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ACRYONYMS

EPA	US Environmental Protection Agency
ER	environmental restoration
GC	gas chromatography
LANL	Los Alamos National Laboratory
MS	mass spectrometry
SMO	Sample Management Office
VOA	volatile organic analysis
VOC	volatile organic compound

1.0 INTRODUCTION

The US Environmental Protection Agency (EPA) introduced SW-846 Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," as part of the SW-846 Update III promulgated in June 1997. EPA no longer allows the use of the previous method (SW-846 Method 5030) for soil samples in site characterization efforts (with the exception of using brass sleeves, discussed in Section 2.2.3 of this document). Method 5035 offers three different techniques for collecting soil samples for the analysis of volatile organic compounds (VOCs) by EPA Method 8260B (gas chromatography/mass spectrometry [GCMS]), EPA Method 8021B (GC only), EPA Method 8015B and total petroleum hydrocarbons-gasoline range organics by EPA guidance 8000. This guidance document summarizes these three options and recommends the technique preferred by the Los Alamos National Laboratory (LANL) Environmental Restoration (ER) Project.

1.1 Option 1 — Weighing the Sample During Field Sampling

This option may be used for low concentration VOCs (<200 g/kg) in soil samples. In the field, a sample is collected directly into a preweighed glass jar containing 5 mL of volatile-free water, a stir bar, and the preservative sodium bisulfate. Five grams of soil are weighed in the field and transferred into the sample vial using clean, dry sampling equipment. Two replicate samples are collected, and a 250-mL plastic bottle also is filled so the laboratory can determine the dry weight of the sample. The samples then are shipped to the laboratory at 4°C.

1.1.1 Option 1 — Advantages

This option has the following advantages:

- Once the sample is sealed, the seal is not broken until analysis, thereby avoiding VOC loss before analysis.
- The chemical preservative sodium bisulfate retards the degradation of some VOCs on the EPA Method 8260 target analyte list.
- The sample has a 14-day holding time.

1.1.2 Option 1 — Disadvantages

This option has the following disadvantages:

- Vials must be preweighed (i.e., weighed before the field-sampling event), and the soil sample must be weighed in the field during the sampling event.
- It is not practical for rock cores.
- Sodium bisulfate may react vigorously with alkaline soils, causing some VOC loss.
- Sodium bisulfate can degrade some VOCs.
- Sodium bisulfate may cause "salting in" or "salting out" (changes in solubility that result in changes in purge efficiency and method sensitivity/applicability) of some VOCs.

- Laboratories have reported an increase of false positive results for acetone and other ketones when they used the preservative sodium bisulfate.
- The sample is weighed and transferred in the field under uncontrolled conditions rather than in the laboratory, where the sample is frozen.

1.1.3 Option 1 — Recommendation

While it is allowed by EPA SW-846 Method 5035, the ER Project does not recommend this option, primarily because of the inconvenience of weighing samples in the field and the problems associated with the preservative sodium bisulfate.

1.2 Option 2 — Methanol Preservation

This option is for high concentration (>200 g/kg VOC) soil samples. A sample is collected in the field directly into a preweighed glass jar containing methanol. Five grams of soil are weighed in the field and transferred into the sample vial. Two replicate samples are collected and a 250-mL plastic bottle is also filled so the laboratory can determine the dry weight of the sample. The samples then are shipped to the laboratory at 4°C.

1.2.1 Option 2 — Advantages

This option has the following advantages:

- Once the sample is sealed, the seal is not broken until analysis, thereby avoiding VOC loss before analysis.
- The methanol preservative retards the volatilization and biodegradation of some VOCs on the EPA Method 8260 target analyte list.
- The methanol-preserved sample has a 14-day holding time.

1.2.2 Option 2 — Disadvantages

This option has the following disadvantages:

- Vials must be preweighed (i.e., weighed in advance of the field-sampling event) and the soil sample must be weighed in the field during the sampling event.
- A hazardous substance (methanol) must be used in the field and shipped.
- The detection limit (>200 g/kg VOC) is greater than the VOC concentrations typically encountered at LANL.
- Unused samples must be disposed of as hazardous waste.

