor lead connection exists with portable pump applications that may be susceptible to degradation and loss of power connection to pump.

### **6.4.4 Inertia Pump**

An inertia pump (Figure 12) consists of a riser tube fitted with a one-way foot valve. It is typically operated by hand, but mechanical actuators can also be employed. Inertia pumps are best used with smaller-diameter wells (e.g., recommended for 2-inch or less). Inertia pumps should not be used for VOC analysis or dissolved gases due to the loss of volatiles in the intake line that draws the sample to the land surface. Any VOC groundwater data collected using inertia pumps should be considered biased low and generally will not be used for demonstrating the extent of the contamination, decreasing trends, or site closure decisions.



*Figure 12. Inertia pumps offered by Solinst*

#### Procedures for Use:

- Install the inertia pump in the well. The one-way valve will open and let water into the tubing. The water level within the tube will be the same as the water level (pressure head) in the well.



- Operate the inertia pump by pulling up the tubing with one upstroke. The upstroke action should be rapid enough to cause water to rise, but slow enough and methodical to avoid in-well disturbance. The weight of the water will keep the valve closed during the upstroke. Water will rise above the head level in the well by the length of the pump stroke. Water will momentarily continue to rise after the upstroke stops because of its momentum. On the down stroke, the valve will open to allow more water to be drawn into the tubing. The valve will close at the end of the down stroke. The momentum on the upstroke and the depth to which the tubing is submerged controls the rate at which water is drawn into the tubing.
- As this cycle is repeated, water will rise in pulses corresponding with each pumping stroke.

#### Advantages:

- Very simple to use and is (typically) hand-operated.
- May be used in small diameter wells.
- No pumps are involved (although electronic actuators may be used).
- No decontamination of pump necessary (however, all tubing should be changed between wells if dedicated tubing is not used).
- Can be used for sampling inorganic contaminants.
- Purge and sample with same tubing.
- Less potential for loss of volatile fraction from negative pressure gradient than for sampling methods using pumps that create a vacuum.

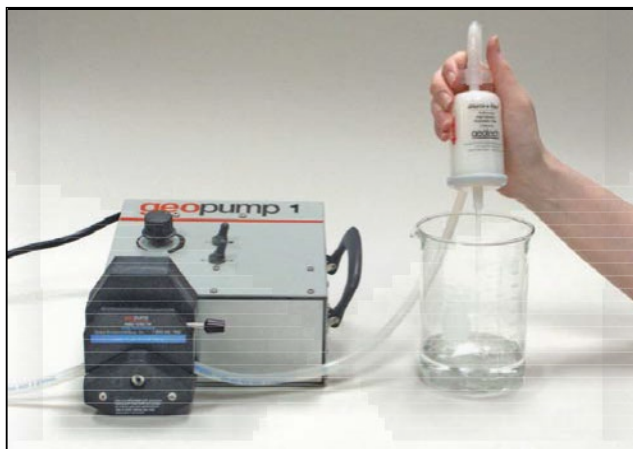
#### Disadvantages:

- Depth limitations are anticipated  $\geq 25$  feet.
- A large annular gap between the inertia pump tubing and the internal diameter of the well casing may facilitate inefficient pumping.
- Unless using an in-line flow through cell for field readings, may not provide reliable or reproducible data for air sensitive parameters e.g., dissolved oxygen, pH, carbon dioxide or iron and its associated forms.
- Assistance from a second person may be required to hold in-line flow through cell when operating the inertia pump.
- Potential for VOC loss during pumping.

### **6.4.5 Peristaltic Pump**

A peristaltic pump (Figure 13) is a self-priming suction lift (negative air pressure) pump utilized at the ground surface, consisting of a rotor with ball bearing rollers. One end of dedicated tubing is inserted into the well and the other end is attached to a short length of flexible tubing, which has been threaded around the rotor, out of the pump, and connected to a discharge tube. The liquid moves totally within the tubing; thus, no part of the pump contacts the liquid. Tubing used for well evacuation may also be used for sample collection. Teflon® or Teflon®-lined polyethylene tubing is recommended for sampling. Silicone tubing is recommended for tubing in contact with the rotors.

The grade of silicone tubing should be appropriate for its intended application. Based upon the required analysis and sampling objectives other materials are acceptable, but should first be approved



*Figure 13. Geopump™ Peristaltic Pump. Photograph with permission from Geotech Environmental Equipment, Inc.*

by CSP in a site-specific work plan. Peristaltic pumps should not be used for volatile analysis or dissolved gases due to the loss of volatiles from the creation of a vacuum in the intake line that draws the sample to the land surface. Any volatile organic groundwater data collected using peristaltic pumps should be considered biased low and generally will not be used for demonstrating the extent of the contamination, decreasing trends, or site closure decisions.

During sampling, the peristaltic pump tubing should remain filled with water to avoid aeration of the sample.

#### Procedures for Use:

- Check tubing at rotor for cracks or leaks, replace if necessary.
- Thread flexible length of tubing through rotor/pump.
- Insert dedicated length of tubing in well and attach to flexible tubing at rotor.
- Tubing depth introduced into the water column should not exceed 12 inches.
- If necessary, add a small stainless steel weight to tubing to aid introduction of tubing into well casing (especially helpful in two-inch diameter wells).
- Attach evacuation line to outlet of flexible pump tubing such that the discharge is directed away from pump and well.
- Engage pump and commence evacuation. Pump speed should be maintained at a rate that will not cause significant drawdown ( $> 0.3$  ft.). After well has been properly purged, begin sampling.
- Collect sample into laboratory cleaned sample bottles.

#### Advantages:

- May be used in small diameter wells.
- Sample does not contact the pump or other sampling equipment other than tubing prior to collection.
- Ease of operation.
- Speed of operation is variably controlled.
- No decontamination of pump necessary (however, all tubing should be changed between wells if dedicated tubing is not used).
- Can be used for sampling inorganic contaminants.
- Purge and sample with same pump and tubing.



### Disadvantages:

- Depth limitation of ~25 feet.
- Potential for loss of volatile fraction due to negative pressure gradient.
- Unless using an in-line flow through cell flow-through-cell for field readings, may not provide reliable or reproducible data for air sensitive parameters e.g., dissolved oxygen, pH, carbon dioxide or iron and its associated forms.

## **6.4.6 Passive Diffusion Bag Samplers**

When confronted with sampling a monitoring well that displays little or virtually no recharge capability during well evacuation (where historic data indicate drawdown exceeds three-tenths of a foot while purging at flow rates that are equal to or below 100 mL per minute), the option to use this no-purge sampling technique may be justified. More appropriately, there may be instances where long-term monitoring during the operation and maintenance phase of remediation justifies their use. Other reasons for using passive diffusion bag samplers (PDB) can also be approved by CSP on a site-specific basis.

PDB samplers cannot be used for all contaminants. They are applicable to a select list of VOCs. For a list of applicable VOCs, refer to the following website:

<https://connect.itcreweb.org/HigherLogic/System/DownloadDocumentFile.ashx?DocumentFileKey=bbc9fc67-b316-4fa1-b8b6-730af5a86850>.

Metals and other organics will not diffuse through the membrane. Additionally, PDB should not be used for semi-volatile contaminants and petroleum hydrocarbons (GRO, DRO, and RRO). Due to the limited number of contaminants PDB samplers are capable of detecting, these devices should not be used for initial investigations where a more complete understanding of the contaminants of concern remains to be determined. PDB samplers may be applicable to sites where adequate characterization has determined that applicable VOCs are the only groundwater contaminants of concern.

In addition, samplers should be cautious when using PDB samplers in wells where the vertical distribution of contaminants has not been determined. Multiple PDB samplers are recommended in wells when the well screen or saturated portion of the borehole is greater than 5 feet, following the general recommendation that a single PDB should not represent more than 5 feet of the water column. In an uncontaminated sentinel well, contaminants might enter the well in a stratified manner that could elude a single PDB. Therefore, the conservative approach for a sentinel well would be to deploy multiple bags as appropriate.

PDB samplers are made of low-density polyethylene plastic tubing (typically 4 mL), filled with laboratory grade (ASTM Type II) deionized water and sealed at both ends (Figure 13). The samplers are typically about 18 to 24 inches in length and can hold from 220 mL to 350 mL of water. Vendors can usually modify the length and diameter of a sampler to meet specific sampling requirements.

Teflon® coated stainless steel wire is preferable for deploying the samplers in the well. Teflon® coated stainless-steel wire can also be reused after proper decontamination. As an alternative to Teflon® coated stainless steel wire, synthetic rope may be used as the deployment line for single-use applications if it's low stretch, non-buoyant, and sufficiently strong to support the weight of the sampler(s). An example of acceptable rope would be uncolored (white) 90-pound, 3/16-inch braided polyester. Extreme care should be exercised when using rope as a deployment line in deep wells due to the potential for the deployment line to stretch, which may result in the improper location of the PDB sampler within the well screen or open hole of the well. Deployment lines consisting of material other than Teflon® coated stainless steel wire may not be used in another well and should be properly disposed of after a one-time use.

The sampler is positioned at the desired depth interval in the well by attachment to a weighted deployment line and left to equilibrate with the water in the well. Many VOCs equilibrate within 48 to 72 hours; however, the minimum equilibration period for PDB should be two weeks. This allows the formation of water and well water to re-stabilize after deployment of the samplers, and to allow diffusion between the stabilized well water and the PDB sampler to occur. In low-yielding formations additional time may be required for the well to re-stabilize.

If quarterly sampling is being conducted, it is acceptable to leave PDB samplers in the well for up to three months so that samplers can be retrieved and deployed for the next monitoring round during the same mobilization. Unfortunately, data are currently unavailable to support longer deployment periods (i.e., semi-annual, or annual). Leaving samplers in a well for longer than three months is not recommended unless data are provided to CSP's satisfaction that longer deployment provides representative data. Additionally, PDB are susceptible to damage by freezing and therefore, are not recommended if freezing conditions are anticipated to be present in the well. If future data become available which demonstrate longer deployment timeframes are appropriate, this condition will be modified.



Figure 14. Eon PDB Sampler with accessories (Photograph by J. Schoenleber)

#### Advantages:

- Purge water associated with conventional sampling reduced or eliminated.
- The devices are relatively inexpensive.
- Simple deployment and recovery reduces the cost and the potential for operator error.
- Monitoring well stability parameters are not required which reduces associated cost.
- PDB samplers are disposable.
- The stainless steel weights and Teflon® coated wire are the only pieces of equipment needing decontamination.
- Quick deployment and recovery is a benefit when sampling in high traffic areas.
- Multiple PDB samplers can be deployed along the screened interval or open borehole to detect the presence of VOC contaminant stratification.
- Has been shown to deliver accurate dissolved oxygen measurement.
- Alkalinity conditions in the well are not transferred across the membrane.
- Effervescence associated with HCL preservation is avoided.

#### Disadvantages:

- PDB samplers provide a time-weighted VOC concentration that is based on the equilibration time of the particular compounds; usually that period is two to three days. This is a limitation if sampling objectives are to identify contaminant concentrations at an exact moment the sample is collected. The time-weighted nature of the PDB may be a factor in comparison with low-flow sampling if concentrations have been shown to be highly variable over time.
- PDB samplers are limited to specific VOC contaminants.
- PDB samplers work best when there is unrestricted horizontal movement of groundwater through the well-screen or open hole. If filter packs or screens are less permeable than the surrounding formation, groundwater flow lines may not enter the well and PDB samples may not be able to provide a representative sample.
- As with low-flow samples, PDB samplers represent a specific depth interval within the water column.
- Contamination migrating above or below the targeted depth interval will not be detected.
- Difficult to measure water quality parameters.
- In some cases, heavy iron or biofouling of the bag may inhibit sampler performance.
- PDB may burst in freezing conditions.

### **6.4.7 Direct Push Technology**

Use of DP technology to obtain ground water samples via temporary well points is widely accepted. While various manufacturers make and distribute their own groundwater equipment and accessories, the same general principles still apply when collecting ground water samples. Direct push wells installed without proper filter packs and annular seals should not be used for long-term monitoring (e.g., more than one sampling event). However, they may be appropriate for collecting grab samples during site characterization, as long as the well is properly developed prior to sampling. Groundwater data collected from monitoring wells that have not been developed are generally biased and of limited utility. In general, such data should not be used to compare to DEC cleanup levels, but

may be used for field screening and characterization of nature and extent if approved by the CSP project manager in a site-specific work plan. Direct push wells installed with proper filter packs and annular seals may be approved by CSP for long-term monitoring if they are developed and sampled in accordance with *CSP Monitoring Well Guidance* (DEC, September 2013). As with any monitoring well, all DP monitoring wells and well points will need to be decommissioned in accordance with the *CSP Monitoring Well Guidance* (DEC, September 2013).

Direct push technology can also be used for high resolution site characterization or used to collect hydrogeologic data. Several specialized tools have been designed to collect data that can be used in modeling groundwater aquifers for contaminant fate and transport studies used to develop site-specific remediation systems. Some useful tools and associated applications are listed below.

#### **6.4.7.1 Plume Delineation**

Light-induced fluorescence (LIF) and ultraviolet optical screening tool (UVOST) uses laser-induced fluorescence to measure NAPL petroleum contamination in-situ. A probe emits ultraviolet light through optical fibers in a direct push steel rod. Light causes polycyclic aromatic hydrocarbons associated with petroleum to excite and fluoresce, emitting different wavelengths depending on the type of fuel. The wavelength signal response is transmitted through a fiber line on the probe and analyzed in real time at the surface in a fluorescence versus depth log. Fluorescence is quantified by percentage of relative emittance (%RE), which is compared to a known standard. By quantifying the %RE, the data can be compared to laboratory analytical correlation samples measured in parts per million (ppm).

Optical Image Profiler (OIP) can also be used to map the vertical and horizontal distribution of LNAPL fuels and light oil in soil based on the fluorescence from PAH contained in fuels or oils. The OIP system relies on an ultraviolet (UV) light emitting diode to produce fluorescence from fuel impacted soils. When PAH in LNAPL are present, they absorb UV light and subsequently produce a fluorescence which is captured by an onboard camera. Acquisition software is then utilized to analyze each pixel of images taken for the presence of fluorescence. Images without significant fuel presence will appear dark under the UV light source. The percent fluorescence is then logged in accordance with depth.

Membrane Interface Probes (MIPs) can be used in saturated and unsaturated zones for the detection and measurement of volatile organic compounds (VOC) in the subsurface. The MIP is an interface between contaminants in the subsurface and detectors at the ground surface. A heated probe fitted with a permeable membrane is pushed to depth through the soil. Volatile organic compounds in the subsurface diffuse across the membrane and enter into a carrier gas stream. The VOC are transported through the carrier gas stream to gas phase detectors at the ground surface for measurement. The MIP is also fitted with an electrical conductivity measurement tip to provide data to indicate probable lithology. A simultaneous log of soil conductivity and VOC results is produced.

These tools are typically used for site characterization of source area soils. However, the data can be used to show concentration representations in three dimensions where transport of contaminants occurs with groundwater flow. The CSP will consider approval of LIF/UVOST, OIP, and MIP on a site-specific basis. Laboratory analytical correlation samples are required.

#### **6.4.7.2 Electrical Conductivity**

Electrical conductivity probes (ECP) measure electrical conductance of unconsolidated soil formations and any contaminated fluids. High electrical conductivity is associated with the finer grain sizes (clays) while lower conductivity is generally indicative of coarser grained sediments such as sands.

