
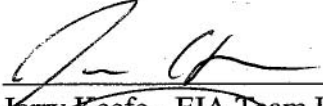


**STANDARD OPERATING PROCEDURE
FOR SOIL, SEDIMENT, AND SOLID WASTE SAMPLING**

The Office of Environmental Measurement and Evaluation
EPA New England - Region 1
11 Technology Dr
North Chelmsford, MA 01863

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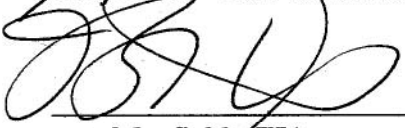
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Attachments:

Sample Label and Custody Seal
 Field Data Sheet
 Chain of Custody Form

1.0 Scope & Application:

- 1.1 This Standard Operating Procedure is applicable to the collection of representative soil, sediment, and solid waste samples. Additional sediment collection techniques can be found in the Sediment Sample Collection Methods-SOP-BiologySection #2.25, Rev. 3, 12/22/98.
- 1.2 Soil samples from drums are addressed in the Drum Sampling SOP.
- 1.3 Sampling using the geoprobe are addressed in the Geoprobe SOP.
- 1.4 Field screening is not addressed in this SOP.
- 1.5 Sampling variations and modifications are possible though the Quality Assurance Project Plans (QAPP) for that site depending on the data quality objectives (DQOs).

2.0 Summary of Method:

- 2.1 The method described in this SOP describes the collection of a solid sample to minimize interferences, cross-contamination, and to maximize the representation that analytical results will reflect actual site conditions.

3.0 Definitions:

- 3.1 **Bottle Blank:** **Analyte-free** water is collected into a sample container, of the same lot as the containers used for the environmental samples. This evaluates contamination introduced **from** the sample **container(s)** from a common lot.
- 3.2 **Equipment/Rinse/Rinsate Blanks:** A sample that is collected by pouring over or running analyte-free water through the sample collection equipment after decontamination and before sample collection. The sample is collected in the appropriate sample container with the proper preservative, identical to the samples. This represents background contamination resulting from the field equipment, sampling procedure, sample container, preservative, and shipment.
- 3.3 **Field Blank:** In the field, analyte-free water is collected into a sample container with preservatives. The sample containers are the same lot used for the environmental samples. This evaluates contamination introduced from the sample **container(s)** with applicable preservatives. Field blanks are not used for volatile samples.

- 3.4 **Field Replicates/Duplicates:** Two or more samples collected at the same sampling location. Field replicates should be samples collected side by side or by collecting one sample and immediately collecting the second sample. Field replicates represent the precision of the whole method, site heterogeneity, field sampling and the laboratory analysis.
- 3.5 **Field Split Samples:** Two or more representative subsamples taken from one environmental sample in the field. Prior to splitting, the environmental sample is homogenized to correct for sample heterogeneity that would adversely impact data comparability. Field split samples are usually analyzed by different laboratories (interlaboratory comparison) or by the same laboratory (intralaboratory comparison). Field splits are used to assess sample handling procedures from field to laboratory and laboratory's comparability.
- 3.6 **Filter Blank:** In the field, analyze-free water is passed through a filter and collected into the appropriate sample container. The filter blank is then preserved. This procedure is identical to the sample collection.
- 3.7 **Laboratory Quality Samples:** Additional samples will be collected for the laboratory's quality control: matrix spike, matrix spike duplicate, laboratory duplicates, etc.
- 3.8 **Proficiency Testing (PT)/Performance Evaluation (PE) Sample:** A sample, the composition of which is unknown to the laboratory or analyst, provided to the **analyst** or laboratory to assess the capability to produce results within acceptable criteria. This is optional depending on the data quality objectives.
- 3.9 **Shipping Container Temperature Blank:** A water sample that is transported to the laboratory to measure the temperature of the samples in the cooler.
- 3.10 **Trip Blanks:** A sample collected at the laboratory using **analyte** free water in the appropriate sample container with the proper preservative, taken out to the field, and returned to the laboratory for analysis without being opened. Trip blanks are generally for volatile organic compounds, low level metals, and gasoline range hydrocarbon samples. Used to assess contamination introduced during sample transport.
- 4.0 Health and Safety Warnings:**
- 4.1 When working with potentially hazardous materials or situations, follow EPA, OSHA, and specific health or safety procedures.
- 4.2 All proper personal protection clothing and equipment is to be worn.

