

STATE OF CONNECTICUT
DEPARTMENT OF ENVIRONMENTAL PROTECTION

GUIDANCE FOR COLLECTING AND PRESERVING SOIL AND
SEDIMENT SAMPLES FOR LABORATORY DETERMINATION
OF VOLATILE ORGANIC COMPOUNDS

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1.0 INTRODUCTION

1.1 Background

Volatile organic compounds (VOCs) are lost from soil and sediment samples (hereinafter referred to as soil samples) due to volatilization and biodegradation during collection, storage and analysis. This leads to low-biased results. Some commonly used techniques are prone to relatively large losses and results that are potentially biased quite low. Such techniques involve collection of disturbed soil samples and storage in soil jars without airtight seals. This guidance describes soil sample collection and preservation techniques designed to minimize such losses. Additional discussion on the background of this issue, sample collection considerations, and current preservation options, is presented in a paper entitled, *Rationale for Preservation of Soil and Sediment Samples for Determination of Volatile Organic Compounds* (October 2005, Connecticut Department of Environmental Protection Laboratory Quality Assurance and Quality Control Workgroup).

This guidance is presented in three parts. The first part (Section 1.0) discusses expectations regarding the use of the sampling and preservation techniques described herein, the effective date of this guidance, and how data generated using other techniques should be evaluated. The second part (Sections 2.0 to 4.0) describes the field techniques associated with the currently recommended procedures. The final part (Section 5.0) consists of typical questions that might arise regarding this guidance and the respective answers.

1.2 Expectations Regarding Currently-Recommended Procedures and Use of Data Generated Using Other Procedures

The DEP expects that all soil samples collected in, or for use in, Connecticut for the purpose of laboratory analysis for VOCs on or after March 1, 2006, the effective date of this guidance, be collected and preserved in accordance with the procedures described in this guidance document.

DEP anticipates that the vast majority of data generated before the effective date of this guidance (hereinafter referred to as “old data”) will be acceptable without the need for resampling and analysis for VOCs. However, environmental professionals should evaluate the old data in the context of their site-specific conceptual model to determine whether potentially low-biased results for VOCs that may be present in soil at the site would create a significant data gap or whether the possible low bias might result in a potential significant risk to human health. Based on this evaluation, additional investigation and/or remediation may be warranted.

2.0 SOIL SAMPLING TECHNIQUES FOR LABORATORY DETERMINATION OF VOCs

2.1 Statement of Purpose

Soil/sediment samples collected for laboratory determination of volatile organic compounds require special handling to prevent loss of VOCs leading to low-biased results. Historic sample collection procedures and associated handling of samples both in the field and in the laboratory may have lead to underestimation of VOC concentrations. This guidance provides environmental professionals with procedures designed to minimize losses of VOCs from soil samples. It is strongly encouraged that environmental professionals also read the background document entitled, *Rationale For Preservation Of Soil and Sediment Samples For Determination Of Volatile Organic Compounds*, (the “White Paper”) that was prepared by the Connecticut DEP QA/QC Workgroup and is currently posted on the EPOC website (www.epoc.org).

2.2 Overview of Sampling Approach

The soil sample collection procedure for determination of VOCs is a two-step process:

Step 1 – Collect an undisturbed soil sample, as defined below, from the subsurface, or expose the targeted area from where a sub-sample for laboratory analysis will be collected,

Step 2 – Collect a representative sub-sample from the undisturbed sample or directly from the exposed subsurface.

2.3 Collection of Undisturbed Samples

Undisturbed samples are those for which the sampling device minimizes break-up of the structure of the soil to the extent practicable. Undisturbed samples can be collected using such techniques as:

- Coring, such as the methods utilizing split-spoon sampling devices, MacroCores™, and large-bore direct-push samplers;
- Bulk sampling, for example, undisturbed soil volumes collected using a backhoe bucket from sidewalls of trenches and excavations where direct access to the sampling location (sidewall or bottom) is not safe; and
- Direct collection of sub-samples from the subsurface.