#### **6.4.7.3 Hydraulic Conductivity**

Hydraulic conductivity probes (HPTs) may be used to help define subsurface lithology, assess formation permeability and hydro-stratigraphy at high resolution (down to the centimeter scale). QA testing of the sensors is required to verify performance prior to field use and a QA log should be submitted with all data reports.

#### **6.4.7.4 Cone Penetrometer Test**

A Cone Penetration Test (CPT) is commonly used to determine the subsurface stratigraphy in-situ and to estimate geotechnical parameters of the materials present. Cone penetrometer tests are a quasi-static penetration test, meaning that the cone is pushed at a slow rate rather than driven with a hammer or rotary drilling. The CPT is designed to evaluate subsurface conditions based primarily on the resistance to penetration encountered by the cone tip. Resistance measurements are also recorded for the cone sleeve, or shaft. The use of CPT tools in combination with DP technology allows for continuous data at high resolution, repeatable penetration results, and cost savings over more traditional boring and sampling methods.

#### **6.4.7.5 High-Resolution Piezocone**

The High-Resolution Piezocone is a sensor probe that converts pore pressure to water level or hydraulic head. Piezocone penetrometer tests are highly effective for identifying sand, silt, and clay layers, as well as determining pore pressure. The piezocone can simultaneously collect soil type and hydraulic conductivity data. Piezocone penetrometer tests are also moderately effective for determining other geotechnical engineering properties including friction angle, undrained shear strength, density index, permeability, and horizontal stress.

##### Advantages:

- Relative ease of collecting minimally disturbed groundwater samples at depth.
- Ability to collect hydrogeological data while installing monitoring well.

##### Disadvantages:

- Decontamination of reusable equipment is required between boreholes.
- For decommissioning, grouting starting from the bottom of the boring and completed at the surface is recommended.
- Depending on casing diameter, some groundwater sampling equipment may not be applicable.

Guidance on the construction of temporary wells installed via direct push technology can be referenced through ASTM D6001-96, *Direct Push Water Sampling for Geoenvironmental Investigations* (ASTM, 2002).

#### 6.4.8 HydraSleeve™

The HydraSleeve™ groundwater sampler consists of three basic components: the sampling sleeve, a stainless steel weight, and a self-sealing valve. The sleeve comes in various volumes and sizes to account for different well diameters and sampling needs. The HydraSleeve™ is typically used for no-purge sampling, but can be used for purged wells also.



Figure 15. 1.5-inch Hydra Sleeve™ and stainless steel weight (1 liter capacity)

##### Procedures for use:

- Attach the weight to the bottom of the flattened sleeve and attach a cord to the top.
- Lower the sleeve to the desired sampling interval.
- Pull sharply on the cord to initiate sample collection.
- Retrieve sampler, insert discharge straw, and fill sample containers as needed.

##### Advantages:

- Disposable, simple to use and inexpensive.
- Effective in sampling low yield wells.
- Can sample discrete intervals.
- Sleeves can be deployed in-line to create a vertical contaminant profile.

##### Disadvantages:

- Larger sample volumes are difficult to retrieve.

#### 6.4.9 Snap Sampler™

The Snap Sampler™ is designed to collect representative groundwater samples in-situ without purging. The sampler utilizes a double ended cap to close the bottle while it is submerged in the well, eliminating the need to transfer the sample to another container. Samplers are typically leased from the manufacturer or purchased and dedicated to a particular well.



Figure 16. Snap Sampler™ VOA Vial

#### Procedures for use:

- Snap Sampler™ container is placed within the Snap Sampler™ and the Snap Caps™ are attached in the open position.
- Sampler is lowered into a well to the desired interval using the trigger tubing which contains the trigger line and is attached to a docking station at the wellhead.
- Pull trigger line to close sampler and retrieve.
- If necessary, preservative is added to a specialized cavity in one of the Snap Caps™.

#### Advantages:

- Capable of sampling discrete intervals.
- Minimal disturbance if allowed to equilibrate prior to sample collection.
- Can be deployed in-line to create a vertical contaminant profile.

#### Disadvantages:

- Limited sample volume depending on type and well diameter.
- Can only be used in wells that are two inches in diameter or greater.
- Fixed trigger length generally means each trigger line is dedicated to a specific well.
- Not all analytical laboratories are equipped to analyze Snap Sampler™ bottles.

### **6.4.10 Bottom Fill Bailer**

Bailer design is simple and versatile, consisting of a cylindrical length of Teflon®, HDPE or stainless steel with a check valve at the bottom. Bailers (Figures 17 and 18) are available in numerous dimensions to accommodate a wide variety of well diameters. Their relative low cost allows them to be utilized for a one-time use per well per sampling episode.

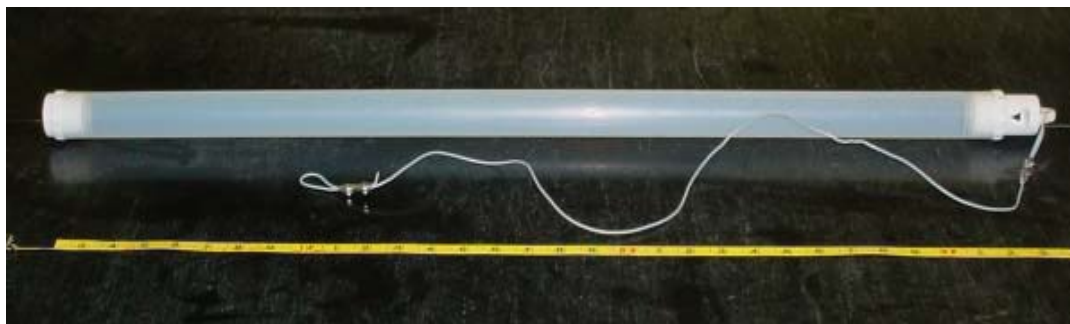


Figure 17. Bottom fill bailer with Teflon® coated stainless steel leader (Photograph by J. Schoenleber)



The bailer, line, and any other equipment entering the well, should be new or laboratory-cleaned and handled with new surgical gloves to prevent cross contamination. Surgical gloves should be changed between each sample location. Clean sampling equipment and any other objects entering the well should not be allowed to contact the ground or any other potentially contaminated surfaces (e.g., gasoline-fueled generators). If this should occur, that item will not be placed in the well or utilized for sampling unless properly decontaminated. It is always good practice to have extra laboratory-cleaned bailers available at the site. Additionally, bailers and sample bottles should be physically separate from pumps or generators during transport and storage.



Figure 18. Teflon® constructed bailer with Teflon® ball check valve (Photograph by J. Schoenleber)

Disposable bailers are typically decontaminated by the manufacturer and should be provided in a sealed polyethylene bag. The manufacturer should be prepared to provide certification that the bailers are clean and state in writing the methods used to achieve decontamination. These bailers may then be acceptable for use depending on site-specific objectives and conditions.

Bailers, even when carefully handled, result in some disturbance of the groundwater in the well, therefore, bailers should not be used as the only method for measuring petroleum product thickness in wells. Samples collected with bailers should be recovered with a minimal amount of aeration. This can be accomplished if care is taken to gradually lower the bailer *until* it contacts the water surface and is then allowed to fill as it slowly sinks in a controlled manner. However, despite the care taken to control aeration during the fill process, filling and emptying the bailer *will* alter dissolved oxygen concentrations. Due to these reasons (operator induced turbulence and air exposure) this device cannot be relied upon to deliver accurate and reproducible measurements of any air sensitive parameter including, but not limited to, dissolved oxygen, pH, carbon dioxide, iron, and its associated forms (ferric and ferrous).

When a bailer is used for sample collection, VOC analytical results may be biased low (due to aeration) and metal analytical results may be biased high (due to turbidity). For this reason, bailers are not recommended for VOC or metals sample collection.

#### Procedures for Use:

- Allow sufficient time after purging for the well to equilibrate and fines to settle. If full recovery exceeds one hour, collect samples as soon as the well has recharged to 80% its pre-purged volume, when practical.
- Fit reusable bailers with a new bailer line for each well sampled; the bailer and line may be handled only by personnel wearing clean disposable gloves.
- Lower the bailer slowly to minimize disturbance of the well and water column.
- The leader or bailer line that comes in contact with the water should be new or decontaminated.



- Prevent the bailing line from contacting the outside of the well, equipment, and clothing.
- Obtain samples as close as possible to the water level/air interface, unless analysis indicates that contamination is at a different depth.
- Lift the bailer slowly and transfer the contents to a sample container with a minimum of disturbance and agitation to prevent loss of volatile compounds.
- Dedicating a bailer and leaving it in a well for long-term monitoring is not allowed due to the potential risk of accumulated contamination.

#### Advantages:

- No external power source required.
- Economical enough that a separate laboratory-cleaned bailer may be used for each well, therefore eliminating cross contamination.
- Available in Teflon®, HDPE, or stainless steel construction.
- Disposable bailers acceptable when material of construction is appropriate for contaminant.
- Simple to use, lightweight, portable.

#### Disadvantages:

- Limited volume of sample collected.
- Not appropriate as the sole means for measuring petroleum fuel product thickness in monitoring well.
- Unable to collect discrete samples from a depth below the water surface (vertical delineation).
- Field cleaning not acceptable.
- Reusable polyethylene bailers are not acceptable sampling devices for chemical analysis.
- Ball check valve function susceptible to wear, dimension distortion and silt buildup resulting in leakage in reusable bailers. This leakage may aerate succeeding sample and may gather unwanted material by rinsing unwanted material from well casing.
- Cannot provide reliable or reproducible data for air sensitive parameters, e.g., dissolved oxygen, pH, carbon dioxide or iron and its associated forms.
- Volatile organic analytical results may be biased low (due to aeration) and metals results may be biased high (due to turbidity).

### **6.4.11 Sterivex™ filters**

A Sterivex™ filter is a sterile in-line filtration apparatus (typically with a 0.22 µm pore size to capture microbial cells) that is connected to tubing and a submersible or low-flow pump. This approach should only be used for the evaluation of environmental molecular diagnostics in groundwater samples.

Groundwater is pumped through the filter which captures microbial biomass for identification, enumeration, or activity assessments, typically via extraction of genetic material (e.g., nucleic acids) and molecular biological analyses in the laboratory. An alternative to using Sterivex™ filters is to collect sufficient groundwater (typically 1 liter or more) for microbial analyses. Care should be given

to ensure that groundwater samples are processed and transported under the appropriate sample preservation conditions for the biomolecule being analyzed (e.g., DNA, RNA, or whole cells).

#### Procedures for use:

- Connect the Sterivex™ filter inlet to a Luer lock fitting and to the end of the Teflon tubing with a clamp.
- Filter groundwater into a graduated cylinder to record the volume of groundwater filtered.
- If less than the desired volume (typically one liter) is filtered prior to clogging, then a second filtration unit may be used.
- When done sampling groundwater, push the remaining water through the Sterivex™ filter using a sterile 10-mL syringe.
- Add preservative/stabilizing agent to the Sterivex™ filter if sampling for mRNA.
- Cap both ends of the filter (one with the Luer lock, the other with a rubber cap provided by the laboratory).
- Place the Sterivex™ filter into a sterile container (e.g., 100-mL Falcon plastic centrifuge tube) and transport to laboratory (typically at 4° Celsius) for analyses.

#### Advantages:

- Can be integrated with the same submersible/low-flow groundwater sampling methods for VOC.
- Shipping filters in lieu of groundwater samples decreases shipping costs and decreases laboratory extraction costs.
- Increases the likelihood of collecting suspended particles and attached microorganisms in groundwater.

#### Disadvantages:

- Sterivex™ filter may clog before the desired volume of groundwater is filtered. (Be sure to record the volume of groundwater filtered through each filter.)

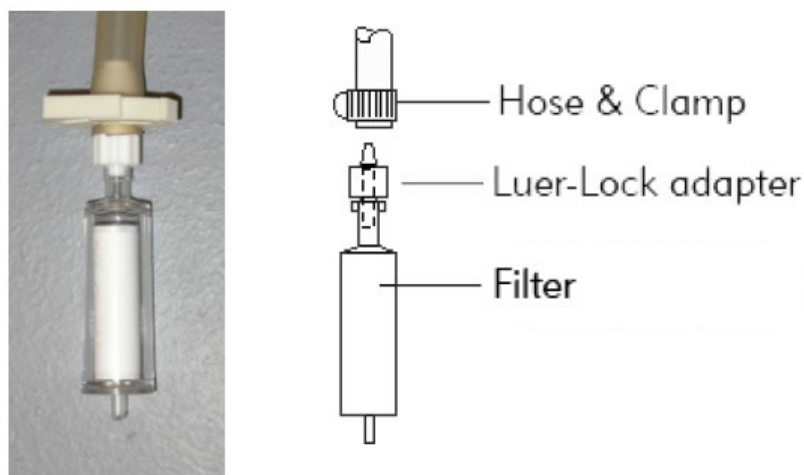


Figure 19. Sterivex™ filter and filter fittings used to collect microbial biomass from groundwater (Figure courtesy Microbial Insights, Inc., Knoxville TN)

### 6.4.12 Bio-Traps™

A Bio-Trap™ is a passive microbial sampling device used to assess in-situ microbial activity. It contains a solid composite matrix of powdered activated carbon and Nomex® material called Bio-Sep® beads. This matrix can be amended with <sup>13</sup>C-labeled compounds or electron donors or acceptors, prior to deployment and incubation in groundwater. Bio-Traps™ are typically deployed for several weeks, retrieved and transported to a laboratory (typically at 4°C), and analyzed using a variety of EMD methods. Bio-Traps™ configurations may also be customized to individual contaminated sites conditions; for example, Bio-Traps™ may be amended with contaminants having stable isotopes to demonstrate that biodegradation is occurring at a site. Bio-Traps™ may also be deployed within bailers that effectively seal the sampling devices from the external ambient groundwater environment, once deployed. This configuration may be useful where the capturing and analyzing metabolic by-products (such as methane or carbon dioxide) may be of interest.

#### Procedures for use:

- Discuss sampling goals for using Bio-Trap™ samplers with the laboratory provider prior to use.
- Deploy the sampling devices into monitoring wells at the desired depth.
- Retrieve samplers after sufficient deployment duration to allow microbial biofilm formation (typically 30 to 90 days).
- Transport to laboratory (typically at 4°C) for analyses.

#### Advantages:

- Simple to deploy and retrieve.
- Can be configured to answer unique site-specific questions.
- Bio-Traps™ can be amended with electron donors or inorganic amendments to bio-stimulate the microbial community and answer site-specific questions or screen remedial alternatives.
- Bio-Traps™ can be amended with <sup>13</sup>C-labeled contaminants to confirm biodegradation is occurring at a site.
- Can potentially provide more accurate temporal and spatial representation of the subsurface microbial community than from groundwater grab samples.

#### Disadvantages:

- Require additional field visits for deployment and retrieval.
- Some <sup>13</sup>C-labeled contaminants may be expensive to synthesize.