- 4.3 When sampling lagoons or surface impoundments containing known or suspected hazardous substances, take adequate precautions. The sampling team member collecting the sample should not get too close of the edge of the impoundment, where bank failure may cause them to lose their balance.
- 4.4 Follow the Boat Safety SOP when conducting sampling from a boat.
- 4.5 Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.
- 5.0 Interferences:**
- 5.1 Interference may result from using contaminated equipment, solvents, reagents, sample container, or sampling in a disturbed area.
- 5.2 Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of the sampling equipment is necessary.
- 5.3 All sampling equipment must be routinely demonstrated to be free from contaminants under the conditions of the analysis by running equipment blanks.
- 6.0 Personnel Qualifications:**
- 6.1 All field samplers working at Superfund sites are required to take a 40 hour health and safety training course and an annual refresher course prior to engaging in any field activities.
- 6.2 The field sampler should be trained by an experienced sampler before initiating the procedure.
- 6.3 All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function.
- 7.0 Equipment and Supplies:**
- 7.1 Shovel, post-hole digger, auger, split spoon corer, core barrel sampler
- 7.2 Ruler, yard stick, or measuring tape
- 7.3 Nitrile, latex, or neoprene gloves, boots, Tyvex suits

- 7.4 New or cleaned Teflon or stainless steel scoop or spatula
- 7.5 Plastic syringe with a cut off top, 5 or 10 mL, TerraCore or equivalent sampler
- 7.6 Cleaned stainless steel mixing bowl for sample homogenization and compositing
- 7.7 Pre-cleaned glass containers with Teflon lined caps, wide-mouth jars in various sizes and 40 mL VOA vials for VOA, VPH samples
- 7.8 Zip lock bags, flexible foam bottle jackets
- 7.9 Ice coolers, Coleman or equivalent
- 7.10 ThermoSafe Portable Chest, dry ice cooler, Polyform, 390 or equivalent
- 7.11 Portable refrigerator/freezer, Elgin, model 70 or equivalent
- 7.12 Ice
- 7.13 Refrigerant Gel Packs, -23 °C, Polyfoam, model 427 or equivalent
- 7.14 Top load balance, 0.1g
- 7.15 Decontamination Supplies: brush, gloves, detergent, water, and isopropyl alcohol
- 7.16 Site log book, field data sheets, custody seals, GPS, and chain of custody forms

8.0 Sample Collection:

- 8.1 Decontaminate or pre-clean equipment, and ensure that it is in working condition. The analytes of concern should be considered when choosing sampling equipment and the material from which it is manufactured.
- 8.2 Prepare a schedule and coordinate with the staff, clients, and regulatory agencies.
- 8.3 Perform a general site survey prior to the site entry in accordance with the Health and Safety Plan.
- 8.4 If possible, use stakes, flags, or marks to identify all sampling locations identified in the QAPP. The proposed locations may be adjusted based on site access, property boundaries, surface discoloration, odors, and surface obstructions. Major modifications

need to have prior approval from the project manager.

- 8.5 At the sampling location, remove twigs, rocks, and loose debris from the sample location.
- 8.6 Clean the sampling equipment. This will vary analytical parameters and equipment. Typically, the first wash is with soapy water and rinsed with water. In some cases isopropyl alcohol is used. Isopropyl alcohol is a solvent waste and must be collected and return to OEME for disposal. This process must be done in-between samples to prevent cross contamination, if non dedicated equipment is used.
- 8.7 The sample depth is based on the site investigation and is specified in the QAPP. A clean pair of gloves should be worn during the sampling activity to minimize cross-contamination between sample locations and sampling equipment. To minimize interference, the Nitrile glove is used for organic compound investigations and latex gloves can be used for metals and nutrient sampling. Dig or drill to the required depth minimizing the soil disturbance as much as possible. The subsurface sample must be intact for the depth being investigated. Measure and record the depth of the sample from the top of the surface.
- 8.8 Sample for volatile organic analyses must be collected before homogenization and from an undisturbed area. The details for the VOA and VPH vial preparation and sampling are in Appendix A.
- 8.9 If possible, collect approximately 4 times the required amount at the specified depth in a mixing bowl, Table 1. Remove large rocks (greater than 1 cm) and twigs. Field mixing is essential for a representative sample. Homogenize the material to be sampled by mixing at least three times. The representative sample is a horizontal slice of the mixture. The order of collection is based on the contamination's volatility: (VOAs, VPH) BNA, TPH, pesticides, PCBs, nutrients, and metals.