When collecting samples for laboratory determination of VOCs, the device used to collect the undisturbed soil sample must be removed as soon as possible from the subsurface; and most importantly, **the sub-samples that are intended for VOC determination must be collected as soon as possible (ideally within five minutes of collection of the undisturbed sample) to reduce loss of VOCs due to volatilization.**

Attempts must be made to further minimize loss of VOCs by managing the sample collection environment (i.e., limiting sun, wind, heat, etc.).

Planning and careful preparation are critical for a successful sampling event. Checklists should be used to ensure that all necessary equipment and supplies are present and in proper working order and that the following conditions are achieved:

- Undisturbed soil to be collected for sub-sampling should be collected in a manner that controls the acquisition of the samples such that they do not “stack up” awaiting logging and sub-sampling;
- Cores should not be stored in large-diameter, small-diameter sampling devices or capped liners (brass, acetate, lexan, polycarbonate etc.);
- Cores should not be exposed to extreme weather conditions, such as direct sunlight, rain and wind, and sub-sample collection should occur in an area that minimizes exposure to the elements (e.g., under cover, shady areas); and
- Undisturbed soil samples cannot be transferred from the core sampler to a secondary container (empty sample bottle, ziplock bag, aluminum foil, or sampling bowls) for future sample collection.

Research has shown that leaving samples in core tubes, split-spoons, covered liners, or intermediate containers will lead to VOC losses and will thus yield poor quality data. Background information on this topic has been provided in the White Paper.

To the extent practicable, undisturbed samples should always be collected. However, in some cases, collection of a disturbed sample may be necessary to characterize source areas or other critical locations. If disturbed soil samples must be collected, the rationale for collecting such disturbed samples must be provided. However, under no circumstances should a sub-sample be collected from a disturbed sample that was previously used for field-screening purposes.

2.4 Collection of the Soil Sub-Sample for Determination of VOCs

Sub-samples are those samples that are submitted to the laboratory for analysis for VOCs. Sub-sampling of the undisturbed soil sample must be performed using a dedicated or decontaminated small-diameter sampler. Sub-samples must be collected as soon as possible from the undisturbed sample (ideally within five minutes) after the undisturbed soil sample is collected.

3.0 OVERVIEW OF SUB-SAMPLING DEVICES

Sub-sampling of the large-diameter or bulk sampling device for VOCs must be performed with the use of a dedicated or decontaminated small-diameter core sampler. The small-diameter core sampler must be capable of collecting the required amount of sample from the large-diameter sampling device (e.g., split-spoon or soil corer) or from freshly exposed subsurface soils. The small-diameter core sampler must be capable of delivering the sample quickly and directly into the sample container without disturbing the native soil structure.

Small-diameter core samplers used in the collection of samples for VOCs must be constructed of non-reactive materials that will not sorb, leach or alter the concentration of VOCs in the sample. Examples of these materials are stainless steel, glass and brass. Other materials, such as Viton[®], PTFE and some ridged plastics, which have demonstrated limited absorptive or diffusive passage of VOCs, can be used as long as the contact time between the sample and the sampler is minimized, or the materials are used for an airtight seal of the sampler. It is important to note that some syringes may be coated with glycerin and should not be used.

There are many acceptable small-diameter core samplers. Product names are included in this guidance document for illustrative purposes and do not indicate an endorsement or requirement by the DEP for the environmental professional to use a specific product. It is the responsibility of the environmental professional to document the rationale that explains the suitability of the selected small-diameter core sampler. Currently available small-diameter core samplers include a modified 10-milliliter (ml) disposable plastic syringe, a Purge and Trap Soil Sampler[®], En Core[®] samplers, Easy Draw Syringe[®] or other small-diameter tube/plunger sampler.

The small-diameter core sampler should fit inside the mouth of the sample container to avoid loss of sample, prevent damage to the sealing surfaces or container threads and ease the soil transfer process.