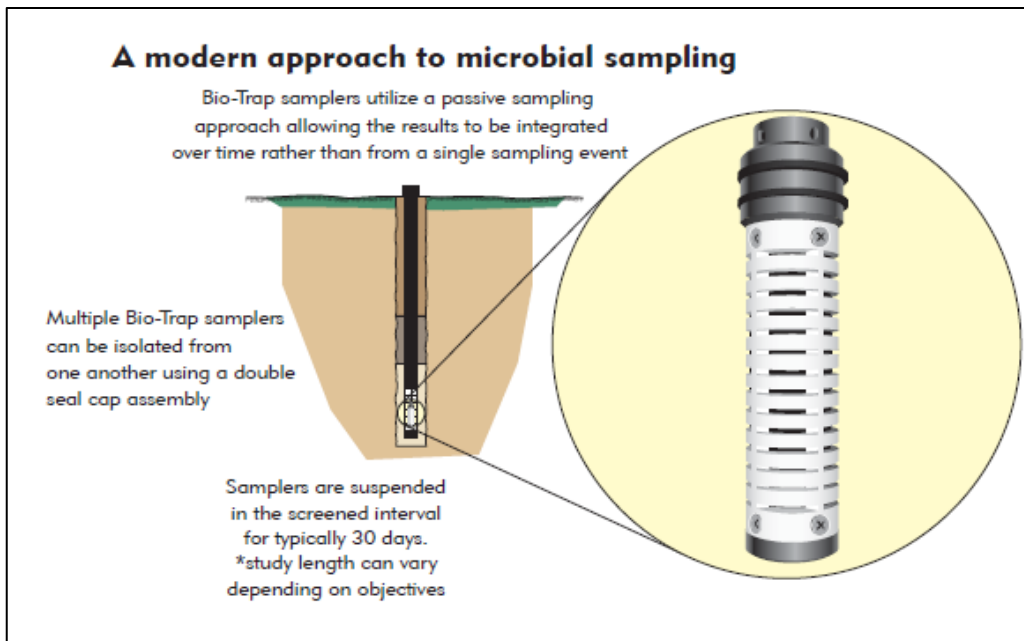


Figure 20. Bio-Trap™ passive microbial sampling device (Figure courtesy Microbial Insights, Inc., Knoxville TN)

### 6.4.13 Purge Techniques

Purging is the process of removing stagnant water from a monitoring well prior to sampling, causing it to be replaced by groundwater from the adjacent formation. Prior to purging, three measurements need to be recorded: the inside diameter of the well, the depth to water in the well, and the depth to the bottom of the well. With that information, the volume of the water in the well casing needs to be calculated and recorded. The table below can be used to help calculate the volume of water in the well casing:

Table 3. Volume of water in well casing

Casing Inside Diameter in Inches	Gallons per Foot of Water
½ (0.5)	0.01
1	0.04
2	0.16
3	0.37
4	0.65
5	1.02
6	1.47
7	2.00
8	2.61
9	3.31
10	4.08
11	4.93
12	5.88

When purging monitoring wells prior to sampling:

- remove at least one casing volumes, **or**
- monitor water quality parameters until a minimum of three (minimum of four if using temperature as an indicator) of the parameters listed below stabilize, **or**
- for low yield wells, the entire well casing is evacuated.

All water quality parameters, except turbidity, should be obtained using a flow-through-cell and turbidity measurements should be obtained before the water enters the flow-through-cell. Additionally, water quality parameters shall be measured and recorded in a field log while purging monitoring wells.

In order to collect representative groundwater samples, CSP recommends that groundwater be purged and sampled using low-flow techniques. For low-flow sampling, the goal is minimum drawdown (<0.3 feet) during purging. The water level should be measured at each interval that the water quality parameters are measured and recorded on the field log. Flow rate should be between 50 and 500 mL/min. Temperature and pH, while commonly used as purging indicators, are insensitive in distinguishing between formation water and stagnant casing water; nevertheless, these are important parameters for data interpretation purposes and should also be measured and recorded. Additional details about low-flow sampling procedures are provided in EPA's *LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS* (EPA, September 2017).

Water quality parameters are considered stable when three successive readings, collected 3-5 minutes apart, are within:

- $\pm 3\%$  for temperature (minimum of  $\pm 0.2^{\circ}\text{C}$ );
- $\pm 0.1$  for pH;
- $\pm 3\%$  for conductivity;
- $\pm 10$  mv for redox potential;
- $\pm 10\%$  for dissolved oxygen (DO); and
- $\pm 10\%$  for turbidity.

A minimum of three (minimum of four if using temperature as an indicator) of these parameters should be monitored and recorded. Low flow purging and sampling are particularly useful for wells that purge dry or take one hour or longer to recover. If a well is low yield and purged dry, do not collect a sample until it has recharged to approximately 80% of its pre-purge volume, when practical. Collection of groundwater samples for EMD analyses should occur after geochemical stabilization.

#### **6.4.14 No Purge Techniques**

No purge groundwater sampling is a method for obtaining groundwater samples without purging the well beforehand. Under certain site conditions no purge sampling may not provide representative groundwater data, so it is necessary to demonstrate, in accordance with a CSP approved site-specific work plan, that no purge sampling will provide similar results to purge sampling at the site. This would include collecting and analyzing both no purge and purge samples from each monitoring well during the same sampling event.

No purge sampling may not be used for the initial groundwater monitoring event at a site unless it is done in conjunction with purge sampling during that sampling event.

Additional information on no purge sampling can be found at the website: <https://clu-in.org/characterization/>.

## **6.5 Passive Groundwater Sampling**

Passive groundwater sampling allows a sample to be collected from a discrete location without active media transport induced by pumping or purge techniques. Passive technologies rely on the sampling device being exposed to media in ambient equilibrium during the sampler deployment period. For example, well water is expected to be in natural exchange with the formation water.

### **6.5.1 Passive Groundwater Sampling for Microorganisms**

Passive samplers for groundwater microorganisms (and EMD analyses) are typically deployed in-situ within an aquifer environment for several weeks, and rely on formation of microbial biofilms that develop on or within a solid matrix of the sampler. Passive samplers can also be amended with substrates (e.g., electron donors or acceptors, stable isotopes, or microbial cultures) prior to deployment as part of biostimulation or bioaugmentation strategies.

The ITRC's website describes various tools and techniques for passive groundwater sampling and use of passive samplers as part of EMD analyses. Refer to the ITRC website at: <https://www.itrcweb.org/Guidance> for current guidance.

## **7.0 Air Sampling**

### **7.1 Air and Soil Gas Sampling**

Vapor intrusion is the migration of volatile chemicals from a subsurface vapor source into overlying buildings. See CSP's *Vapor Intrusion Guidance*, (DEC, November 2017) for more specific guidance for evaluating and responding to a vapor intrusion exposure pathway at contaminated sites. Procedures for air and soil gas sample collection and analysis are provided in CSP's *Vapor Intrusion Guidance* (DEC, November 2017). Evaluation of soil gas or air by methods not described in the *Vapor Intrusion Guidance* (DEC, November 2017) can be approved by CSP on a site-specific basis.

## **8.0 Surface Water Sampling**

Identification of surface water bodies or seeps that are hydrogeologically connected to groundwater is an important component of site characterization.

Surface water sampling methods can be defined in many ways. In general, water can be collected by two general methods: by hand collection, or by use of sampling equipment designed for obtaining water samples from specific depths of the water column. (Alternative classification can include isokinetic and non-isokinetic, depth-integrated or non-depth-integrated sampling methods, as defined by U.S. Geological Service, depending on the environment sampled and type of sample collected.)

Hand collection is obviously limited to surface waters or just under the water's surface, whereas specialized sampling equipment may obtain individual water samples from depths of many meters. Examples of water sample collection equipment include those common to limnological and oceanographic applications, such as: a Van Dorn sampler, a Kemmerer Bottle, a Nansen or Niskin bottle, or other samplers capable of sampling at specific depths (often with messengers or manipulating a trigger line and mechanism). Deviations from this section may be approved by CSP on a site-specific basis but should be clearly identified and discussed in the work plan and report.

Other surface water sampling devices may include a dip sampler to obtain samples from an outfall pipe or areas difficult to access; bailers, hand-held bottles or even buckets; and automatic samplers deployed to collect either grab or composite samples at specific depths, flow rates, or points in time. Additional samplers may also be available for specialized applications (e.g., Biological Oxygen Demand (B.O.D.)). The United States Geological Survey (USGS) has also developed a specialized sampler specific to collecting VOC in stream water (manufactured by WILDCO, Inc.; see Figure 21).

CSP recommends surface water samples be collected unfiltered. If filtering is approved by CSP in a site-specific work plan, both filtered and unfiltered surface water samples will need to be collected and analyzed so the effects (bias) of the filtering process on the contaminant concentrations can be evaluated.

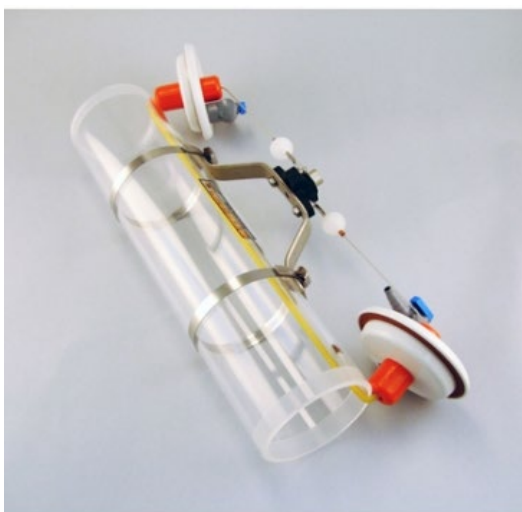


Figure 21. Van Dorn water sampler on left, Kemmerer bottle sampler on right (photos courtesy of Wildco, Inc.).

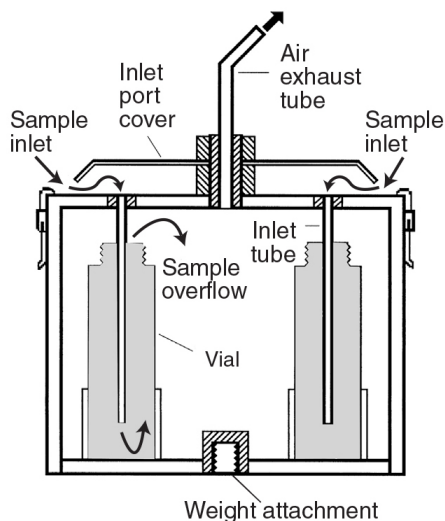


Figure 22. VOC sampler that holds four VOA vials from *Field Guide for Collecting Samples for Analysis of Volatile Organic Compounds in Stream Water for the National Water-Quality Assessment Program* by Larry R. Shelton, U.S. Geological Survey, *Open-File Report 97-401* (USGS, 1995). Image from [http://water.usgs.gov/owq/FieldManual/Chapter2/Chapter2\\_V3-1.pdf](http://water.usgs.gov/owq/FieldManual/Chapter2/Chapter2_V3-1.pdf).

The CSP recommends sampling surface waters following procedures and guidelines established by USGS, and documented in the *National Field Manual for the Collection of Water- Quality Data* (USGS, June 2018).

As with groundwater sampling, field measurements (e.g., temperature, dissolved oxygen, pH, water hardness, etc.) should also be collected at the time of surface water sample collection, following the guidelines in Section 12 of this document. Many of these can be collected with the use of multi-parameter probes or instrumentation. Other parameters or information (e.g., stream discharge, light penetration to determine euphotic zone, etc.) may also be necessary when collecting water samples for biological indicators, or analyte-specific analyses.

#### Procedures for Surface Water Sampling:

Consult the guidance documents above to select sampling equipment based on project objectives, sampling strategy and type of surface water body anticipated. In particular:

- Determine the point of compliance or location where any release enters the surface water body. (e.g., sampled before dilution by surface water body). For groundwater that is discharging to surface water, compliance will be measured in the groundwater as close as practicable to the surface water discharge.
- Determine how samples are to be collected; e.g., by hand, specialized sampling equipment, or through ice.
- Determine what kind of (and how often) ancillary data should be collected.
- Include the appropriate number of sample blanks and other quality control samples to be collected.
- If using in-situ samplers and sensors, be sure instrumentation is properly calibrated before deployment or use.



- Follow Appendix E for use of appropriate sampling containers, depending on the analyses of interest.
- All sample containers should be pre-cleaned by the laboratory prior to filling with sample.
- The sampler and supporting equipment should be rinsed thoroughly with water at the sampling location between samples, and rinsed with water from the next sampling location before collecting a sample from the new location. More rigorous equipment decontamination may be necessary if highly contaminated sites are sampled or if low level contaminants are a concern. To reduce the probability of cross-contamination of samples, sample relatively clean sites first and then subsequently sample more contaminated locations.
- Consider employing “Clean Hands/Dirty Hands” sampling techniques (USGS, June 2018) (e.g., dedicated sampler and dedicated logistical support).
- If anaerobic conditions need to be maintained, then the water in sampler and/or sample bottle should not be exposed to air after collection, and shall be collected and capped at depth.
- When collecting surface water into sample bottles that contain preservative (e.g., pre-loaded), it may be most practical to sub-sample from a sampling device (which could be another sample bottle used as a ladle) and fill the sample container containing preservative until no headspace is apparent, instead of attempting to sample and cap at depth, and potentially losing preservative.
- Follow guidelines in Appendix E for surface water sample storage and preservation, depending on analyses to be performed.
- For toxicity testing, store unpreserved at 4°C for not longer than 24 hours, unless the test method dictates otherwise.
- For compliance with or to demonstrate impairment of Alaska Water Quality Standards, Water Hardness, and Water Quality Criteria for Toxics and Other Deleterious Substances (Alaska Administrative Code, 18 AAC 70), sufficient numbers of samples may need to be collected to adequately describe the frequency and duration of a criterion or standard compliance or exceedance. Sufficient sampling may also need to occur to demonstrate impairment representative of chronic or acute exposure (e.g., sampling to determine 1-hour, 24-hour, or 4-day average concentration).
- Sufficient sample volumes may need to be collected to determine both total and dissolved analytes.
- In some cases, surface waters samples may be composited prior to analysis. ***In all cases, samples collected for VOC analysis should be sub-sampled and collected directly from the water sampler before mixing the sample to minimize volatilization of contaminants.***

## 9.0 Sediment Sampling

### 9.1 Sediment Grab Sampling and Core Sampling

Depending on project objectives, sediment sampling can range from collecting sediments with simple hand tools (such as a shovel) to the use of mechanical equipment common to oceanographic and limnological investigations. Sediment samples can be collected from different environments including streams, marine inter-tidal areas, or sub-tidal areas. The types of sampling conducted is dependent on the data quality objectives. Two common forms of sediment sampling will be discussed in this guidance: grab sampling and core sampling. For more information on sediment sampling techniques, equipment and procedures, consult *Methods for Collection, Storage and Manipulation of Sediments*

*for Chemical and Toxicological Analyses* (EPA, October 2001). Deviations from this section may be approved by CSP on a site-specific basis but should be clearly identified and discussed in the work plan and report.

Sediment grab samplers commonly employ the use of simple mechanical jaws with a trigger mechanism that remain open during descent through the water column, but closes the sampler upon pulling up during ascent. Common examples include the Ponar and Van Veen grab samplers, pictured in Figures 23a and 23b. Grab samplers exist in a variety of configurations, and are recommended for collecting surficial sediments where depth profiling is not required. In contrast, dredge samplers (Figure 23c) are commonly used to collect benthos (i.e., macroinvertebrates or other aquatic organisms living in sediment). Because dredge sampling typically disrupts the sediment profile and potentially alters pore water integrity, and a loss of fine grain sediment may occur, dredge sampling is not typically used to characterize sediments and should be approved by CSP on a site-specific basis. Many grab samplers are also marketed as dredge samplers, as they may also be appropriate for benthos collection.

