Washington State Department of Ecology

Spill Prevention, Preparedness, & Response Program

Standard Operating Procedure for Collecting Soil or Sediment Samples for Gasoline Spills

Version 1.0

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Please note that the Washington State Department of Ecology's Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical and administrative experts. Their primary purpose is for internal Ecology use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. Distribution of these SOPs does not constitute an endorsement of a particular procedure or method.

Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the Department of Ecology.

Although Ecology follows the SOP in most instances, there may be instances in which Ecology uses an alternative methodology, procedure, or process.

SOP Revision History

Revision Date	Rev number	Summary of changes	Sections	Reviser(s)
09-30-2011	1.0			

Washington State Department of Ecology

Spill Prevention, Preparedness, & Response Program

Standard Operating Procedure for Collecting Soil or Sediment Samples for Gasoline Spills

1.0 Purpose and Scope

1.1 This document is the Spill Prevention, Preparedness, & Response (Spills) Program Standard Operating Procedure (SOP) for collecting soil or sediment samples for gasoline spills.

1.2 **Objectives** – 1) To determine the concentration of gasoline in soil or sediment associated with a spill. 2) To document exposure of organisms present in sediment and validate toxicity models. 3) To maintain the integrity of the samples during sampling, transport, and storage.

1.3 The main purpose for collecting soil or sediment samples for gasoline spills is to document the concentrations of gasoline and/or gasoline components in the soil or sediment and exposure of natural resources to determine if the resources have been injured. Concentrations in soil can also be used to estimate spill and recovery volumes. To be legally valid, samples must be carefully collected to prevent contamination and minimize loss of volatile components, and strict chain-of-custody must be maintained throughout collection, transport, and analysis.

1.4 Two analyses are typically conducted on soil or sediment samples collected for gasoline spills; Total Petroleum Hydrocarbons (TPH) and Benzene-Toluene-Ethybenzene-Xylene (BTEX). Volatile Organics Analysis (VOA) samples can also be collected, but this analysis is more expensive and is usually not necessary.

2.0 Applicability

2.1 This procedure is based on EPA Method 5035A, which sets forth the requirements and procedures applicable to the collection and preparation of soil samples for volatile organic compound (VOC) analysis. This method allows for either laboratory or field preservation of the soil samples, but this SOP only uses laboratory preservation. Laboratory preservation is easier than field preservation and only requires the use of an appropriate core sampling device (airtight capsules) that can be sealed and shipped to the laboratory for preservation within 48 hours.

3.0 Definitions

3.1 EPA – Environmental Protection Agency.
3.2 NOAA – National Oceanic and Atmospheric Administration.
3.3 TRAP – Trustee Resource Assessment and Protection.

3.4 NRDA – Natural Resource Damage Assessment.
3.5 SOSC – State on Scene Coordinator.

4.0 Personnel Qualifications/Responsibilities

4.1 Any Ecology employee that expects to collect oil spill related samples must attend appropriate training from a Spills Program Sampling Specialist. Gasoline spill soil or sediment sample collection can be conducted by any employee that has completed basic and intermediate sampling training and has collected at least one quantitative field sample under the supervision of an experienced sampler. All Spills Program employees (including managers and administrative staff) are encouraged to attend basic sampling training. Due to safety requirements, at least two people are recommended for any field sampling and anyone that has taken the training, with or without field experience, can assist an experienced sampler.

5.0 Equipment, Reagents, and Supplies

5.1 Equipment

5.1.1 **If available, use En Core, Core N' One, or other equivalent airtight core samplers**
5.1.2 For the TPH analysis, use one core sampler and one 2 or 4 oz. jar (with septum cap).
5.1.3 For the BTEX analysis, use two core samplers and one 2 or 4 oz. jar (with septum cap).
5.1.4 For the VOA analysis, use three core samplers and one 2 or 4 oz. jar (with septum cap).
5.1.5 When using core samplers, separate samples must be collected for each analysis.

5.1.6 The jar samples are used to calculate water content (the results are reported as percent solids, dry weight).

5.1.7 If core samplers are not available, the samples can be collected in certified clean glass 4 oz. jars (with septum cap).

5.1.8 GPS (set datum to WGS-84, Latitude/Longitude to decimal degrees).

5.1.9 Digital camera (synched to GPS time).

5.1.10 Ice chests with wet or blue ice (preferably equipped with chain of custody security cables).

5.2 Reagents

5.2.1 Pesticide grade acetone and hexane for decontamination.

5.3 Supplies

5.3.1 Sample tags and labels
5.3.2 Field notebook
5.3.3 Chain-of-custody seals
5.3.4 Chain-of-custody forms

5.3.5 Nitrile disposable gloves
5.3.6 Ziploc bags
5.3.7 Paper towels
5.3.8 Stainless steel spoons or spatulas
5.3.9 Liquinox or equivalent soap
5.3.10 Aluminum foil
5.3.11 Cleaning brush
5.3.12 Distilled water

6.0 Summary of Procedure

6.1 Sample Collection Procedure

6.1.1 Safety is of greatest concern. Be aware of physical and chemical hazards at the site. Get a safety briefing before entering the area. Do not enter confined spaces unless they have been determined to be safe. Use recommended safety equipment and procedures.