3.1 Procedure for Obtaining Test Samples to Determine Sub-Sample Volume

The purge and trap laboratory procedure used to determine volatile organic compounds requires approximately equal amounts of soil and liquid to be used in the analysis. If the ratio of soil to liquid is too high, the soil will not be adequately dispersed in the liquid, leading to poor results. If the amount of soil is too low, the detection limits will be increased, potentially rendering the results to be of limited use. It is better to use a slightly lower weight of soil than a higher weight of soil, as the regulatory limits are, in general, significantly higher than the typical laboratory reporting limit for volatile analytes.

The small-diameter core sampler must be able to deliver a minimum of 5 grams of sample ($\approx 3 \text{ cm}^3$ of sample, assuming a density of 1.7 g/cm^3) into a 40-ml VOA vial. While most small-diameter core samplers can only be used for sampling and placement

into the appropriate sample containers, only the En Core[®]-type samplers can be used for sampling, storage, and transportation of the sample to the laboratory. Small-diameter core samplers should be selected based upon the properties of the matrix, the type of preservation method (field vs. lab), and personal preferences.

It is important that the small-diameter core sampler provide the required mass of sample material. As such, a test sample (of similar matrix to that being sampled) may be collected and weighed to determine the amount of soil needed to obtain the required mass of sample material for each type of small-diameter core sampler and analytical method.

The procedure for obtaining a test samples is as follows:

1. Using a small electronic portable scale with an accuracy of 0.1 grams, weigh the empty small-diameter core sampler (e.g., disposable syringe) to the nearest 0.1 grams. The scale must be calibrated before use and intermittently checked during the day to ensure accurate weight measurement. Calibration information must be recorded in the field logbook. A translucent cover can be placed over the scale during the weighing process to negate variations caused by wind.
2. Push the small-diameter core sampler test sample into the matrix to collect the required mass of material (3 cm³ should yield approximately 5 grams of sample [wet weight]).
3. Wipe clean any soil adhering to the outside of the small-diameter core sampler before weighing.
4. If the weight is above the required amount, remove excessive soil by extruding a small portion of the core and cutting it away with a decontaminated trowel or spatula. If the weight is below the weight limit, obtain additional soil by reinserting the small-diameter core sampler into the soil core. Re-weigh after each addition or removal of sample from the small-diameter core sampler until the target weight is attained. Note the sample volume and amount in the small-diameter core sampler.
5. Discard the test sample appropriately.
6. Use the volume of the test sample as a guide in collecting the appropriately sized sub-sample of a similar matrix. Additional test samples should be weighed whenever a change in the matrix is observed.

3.2 Overview of Procedure for Collection of Sub-Samples

The goal of soil sampling for the purposes of evaluating concentrations of contaminants in soil is to obtain a representative soil sample in accordance with the data quality objectives for the project. Often, this is accomplished using an appropriate small-diameter core sampler.

Different sample matrices (e.g., sand, gravel, clay, fill) will be encountered and may warrant slightly different sub-sampling field techniques. The goal for all techniques is to collect the sub-sample as quickly as possible while minimizing disruption.

Environmental professionals should use good judgment as to how to handle samples that do not fit into the samplers and must describe the rationale for any deviations from this guidance.

The procedure for obtaining soil sub-samples is as follows:

1. Once the sampling interval has been selected, trim off the exposed surface of the matrix to expose a fresh surface. A loss of VOCs from the surface of the matrix will occur even if the matrix has been exposed for a short period of time (during screening, etc.). Removal of the unwanted surficial material can be accomplished by scraping the matrix surface with a decontaminated spatula or trowel. Soil sampling must commence immediately once a fresh surface has been exposed.
2. Using the test sample as a guide, push the small-diameter core sampler into the matrix to collect a volume of material that will yield the required mass of sample (wet weight) as determined by the analytical method.
3. Depending upon the texture, depth or moisture content, insert the small-diameter core sampler straight into the matrix, on an angle. Multiple insertions can be made to obtain the required sample weight.
4. After sample collection, wipe the outside of the small-diameter core sampler to remove any excess material adhering to the barrel.
5. Immediately open the sample container and extrude the soil core into the sample container that will be submitted to the laboratory. Avoid splashing any preservative, if present, out of the sample container by holding the container at an angle while slowly extruding the soil core into the sample container. Do not immerse the small-diameter core sampler into the preservative. If an En Core[®]-type sampler is to be used for storage and shipment, prepare the sampler for shipment according to manufacturers instructions.
6. Collect the required number of sample containers or En Core[®]-type samplers based on the chosen preservation and analytical methods, as discussed in the subsequent section on soil preservation methods.
7. Include an additional sample for determination of soil moisture content and sample screening.
8. Ensure the threads and cap of the sample container or En Core[®]-type sampler are free of soil particles. Use a clean paper towel to remove soil particles from the threads and sealing surface of the sample container or En Core[®]-type sampler.

The presence of soil particles will compromise the container's seal and may result in loss of preservative or VOCs. This loss ultimately may invalidate the sample analysis. Always make sure the sample lid is firmly secure. The laboratory should flag samples that appear to have a poor seal so that the potentially low biased data can be evaluated relative to the project's data quality objectives.

9. Record the laboratory and field identification numbers in the field notes and on the chain of custody. Record the sample identification information on the sample container using a suitable marker. Container labels with wire or rubber band attachments can be used, provided they can be removed easily for sample weighing. Do not attach any additional adhesive-backed labels or tape to the sample containers unless requested by laboratory or specified in manufacturer instructions. This will increase the weight of the sample container and the laboratory will not be able to determine the sample weight.
10. After sample collection, immediately return the containers to an iced cooler. Sample containers from different locations should be placed in separate ziplock bags to help avoid cross-contamination. The laboratory sample number or field sample identification number may be placed on the bag and cross-referenced on the chain of custody. The laboratory performing the analysis will determine the sample weight.

4.0 PRESERVATION OF THE SOIL SAMPLE

4.1 Overview of the Soil Preservation Procedure

The preservation of samples for VOC analysis can be initiated either at the time of sample collection or in the laboratory. This section deals with the preservation of soil samples in the field using chemical and physical preservation methods.

It is important that the laboratory analytical methods, field preservation methods, appropriate sample containers and sample holding times are determined prior to mobilizing to the field. It is also necessary to consider that additional sample containers maybe required for various quality control/ quality assurance (QA/QC) samples such as matrix-spike and matrix-spike duplicates (MS/MSD). The number of extra containers required varies by laboratory and analytical procedure.

In addition to the various chemical preservation methods, samples must be physically preserved (e.g., iced or frozen) in the field immediately upon sample collection. It is important to match up the correct physical preservation method with the appropriate sample container and field chemical preservation method. According to USEPA Contract Laboratory Protocol (CLP) Guidance for Field Samplers, the physical preservation methods are described as:

Iced – soil and sample containers are cooled to $4^{\circ} \pm 2^{\circ}\text{C}$.

Frozen – soil and sample containers are cooled to between -7° to -15°C .

Sample containers that will be frozen should be placed on their side prior to freezing process to prevent breakage. Additional aliquots for screening and moisture determination need only be iced and kept cooled at $4^{\circ} \pm 2^{\circ}\text{C}$; these sample containers should not be frozen. **Sample containers and En Core[®]-type samplers should not be frozen below -20°C , as the integrity of the container seals, o-rings and septum may be compromised by the freezing, resulting in the loss of VOCs upon thawing of the sample.**

En Core[®]-type samplers with soil containing greater than approximately 20% moisture should not be frozen. Greater than 20% moisture is extremely moist and is easy to spot with visible pooling of liquid on the sample surface. In most cases involving soil sampling, greater than 20% moisture is unusual.