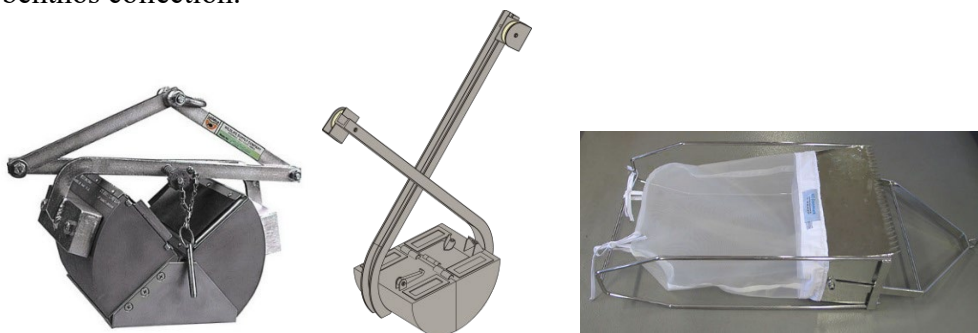


Figure 23: a) Ponar grab sampler; b) Van Veen grab sampler; c) rectangular dredge sampler. Image sources: a) Wildco Supply; b) Wikipedia; c) KC-Denmark A/S Research Equipment.)

Sediment core samplers typically consist of a hollow metal or plastic pipe that serves as the core barrel, in which a removable plastic liner or core tube fits and retains the sediment sample. Core samples are recommended when vertical sediment profiles, historical depositional analyses, or maintenance of oxygen-free environments in sediments is required. Core samplers can be simply hand-operated by pushing into sediments, or weighted or mechanical attachments (often deployed from a vessel) can be used to facilitate sediment penetration and collection (e.g., vibracorer, box corer, etc.). Additional configurations may include a valve and core catcher to retain the sediment sample; driving tips and core cutter for penetration of the sediment; piston-driven impact or vibration mechanisms to increase penetration of the corer into the sediment; and stabilizing fins to ensure vertical descent of the corer. Most core samplers do not work well in sandy sediments or in extremely soft (high water content) sediments. In these situations, use of grab samplers may be more appropriate. In cases where metals are contaminants of concern, plastic liners should be used to avoid contact of the sediments with stainless steel.

Sediment collection techniques (most commonly core sampling) can also be used to collect sediment pore water samples, via post-sampling processing by centrifugation or various sediment “squeezing” techniques. These additional sample processing steps increase the potential to alter sediment pore water chemistry by causing increases in ammonia, sulfide, and dissolved organic carbon (DOC) concentrations, as compared to those collected via passive sampling methods (e.g., peepers). Other

constituents, such as salinity, dissolved inorganic carbon, sulfide, and sulfate may also change if oxidation is not prevented. If sediments are anoxic (which is common), it may be necessary to maintain anoxia during sediment sampling and processing, depending on project objectives. When anoxic sediments are exposed to air, volatile sulfides will be lost, which may increase the availability of sulfide-bound metals. In addition, iron and manganese oxyhydroxides can quickly form and readily complex with trace metals, and alter metal-related toxicity. Generally, if total metal concentrations are of interest for regulatory adherence, maintaining anoxic conditions may be unnecessary. However, if project objectives call for determining various metal species, or if sediment pore water is to be extracted for bioavailability determinations, then anoxic conditions need to be maintained, and all steps involved in sediment sample processing should be conducted in inert atmospheres (e.g., glove box with argon or nitrogen gas), or by limited contact with the atmosphere to prevent oxidation (and subsequent sorption/precipitation) of reduced metals or organic contaminants. Light (photochemistry) and temperature may also impact sediment chemistry by causing changes in metal speciation or DOC concentrations. Sediment samples should be immediately placed and kept in the dark, at less than 6°C for transport. Follow Appendices D and E below for proper preservation and storage/hold time requirements.

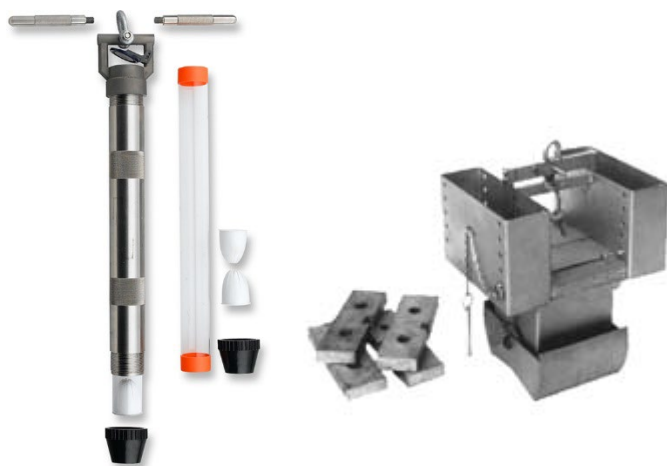


Figure 24. a) Sediment hand core sampler; b) box corer (image sources: a) Forestry Suppliers, Inc.; b) Wildco)

The optimal sediment collection method will depend upon the purpose of the sample (i.e., intended analysis), characteristics of the sediment, and the contaminants of concern. Table 4 provides guidance on sediment sample volumes required for common environmental analyses. CSP requests that any sediment sampling strategy, equipment, and procedures, as well as handling and processing steps, be adequately described in a work plan for contaminated site assessment or remedial evaluation.

Table 4. Typical Sample Volumes for Various Sediment Analyses

Sediment Analysis	Minimum Sample Volume
Inorganic chemicals	100 mL
Non-petroleum organic chemicals	1 L
Other chemical parameters (e.g., total organic carbon, moisture content)	300 mL
Particle size	230 mL
Petroleum hydrocarbons <sup>1</sup>	250-1000 mL
Acute and chronic whole sediment toxicity tests <sup>2</sup>	1-2 L
Bioaccumulation tests <sup>3</sup>	15 L
Benthic macroinvertebrate assessments	8-16 L
Pore water extraction	2 L
Elutriate (aqueous extraction of suspended sediments) preparation	1 L

<sup>1</sup> The maximum volume (1,000 mL) is required only for oil and grease analysis; otherwise, 250 mL is sufficient for AK 101, 102, and 103 analyses. BTEX and VOC analysis will require additional sediment volumes; see Appendix D.

<sup>2</sup> Amount needed per whole sediment test (i.e., one species) assuming 8 replicates per sample and test volumes specified in EPA's Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates (EPA, March 2001) .

<sup>3</sup> Based on an average of 3 L of sediment per test chamber and 5 replicates specified in EPA's *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates* (EPA, March 2000).

### Procedures for Sediment Sampling:

Consult EPA's *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (EPA, October 2001) for selecting sampling equipment based on project objectives, sampling strategy and type of sediment material anticipated (i.e., coarse or fine-grained). The depth of sediment sampling is dependent on the project objectives (e.g., whether vertical profiling is necessary). Issues that determine the appropriate depth of sampling include: regulatory objectives (e.g., depth of dredging for sediment remediation), need to characterize sediments at depth (e.g., materials to be dredged versus shallow depositional areas in some superfund sites), historical comparisons, sediment deposition rates, and/or time period of contamination.

Generally, grab samples should be collected if large sediment volumes, large sediment sizes, or greater surficial surface area is to be sampled, and vertical depth profiles or maintenance of anoxic sediment conditions are not required. Winching systems for sampling from vessels and maintaining appropriate sampler control during descent and ascent may be necessary in some situations.

Using Table 4 as guidance, collect sufficient sediment volume necessary for project objectives and analyses. Collect a minimum volume of sediment and store in glass bottles or high density polyethylene (HDPE) or polytetrafluoroethylene (PTFE) containers with appropriate preservatives, depending on the chemical analysis, as outlined in Appendix D. Following collection methods outlined in EPA's *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (EPA, October 2001), samples should be visually inspected to ensure that:

- The sampler is not overfilled so that the sediment surface is touching the top of the sampler;
- Overlying water is present (indicates minimal leakage). This overlying water should be removed prior to processing and storage by siphoning, not decanting;
- The overlying water is clear or not excessively turbid;
- The sediment-water interface is intact and relatively flat, with no sign of channeling or sample washout;
- The desired depth of penetration has been achieved; and
- There is no evidence of sediment loss (e.g., incomplete closure of the sampler, penetration at an angle, or tilting upon retrieval).

All containers should be pre-cleaned prior to filling with sample. Purge containers with inert gas (e.g., nitrogen) prior to and after filling if anoxic conditions should be maintained.

Sediment samples collected in the field should be stored in containers without headspace at less than 6°C and in the dark to minimize changes in contaminant bioavailability.

The sampler and equipment should be rinsed thoroughly with deionized water at the sampling location between samples, and rinsed with deionized water from the next sampling location before collecting a sample from the new location. More rigorous equipment decontamination might be necessary if highly contaminated sites are sampled or if low-level contaminants are a concern. To reduce the probability of cross-contamination of samples, sample relatively clean sites first and then subsequently sample more contaminated locations.

If a project involves evaluation of metal contamination, or if anaerobic conditions need to be maintained for other reasons, it might be necessary to homogenize, sub-sample, and composite samples in an oxygen-free glovebox or other suitable apparatus.

Be sure to record the following in the field notes:

- Latitude and longitude coordinates of sample location, if possible;
- Date and time of sampling;
- Water depth and the sampling penetration depth;
- Possible sample contamination, equipment failure, unusual appearance of sediment integrity, inability to control vertical descent of the sampler;
- Estimate of quantity of sediment recovered by a grab sampler, or length and appearance of recovered cores;
- Description of the sediment including texture and consistency, color, presence of biota or debris, presence of oily sheen, changes in sediment characteristics with depth, and presence/location/thickness of the redox potential discontinuity layer (a visual indication of black is often adequate for documenting anoxia); and
- A photograph of the sample is desirable, especially longitudinally-sectioned cores, to document stratification.

Core samples should be collected if depth profiling, historical analysis, or reduced oxygen exposure is required. Following collection methods outlined in EPA's *Methods for Collection, Storage and*

*Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (EPA, October 2001), core sampling procedures ensure:

- The core sampler was not inserted at an angle or tilted upon retrieval.
- The core collected the required depth to meet the study objectives, with no loss of sediment.
- The volume of overlying water in sediment samples should be minimized to reduce the potential for re-suspension of surface sediments during transport.
- Care should be taken to retain the surficial floc overlying a core sample.
- Core samples may be best shipped as intact core samples, using the core sampler tube liner as a shipping container. Prior to transport, headspace in the core liner should be filled with site water and both ends of the liner should be completely sealed. Cores should be secured in an upright position during transport to minimize disturbance of the sediment.

Processing sediment samples in the field or laboratory may also involve homogenization, sieving, and other manipulations prior to chemical analyses. ***In all cases, samples collected for VOC analysis should be sub-sampled and collected directly from the core sampler or grab sampler, before mixing the sample, to minimize volatilization of contaminants.***

Advantages:

- Sediment grab and core sampling is typically simple to perform (particularly if bathymetric information is available prior to sampling).
- Sediment pore water can be extracted from core samples.
- Bioavailable concentrations of contaminants may be determined from pore water.

Disadvantages:

- Samples are snap-shot of conditions at the time of sampling; not time-integrated.
- Some sites may require use of boats or vessels to sample sediment locations.
- Core samplers typically have a small surface area and may require multiple samples to represent site conditions.
- Ex-situ sediment pore water extraction is known to change pore water chemistry to some degree (although such changes may be minimized or controlled with proper handling and processing).

## 9.2 Sediment pore water sampling

Sediment pore water sampling can be used to characterize groundwater transport and discharge to surface water bodies, identify sediment contamination and exposure pathways, and evaluate ecological risk. Pore water can be collected from sediment samples, or via active sampling methods, or by the use of passive sampling methods. Pore water sampling also commonly involves sampling from the groundwater-surface water interface, so CSP groundwater cleanup levels and Alaska Water Quality criteria and standards may apply when reporting the data. Refer to the CSP's *Regulatory Approach to Managing Contamination in Hydrologically Connected Groundwater and Surface Water* (DEC, April 2011).

## 9.2.1 Extracting sediment pore water from sediment samples

### 9.2.1.1 Extraction via centrifugation

Sediment pore water can be extracted *ex-situ* from sediment samples collected as either grab samples or core samples; the latter can be collected in a manner to preserve anoxic sediment conditions. Centrifugation is generally the preferred laboratory method for the extraction of interstitial water. The following guidelines are applicable for centrifugation:

- Extract sediment pore water as soon as possible.
- Interstitial water that has accumulated on the surface of the homogenized sediment sample should be mixed into the sediment before the sample is partitioned among centrifuge bottles.
- Sediments should be centrifuged at high speed (e.g., 8,000-10,000 x g force) for 30 minutes.
- Centrifuging should be conducted at 2-6°C to minimize temperature-mediated biological and chemical processes.
- Extracted sediment pore water should be preserved immediately for chemical analyses or analyzed as soon as possible after extraction, unpreserved. For toxicity testing, store at 2-6°C for not longer than 24 hours, unless the test method dictates otherwise.
- Filtration should be avoided unless required by a test method because it might reduce interstitial water toxicity. Double (serial) centrifugation (low speed followed by high speed) should be used instead.
- If filtering is required by a test method, pre-treated filters should be used to reduce potential contamination.

### 9.2.1.2 Extraction via other methods

Additional methods of obtaining sediment pore water include sediment squeezing, vacuum filtration, gas pressurization and displacement. Generally, these methods are known to alter sediment pore water chemistry and characteristics by causing changes in equilibrium from pressure, temperature, and gradient changes. These methods should not be used for obtaining sediment pore water.

## 9.2.2 Active sampling methods for pore water

The CSP defines active pore water sampling as that which involves the use of pumps and PushPoint™ samplers, temporary well points, soil vapor implants (commonly installed with a slide hammer or fence post driver), or small-diameter pre-packed wells to provide discrete, single-point-in-time (i.e.,



Figure 25. Photo showing vapor implant (top) and Henry Sampler (bottom) push points; courtesy of Chris Eckley, EPA, Region 10

snap-shot) samples. Installation is commonly performed using hand tools, although larger mechanical equipment may be used for deeper installations. Although many variations of push points, well points and sampling apparatus exists, CSP recommends the use of one-inch inner-diameter (I.D.) well points to facilitate the use of submersible or inertia pumps to minimize the loss of VOC during sampling. However, CSP also recognizes that EPA guidance titled: *Operating Procedure: Pore Water Sampling* (EPA, May 2020) that describes the use of low-flow peristaltic pumps to collect pore water samples. In

this particular methodology, the low-flow pump is stopped after drawing pore water into the tubing, and the portion of tubing connected to the well point is removed (without losing the water in the tubing), and the samples are collected from this lower portion of tubing, again to minimize loss of VOC from the sample. This approach allows the use of smaller diameter points and tubing. Once collected, samples of pore water collected by active methods can be handled, processed and analyzed with methods established for groundwater and/or drinking water samples.

