1.0 Scope and Applicability

U.S. EPA SW-846 Methods 5035 and 5035A, Closed-System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples (U.S. EPA 1996 and 2002), describe a closed system purge and trap process for analyzing volatile organic compounds (VOCs) in solid materials such as soil, sediment, and wastes. These procedures, which include field methods for sample collection, preservation, and handling, may be used in conjunction with any appropriate gas chromatographic procedure such as U.S. EPA SW-846 Methods 8260, 8021 or 8015.

This FSOP describes field procedures used by Division of Environmental Response and Revitalization (DERR) remedial response personnel to collect soil and other solids for VOC analysis that conform to the sample collection, preservation and handling procedures acceptable under Methods 5035 and 5035A. Field procedures that are compliant with Methods 5035 and 5035A are preferred to bulk sampling procedures (e.g., using a sampling spatula to manually fill unpreserved laboratory supplied containers with soil) and should be used by DERR personnel whenever possible. DERR recognizes, however, that certain regulatory or laboratory certification programs may not currently allow or support the use of Method 5035 or 5035A procedures. These programs may require bulk sampling of soil or other solids for VOC analysis. For such situations, consult FSOP 2.1.6, Soil Sample Collection for Volatile Organic Compound Analysis by Bulk Sampling Methods.

Several sample collection and preservation methods are described in Methods 5035 and 5035A. The methods specify field or laboratory preservation of samples in one or more solvents including methanol, sodium bisulfate, and organic-free reagent-grade water or laboratory preservation of the sample by freezing. Two general methods are described in this FSOP. One involves collection of the sample followed by immediate field preservation, and the other method describes collection of the sample in a headspace-free sample device and delivery to the analytical laboratory for preservation or analysis within 48 hours of sample collection. Other collection and preservation methods detailed in Methods 5035 and 5035A may also be acceptable on a case-by-case basis depending on project data quality objectives (DQOs) (U.S. EPA 1999).

2.0 Definitions

Not applicable

3.0 Health and Safety Considerations

3.1 Handle flammable or toxic solvent preservatives such as methanol or sodium bisulfate carefully.
3.2 Wear appropriate personal protective equipment as specified in the project health and safety plan (HASP) while conducting sampling activities. Wear appropriate eye protection, gloves and other splash protection as appropriate when handling solvent preservatives.

3.3 Conduct air monitoring as specified in the project HASP during sampling activities. Refer to Table 1 of FSOP 1.1, Initial Site Entry.

4.0 Procedure Cautions

4.1 Be familiar with all relevant program requirements, laboratory capabilities or certification requirements and project DQOs to determine that the procedures described in this FSOP are appropriate for the sampling event.

4.2 Consult with the laboratory prior to sample collection to determine appropriate sample collection, preservation, shipping and handling and holding time requirements as these requirements may vary between laboratories. Be sure to determine in advance if there are any significant differences in laboratory detection limits between methods.

4.3 If a headspace-free sampling device such as the En Core® Sampler is used, then the sample must be preserved within 48 hours of collection by the laboratory. Samples should be delivered to the laboratory as soon as possible, and the laboratory needs to receive advance notice of sample arrival. This is especially critical for Saturday delivery.

4.4 Preserve and containerize laboratory samples as soon as possible. Steps should be taken to minimize headspace screening, handling, or other manipulation of samples collected for laboratory analysis prior to sample preservation or containerization. For example, don’t submit material from headspace screening for laboratory analysis, and don’t allow soil cores to sit for an extended period prior to containerizing the sample.

4.5 If a field or laboratory solvent preservation method is used, an additional unpreserved portion of the sample must also be submitted to determine the VOC concentration on a dry weight basis.

4.6 For samples to be submitted to the laboratory, the sample device should never be reused (i.e., soil samples from multiple sampling locations should not be collected with the same device). However, multiple aliquots of sample from the same location may be collected (into separate vials) using a device such as the Terra Core® sampler or EasyDraw Syringe®.