6.1.2 Develop a sampling plan. Identify the number and type of samples that are to be collected, the type of containers that will be used, and unique identifiers for each sample. Make a drawing of the area being sampled, including points of reference and the location of each site where samples are collected so the sites could be located later if necessary. Identify the sampling procedure(s) that will be used. Include any preservation techniques used for the samples, the type of analyses that will be done, and the laboratory that the samples will be sent to. The plan can be recorded in a field notebook or on a separate piece of paper; there is no required form or specific format that must be followed. Each plan will be different depending on the number and type of samples being collected and the complexity of sampling site.

6.1.3 En Core Sampling Procedure (En Novative Technologies, inc.)

6.1.3.1 Remove the core sampler from the foil pouch (wear gloves to prevent contamination).
6.1.3.2 Insert the core sampler into the T-handle.
6.1.3.3 Remove the cap.
6.1.3.4 Press the core sampler into the soil (preferably from the freshly exposed side of a hole or trench to minimize loss of volatile components from the exposed soil surface) until the o-ring on the core sampler stem is visible through the first hole on the T-handle.
6.1.3.5 Use a clean paper towel to wipe off the sides of the core sampler (to ensure that the cap seals properly).
6.1.3.6 Replace the cap, twisting 90 degrees to lock in place.
6.1.3.7 Remove the core sampler from the T-handle.
6.1.3.8 Twist the core sampler stem counter-clockwise to lock the stem and piston in place.
6.1.3.9 Write the sample number on the foil pouch and the gummed label (using a waterproof pen), place the gummed label on the cap of the core sampler.
6.1.3.10 Place the core sampler back into the foil pouch and store on ice.

6.1.4 **Core sampling procedure using ESS Core N' One capsules & Handle**

6.1.4.1 Remove capsule from zip lock and insert prongs into the slots of the CNO handle. Make a 1/4 turn to the right to lock the capsule in place.

6.1.4.2 Unscrew the capsule cap and bore the beveled edge opening into the soil. You can determine if you have taken a full (~5 gram) sample by holding the capsule up to the light. The dark shading of the soil should be at the top level of the cap threads.

6.1.4.3 Wipe any soil from the cap threads and screw the cap tight. The edge of the cap should touch the center rib of the capsule.

6.1.4.4 Label zip lock bag with sample number. Insert the capsule into the zip lock bag for transport to the laboratory.

6.1.4.5 The core sampling procedure does not work well with samples that have a lot of small rocks or pebbles, coarse sand, or muddy soils. In this case, it may be better to sub-sample a grab of soil with a clean stainless steel spoon (see jar sampling method below). If the material being sampled tends to fall out of the core sampler as it is pulled from the soil or sediment before the cap can be put on, a spoon or spatula can be used to hold the material in the core sampler until it is moved to a vertical position. A spoon or spatula can also be used to fill the core sampler.

6.1.4.6 **Ship samples to laboratory as soon as possible.** Samples must be kept in the dark and at 4° ± 2°C during shipment.

6.1.4.7 **The core samples must be preserved and/or extracted within 48 hours of collection** (the samples must be submitted to the lab allowing time within the 48 hours for the preservation or extraction process. Contact the lab at 360-871-8800 for coordination **prior to sampling**).

6.1.4.8 **If core samplers are not available or they cannot be delivered to the lab and extracted within 48 hours, use the following procedure:**

6.1.5 **Jar Sampling Procedure** (also use for jars collected with the core samplers for percent solids).

6.1.5.1 Use a pre-cleaned stainless steel spoon to quickly transfer the soil directly into 4 oz. jars (with septum cap).

6.1.5.2 Collect soil from the freshly exposed side of a hole or trench to minimize loss of volatile components from the exposed soil surface.

6.1.5.3 Fill the jars completely full (no head space).

6.1.5.4 Fill the jars and replace the cap as quickly as possible to reduce loss of volatile components.

6.1.5.5 If using the jar sampling procedure (in place of the core samplers), one sample can be used for both the NWTPH-Gx and the BETX analyses if desired.

6.1.5.6 Label each container (foil pouch, Ziploc bag, or jar) with an adhesive label directly applied to the container. A paper tag should be attached to jars with an elastic band. Use a waterproof pen or marker to record the project or incident name, a station identifier, a unique sample identifier, the collection date and time, the sample type (TPH, BTEX, or VOA), and the sampler's name or initials on each label. The adhesive

label should be filled out and applied to the container prior to sample collection.

6.1.5.7 After labeling, place each sample jar in a separate Ziploc bag to reduce the chance of contamination should a container leak or break.