This sampling method is likely to capture colloidal material, onto which contaminants of concern may adhere (e.g., PAH, metals, etc.). Colloids may or may not be removed during laboratory extraction and analysis, depending on project objectives and laboratory procedures used. It may be important to consider colloids when interpreting pore water data or comparing to other sampling methods (e.g., passive sampling). Consult the CSP Project Manager for more project-specific guidance.

#### Procedures for Use:

- Assemble the drive rod (and guard rod if using one), drive point, tubing and slide hammer (if using a PushPoint™, no assembly may be necessary).
- Insert the assembly at the sampling location and advance to the desired depth (often the groundwater-surface water interface). It may be necessary to use a flange if deploying through a surface water column to prevent intrusion of surface into the pore water sample.
- Remove the drive or guard rod.
- Purge and sample by using a pump inserted into the rod assembly or with a low-flow peristaltic pump.
- Remove the drive rod and pull tubing from buried well point or implant, or remove entire PushPoint™.
- An alternative is to install one-inch I.D. pre-packed screened monitoring wells (with direct push equipment or via trenches dug to just above the ground smear zone), and develop and sample as for drive points.

#### Advantages:

- Inexpensive and easy to use.
- Multiple equipment choices and configurations are available for sample collection.

#### Disadvantages:

- Push point or implant is expendable.
- Frozen soils or gravely sediments or subsurface refusal may limit desired depth to collect pore water.
- Well point or implant screen may clog with sediment during sampling.
- Samples are snap-shot of conditions at the time of sampling; not time-integrated.

## **9.2.3 Passive sampling methods for pore water**

### **9.2.3.1 Equilibrium-based samplers**



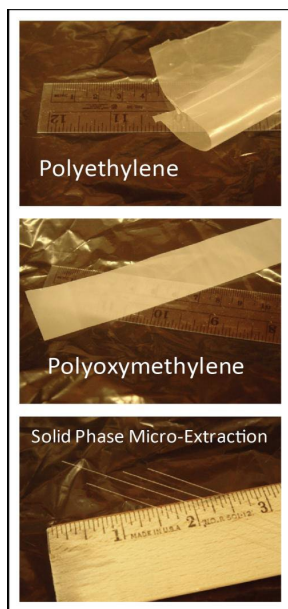


Figure 26. Polymer passive sampling materials; source: EPA 2012

One form of passive sampling methods involves the use of equilibrium-based polymer sampling devices, which are typically deployed in-situ for extended lengths of time (e.g., 30 days or more). Under equilibrium, the contaminant composition of the sampler water will match that of the surrounding pore water. Common polymer passive sampling materials include polyethylene (PE), polyoxymethylene (POM), and fiber optic cable coated with polydimethylsiloxane; also known as a Solid Phase Micro Extraction (SPME) sampler. These passive samplers are commonly used to evaluate hydrophobic chemicals in the dissolved phase, and to determine bioavailability of contaminants. EPA's guidance further describes the theory and practical applications of polymer-based passive samplers for sediment pore water; see *Guidelines for Using Passive Samplers to Monitor Organic Contaminants at Superfund Sediment Sites* (EPA, December 2012).

Additional equilibrium-based sampling devices include Peepers (also called in-situ dialysis samplers), which are commonly a rigid material sampler with openings (with many configurations) that exposes a permeable membrane that separates a volume of water (commonly deionized and deoxygenated water) from the pore water environment it is sampling.

Passive Diffusion Bag samplers (mentioned in groundwater sampling equipment) can also be deployed in protective screened housings for deployment in sediments. Peepers are similar to PDB samplers, but may be constructed of different membrane materials (e.g., polysulfone or cellulose), and were originally designed to sample the groundwater-surface water interface.

When equilibrium-based passive samplers are used, CSP requires an approach to ensure samplers have achieved equilibrium with their environment during deployment. This may include a sufficiently

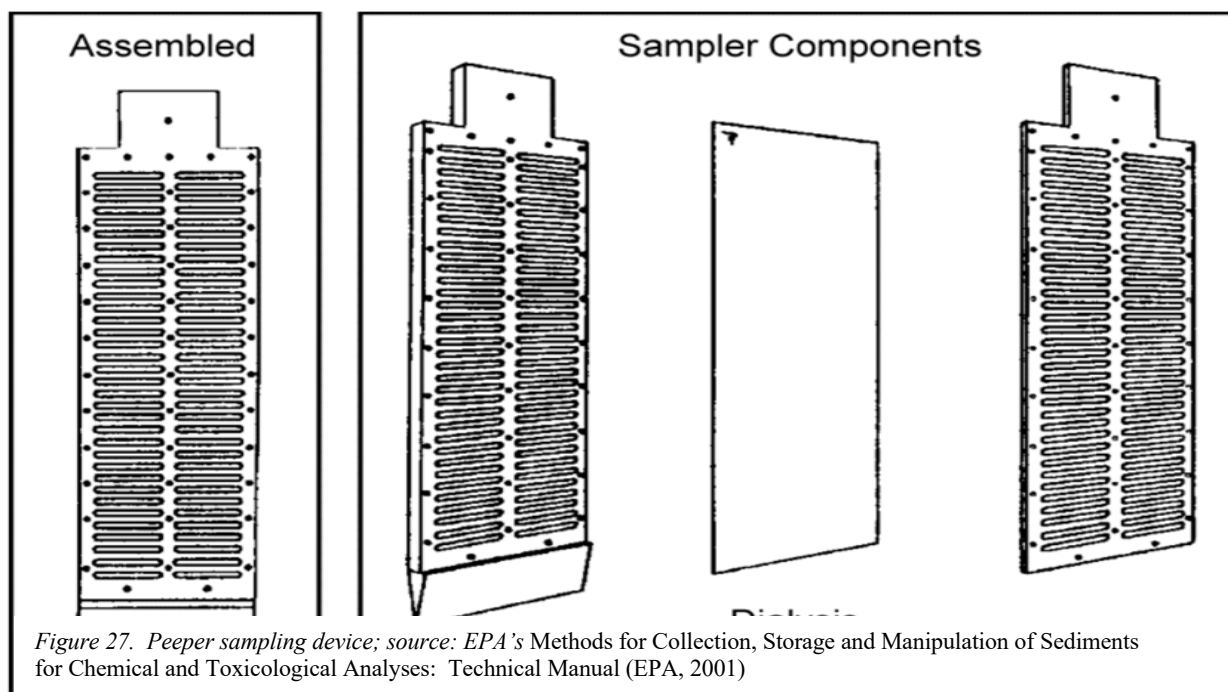


Figure 27. Peeper sampling device; source: EPA's Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual (EPA, 2001)

long duration of deployment (e.g., 45 days, or duration based on empirical evaluation) or use of Performance Reference Compounds (PRC), which can be used to determine if the sampling device has achieved equilibrium with its environment. For example, known quantities of a PRC can be pre-loaded into samplers prior to deployment, and their loss can be quantified after retrieval and used to demonstrate the sampling device has achieved equilibrium, or to estimate concentrations of contaminants under modeled equilibrium conditions. Because of the uncertainty of equilibrium conditions in the environment and how well the PRC mimic behavior of contaminants of concern,

CSP may require the inclusion of PRC during the use of passive sampling on a case-by-case basis.

Determining dissolved phase concentrations of contaminants requires use of dissolved phase partition coefficients ( $k_d$ ) for each sampling device (material) and contaminant being sampled, that can be calculated from the contaminants  $K_{ow}$ , determined empirically by the laboratory, or may be reported in the scientific literature.

### 9.2.4 Sorbent-based Samplers

Additional passive samplers that are sorbent-based diffusion samplers are available for sampling VOC in sediment pore water. These include the GORE Module (also known as the GORESORBER™ Module), devices containing activated carbon (such as the Bio-Trap™), the Polar Organic Chemical Integrative Sampler (POCIS™), and Semi-permeable Membrane Devices (SPMDs). Semi-permeable Membrane Devices are similar to the PDB samplers, but are filled with triolein (a fatty acid compound used to simulate the bio-concentration of contaminants into lipid tissues of aquatic organisms). Semi-permeable Membrane Devices may be used to sample sediment pore water in some situations, but they are more commonly used to sample the overlying water column. The range of contaminants the various sorbent-based sampling devices can sample may also include more than VOC. Sorbent-based passive samplers accumulate contaminants over the duration of the deployment time. As with polymer samplers used for hydrophobic contaminants, PRC may be incorporated into

sorbent-based passive samplers use to evaluate sampling rates during deployment.



Figure 28. SPMD wound on sampling apparatus; source EST Labs, Inc.

Use of some passive samplers may require patent rights to be observed and may be only available for purchase and/or analysis from sole source vendors and laboratories. Analytical laboratories that are not otherwise approved to perform sample analysis for contaminated sites in Alaska may offer analysis of specific passive samplers and may be approved by CSP on a case-by-case basis.

#### Procedures for Use:

- Use of passive samplers requires solvent cleaning prior to deployment (and possibly loading with a PRC), so coordination with the analytical laboratory is necessary beforehand.

- In addition to samplers, hardware for deployment should be clean and free of petroleum hydrocarbons.
- Deployment in the field can utilize many different configurations for protection and securing passive samplers for later retrieval. Consult EPA's *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual* (EPA, October 2001) for additional guidance on deployment variations.
- Utilize blanks; including fabrication, field (exposed to surrounding air during sampler deployment and retrieval), trip (remaining in sealed container; should include sampler and solvent blanks), and laboratory blanks. If PRC are used with the passive samplers, the trip blanks also should be spiked with the PRC.
- Remove colloids, biofilms, and debris from the surface of passive samplers by rinsing with clean (distilled or deionized) water or site water. If using tweezers to handle sampler materials, be sure to clean them of colloids or biofilm material before handling new samplers.
- After rinsing, place the samplers back into vials or wrap in aluminum foil and seal in ZipLock™ bags without delay. If using vials preloaded with solvents in the field, be aware of shipping and handling restrictions with various freight and air carriers.
- **Important note:** many passive samplers can sample the air as well as the sediment pore water you may be targeting; it is important to handle them carefully by keeping them away from common field gear, such as outboard motors and gas cans, and vehicle exhaust, etc.

#### Advantages:

- Polymer samplers are typically inexpensive, easy to use, and very durable.
- May eliminate the costs associated with purge water/IDW disposal.
- Time-integrated (e.g., time-weighted average) samples may be obtained.
- Very low detection limits (ng/mL) are possible with many passive samplers.
- Partition coefficients may be readily available to determine dissolved phase concentrations of contaminants.
- Bioavailable concentrations of contaminants may be determined.

#### Disadvantages:

- Non-polymer based passive samplers may be more costly than polymer samplers, and limited laboratory analysis may be available.
- A minimum of two field visits is required. A third field trip may be required if samplers cannot be retrieved during the first retrieval attempt.
- Extended deployments (e.g., 30 to 45 days), or samplers readily visible for retrieval may increase the risk of vandalism or theft.

### **9.2.5 Passive sampling sediment pore water for metal contaminants**

As with hydrophobic organic compounds (HOCs), pore water concentrations of metals may be useful to predict the bioavailability of metals to aquatic organisms or provide a more relevant exposure metric than bulk sediment metal concentrations. Metals dissolved in pore water are also often the partitioned component of sediments that are chemically available for reactions (e.g., mercury

methylation). Sampling pore water for metals can occur by active or passive sampling methods. Active methods include using sediment core sampling or grab sampling (e.g., ponar dredge). Active sampling is typically followed by pore water extraction via centrifugation or sediment squeezing. Passive sampling for metals is less established than for HOCs, and there is a lack of clear guidance on sampling protocols, calibration methods, and data interpretation for many metals. Peepers are probably the most common equilibrium-based sampling approach for metals, but other equilibrium sampling devices (e.g., Gellyfish) are also available; see Table 5. Metals that have been sampled with passive samplers include silver, cadmium, chromium, copper, nickel, lead, zinc, as well as iron and manganese. When using passive samplers for dissolved metals in sediment pore water, it is recommended that additional sediments and pore water characteristics and parameters also be determined; these include: acid volatile sulfide (AVS), sediment total organic carbon (TOC), oxidation-reduction potential (ORP), pH, dissolved organic carbon (DOC), dissolved oxygen, temperature, and grain size. This information will be useful in identifying vertical transition zones from oxic to anoxic conditions in sediments and sediment pore water, and the extent of chemical interactions and potential metal complexes that may form in pore water (e.g., reactions with sulfate, carbonate, DOC, etc.). When using passive samplers (such as Peepers) for dissolved metals in sediment pore water, sampling devices should be de-oxygenated prior to deployment (e.g., water within the sampling device should be purged with Argon, or other inert gas).

When using active sampling methods and extracting pore water from sediments (e.g., centrifugation), pore water is filtered through 0.45 µm filters to remove colloidal materials. However, colloids may also be important to consider for bioavailability determinations, depending on project objectives. A decision may be made to eliminate filtering as a sample processing step, or to utilize filters with larger-diameter pore spaces to allow colloids to pass through. Consult with the CSP Project Manager for further guidance. Use of PRC is not common practice when using passive sampling methods for dissolved metals in sediment pore water.

Table 5. Summary of passive sampling media and configurations for organic and metal analytes

Passive Sampling Media	Configuration	Target Analytes
Polydimethylsiloxane (PDMS)	Coated fiber, vial	HOCs
Polyethylene (PE)	Film/sheet, tube	HOCs
Polyoxymethylene (POM)	Film/sheet	HOCs
Ethylvinylacetate (EVA)	Coated vial	HOCs
Silicone rubber (SR)	Sheet, ring	HOCs
Gels	Diffusive gradient thin film (DGT)	Metals
Resin impregnated polyacrylamide gel	“Gellyfish”	Metals
Metal-chelating media	Disk/membrane	Metals
Water-filled equilibration cell	“Peeper”	Metals

Note: HOCs = Hydrophobic organic compounds.

Adapted from: Parkerton *et al.* 2012. Guidance on passive sampling methods to improve management of contaminated sediments: Summary of a SEATAC Technical Workshop: Pensacola, Florida. Society of Environmental Toxicology and Chemistry (SEATAC).

## 10.0 Fish Tissue

Persistent environmental contaminants, such as per- and polyfluoroalkyl substances, polychlorinated biphenyls (PCB), pesticides, and heavy metals such as mercury have been found to bioaccumulate and

have been detected in fish posing ecological and human health risks. Once released, certain hazardous substances can biomagnify up the food chain, sometimes resulting in high concentrations in apex predators. The laboratories that accept fish tissue samples for contaminant analysis include commercial laboratories and the DEC Environmental Health Laboratory.

Please refer to DEC's Division of Environmental Health *Field Manual for the State of Alaska Fish Monitoring Program* (DEC, March 2017) for guidance on fish species selection, identifying fish sex, and sampling design for collecting representative fish samples for contaminant analysis. Fish samples that are submitted to the DEC Office of the State Veterinarian should follow the most current *Quality Assurance Project Plan* for the State of Alaska Fish Monitoring Program. Other resources available that provide guidance on fish sampling include:

- American Fisheries Society's *Standard Methods for Sampling North American Freshwater Fishes* (Bonar et al., August 2009) publication located at: <https://fisheries.org/bookstore/all-titles/professional-and-trade/55059c/>
- State of Washington *Standard Fish Sampling Guidelines for Washington State Ponds and Lakes* (Bonar et al., 2000) located at: <https://wdfw.wa.gov/sites/default/files/publications/00455/wdfw00455.pdf>
- EPA Region 4, *Fish Field Sampling Operating Procedure* (EPA, April 2020) located at: <https://www.epa.gov/sites/production/files/2015-06/documents/Field-Fish-Sampling.pdf>

CSP recommends collaborating with the Alaska Department of Fish and Game (ADF&G) and/or the United States Fish and Wildlife Service (USFWS) for assistance on developing work plans that target fish life stages and species and to address any licensing or permitting requirements.