In addition, the use of dry ice to freeze samples immediately upon sample collection or for use during shipment is not recommended. Dry ice, which is at a temperature of -78.5°C , will lower the temperature of the sample container below the design specifications, causing damage to the glass, septum, seals, o-rings, and cap. In addition, dry ice has specific handling, storage and shipping requirements that outweigh its usefulness to the field sampling team.

4.2 Sub-Soil Sample Collection Procedures

When collecting soil sub-samples for determination of volatile organic compounds, up to four types of samples may be required:

- A high-concentration-level sample
- A low-concentration-level sample
- An SPLP/TCLP sample
- A sample for percent solids determination

Additional samples may be necessary for matrix spikes and matrix spike duplicates. Field and trip blanks also may be required.

An overview of the various options for sample collection procedures is attached as Figure 1.

4.3 High-Concentration Sub-sample Collection Procedures

There are two options for collection of the high-concentrations sample: collection of the sample in a methanol preserved VOA vial or using En Core[®]-type samplers.

4.3.1 High-Concentration Sample Option 1, Methanol Preservation

Supplies:

Electronic field balance accurate to 0.1 grams

Minimum of one VOA vial (40 ml), pre-weighed and containing 5 or 10 mls of methanol
Sub-sampling device

1. Label the vials as appropriate. Do not add more weight (e.g., excessive labels) to the pre-weighed vials.
2. Weigh the vials to confirm the recorded vial weight.
3. Select the area to be sampled as soon as possible after the soil is exposed.
4. Obtain a test sample, using the coring device and field balance, to determine approximately how much volume of soil will yield equal grams of soil to methanol (5 or 10 ± 1 grams). This step may be skipped when the amount of soil needed for a particular matrix at a site has been determined.
5. Scrape away the surface material from the area to be sampled to expose fresh soil.
6. Rapidly insert the syringe into the soil to obtain the sample. Quickly extrude the sample into the vial containing the methanol. Wipe off the threads and cap; seal the vial.
7. Using the field balance, weigh and record the weight of the vial. A record of the weight must be submitted with the samples to the laboratory.
8. Place sample in cooler with ice.
9. Collect separate sample for percent solids, if necessary.

4.3.2 High-Concentration Sample 2, Use of En Core[®]-Type Samplers

Supplies:

One 5 or 10 -gram En Core[®]-type Sampler

1. Label the sample as appropriate.
2. Select the area to be sampled as soon as possible after the soil is exposed.
3. Scrape away the surface material from the area to be sampled to expose fresh soil.
4. Rapidly insert the sampler into the soil to obtain the sample. Quickly wipe the contact areas to remove any soil particles, close and seal the device.
5. Place devices in resealable pouch, place in cooler on ice.
6. Collect separate sample for percent solids, if necessary.
7. Samples must be frozen, preserved or analyzed within 48 hours of collection.

4.4 Low-Concentration Sub-Sample Collection Procedures

There are four options for collecting low-concentration soil samples:

- collection in VOA vials containing water,
- collection in empty VOA vials,
- collection in VOA vials containing sodium bisulfate, and
- collection using En Core[®]-type devices.

All of the procedures using VOA vials are essentially the same, except for the media contained in the vial. It should be noted that sodium bisulfate preservation might lead to formation of acetone in samples containing high amount of humic material. Additionally, certain analytes, such as styrene, vinyl chloride, trichloroethene (TCE), may be decomposed by the bisulfate, leading to low- biased results. Also carbonate rich soils may effervesce. The effervescing will result in significant losses of VOCs, and in such cases the sodium bisulfate cannot be used. Environmental professionals should use caution in using this preservation technique. **The use of sodium bisulfate can be an acceptable option, so long as the limitations discussed in this guidance are considered on a site-specific basis**

4.4.1 Low-Concentration Sample Option 1, Collection in VOA Vials Containing Water

Supplies:

Electronic field balance accurate to 0.1 grams

2 VOA vials (40 ml), pre-weighed and containing 5 mls of water and a magnetic stir bar

Sub-sampling device

1. Label the vials as appropriate. Do not add more weight (e.g., excessive labels) to the pre-weighed vials.
2. Select the area to be sampled as soon as possible after the soil is exposed.