4.7 Samples collected using the Terra Core® sampler, EasyDraw Syringe® or like coring device should be calibrated to ensure that the proper amount of sample material is collected. This may be achieved by adjusting the sampler to the soil density, per location, necessary to achieve 5.0 (+/- 0.5) grams of sample. Some samplers have calibrations on the cylinder of the sampler (e.g., EasyDraw
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Syringe®, etc.). Alternatively, collect several trial samples with a clean plastic syringe. Weigh each trial sample and note the length of the soil column in the syringe. Use these data to determine the length that corresponds to 5.0 +/- 0.5 g. Discard each trial sample.

4.8 Methods 5035 / 5035A state that when practical, pre-prepared vials containing methanol should be weighed on the day of sampling to ensure that no solvent has been lost since the time of container preparation. Vials with > 0.01 g less methanol than noted on the vial should be returned to the laboratory for disposal and not used for sampling.

4.9 Use a portable analytical balance for confirming the weight of sample aliquots and pre-preserved sample vials. Limitations of using portable balances may include imprecise readings due to lack of a stable and sheltered location for the balance (e.g., mobile laboratory, fixed building, etc.) and variability in instrument precision or calibration standard weights between the laboratory’s and Ohio EPA’s instruments or standards. Balances should be calibrated in the field on a daily basis using an appropriate standard weight.

4.10 Always wear clean sampling gloves before collecting each sample.

4.11 Some of the procedures described in this FSOP may not be appropriate for Bureau of Underground Storage Tank Regulations investigations or Targeted Brownfield Assessments. Check program and laboratory certification requirements as well as laboratory capabilities prior to sampling.

4.12 The procedures listed in this FSOP are not acceptable for Toxicity Characteristic Leaching Procedure (TCLP) sampling.

4.13 Non-cohesive sample material (e.g., dry sand, sediments/sludges with a high moisture content, etc.) sampled using En Core®, Terra Core® or similar devices should be collected differently than cohesive or consolidated materials, refer to Section 7.2.3. Alternate methods should be considered based on work plan and DQOs.

4.14 Aggregate, cemented material or material larger than the diameter of the sampler cannot be effectively collected using En Core®, Terra Core® or similar sampling devices. These materials should be collected using an alternate sampling technique.

4.15 If samples containing methanol are to be shipped by common courier (e.g., UPS, FedEx), air or ground, ensure that applicable U.S. DOT and/or IATA regulations are followed. The shipping of methanol is regulated by U.S. DOT, Title 49 of the Code of Federal Regulations (Parts 171 through 180).
5.0 Personnel Qualifications

Ohio EPA personnel performing field sampling activities must meet DERR’s qualifications for performing work at uncontrolled hazardous waste sites.

6.0 Equipment and Supplies

The following is a list of equipment and supplies that may be required depending on the selected sampling method:

6.1 Appropriate sample handle for En Core® (T-Handle) or EasyDraw Syringe® (PowerStop Handle®)
6.2 Chain-of-custody form
6.3 Dry weight containers
6.4 En Core® samplers or similar headspace-free sample collection devices
6.5 Ice
6.6 Paper towels
6.7 Preservative
6.8 Sample cooler
6.9 Sample labels
6.10 Sampling gloves
6.11 Sealable plastic bags
6.12 Terra Core® or EasyDraw Syringe® samplers or similar sample collection devices
6.13 Trip blanks
6.14 Water-proof markers and pens
6.15 Analytical field balance and calibration weights
6.16 Pre-preserved/pre-tared sample containers, including stir bar (as applicable)

7.0 Procedures

7.1 Collection and solvent field preservation of samples followed by laboratory analysis:

7.1.1 Obtain a new unused sampler and remove the end cap. Seat the plunger on the Terra Core® or place the EasyDraw Syringe® into the PowerStop Handle® per the manufacturer’s directions (Recommended Use of Terra Core®, Recommended Use of the EasyDraw Syringe® and The PowerStop Handle®, attached)

7.1.2 Expose the soil to be sampled by scraping the surface with a clean spatula or spoon. Push the device into the soil until the sample chamber is full and then extract the device.

7.1.3 Wipe any soil or debris from the outside of the sampler with a clean paper towel. Rotate the plunger 90 degrees to align with the slots in the body of the sampler.
7.1.4 Place the mouth of the sampler into a laboratory-supplied vial containing
the appropriate solvent preservative and extrude the sample into the vial
by pushing the plunger down. Replace the cap on the vial immediately
and gently swirl (do not shake) the vial to saturate the entire sample.