## **10.1 Fish Sampling, Handling, and Delivery Procedures For Contaminant Analysis**

On the day of collection, collector name, address, date, location (latitude and longitude) of fish capture, species, and number of each species kept should be documented. Fish capture shall comply with State and Federal laws including licensing and permitting requirements. Every effort should be made to collect fish that are representative of a given location; therefore, samples should be collected from different depths and locales within a lake or stream. The species collected will depend on the data quality objectives of the project. The species life stage, sex, as well as geographical and seasonal life history parameters may all need to be considered during sample planning. Consult with ADF&G or USFWS for more information on targeting fish species and fish capture methods and approaches. A minimum of three individuals of the target species should be collected and a total sample mass of fish species should be at least 10 grams. Some contaminant concentrations tend to increase with increasing fish size, thus collecting fish of different lengths should be considered. If ecological risk assessment is the project goal, targeting species that piscivorous birds and mammals consume should be considered. Compositing of fish samples can be approved depending on the data quality objective and provided fish are collected of similar size (i.e., smallest fish within 75% of largest fish as measured by fork length). If the goal of the study is to determine the variability of contaminants in fish populations, then compositing is not recommended. The collection of field duplicates fish tissue samples is not required.

Care in handling of fish during collection is needed to ensure specimens are not being contaminated. This includes handling fish with latex or nitrile gloves, as appropriate, and storing fish in a sealed clean plastic bag. Samples should be collected at a clean location (e.g., avoid bilge water or boat exhaust locations). Rinse fish in ambient water to remove debris. Sample and analysis of the whole fish is the common approach but will ultimately depend on the data quality objectives. It is recommended that the chosen commercial laboratory fillet or cut fish prior to analysis. If captured fish is cut or filleted in the field to meet data quality objectives, then decontamination procedures should be conducted and approved by CSP. Fish tissue should be placed in a food grade resealable clean plastic bag. Non-lethal forms of fish capture for collecting fish tissue can be approved by CSP to meet site-specific data quality objectives. It's important that each fish be placed in its own individual clean plastic bag to avoid cross contamination. For small fish to be analyzed as a composite, put all fish for the composite sample in a single resealable clean plastic bag. The resealable plastic bag should be labeled with the date, species, and sample number. Fish samples should be kept as cool as possible immediately after harvesting. Fish should be put on ice or gel packs and either shipped immediately to the laboratory, or frozen within 12 hours. As soon as possible, fish samples should be frozen to a minimum of  $-20^{\circ}\text{C} \pm 5^{\circ}\text{C}$ . Temperature logs of the freezer should be kept to document freezer did not fail during fish storage. Electronic loggers may also be required for coolers to ensure samples haven't been exposed to excessive temperatures.

Once frozen, the fish samples can be submitted to the laboratory and analyzed in accordance with an approved method identified in the project's QAPP or work plan. A fish collection record is provided in Appendix G of this document. Fish samples should be delivered to laboratories under standard chain of custody procedures with custody seals applied to coolers if samples are not hand delivered. Laboratories selected for analyzing fish samples should be listed in CSP work plans for review and approval.

The following data should be recorded on a fish tissue sampling form:

- Project name;
- Qualified Environmental Professional responsible for collecting tissue samples including name, address, phone number, and email;
- Tissue sample identifier or Tag Number;
- Species identification;
- Collection date;
- Sample location (latitude and longitude coordinates) and description of capture vicinity;
- Length (use fork length); and
- Weight- measurements should be collected as soon as possible after collection with calibrated and protected instruments (e.g., from wind).

If sampling equipment is to be reused, it should be thoroughly cleaned with detergent, rinsed in isopropanol, and washed with distilled water before each specimen is processed. Original copies of Chain of Custody and tissue sampling forms should accompany delivery tissue to the laboratory.

## **10.2 Special Requirements for Per- and Polyfluoroalkyl Substances in Fish**



Contact the laboratory performing the analysis, they may require that you use laboratory-supplied sample containers for sample collection. In general, do not use any materials containing Teflon or polytetrafluoroethylene (PTFE). Post-it notes should not be used on sample containers. Avoid materials containing waterproof coatings, coated Tyvek clothing, and anything containing “fluoro” in the name. Avoid plastic materials other than HDPE. Sample containers should be specified in the analytical method and certified PFAS-free. Staff should avoid cosmetics, moisturizers, hand creams and similar products on day of sampling. Gloves worn should be powder free nitrile. Aluminum foil is not recommended to be used during the sampling process, and if it is used, it should be certified PFAS-free.

If matrix interference is suspected, cleanup procedures may be proposed but should be approved by CSP on a site specific basis. CSP recommends adhering to ITRC PFAS fact sheet on *Sampling Precautions and Laboratory Methods for Per- and Polyfluoroalkyl Substances (PFAS)* (ITRC, August 2020) for sampling precautions, decontamination procedures, and laboratory methods.

## **11.0 Other Environmental Media**

Consult with CSP project manager for specific guidance when sampling other media (e.g., blueberries, shellfish, etc.) as part of bio-monitoring or risk assessment.

## **12.0 Quality Control (QC) Measures**

It is expected that all sampling and field screening activities discussed in this document are performed using standard industry methods and practices. In addition, all sampling and field screening methods are performed using tools and instruments that are either single use (disposable) or are free of contamination and will not contribute to false readings in the field or in the laboratory. Field instruments should be calibrated on a periodic basis and documented in a field record or logbook. Deviations from this section may be approved by CSP on a site-specific basis but should be clearly identified and discussed in the work plan and report.

### **12.1 Field Documentation**

Document all field readings, sample locations, and field observations in a field record or logbook. Logbooks or field records should be bound books that are permanently assigned to a specific project. Field forms and camera may also be used for field documentation in a variety of activities. Field forms include borehole logs, well construction, well sampling, site safety and health plan forms, etc. It is not necessary to duplicate information recorded on a field form into the logbook. All logbooks and field form entries should be printed legibly using a waterproof pen. All field forms should be completed in full on a daily basis. Entries to the field logs should include the following items if applicable:

- Project name/Site ID/Client/Page Number.
- Date.
- Weather, site conditions, and other salient observations.

- Full name of on-site personnel, affiliations and project title e.g., team leader (including all visitors).
- Daily objectives.
- Time and location of activities.
- Field observations and comments.
- Deviations from the CSP site-specific approved work plan.
- Photographic log (photographic name, roll or frame number, description of photograph, date, and time).
- Site sketches with reference to north direction, sample and field screening locations and depths, and on-site groundwater flow direction.
- Survey and location (latitude and longitude coordinates when possible).
- All field measurements (e.g., leak check results, geochemical parameters, field screening results).
- Daily equipment calibrations and maintenance.
- Sample record (sample identification, date, time, media, number of samples, and location).
- Cleanup or remediation activities (system performance, system calibration or maintenance record, excavation activities and volume of material removed).
- Waste tracking (when, media, how much, destination).
- Soil boring logs will include: blow counts, visual or olfactory observations, diameter of boring, total depth of boring, field screening, cone penetrometer testing, readings, soil type, soil moisture, groundwater depth if encountered, soil log completed using the Unified Soil Classification System, U. S. Soil Conservation Service classification system, or another similar soil classification system.
- If a monitoring well is installed, the following information is required in a well log: well location determined by reference to site benchmark, depth to top and bottom of screened interval, depth to water, soil types, diameter of screened interval, diameter of casing, well construction material, depth of packed filter interval, depth and thickness of seals, type of surface cap, and names of drilling firm and personnel.

Correct erroneous field record or logbook entries with a single line through the error. Do not erase incorrect information. Date and initial revised entries. Logbooks and field forms will be kept in the project file when complete or when not in use. Include complete copies of all field notes and field records in reports submitted to the CSP.

## **12.2 Instrument Calibration**

All field instruments should be calibrated prior to each project according to manufacturer's specifications and instrument calibration should be checked and documented. Certain field screening parameters may require more frequent calibrations depending on site conditions, such as temperature, barometric pressure, etc. Retain a reference copy of manufacturer's operating instructions in the field. All instrument users should be trained in routine maintenance and operation. Calibration standard(s), dates, times and all calibration results should be recorded in the field record or logbook.

## **12.3 Sample Containers and General Sample Collection QC**



Obtain containers from the lab with the appropriate preservative. Sample containers should conform to the specifications in the required laboratory procedure. In cases where EMD may be used, sample containers may be sterile and field personnel may need to practice aseptic technique. Sample container and preservative shipments should comply with USDOT and/or IATA regulations.

Inspect sample containers before transit to the site to ensure that they are undamaged and are tightly sealed. Sample containers should be packaged so that they are secured to prevent damage or tampering in transit to the site. Re-inspect sample containers and lids at the job site. Sample containers that have lost lids or that have been damaged may not be used for sample containment.

Use indelible, waterproof ink to label containers. Document information entered onto the label or container in the field record or logbook.

Ensure that sample container threads and rims are clean before tightening lids. Do not tape lids to jars when collecting samples. Change disposable gloves after each sampling location.

Include the following information on the containers or labels:

- Project name;
- Unique identifying alphanumeric assigned to the sample for laboratory analysis;
- Date and time of collection;
- Sampler's name or initials;
- Requested laboratory analysis; and
- Preservative, as applicable;

All sample numbers need to be unique and the number convention should be discussed in the work plan. Use packing material, such as bubble wrap around glass jars to prevent breakage during transport. Unless specified in a CSP site-specific approved work plan, samples should be placed in a cooler that is kept under 6°C and held in the dark. Samples should be verified that they are properly labeled and that field sample forms including a Chain of Custody are properly filled out. During transport and storage of samples, maintain strict chain of custody and place chain of custody seals on coolers and boxes during transport.

## **12.4 Sample Preservation for Biological EMD Analyses**

Preservation of samples to be analyzed via biological EMD methods depends upon the biomolecule of interest (i.e., whether it is DNA, RNA, or whole cells). Nucleic acids are susceptible to degradation from high temperatures, acid or alkaline conditions, or enzymes that specifically degrade them (e.g., DNases or RNases). Use of preservatives may be appropriate to maintain the integrity of DNA; preservatives are required to stabilize RNA. Preservatives used to stabilize groundwater cations or anions are typically not used. Alternatively, freezing soil cores or groundwater samples at -80° Celsius may preserve nucleic acids for subsequent analysis. Exceptions to freezing samples include EMD that analyze whole cells (e.g., Fluorescence In-situ Hybridization) or use of Bio-Trap™ passive sampling devices. Generally, samples collected for whole cell analysis or via Bio-Trap™ sampling devices will be chilled to 2-6°C and transported via overnight delivery. Consult the laboratory prior to

sampling and transport to ensure holding time problems can be avoided or minimized, or if freezing or use of stabilizing agents is appropriate.

## 12.5 Quality Control Field Sample Collection

A sampling approach that is properly designed and implemented will allow the sampling objectives to be met, avoid confusion in the field, and contribute to the generation of high-quality data necessary to support defensible decision-making. Measures of quality include the appropriateness and accuracy of the sample collection, adherence to sample handling protocols, quality and appropriateness of the laboratory analysis, and representativeness of the data with respect to the study objectives. Quality Control (QC) activities should be documented in field record or logbook. Collect QC samples per the requirements in Table 6. For guidance on laboratory data, see CSP's technical memorandum *Minimum Quality Assurance Requirements for Sample Handling, Report and Laboratory Data* (DEC, October 2019).

Table 6. Minimum Quality Control Requirements

Minimum Field QC Samples	Applicability	Allowable Tolerance
Field Duplicate (Minimum of 1 per every 10 field samples for each matrix sampled for each target analyte, minimum of 1)	All soil and water samples	Relative percent differences (RPD) less than: 30% water, 50% soil
Decontamination or Equipment Blank (1 per set of 20 similar samples, minimum of one)	Per project specifications	Less than the practical quantitation limit
Trip Blank – Water (1 trip blank per analysis and cooler)	All water samples being analyzed for GRO, BTEX, or VOC	Less than the practical quantitation limit
VOC Trip Blank – Soil (1 trip blank per preservation method per set of 20; a minimum of 1 per analysis and cooler)	All soil samples being analyzed for GRO, BTEX, or VOC	Less than the practical quantitation limit
Temperature Blank or Cooler Temperature (minimum 1 per cooler)	All soil and water samples	Less than 6 °C
Field Blank (1 per set of 20, minimum of 1)	Per project specifications. Used for highly contaminated sites with VOC	Less than the practical quantitation limit

## 12.6 Field Duplicate Requirements

Field duplicates provide a measure of the precision of the sampling process and sample heterogeneity and thus are an important quality control parameter to evaluate. A minimum of one field duplicate should be collected for every 10 field samples for each matrix sampled and for each target analyte. Field duplicates should be collected from locations of known or suspected contamination, and duplicate soil and water samples should be collected in the same manner and at the same time and location as the primary sample. For a sampling occurring over multiple days, all field duplicates

should not be collected in one day and the goal should be to collect field duplicates over multiple field days.

Field duplicates should be:

- Submitted as blind samples to the approved laboratory for analysis;
- Given unique sample numbers (or names) and sample collection time; and
- Adequately documented in the field record or logbook.

Field duplicate results should be used to calculate and report a precision value for field sampling quality control according to the following equation:

$$RPD (\%) = \left| \frac{R_1 - R_2}{\frac{(R_1 + R_2)}{2}} \right| \times 100$$

Where:

$R_1$  = Sample Concentration

$R_2$  = Field Duplicate Concentration

An exceedance of the allowable tolerance limits suggests that the precision of the sampling effort is insufficient. Inadequate precision could be due to various issues including poor sampling methodology.

## 12.7 Trip, Field, and Equipment Blank Requirements

The primary purpose of quality control blanks (i.e., trip, field, and equipment blanks) is to trace sources of artificially introduced contamination. Field blanks are a sample of preservative or deionized water poured into the container in the field, and shipped to the laboratory with field samples. Per project specifications, a minimum of one field blank will be collected per 20 samples per matrix. Equipment blanks are samples of analyte-free water poured over or through decontaminated field sampling equipment prior to the collection of environmental samples. Per project specification, a minimum of one equipment blank will be collected per 20 samples per matrix. Exceeding allowable tolerance limits for equipment or field blanks suggests that field contamination may have affected associated sample results.

Trip blanks are a clean sample of a matrix that is taken from the laboratory to the site and then transported back to the laboratory without having been exposed to sampling procedures. One trip blank is required per cooler. Exceeding allowable tolerance limits for trip blanks suggests that contamination was introduced during shipping and field handling procedures.

## 12.8 Equipment Decontamination

Depending on the contaminant, wash water and rinsate solutions may need to be collected in appropriate containers and disposed of properly in accordance with federal, state, and local regulations. Proposed decontamination water management needs to be described in work plans.