Note: The required sample volume is doubled for samples having high moisture (< 25% solids).

- 8.10 In some projects, compositing is needed to achieve a sample averaging of an area, especially in heterogeneous sites. VOA samples for solids is not normally composited. However, high level voa soils samples can be composited directly into a preweighed methanol containing voa vial (Use up to 3 aliquots of soils with 10ml methanol). This is accomplished by collecting equally weighed portions from a spatial grid to cover the identified area. The composite sample is then homogenized, step 8.9.

9.0 Handling, Preservation, and Storage:

- 9.1 The representative sample is transferred and preserved in the appropriate container, Table 1.
- 9.2 Complete the sample label on the sample container with the necessary information.
- 9.3 Cap the container, place a custody seal over the cap (for enforcement samples), and then place the container in a zip lock plastic bag. For VOA samples place vials into zip lock plastic bag and custody seal the bag (enforcement samples).
- 9.4 Record all pertinent data in the site log book and/or on the field data sheet.
- 9.5 Complete the chain of custody form.
- 9.6 Load all the samples into the appropriate shipping container. Do not immerse the samples in water from the ice or freeze samples with high moisture in the gel packs/freezer.
- 9.7 Attach the custody seals to the cooler prior to delivery to OEME Laboratory or shipment to another laboratory.

10.0 Data and Records Management:

- 10.1 All necessary information is recorded on the sample label, custody seal, site log book, field data sheet, and chain of custody. All entries are in ink and corrections are one line strike out with the date and the sampler's initials.
- 10.2 The chain of custody form is signed over to the laboratory with sample log in. A copy is kept with the sampling records.
- 10.3 The sampling data is stored at US EPA - NE, 11 Technology Dr, North Chelmsford, MA for at least 3 years.

11.0 Quality Control and Quality Assurance:

- 11.1 Representative samples are required. The sampler will evaluate the site specific conditions to assure the sample will be representative.
- 11.2 All sampling equipment must be decontaminated prior to use and after each discrete sample. Trip blanks and equipment blanks are used to monitor interferences and

contamination.

- 11.3 All field QC samples requirements in the QAPP must be followed. These may involve trip blanks, equipment blanks, field duplicates, performance evaluations samples, MST SRMs, and the collection of extra samples for the laboratory's quality control, such as matrix and matrix spike duplicates.

12.0 Waste Management and Pollution Prevention:

- 12.1 During field sampling and analysis events there may be hazardous waste produced from the sample collection. The waste must be handled and disposed of in accordance with federal, state, and municipal regulations. Dispose of the hazardous waste produced at the site where the work was performed, if the operating site has proper disposal available. If there is no disposal that meets regulatory requirements, the waste must be transported back to EPA-NE and transferred to the hazardous waste manager for disposal. The sample volume should be minimized to reduce unnecessary waste.

13. References:

- 13.1 Drum samples, EPA Region 1 SOP
- 13.2 Soil Core Sampling using the Geoprobe, EPA Region 1 SOP
- 13.3 Standard Guide for Compositing Sampling and Field Subsampling for Environmental Waste Management Activities, ASTM, D6051-96
- 13.4 Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples, EPA, RCRA, method 5035A

Table 1:

Sample Volume, Container, Preservative, and Holding Times

| Medium/ Matrix | Analytical Parameter | Conc. Level | Sample Volume | Containers (Number, size and type) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time |
|---------------------|-------------------------|----------------|------------------|--|---|----------------------------|
| Soil or Sediment | VOA, VPH | Low | 5 g (3 mL) | 3 * 40 mL VOA vials | 2 vials containing 5 mL water; 1 vial with no preservative; 4 ± 2 °C | 48 hours |
| Soil or Sediment | VOA, VPH | Low | 5 g (3 mL) | 3 * 40 mL VOA vials | 2 vials containing 5 mL water; 1 vial with no preservative; between -7 °C and -20 °C | 14 days |
| Soil or Sediment | VOA,VPH | Medium | 5 g (3 mL) | 2 * 40 mL VOA vials | 1 vials containing 5 mL methanol, 1 vial with no preservative; 4 °C | 14 days |
| Soil or Sediment | BNA | Low | 30 g | 8 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | BNA | Medium | 5 g | 4 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | PAH | Low | 5 g | 4 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Sediment or Soil | Metals | Medium /Low | 200 g | 8 oz glass jar with Teflon cap | none | 6 mos |
| Soil or Sediment | Pesticides | Low | 30 g | 8 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | Pesticides | Medium | 5 g | 4 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | PCBs | Low | 30 g | 4 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | PCBs | Medium | 5 g | 4 oz glass jar with Teflon cap | 4 ± 2 °C | 14 days |
| Soil or Sediment | Mercury | Low | 200 g | 8 oz glass jar with Teflon cap | 4 ± 2 °C | 28 days |

Table 1:**Sample Volume, Container, Preservative, and Holding Times**

| Medium/ Matrix | Analytical Parameter | Conc. Level | Sample Volume | Containers (Number, size and type) | Preservation Requirements (chemical, temperature, light protected) | Maximum Holding Time |
|---------------------------|------------------------------------|------------------------|--------------------------|---|---|-------------------------------------|
| Soil or Sediment | TCLP, SPLP | NA | 400 g | 16 or 32 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 14 days |
| Soil or Sediment | TCLP, SPLP for Mercury | NA | 400 g | 16 or 32 oz plastic container with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 14 days |
| Soil or Sediment | ZHLP | NA | 200g | 8 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 14 days |
| Soil or Sediment | AVS/SEM | NA | 200 g | 8 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 21 days |
| Soil or Sediment | TPH, EPH, oil identification | NA | 5 g | 2 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 14 days |
| Soil or Sediment | TKN | NA | 200 g | 8 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 28 days |
| Soil or Sediment | TOC | NA | 20g | 2 oz glass jar with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 28 days |
| Soil or Sediment | Cyanide | NA | 10 g | 2 oz glass or plastic container with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | 14 days |
| Soil or Sediment | pH | NA | 20 g | 2 oz glass or plastic container with Teflon cap | $4 \pm 2^{\circ}\text{C}$ | ASAP |

Appendix A:

**Samples for Volatile Organic Analysis
(VOAs and VPHs)**

1. Preparation of sample vials

Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

1.1 Low concentration soil samples

1.1.1 Add a clean magnetic stirring bar to each clean vial.

1.1.2 Add 5 mL of organic-free reagent water to each vial.

1.1.3 Seal the vial with the screw-cap and septum seal.

1.1.4 Affix a label to each vial. (The weight of any markings added to the label in the field is negligible).

1.1.5 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

1.2 High concentration soil samples

1.2.1 Add 10 mL of methanol to each vial.

1.2.2 Seal the vial with the screw-cap and septum seal.

1.2.3 Affix a label to each vial. (The weight of any markings added to the label in the field is negligible).

1.2.4 Weigh the prepared vial to the nearest 0.01 g, record the tare weight, and write it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

2. Sample collection

This is the normal procedure, but modifications and variations are acceptable depending on the data quality objectives, site, and compounds of interest. One must collect the sample according to the procedures outlined in the QAPP.

2.1 Low concentration soil samples

2.1.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.

2.1.2 Add about 5 g (2 - 3 cm) of soil to the sample vial containing the 5 mL of water. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap.