1.2.3 Option 2 — Recommendation

While it is allowed by EPA SW-846 Method 5035, the ER Project does not recommend this option. The detection limits using the methanol dilution are greater than the detection limits required for human health

and ecological risk assessments, and greater than the concentrations for VOCs typically encountered at LANL.

1.3 Option 3 — Using the Encore™ Device as a Sampling Device and Container

This option is for either low concentration (<200 g/kg VOC) or high concentration soil samples (>200 g/kg VOC). A sample is collected in the field using the Encore™ device as both a sampling device and as a container for shipment to the laboratory. The sample should be frozen at the LANL Sample Management Office (SMO) as soon as possible after the sample is collected, but always on the day of sampling. The SMO then will ship the frozen Encore™ samples to the laboratory on blue ice at 4°C. The laboratory then will store the Encore™ samples at -13°C as a method of sample preservation. The laboratory must analyze the samples within 14 days of the date of sampling.

The current version of EPA SW-846 Method 5035 states that the Encore™ device may be used as a sample container for up to 48 hr after collection. This time must be extended for the LANL ER Project because of the extra time required for radiological screening of samples. A 14-day holding time for frozen samples is supported by research presented by the Cold Regions Research Laboratory in a recent article (Hewitt 1999, 64970). When using the Encore™ device to sample it is important to coordinate with the SMO and the radiological screening facility, so that time delays in shipping the Encore™ samples to the contract laboratory are minimized.

In the field, Encore™ samples should be held on blue ice at 4°C. At the end of each day, Encore™ samples should be taken to the SMO to be frozen. Frozen samples should be shipped to the contract laboratories on blue ice at 4°C as soon as possible. The contract laboratory must analyze these Encore™ samples within 14 days from the time of sampling.

1.3.1 Option 3 — Advantages

This option has the following advantages:

- This method can be used for both high- and low-concentration samples.
- Use of the Encore™ device as both a sampling device and container for shipping and storage avoids VOC losses in the field.
- The Encore™ is a proven storage device that eliminates the loss of VOCs, especially when the Encore™ device is frozen.
- Transfer of the sample to the analysis vial occurs in the laboratory with the sample frozen, thus avoiding the loss of VOCs.
- Freezing as the method of preservation avoids the inherent problems of using sodium bisulfate or methanol as the preservative.

1.3.2 Option 3 — Disadvantages

This option has the following disadvantages:

- The Encore™ device is not practical for rock cores.
- The sample may have to remain in the Encore™ device longer than 48 hr because of LANL's requirement for radiological screening.

1.3.3 Option 3 — Recommendation

This option is the ER Project's preferred technique for implementing EPA SW-846 Method 5035 for cohesive and noncohesive materials. The Encore™ device will be used as both a sampling device and a sample container or just as a sample container, depending on the material to be sampled:

- Cohesive material (e.g. soil, sediment) — The Encore™ device can be used as both a sampling device and a sample container.
- Noncohesive material (sand, pumice, ash, and loose tephra) — For noncohesive samples the Encore™ device will be used as a sample container (see Section 2.1, Daily Field Blanks, of this guidance document).
- Rock cores — Rock core sampling will vary, depending on the degree of induration of the sample. See the detailed instructions below.

2.0 DETAILED INSTRUCTIONS FOR USE OF THE ENCORE™ DEVICE

2.1 Daily Field Blanks

EPA SW-846 Method 5035 was developed to avoid VOC losses during sampling and analysis. Using SW-846 Method 5035 for many ongoing activities (e.g., quarterly sampling events) may show VOCs where none were detected in the past, or the sites may show elevated VOC concentrations when previous sampling (using EPA SW-846 Method 5030) did not. Therefore it is important to ensure that these new results are valid. To address this concern, daily collection of a field blank is required to assess the potential for sample contamination.