Decontaminate all reusable equipment such as steel tapes, well sounders, transducers, and water quality probes after each sampling point using a stiff brush and a solution of water and laboratory-grade detergent. An appropriate solvent may be used to remove heavy contaminant residues from the sampling tools. If necessary, sampling equipment can be sterilized in the field with chemical disinfectants, (e.g., detergents, hydrogen peroxide, sodium hypochlorite, ethanol, etc.) or heat (flame) sterilization. Rinse tools twice in clean water and again with distilled or deionized water.

Properly collect, store, and dispose of solvent waste and wash water in accordance with hazardous waste regulations, if applicable, and the CSP site-specific approved work plan. Clean drill auger sections, split spoons, and drive hammers that come in contact with bore holes before use and between borings. Scrub tools with a stiff brush in a solution of water and laboratory-grade detergent. High pressure water or steam may also be used.

Visibly contaminated decontamination water for sites with petroleum hydrocarbons may be containerized for off-site shipment, or with CSP site-specific approval, filtered on-site and re-applied directly to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies. If not visibly contaminated, decontamination water may be re-applied directly to the ground surface within site boundaries a minimum of 100 feet away from any drinking water wells and/or surface water bodies, if approved in a CSP site-specific work plan.

Refer to ASTM D5088 *Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites* (ASTM, 2015).

## **12.9 Laboratory Certification**

While a laboratory should assure satisfactory levels of quality control within the laboratory to maintain its status with DEC, the qualified environmental professional should:

- Verify that the laboratory being used is certified by CSP;
- Ensure that analytical testing meets the objectives of the project and regulations;
- Report in any project report any deviation from standard laboratory procedures of which they become aware; and
- Take appropriate corrective actions if questions or problems arise with the laboratory analysis.

## **12.10 Chain-of-Custody and Sample Handling and Shipment**

The purpose of the chain-of-custody is to demonstrate accountability and document sample integrity from the time of sample collection until sample analysis.

CSP requires the following elements of chain-of-custody for sample collection:

- Sample labeling;
- Laboratory receipt forms;

- Field custody form (chain-of-custody form);
- Custody seals on all coolers; and
- Inter-laboratory transfer documentation, if applicable.

Maintain samples according to the holding times and temperatures in Appendix C, Appendix D, and the approved CSP site-specific work plan. The chain-of-custody form should include information on analyses specifying the methods to be performed. Do not place samples into the shipping container unless they are recorded on the chain-of-custody form. Obtain a copy of the shipping manifest if using a lab courier or commercial carrier for sample shipment. Sample coolers/containers should arrive at the lab with an intact and correctly applied custody seal unless the coolers are hand-delivered. If the seal was broken at some point during transport, the reason for breaking the seal, condition of the container contents, the cooler temperature, and anything added to or removed from the container should be documented on the chain-of-custody form. Temperature blanks should be provided for each cooler and measured to ensure preservation requirements are met. If a temperature blank is not provided in a cooler, a cooler temperature measurement should be recorded. The container should then be sealed with a new custody seal. Sample shipments must comply with USDOT and/or IATA regulations. Refer to ASTM D4840 *Guide for Sample Chain-of-Custody Procedures* (ASTM, 2018) and ASTM D6911 *Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis* (ASTM, 2015) for additional information.

### 13.0 Reporting

Following the completion of field work, a report should be submitted to CSP for review. A qualified environmental professional must prepare reports required under 18 AAC 75 and 18 AAC 78. The report needs to include a detailed description of every phase of the sampling effort and methodology and include a discussion of the analytical results. Descriptions of the sampling effort should match the CSP site-specific approved work plan or any deviations that occurred in the field should be explained and documented in the report. The sampling results should be well organized in tabular format. Sample and field screening locations should be depicted on figures with a scale and an arrow pointing north so sample results can be cross-referenced against data tables and other site figures or pictures. The report narrative should define the extent of contamination vertically and horizontally determined by the sampling effort and identify any data gaps that remain. Soil boring logs, monitoring well logs, complete copies of field notes, laboratory data packages, and CSP laboratory review checklists should be included in the report. The report should also include a quality assurance section discussing data quality and usability in accordance with CSP's *Minimum Quality Assurance Requirements for Sample Handling, Report and Laboratory Data* (DEC, October 2019) located at: <http://dec.alaska.gov/spar/csp/guidance-forms/>. Data that has been qualified shall be identified in the report text, tables, and figures. Laboratory data that has been rejected shall not be shown in report tables or discussed in the report results. Rejected and qualified laboratory results should be discussed and explained in the quality assurance section of the report.

Refer to CSP's *Site Characterization Work Plan and Reporting Guidance for Investigation of Contaminated Sites* at: <http://dec.alaska.gov/spar/csp/guidance-forms/> (DEC, March 2017) for further guidance on reporting elements. These may vary on a site-specific basis.

## 14.0 Investigative Derived Waste

In accordance with 18 AAC 75.360(3)(c), a waste management plan is required for handling, transporting, and disposing of investigative derived wastes (IDW). Soil sampling waste should be managed in a manner that does not contribute to further environmental degradation or pose a threat to public health or safety. On-site IDW disposal may be approved by CSP on a site specific basis.

For work conducted at contaminated sites, with CSP approval, contaminated soil cuttings can be managed accordingly:

- Returned to the borehole or spread on the land surface for soil that is not known or suspected to be contaminated.
- Stockpiled or containerized and sampled to determine disposal options if soil comes from an area of suspected contamination due to knowledge of nearby releases or contaminant migration.
- Returning soils that are suspected to be contaminated to a borehole may be evaluated on a site-specific basis, with consideration of the creation of preferential pathways, soil heterogeneity, and other fate and transport concerns.
- At locations downgradient of known or suspected releases, soil from the vadose zone may be treated as uncontaminated (unless evidence suggests it is) and disposed by spreading on the surface nearby. Soil from the smear zone or below should be sampled prior to disposal.

All monitoring well development and purge water should be treated or disposed of using methods described in an approved CSP site-specific work plan.

Proper waste characterization is important to determine whether any RCRA hazardous waste is generated during well development, purging, or sampling, and if so, it must be treated or disposed of in accordance with RCRA and the approved CSP site-specific work plan.

If free product or a heavy sheen are present in development or purge water, then it needs to be treated or disposed of off-site.

If contamination is not visible (i.e., no free product or heavy sheen), the monitoring well purge water may be filtered using a type of water filter system appropriate for the contaminants in the purge water. IDW filtration for on-site disposal should be conducted in accordance with a CSP-approved standard operating procedure that describes the sampling and management of IDW effluent. If granulated activated carbon (GAC) is being used to filter out petroleum constituents and sampling of effluent is not proposed, calculations of GAC breakthrough should be provided to CSP for review and approval before on-site disposal of purge water. The IDW management standard operating procedure should provide details about GAC monitoring and tracking procedures, and plans for final disposition of any spent GAC. CSP may request tracking spreadsheets, or other documentation that demonstrates that the timing of contaminant breakthrough is correctly estimated and that steps are being taken to prevent breakthrough from occurring.

Filtered purge water may be reapplied to the ground surface in an area where the soil contains the same contaminants within site boundaries which is a minimum of 100 feet away from any drinking water wells and/or surface waters, with site-specific CSP approval.

## Appendix A – References

Alaska State Regulation 18 AAC 80.

Alaska State Regulation 18 AAC 75.

Alaska State Regulation 18 ACC 78.

ASTM D1586-08 (2018). *Standard Test Method for Standard Penetration Test (SPT) and Split-Barrel Sampling of Soils*.

ASTM D4840-99 (2018). *Standard Guide for Sample Chain-of-Custody Procedures*.

ASTM D6911-03 (2015). *Standard Guide for Packaging and Shipping Environmental Samples for Laboratory Analysis*.

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ASTM D6001-96 (2002). *Direct Push Water Sampling for Geoenvironmental Investigations*.

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DEC, September 2013. *Monitoring Well Guidance*.

DEC, April 2011. *Regulatory Approach to Managing Contamination in Hydrologically Connected Groundwater and Surface Water*.

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DOD/DOE, September 2019. *Consolidated Quality Systems Manual (QSM) for Environmental Laboratories*: <https://denix.osd.mil/edqw/documents/manuals/qsm-version-5-3-final/>.

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EPA, April 2020. *Fish Field Sampling Operating Procedure; LSASDPROC-512-R5*.

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EPA, September 2014. *3PE: A Tool for Estimating Groundwater Flow Vectors*; EPA/600/R-14/273.

EPA, December 2012. *Guidelines for Using Passive Samplers to Monitor Organic Contaminants at Superfund Sediment Sites. Oswey Directive 9200.1-110 FS*.

EPA, January 2011. *Contract Laboratory Program Guidance for Field Samplers*; EPA 540-R-09-03.

EPA, February 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process*; EPA/240/B-06/001

EPA, December 2002. *Guidance on Choosing a Sampling Design for Environmental Data Collection*.

EPA, September 2002. *Guidance for Comparing Background and Chemical Concentrations in Soil for CERCLA Sites*.

EPA, May 2002. *Ground-Water Sampling Guidelines for Superfund and RCRA Project Managers*.

EPA, October 2001. *Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual*. Office of Water; EPA-823-B-01-002.

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USGS, 1995. *Guidelines for collecting and processing samples of stream bed sediment for analysis of trace elements and organic contaminants for the National Water-Quality Assessment Program*, by Larry R. Shelton and Paul D. Capel. U.S. Geological Survey Open-File Report 94-458, revised 1995.

## Appendix B – Initial Comparison for Selecting the Appropriate Field Screening Method for Petroleum Hydrocarbons

Field Screening Method Categories	Principle Used To Detect & Measure Petroleum Hydrocarbons	Detectable Target Analytes	Effects Caused by Weathered Petroleum Hydrocarbons	Data Quality Objectives	Applicable Concentration Ranges
<i>Colorimetric Wet Chemistry</i>	Colorimetric reagents mix with petroleum hydrocarbons providing a visual response	Petroleum hydrocarbons	Bias is minimal	Qualitative	Yes (petroleum hydrocarbon present) No (petroleum hydrocarbon not present) at a concentration >300 ppm
<i>Headspace Organic Vapor Monitoring</i>	Vapor phase volatile hydrocarbons are ionized or passed through colorimetric reagents for detection	Volatile organic compounds	Low bias due to loss of volatile organic compounds	Semi-quantitative	1.0 to >10,000 ppm
<i>Immunoassay</i>	Some kits are selective for BTEX and aromatic compounds, while other kits are selective for aliphatic compounds	Gasoline, Diesel Fuel, and Heavier Fuels OR Oils	Low bias using test Methods that quantify using BTEX and other aromatic compounds	Quantitative or Semi-quantitative	100 to >25,000 ppm; Semi-quantitative methods provide a greater than/less than to two calibration point concentrations
<i>Infrared Spectrophotometry</i>	Method measures infrared adsorption of C-H bonds present in all organic compounds	Gasoline, Diesel Fuel, and Heavier Fuels or Oils	No bias	Quantitative	100 to >25,000 ppm
<i>Qualitative Physical Screening Methods</i>	Physical properties are used to determine if petroleum hydrocarbons are present	Petroleum hydrocarbons	Bias is minimal	Qualitative	Yes (petroleum hydrocarbon present) No (petroleum hydrocarbon not present)

## Appendix C – Technical and Logistical Screening Method Comparison

Field Screening Method Categories	Factors Affecting Accuracy	Factors Affecting Precision	Training and Required Expertise	Interferences		Waste Byproducts	Logistic Considerations	Comments
				Cause	Effect			
<i>Colorimetric Wet Chemistry</i>	Weathered petroleum mixtures	Soil heterogeneity	Qualified Environmental Professional (QEP) is necessary	Moisture	Low bias from dilution	Petroleum Hydrocarbons Colorimetric reagent mixture	No significant considerations	Test kit literature should be reviewed during the selection process
<i>Headspace Organic Vapor Monitoring</i>	Moisture, weathered petroleum mixtures, operator error	Soil heterogeneity and operator error	QEP is necessary	Moisture and non-target analytes that respond to ionization detector instruments	Erroneous readings	Empty gas cylinders used to store calibration gases	Shipping of USDOT hazardous substances (isobutylene, hydrogen, and methane)	Most ionization detectors are limited by altitudes greater than 4,000 feet and temperatures less than 40°F
<i>Immunoassay</i>	Moisture, weathered petroleum mixtures, multiple petroleum mixtures, operator error	Soil heterogeneity and operator error	Training of QEP is recommended by test kit manufacturers	Moisture and biological organic matter	Low bias High bias	Methanol and Enzymatic reagent wastes	Shipping of USDOT hazardous substances (methanol). Some methods require low temperature preservation	Manufacturer literature should be consulted during the field screening method selection process
<i>Infrared Spectrophotometry</i>	Operator error	Soil heterogeneity and operator error	Trained chemist support necessary	Moisture and biological organic matter	No bias Limited bias with high concentrations	Methanol; hexane; possibly chlorinated solvent wastes	Shipping of USDOT hazardous substances (methanol, hexane, or other chlorinated solvents)	Biological organics and moisture removed during sample extraction process. Excessive quantities of the biological organics may overwhelm the silica gel
<i>Qualitative Physical Screening Methods</i>	Soil adsorption and weathering of petroleum product	Soil heterogeneity	QEP is necessary	Natural organic materials	Limits visibility	Petroleum; soil and water mixture	No significant considerations	Stick Test and Jar Shake Test <sup>(1)</sup> and direct visual observation

### Notes:

<sup>1</sup>The Stick and Jar Shake Tests are described in Appendix A of DEC's *Listing Methodology for Determining Water Quality Impairments from Petroleum Hydrocarbons, Oils, and Grease*, (DEC, 2015)

## Appendix D – Sample Collection Reference Guide – Soil, Sediment, Sludge, Fill Material

Parameter	Analytical Method <sup>(1)</sup>	Container Description (Minimum) [Clear glass may be substituted for amber if samples are protected from exposure to light]	Preservation/ Holding Time
Gasoline Range Organics**	AK101*	4 oz. amber glass, TLS	Methanol preservative, 0° to 6°C / 28 days
Diesel Range Organics	AK102*	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Residual Range Organics	AK103*	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Benzene, Toluene, Ethylbenzene, Xylenes (BTEX) <sup>(4)</sup>	8021B or 8260D	4 oz. amber glass, TLS	Methanol preservative, 0° to 6°C / 14 days
Volatile Organic Compounds (VOC) <sup>(4)</sup>	8260D	4 oz. amber glass, TLS	Methanol preservative, 0° to 6°C / 14 days
Volatile Aliphatic and Aromatic Petroleum Hydrocarbons (VPH)	NWTPH-GX	4 oz. amber glass, TLS	Methanol preservative, 0° to 6°C / 14 days
Extractable Aliphatic and Aromatic Petroleum Hydrocarbons (EPH)	NWTPH-Dx	4 oz. amber glass, TLS	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Dibromomethane 1,2-	8011 or 504.1 or 8260D	4 oz. amber glass, TLS	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
1,4-Dioxane <sup>(6)</sup>	8260D or 8260B	4 oz. amber glass, TLS	Methanol preservative, 0° to 6°C / 14 days
Semi-Volatile Organic Compounds (SVOC)	8270E	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Polycyclic Aromatic Hydrocarbons (PAH) <sup>(2)</sup>	8270E or 8310	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Fraction Organic Carbon	Lloyd-Kahn or 9060 or mod Walkley-Black	4 oz. amber glass, TLC	0° to 6°C / 14 days
Pesticides	8081B or 8270E	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Herbicides	8151A	4 oz. amber glass, TLC	0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Polychlorinated Biphenyls (PCB) <sup>(5)</sup>	8082A	4 oz. amber glass, TLC	0° to 6°C / None, 40 days to analysis of extract (recommended)
Per-and Polyfluoroalkyl Substances (PFAS)	Consult with CS Program for Method, Container, and Holding Times <sup>(8)</sup>		
Metals <sup>†</sup>	6010D or 6020B or 7000 series	100mL Wide mouth HDPE or amber glass jar <sup>(3)</sup> , TLC	None / 6 <sup>(7)</sup> months
Mercury	7470A, or 6010D or 6020B	100mL Wide mouth HDPE or amber glass jar <sup>(3)</sup> , TLC	None / 6 <sup>(7)</sup> months

Notes:

Several of the 7000 series methods have been deleted from SW-846 but these methods may still be approved in a CSP site-specific work plan. Check the laboratory's approval status. The sampling and analysis of soil parameters for alternative cleanup level calculations is discussed in CSP technical memos located at: <https://dec.alaska.gov/spar/csp/guidance-forms/>.