- 2.1.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.
- 2.1.4 Place the vial in a zip lock bag and store samples on ice at 4°C. The VOA sample at 4°C must be delivered to the laboratory before 48 hours. Alternatively, samples can be placed in a foam VOA protector on its side and frozen in a portable freezer or sandwiched in gel packs at -7°C to -20°C. The holding time for the frozen VOA sample is 14 days.
- 2.1.5 Collect at least two replicate samples. This will allow the laboratory an additional sample for reanalysis, if needed. The second sample should be taken from the same soil stratum or the same section of the solid waste being sampled, and within close proximity to the location from which the original sample was collected.
- 2.1.6 In addition, since the soil vial cannot be opened without compromising the integrity of the sample, at least one additional aliquot of sample must be collected for screening, moisture determination, and high concentration analysis (if necessary).
- 2.2 High concentration VOA soil samples:

The use of methanol preservation has not been formally evaluated by EPA and analysts must be aware of three potential problems. First, the use of methanol as a preservative and extraction solvent introduces a significant dilution factor that will raise the method quantitation limit beyond the operating range of the low concentration direct purge-and-trap procedure (0.5-200 $\mu\text{g}/\text{kg}$). The exact dilution factor will depend on the masses of solvent and sample, but generally exceeds 100, and may make it difficult to demonstrate compliance with regulatory limits or action levels for some analytes. Because the analytes of interest are volatile, the methanol extract cannot be concentrated to overcome the dilution problem. Thus, for samples of unknown composition, it may still be necessary to collect an aliquot for analysis by this closed-system procedure and another aliquot preserved in methanol and analyzed by other procedures. Secondly, solid samples with a significant moisture content (>10%) that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the solvent/water mixture. The final problem is that the addition of methanol to the sample is likely to cause the sample to fail the ignitability characteristic, or cause it to become a listed waste, thereby requiring the unused sample volume to be managed as a hazardous waste.

- 2.2.1 Using an appropriate sample collection device, collect approximately 5 g of sample as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere: generally within a few minutes at most. Carefully wipe the exterior of the sample collection device with a clean cloth or towel.
- 2.2.2 Add about 5 g (2 - 3 cm) of soil to the vial containing 10 mL of methanol. Quickly brush any soil off the vial threads and immediately seal the vial with the septum and screw-cap.
- 2.2.3 When practical, use a portable balance to weigh the sealed vial containing the sample to ensure that 5.0 ± 0.5 g of sample were added. The balance should be calibrated in the field using an appropriate weight for the sample containers employed. Record the weight of the sealed vial containing the sample to the nearest 0.01 g.
- 2.2.4 Place the sample in a zip lock bag and store samples on ice at 4°C.

- 2.2.5 Other sample weights and volumes of methanol may be employed, provided that the analyst can demonstrate that the sensitivity of the overall analytical procedure is appropriate for the intended application.
- 2.2.6 The collection of at least one additional sample aliquot is required for the determination of the moisture content.

CUSTODY SEAL

Signature _____

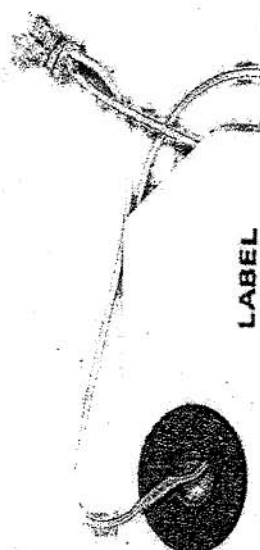
Date _____




CUSTODY SEAL

Date _____

Signature _____



LABEL

SAMPLE

| U.S. ENVIRONMENTAL PROTECTION AGENCY – REGION I BOSTON, MASS. | |
|---|--|
| NAME OF UNIT AND ADDRESS | DATE: YR/MO/DAY |
| | TIME |
| | STATION NO. |
| SOURCE OF SAMPLE | SAMPLE NO. |
| | SUB NO. <input checked="" type="checkbox"/> |
| | PRESERVATIVE |
| SAMPLING CREW (FIRST, INITIAL, LAST NAME) | AMOUNT |
| | ANALYSIS |

Field Log Sheet for Sampling Activity

Project/Site Name: _____

Sampling Location: _____

Sample Type: Soil Sediment Wastewater Drinking water QA or other: _____

Weather Conditions: _____

Sampled By: _____

| Sample # | Date | Time | Sample Depth | Sample/Location Description: | Other Info. |
|----------|------|------|--------------|------------------------------|-------------|
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Signature(s): _____

Chain of Custody Number(s): _____