6.1.5.8 Immediately place all samples in a cooler and keep at 4° ± 2°C.

6.1.5.9 Use packing material, such as bubble wrap, around containers to prevent breakage.

6.1.5.10 Fill out the chain-of-custody form; be sure to record all information for each sample. Record the same information in a field notebook and add specific information about where each sample was collected from, including latitude-longitude, depth below the surface, and a map if necessary. Also include the sampling devices used, container sizes and types, if any preservative was used, and sampler name.

6.1.5.11 Make special notation on the chain-of-custody form about any problems or observations during sampling that could affect the integrity of the sample, such as potential sources of contamination, etc.

6.1.5.12 Maintain strict chain-of-custody during sample storage and transportation.

6.1.5.13 If possible, ship source samples separately from environmental samples to reduce risk of cross contamination.

6.1.5.14 If the airtight capsules (core samplers) are used, the samples must be sent to the laboratory so they can be preserved or analyzed within 48 hours. Once the samples have been preserved, the holding time is 14 days. If the sample is collected in a 4 oz. jar, the holding time is 7 days at d6°C; do not freeze. Samples collected for percent solids analysis have a 7 day holding time at d6°C.

6.2 Analytical Methods

6.2.1 **Total Petroleum Hydrocarbons** (TPH). As the name implies, this analysis is used to estimate the total amount of gasoline range petroleum hydrocarbons in a sample. This analysis is called NWTPH-Gx at the Manchester Laboratory and samples are analyzed using purge and trap with gas chromatography and a flame ionization detector (FID).

6.2.2 **Benzene-Toluene-Ethybenzene-Xylene** (BTEX). BTEX are compounds in most petroleum products that are highly soluble in water and cause most of the acute toxicity, but also break down or evaporate quickly. BTEX samples are analyzed using purge and trap with capillary gas chromatography and a photoionization detector (PID).

6.3 Decontamination Procedure

6.3.1 Wash with soap (Liquinox or equivalent) and water (preferably hot), scrubbing all surfaces thoroughly with the cleaning brush. If the item being cleaned is heavily

contaminated (oiled), this step should be repeated after washing to remove gross contamination.

6.3.2 **Procedure**:

6.3.2.1 Rinse with tap water and then triple rinse with distilled water.
6.3.2.2 Rinse with acetone.
6.3.2.3 Rinse with hexane.
6.3.2.4 Allow to air dry.
6.3.2.5 If item is not used immediately, wrap in solvent rinsed aluminum foil.

7.0 Records Management

7.1 Sampling Plan Template – Use this form as an aid for developing a complete and comprehensive sampling plan. This form is not required for developing a sampling plan, but provides ready access to guidelines and reminders.

7.2 Sampling Documentation Form – Use this form as an aid and reminder for recording complete and comprehensive sampling information, and provides a single place to record information for multiple samples.

7.3 Both forms are available at:
http://teams/sites/SPPR/response/trap/Sampling/Forms/AllItems.aspx (SharePoint)
or X:\Spills_Program\Response Section\TRAP-NRDA\Sampling\

7.4 Oil Spill Chain-of-Custody Form (ECY 050-42 (11/01))

8.0 Quality Control and Quality Assurance Section

8.1 There are no QA/QC requirements specific or unique to this procedure. Samples collected for these analyses cannot be split in the field. If splits are requested, the samples must be submitted to the laboratory and the laboratory can be asked to split the samples. Typically, the laboratory will extract the samples and then split the extracts. The laboratory will usually provide splits directly to the requester or their laboratory if the sample contains enough material.

9.0 Safety

9.1 Sample collection can present some unusual circumstances that could have equally unusual associated safety hazards. Samplers should consult with the Safety Officer or SOSC and review the incident safety plan or Hazard Assessment Worksheet (HAW) prior to developing a sampling plan so known hazards can be avoided. Samplers should also be aware that sampling will often take place in areas that may not be adequately addressed in the safety plan or HAW. If there is any question, then a separate HAW should be prepared prior to sampling. A Sampling HAW is available at: http://teams/sites/SPPR/response/trap/default.aspx (SharePoint) or X:\Spills_Program\Response Section\TRAP-NRDA\Sampling, that includes action levels appropriate for sampling various petroleum products.

9.2 Some hazards that could be associated with sampling are:
9.3 Low areas that could collect fumes (vapors) or have reduced oxygen levels (confined space)
9.4 Higher concentrations of fumes at ground level where samples are collected
9.5 General water hazards when collecting samples on a shoreline or from a boat or dock, etc.
9.6 Increased risk of slips, trips, and falls
9.7 Traffic when sampling near a highway
9.8 Dangerous animals
9.9 Exposure to elements (hot or cold)
9.10 Eye damage from splashes or brush/branches

10.0 References

10.1 NOAA, 1993. Sampling and analytical methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Water Projects, 1984-1992. Volume IV, Comprehensive descriptions of trace organic analytical methods. Lauenstein, G.G. and A.Y. Cantillo (eds.). NOAA Tech. Memo NOS ORCA 71, Silver Spring, MD. 181 pp.