7.1.5 Complete a sample label on the vial. (Note: labels should be affixed to
the vials prior to weighing/reweighing the vials. Labels affixed after filling
and weighing of the vials may introduce a sample weight error).

7.1.6 Place the vial with the preserved sample in a locking plastic bag and
place in a cooler with ice.

7.1.7 Repeat Steps 7.1.1 through 7.1.5 to collect as many vials per sample as
directed by the laboratory for VOC analysis.

7.1.8 Collect a portion of the soil sample in the same manner as above (Steps
7.1.1 through 7.1.3) and extrude the sample into an unpreserved vial or
container (e.g., 40mL or 60mL VOA vial) for laboratory determination of
percent moisture. This data is needed for the laboratory to determine
VOC concentrations on a dry weight basis.

7.1.9 Immediately place filled sample containers into a sample cooler chilled to
4°C and including trip blank samples. Samples collected by Methods
5035 / 5035A should be segregated from samples with gross
contamination or free product and packed in separate coolers.

7.2 Collection of samples followed by laboratory preservation and analysis with an
En Core® Sampler or similar headspace-free sample device:

7.2.1 Obtain a new sampler and place the sampler in the T-Handle per the
manufacturer’s directions (Disposable En Core® Sampler Sampling
Procedures, attached).

7.2.2 For cohesive material, use the handle to push the sampler into the soil
until the body is completely full and the O-ring rests against the tabs.
Remove the sampler and wipe any excess soil from the sampler’s exterior
with a clean paper towel or wipe.

7.2.3 The En Core® Sampler is not recommended for non-cohesive sample
material (e.g., dry sand, sediments/sludge with a high moisture content).
Other sampling methods should first be considering when establishing the
project’s data quality objectives.

7.2.4 If the En Core® Sampler is chosen as the sampling method for non-
cohesive material, push the sampler plunger down into the O-ring until it
rests against the tabs. Depress the locking lever on the handle and place
the sampler, plunger end first, into the handle, aligning the slots on the
device body with locking pins in the handle. Turn the sample upside down and fill with a clean spatula or other device.

7.2.5 Cap the sampler body while it is still in the handle. Push the cap flat and twist to lock.

7.2.6 Remove the sampler from the handle by depressing the locking lever on the handle while twisting and pulling the sampler from the handle.

7.2.7 Lock the plunger by rotating the plunger rod counterclockwise until the wings are firmly resting against the tabs.

7.2.8 Attach a sample label to the sample device and place the sampler(s) in a locking plastic bag. Place the bag in a cooler with ice.

7.2.9 Repeat the above steps to collect as many sampling devices per sample as directed by the laboratory.

7.2.10 Collect at least one portion of sample in a sample device or container (e.g., 2oz jar, 40mL or 60mL VOA vial) for laboratory dry weight determination of the sample.

7.2.11 Immediately place filled sample containers into a sample cooler with ice (4°+/-2°C). Samples collected for high-level VOC analysis should be segregated from samples collected by Methods 5035 / 5035A.

7.2.12 Deliver the samples to the laboratory within 48 hours of collection. Ensure that there is sufficient ice in the cooler for preservation during sample shipment.

7.3 Follow all applicable criteria in FSOP 1.5, Sample Custody and Handling, when handling or shipping/transporting soil samples to the analytical laboratory.

8.0 Data and Records Management

Refer to FSOP 1.3, Field Documentation.

9.0 Quality Control and Quality Assurance

Quality assurance / quality control (QA/QC) samples will depend on the site-specific work plan, DQOs, or laboratory requirements. QA/QC requirements need to be determined prior to ordering sample containers or devices. Trip blank samples should be included in each cooler which holds samples to be analyzed for VOCs. Inclusion and analysis of trip blanks imparts information on potential contamination of samples during sample, handling and field conditions by accompanying samples during mobilization, sampling, demobilization and shipment operations.
10.0 Attachments

Chemisphere, Inc., Recommended Use of the EasyDraw Syringe® and The PowerStop Handle®