The daily field blank should be prepared from a certified volatile-free sand (which may be obtained from Environmental Resource Associates, 1-800-ERA-0122) or volatile-free water (which may be obtained from the SMO). If the volatile-free sand is used daily, the sampler must transfer enough volatile-free sand into a clean 4-oz. glass jar to fill two 5-g Encore™ samplers. If the volatile-free water is used, the sampler should collect two field blanks using 5 mL of volatile-free water in each 40-mL volatile organic analysis (VOA) vial. Either type of field blank can be frozen and shipped with the samples. For water field blanks, the glass 40-mL VOA vial must be placed horizontally in the freezer to eliminate the possibility that water expansion will break the vial upon freezing. Field blanks should be opened in the field such that they are subjected to the same conditions as the field sample. For example, when boreholes are drilled, the field blanks should be opened and placed near the core when the core is opened, photographed, and sampled, thereby exposing the field blank to exhaust fumes (from drilling equipment) if the core sample is exposed to exhaust fumes.

Two field blank samples consisting of two Encore™ devices filled with either volatile-free sand or volatile-free water should be taken each day of sampling. Because of the expense of volatile-free sand, a 250-mL plastic bottle of the volatile-free sand will not be sent to the contract laboratory for soil-moisture analysis. The contract laboratories will be instructed to report these sand field blanks as 100% solids when they report VOCs.

2.2 Sample Collection in the Field

In preparing to sample soils for VOC analyses (EPA Method 8260, EPA Method 8021B, and Total Petroleum Hydrocarbons-Gasoline Range Organics [EPA Guidance 8000]) coordination with the

radiological screening facility and the SMO is critical. Prearranging with the radiological screening facility and coordination with the SMO for sample shipment will minimize delays in the Encore™ samples arriving at the laboratory. The Encore™ device can be obtained through the SMO. The Encore™ T-handle used to provide the leverage needed to properly sample solid materials can be checked out from the SMO.

EPA SW-846 Method 5035 recommends using three Encore™ devices at sites where there is no history on the VOC contamination; one for a low-concentration analysis, one for a high-concentration analysis (if dilution is required to bring target analytes within the dynamic range of the analytical instrument), and one spare in case of difficulties at the analytical laboratory. A 250-mL plastic bottle of soil also should be collected so the laboratory may determine the moisture content of the soil in order to report VOC sample results for the dry weight of the soil. If high VOC concentrations (>200 g/kg VOC) are known to be not present, two Encore™ devices will suffice.

2.2.1 Taking a Sample in Cohesive Soil

Refer to Figures 2.2-1 through 2.2-5 when you use the following instructions. (All figures appear at the end of this document.)

1. The Encore™ device will come out of the package with the plunger rod down and the plunger bottom at the body of the coring body (Figure 2.2-1).
2. If the plunger bottom is not at the bottom of the coring body, push the plunger rod down until the small o-ring rests against the tabs. With the plunger rod in the down position, the Encore™ device is ready to be loaded into the Encore™ T-handle (shown in Figure 2.2-2).
3. Depress the locking lever on the Encore™ T-handle and place the coring body (plunger end first) into the open end of the T-handle. Align the slots on the upper portion of the coring body with the locking pins on the T-handle and twist the coring body clockwise until the pins are locked in the slots. Ensure that the sampler is locked into the handle by pulling straight down on the coring body.
4. The coring body now can be pushed into a soil sample. Using the T-handle, push the coring body into the soil until the coring body is completely full. When the coring body is full the small o-ring at the top of the plunger rod will be centered in the T-handle viewing hole (the T-handle has two viewing holes: one for the 5-g Encore™ device and one for the 25-g Encore™ device) (Figure 2.2-2). Remove the sample from the soil, and wipe excess soil from the coring body using your clean glove or a clean wipe. The Encore™ cap should now be removed from the package and fitted onto the coring body while the coring body is still on the T-handle. Push the cap over the flat area of the ridge on the coring body and twist to lock the cap in place (Figure 2.2-3). The cap must be properly sealed to seal the Encore™ sample (Figure 2.2-4).

To prepare the sample for shipment, remove the sampler from the T-handle by depressing the locking lever on the T-handle while twisting and pulling the sampler from the handle. The plunger rod now can be locked in place by inserting the plunger rod in the small slot on the top of the T-handle and rotating the extended plunger rod fully counterclockwise until the wings rest firmly against the tabs (Figure 2.2-5).