1. Unless otherwise noted, all preparation and analytical methods refer to the most current of EPA's Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846.
2. Naphthalene can be analyzed by 8021B or 8260D, if naphthalene is the only PAH contaminant of concern.
3. HDPE, High Density Polyethylene or amber glass sample collection bottles, certified clean for trace metals analysis.
4. May be analyzed out of AK101 methanol preserved sample.
5. PCB should be prepared using extraction method 3540C or 3550C.
6. High temperature sample preparation techniques by EPA Method SW-846 may be required to improve the recovery and achieve lower detection limits.
7. If bioassays or toxicity testing is to be conducted with metals, then anoxia may need to be maintained, and analyses should occur within 24 hours after sample collection, unless the test method dictates otherwise. Consult the CSP Project Manager for more project specific guidance.
8. Sampling and preservation considerations are discussed in ITRC's *Sampling Precautions and Laboratory Methods for Per- and Polyfluoroalkyl Substances (PFAS)* (ITRC, August 2020).

<sup>†</sup>Hexavalent Chromium can be analyzed with EPA methods 7199 (modified) or 7196A.

## Appendix E – Sample Collection Reference Guide – Groundwater, Surface Water<sup>7</sup>, Marine Water, Drinking Water<sup>7</sup>, Wastewater

Parameter	Analytical Method <sup>(1,3,7)</sup>	Container Description	Preservation/ Holding Time
Gasoline Range Organics (GRO)	AK101*	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Diesel Range Organics (DRO)	AK102*	min. 100 mL <sup>(2)</sup> - 1 L amber glass, TLC	HCL to pH less than 2 / 0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Residual Range Organics (RRO)	AK103*	min. 100 mL <sup>(2)</sup> - 1 L amber glass, TLC	HCL to pH less than 2 / 0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Benzene, Toluene, Ethylbenzene, Xylenes (BTEX)	8021B or 8260D	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Volatile Organic Compounds (VOC)	8021B or 8260D	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Volatile Aliphatic and Aromatic Petroleum Hydrocarbons (VPH)	NWTPH-Gx	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Extractable Aliphatic and Aromatic Petroleum Hydrocarbons (EPH)	NWTPH-Dx	min. 100 mL <sup>(2)</sup> - 1 L amber glass, TLC	HCL to pH less than 2 / 0° to 6°C / 14 days to extraction, 40 days to analysis of extract
Total Aromatic Hydrocarbons (TAH)	EPA 8260D	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Total Aqueous Hydrocarbons (TAqH)	EPA 8260D & 8270E	Duplicate or Triplicate 40 mL VOA, TLS & 1 L amber glass, TLC	HCL to pH less than 2 / 0° to 6°C / 14 days & 0° to 6°C / 7 days to extraction, 40 days to analysis of extract
Dibromomethane 1,2-	EPA 8011 or EPA 504.1 or EPA 8260D	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
1,4-Dioxane <sup>(8)</sup>	8260D	Duplicate or Triplicate 40 mL VOA, TLS	HCL to pH less than 2 / 0° to 6°C / 14 days
Semi-Volatile Organic Compounds (SVOC)	8270E	1 L amber glass, TLC	0° to 6°C / 7 days to extraction, 40 days to analysis of extract
Polycyclic Aromatic Hydrocarbons (PAH) <sup>(5)</sup>	8270E or 8310	1 L amber glass, TLS	0° to 6°C / 7 days to extraction, 40 days to analysis of extract
Pesticides	8081B or 8270E	1 L amber glass, TLC	0° to 6°C / 7 days to extraction, 40 days to analysis of extract
Herbicides	8151A	1 L amber glass, TLC	0° to 6°C / 7 days to extraction, 40 days to analysis of extract
Polychlorinated Biphenyls (PCB) <sup>(6)</sup>	8082A	1 L amber glass, TLC	0° to 6°C / None, 40 days to analysis of extract (recommended)
Per-and Polyfluoroalkyl Substances (PFAS) <sup>(10)</sup>	EPA 537.1 EPA 533, 8327, EPA 3510	1 L HDPE with unlined no Teflon lid	0° to 6°C / 7 days to extraction, 30 days to analysis of extract
Metals <sup>†</sup>	6010D or 6020B or 7000 series	min. 100 mL HDPE <sup>(4)</sup>	HNO <sub>3</sub> to pH less than 2 <sup>(9)</sup> / 6 months max. total holding time



Parameter	Analytical Method <sup>(1,3,7)</sup>	Container Description	Preservation/ Holding Time
Mercury	7470A, 6010D or 6020B	min. 100 mL HDPE <sup>(4)</sup>	HNO <sub>3</sub> to pH less than 2 / 28 days max. total holding time

Notes:

- Several of the 7000 series methods have been deleted from SW-846 but these methods may still be approved in a CSP site-specific work plan. Check the laboratory's approval status. For method 6020B, a gold preservative stock solution for mercury (100 µg/mL Au) may be used. Purchase as a commercially prepared, high-purity solution of AuCl<sub>3</sub> in dilute HCl matrix.
- Minimum (100 mL) is listed for the modified "small volume" method. This requires a separate lab approval and is designated AK102-SV or AK103-SV. Verify the laboratory approval status for this method.
- Sample collection and laboratory analyses for water collected from drinking water sources must be done in accordance with 18 AAC 80 and appropriate drinking water methods.
- HDPE, High Density Polyethylene sample collection bottles, certified clean for trace metals analysis.
- Naphthalene can be analyzed by 8021B or 8260D, if naphthalene is the only PAH contaminant of concern.
- PCB should be prepared using method 3510C, 3535A, or 3520C.
- TAH (summation of benzene, toluene, ethylbenzene, and xylenes) and TAqH (summation of PAH and BTEX) may need to be evaluated if groundwater is hydrologically connected to surface water.
- High temperature sample preparation techniques by EPA Method SW-846 may be required to improve the recovery and achieve lower detection limits.
- If total metals are analyzed, then acidification is acceptable. However, if metals are to be speciated, or if bioassays or toxicity testing is to be conducted with metals, then samples should not be acidified. Instead, store at 4°C in the dark and maintain anoxia. Conduct toxicity analyses within 24 hours of sample collection, unless the test method dictates otherwise. Consult the CSP Project Manager for more project-specific guidance.
- Sampling and preservation considerations are discussed in ITRC's *Sampling Precautions and Laboratory Methods for Per- and Polyfluoroalkyl Substances (PFAS)* (ITRC, August 2020).

<sup>†</sup>Hexavalent Chromium can be analyzed with EPA methods 7199 or 218.6.

Legend:

Appendix D and E: PAH = acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, ideno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene VOA = Volatile Organic Analysis TLC = Teflon® lined screw caps TLS = Teflon® lined septa sonically bonded to screw caps

## Appendix F – Determination of Sampling and Lab Analysis for Source Areas in Soil and Groundwater, and Recommended Sampling Materials

Product Type Test Methods <sup>(9)</sup>	<u>GRO</u> <sup>(1)</sup> AK101	<u>DRO</u> AK102	<u>RRO</u> AK103	<u>BTEX</u> <sup>(1,2)</sup> EPA 8021B EPA 8260D	<u>SVOC</u> <sup>(3,4,5,12)</sup> EPA 8260 <sup>(3)</sup> EPA 8270E EPA 8310VOC	Other <u>VOC</u> <sup>(1,2,3)</sup> EPA 8021B EPA 8260DVOC	<u>EDB</u> <sup>(7)</sup> <u>1,2-DCA</u> EPA 8260D <sup>(1,6)</sup>	<u>Dioxins</u> EPA 8290	<u>MTBE</u> <sup>(13)</sup> EPA 8260D	<u>PCB</u> EPA 8082A	<u>Metals</u> <sup>(8)</sup> EPA 6010D EPA 6020B, or 7000 series	<u>PFOS/</u> <u>PFOA</u> <sup>(10)</sup> EPA 537.1, EPA 533, EPA 8327, EPA 3510
Recommended Sampling Materials <sup>(11)</sup>	Glass, Teflon, HDPE, or Stainless Steel	Glass, Teflon, or HDPE								HDPE or Stainless Steel		HDPE
Site COPCs												
Leaded Gasoline	required			required	required	required	required		may be required by PM		Total lead only	
Aviation Gasoline	required			required	required	required	required		may be required by PM		Total lead only	
Unleaded Gasoline	required			required	required	required			may be required by PM			
JP-4, Kerosene, Jet B	required	required		required	required	required						
Diesel #1 or Arctic Diesel	required	required		required	required	required						
#2 Diesel	required	required		required	required	required						
JP-5, JP-8, or Jet A	required	required		required	required	required						
#3-#6 Fuel Oils or Bunker C	may be required by PM	required	required	required	required	required						
Crude Oil	required	required	required	required	required	required					may be required by PM	
Waste oil, used oil, or unknowns	required	required	required	required	required	required	required	may be required by PM	may be required by PM	required	required	

Product Type Test Methods <sup>(9)</sup>	<u>GRO</u> <sup>(1)</sup> AK101	<u>DRO</u> AK102	<u>RRO</u> AK103	<u>BTEX</u> <sup>(1,2)</sup> EPA 8021B EPA 8260D	<u>SVOC</u> <sup>(3,4,5,12)</sup> EPA 8260 <sup>(3)</sup> EPA 8270E EPA 8310VOC	Other VOC <sup>(1,2,3)</sup> EPA 8021B EPA 8260DVOC	EDB <sup>(7)</sup> 1,2-DCA EPA 8260D <sup>(1,6)</sup>	<u>Dioxins</u> EPA 8290	<u>MTBE</u> <sup>(13)</sup> EPA 8260D	<u>PCB</u> EPA 8082A	<u>Metals</u> <sup>(8)</sup> EPA 6010D EPA 6020B, or 7000 series	PFOS/ PFOA <sup>(10)</sup> EPA 537.1, EPA 533, EPA 8327, EPA 3510
Recommended Sampling Materials <sup>(11)</sup>	Glass, Teflon, HDPE, or Stainless Steel	Glass, Teflon, or HDPE								HDPE or Stainless Steel		HDPE
Site COPCs												
Landfills	required	required	required	required	required	required	required	required	required	required	required	required
Dry Cleaners						required	may be required by PM					may be required by PM
Burn Pit					may be required by PM			required				
Shooting Range					may be required by PM						required	
Fire Training Facilities, fires, and facilities where Aqueous Film Forming Foam (AFFF) was used						may be required by PM						required

**Notes:**

- AK101 (GRO) soil samples must be preserved in methanol.
- EPA 8260D may be required to evaluate non-BTEX volatile petroleum hydrocarbons, such as 1,2,4- and 1,3,5-trimethylbenzene, butylbenzene(s), etc. for vapor intrusion or other applicable pathways to protect human health and the environment.
- Naphthalene can be analyzed by 8021B or 8260D, if naphthalene is the only PAH contaminant of concern.
- PAH must include acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(g,h,i)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, naphthalene, phenanthrene, and pyrene.
- For each petroleum hydrocarbon source area, PAH analysis should be performed on a sufficient percentage of the samples of the most likely contaminated locations based on field readings/site observations/previous sampling data to determine if PAH are contaminants of concern. For each source area this would include the most likely contaminated location in the excavated soil and the most likely contaminated excavation location of what is left in the excavation. If PAH concentrations are less than applicable cleanup levels, further PAH analysis is generally not required. PAH should be sampled in groundwater if soil sample concentrations are above applicable cleanup levels and groundwater sampling is required.
- EPA 8260D is required for the analysis of 1,2-Dichloroethane (1,2-DCA). EPA 8011 or EPA 504.1 can be used when evaluating Dibromomethane 1,2- (EDB). EDB soil samples should be collected with zero headspace and cooled to less than 6°C.
- EDB and 1,2-DCA are lead scavengers and may be present following an avgas spill or a gasoline spill that occurred prior to January 1, 1996. For each source area, EDB and 1,2-DCA analysis should be performed on a sufficient percentage of the samples of the most likely contaminated locations based on field readings/site observations/previous sampling data to determine if EDB and 1,2-DCA are contaminants of concern. For each source area this would include the most likely contaminated location in the excavated soil and the most likely contaminated excavation location of what is left in the excavation. In general, analyzing 10% of the samples for

each source area is a sufficient number for site characterization. If EDB and 1,2-DCA concentrations are less than applicable cleanup levels, further EDB and 1,2-DCA analysis is generally not required. EDB and 1,2-DCA should be sampled in groundwater if soil sample concentrations are above applicable cleanup levels and groundwater sampling is required.

8. Metals must include arsenic, cadmium, chromium, mercury, and lead, unless otherwise noted. For CERCLA sites or characterizing waste include silver, barium, and selenium along with arsenic, cadmium, chromium, mercury, and lead. Lead should be analyzed for avgas spills or gasoline spills that potentially occurred prior to January 1, 1996. For shooting ranges include antimony, copper, tungsten, nickel, cobalt, bismuth, tin, iron, and chromium in addition to lead.
9. TAH and TAqH may also be required for groundwater that is in contact with surface water.
10. To prevent interference and adsorption, Teflon and glass must not be used during sampling or storage for samples destined for analysis of perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA).
11. Samples should be collected using the specified materials above (i.e., tubing, bladders, containers, etc.) to prevent any sampling bias.
12. Low level 1,4 dioxane in water samples can be quantified with SW8270E with liquid-liquid extraction and isotope dilution.
13. MTBE has been used in gasoline since 1979. Ten percent of soil and groundwater samples should be analyzed for MTBE.

## Appendix G – Fish Collection Record

page \_\_\_\_ of \_\_\_\_

### FISH COLLECTION RECORD

Project Name \_\_\_\_\_

Collections made by (include all crew) \_\_\_\_\_

Sampling Method: ☐ Electrofishing ☐ Gill netting ☐ Trap netting ☐ Trawling ☐ Seining ☐ Angling ☐ Other \_\_\_\_\_

Preservation Method: ☐ Freezing ☐ Other \_\_\_\_\_ Notes: \_\_\_\_\_

SAMPLE OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX	LENGTH (mm)	WEIGHT (g)	REMARKS