En Novative Technologies, Inc., Disposable En Core® Sampler Sampling Procedures

En Novative Technologies, Inc., Recommended Use of Terra Core®

11.0 References

FSOP 1.1, Initial Site Entry

FSOP 1.3, Field Documentation

FSOP 1.5, Sample Custody and Handling

FSOP 2.1.6, Soil Sample Collection for Volatile Organic Compound Analysis by Bulk Sampling Methods

U.S. DOT, Title 49 CFR, Parts 171 through 180


U.S. EPA, September 1999, Soil Sampling for Volatile Compounds, Region 9 Laboratory Field Sampling Guidance Document #1210

Recommended Use Of The Terra Core®

**NOTE:** The Terra Core® Sampler is a single use device. It cannot be cleaned and/or reused.

**Step 1**
Have ready a 40ml glass VOA vial containing the appropriate preservative. With the plunger seated in the handle, push the Terra Core® into freshly exposed soil until the sample chamber is filled. A filled chamber will deliver approximately 5 or 10 grams of soil.

**Step 2**
Wipe all soil or debris from the outside of the Terra Core® sampler. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler.

**Step 3**
Rotate the plunger that was seated in the handle top 90° until it is aligned with the slots in the body. Place the mouth of the sampler into the 40ml VOA vial containing the appropriate preservative and extrude the sample by pushing the plunger down. Quickly place the lid back on the 40ml VOA vial.

**Note:** When capping the 40ml VOA vial, be sure to remove any soil or debris from the top and/or threads of the vial.
Recommended Use Of
The EasyDraw Syringe® and The PowerStop Handle®

Step 1 - Load Sampling Device

Insert the EasyDraw Syringe® into the appropriate slot on the Powerstop Handle® and remove end cap from syringe.

- If sampling for EPA Method 5035A low-level Protocol, insert syringe into one of the three 5 gram positions. Use the heavy position for dense clay, the light position for dry sandy soil, and the medium position for all others.
- There are also three 10 gram and one 13 gram positions to accommodate other methods.

Step 2 - Collect Sample

Push the EasyDraw Syringe® into freshly exposed soil. Continue pushing until the soil column inside the syringe has forced the plunger to the stopping point. Wipe all debris from the outside of the EasyDraw Syringe®. The soil plug should be flush with the mouth of the sampler. Remove any excess soil that extends beyond the mouth of the sampler. The EasyDraw Syringe delivers approximately 5, 10 or 13 grams. Actual weight will be determined at the laboratory.

Step 3 - Eject Sample Into Vial

Remove the syringe from the Powerstop Handle®. Insert syringe into open end of a pre-tared 40-ml vial containing the appropriate preservative. Extrude the sample into the vial by pushing on the syringe plunger. Avoid getting dirt on the threads of the 40-ml vial.

Cap vial immediately and put on ice or in an environment maintained at 4° C.

Option: Refill EasyDraw Syringe® and cap for use as a dry weight container.
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Disposable En Core® Sampler

Sampling Procedures

Using The En Core® T-Handle

Top

En Core®

Bottom

PREPARING SAMPLER FOR SHIPMENT:
5. Remove the capped Sampler by depressing locking lever on T-Handle while twisting and pulling Sampler from T-Handle.
6. Lock plunger by rotating extended plunger rod fully counter-clockwise until wings rest firmly against tabs (see plunger diagram).
7. Attach completed tear-off label (from En Core Sampler bag) to cap on coring body.
8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.

NOTE:
1. En Core® Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.
2. En Core® Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product.
3. En Core® Sampler must be used with En Core® T-Handle and/or En Core® Extrusion Tool exclusively. (These items are sold separately.)

BEFORE TAKING SAMPLE:
1. Hold coring body and push plunger rod down until small o-ring rests against tabs. This will assure that plunger moves freely.
2. Depress locking lever on En Core T-Handle. Place coring body, plunger end first, into open end of T-Handle, aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle. Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

TAKING SAMPLE:
3. Turn T-Handle with T-up and coring body down. This positions plunger bottom flush with bottom of coring body (ensure that plunger bottom is in position). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in T-Handle viewing hole. Remove Sampler from soil. Wipe excess soil from coring body exterior.
4. Cap coring body while it is still on T-handle. Push cap over flat area of ridge and twist to lock cap in place. CAP MUST BE SEALED TO SEAL SAMPLER (see diagram).