2.2.2 Taking a Sample in Noncohesive Soil

To sample sand or noncohesive material, pull the Encore™ plunger rod back to form an o-ring seal on the back end of the Encore™ body. Lock the plunger by rotating the extended plunger rod until the plunger rod wings rests firmly against the Encore™ tabs. Once the plunger is locked, scoop or push the sample into the sampler using a sampling scoop or other appropriate sampling equipment. Install the sample cap

such that the cap's locking arm grooves seat over the coring body ridge. This action keeps the sample in a handspace-free state for shipment to the laboratory.

2.2.3 Taking a Sample from Rock Cores

For nonwelded or moderately welded tuff it may be possible to use the Encore™ as both a sampling device and a sample container. The Encore™ device has a cutting edge at the bottom of the coring body. This cutting edge can be used to sample less-indurated tuff. While it is being held at a 90-degree angle to the nonwelded tuff core, this cutting edge must be rotated clockwise and then counter clockwise and worked into the tuff. To sample indurated rock cores and basalt it will be necessary to use sealed brass-sleeved cores to sample for VOCs.

VOCs in the tuff will be in the vapor phase because they do not absorb on the tuff. Therefore, the sampler must not disturb the material during sampling for VOCs in tuff. If the tuff is too welded to use the Encore™ as a sampling device, the sampler must not breakup the tuff and transfer it to the Encore or VOCs will be released. In densely welded tuff, brass-sleeved cores should be used to sample for VOCs. Pore-gas sampling using EPA Method TO-14, "Determination of Volatile Organic Compounds (VOCs) in Ambient Air Using SUMMA® Passivated Canister Sampling and Gas Chromatographic Analysis," also may be appropriate for sampling VOCs in densely welded tuff.

3.0 LANL CHAIN-OF-CUSTODY SEALS

After being used for sample collection, each Encore™ sample device should be placed back into its original bag and a LANL chain-of-custody seal should be used to cover the seal on this bag.

4.0 REFERENCES

Hewitt, A. D., September/October 1999. "Frozen Storage of Soil Samples for VOC Analysis," *Environmental Testing & Analysis*, Volume 8, Number 5, p. 18. (Hewitt 1999, 64970)

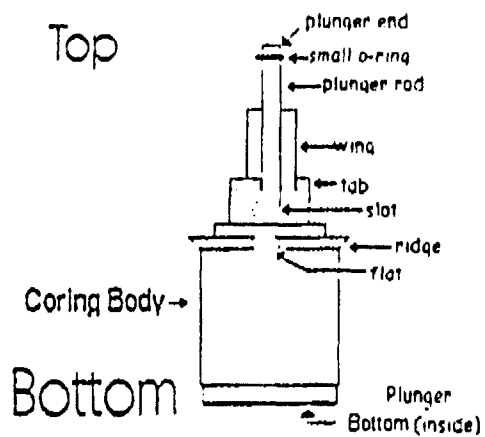


Figure 2.2-1 Encore sampler

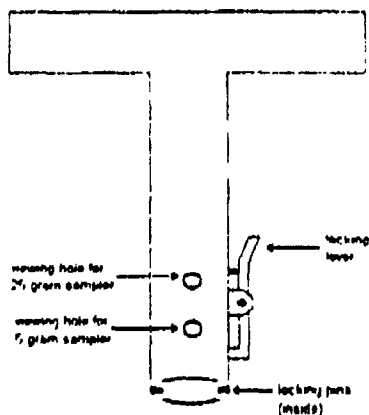


Figure 2.2-2 Encore T-handle

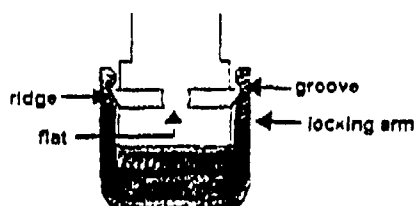


Figure 2.2-3 Sampler correctly capped

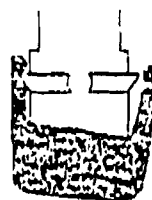


Figure 2.2-4 Sampler incorrectly capped

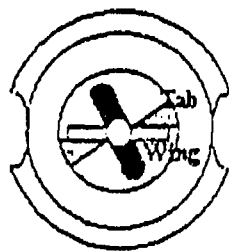


Figure 2.2-5 Plunger correctly locked

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