3. Obtain a test sample, using the coring device and field balance, to determine approximately how much volume of soil will yield 5 grams of soil. Note that the sample weight should be within 1 gram of the nominal weight, e.g. 5 ± 1 gram. This step may be skipped when the amount of soil needed for a particular matrix at a site has been determined.
4. Scrape away the surface material from the area to be sampled to expose fresh soil.
5. Rapidly insert the syringe into the soil to obtain the first 5-gram sample. Quickly extrude the sample into one of the two vials containing the water. Wipe off the threads and cap; seal the vial.
6. Repeat steps 4 & 5 for the second vial containing water.
7. Using the field balance, weigh and record the weight of each vial. A record of the weight must be submitted with the samples to the laboratory.
8. Place all samples in cooler with ice.
9. Collect separate sample for percent solids, if necessary.
10. Samples must be frozen or analyzed within 48 hours of collection.

4.4.2 Low-Concentration Sample Option 2, Collection in Empty VOA Vials

Supplies:

Electronic field balance accurate to 0.1 grams
2 VOA vials (40 ml), pre-weighed containing a magnetic stir bar
Sub-sampling device

1. Label the vials as appropriate. Do not add more weight (e.g., excessive labels) to the pre-weighed vials.
2. Select the area to be sampled as soon as possible after the soil is exposed.
3. Obtain a test sample using the coring device and field balance, to determine approximately how much volume of soil will yield 5 grams of soil. Note that the sample weight should be within 1 gram of the nominal weight, e.g. 5 ± 1 gram. This step may be skipped when the amount of soil needed for a particular matrix at a site has been determined.
4. Scrape away the surface material from the area to be sampled to expose fresh soil.
5. Rapidly insert the syringe into the soil to obtain the first 5-gram sample. Quickly extrude the sample into one of the two vials. Wipe off the threads and cap; seal the vial.
6. Repeat steps 4 & 5 for the second vial.
7. Using the field balance, weigh and record the weight of each vial. A record of the weight must be submitted with the samples to the laboratory.
8. Place all samples in cooler with ice.
9. Collect separate sample for percent solids, if necessary.
10. Samples must be frozen or analyzed within 48 hours of collection.

4.4.3 Low-Concentration Sample Option 3, Collection in VOA Vials Containing Sodium Bisulfate

Supplies:

Electronic field balance accurate to 0.1 grams

2 VOA vials (40 ml), pre-weighed containing 5 ml sodium bisulfate solution and a magnetic stir bar

Sub-sampling device

1. Label the vials as appropriate. Do not add more weight (e.g., excessive labels) to the pre-weighed vials.
2. Select the area to be sampled as soon as possible after the soil is exposed.
3. Obtain a test sample using the coring device and field balance to determine approximately how much volume of soil will yield 5 grams of soil. Note that the sample weight should be within 1 gram of the nominal weight, e.g. 5 ± 1 gram. This step may be skipped when the amount of soil needed for a particular matrix at a site has been determined.
4. Scrape away the surface material from the area to be sampled to expose fresh soil.
5. Rapidly insert the syringe into the soil to obtain the first 5-gram sample. Quickly extrude the sample into one of the two vials containing the bisulfate solution. Wipe off the threads and cap; seal the vial.
6. Repeat steps 4 and 5 for the second vial.
7. Using the field balance, weigh and record the weight of each vial. A record of the weight must be submitted with the samples to the laboratory.
8. Place all samples in cooler with ice.
9. Collect separate sample for percent solids, if necessary.

4.4.4 Low-Concentration Sample Option 4, Collection Using En Core[®]-Type Devices

Supplies:

Two 5-gram En Core[®]-type sampling devices

1. Label the sample as appropriate.
2. Select the area to be sampled as soon as possible after the soil is exposed.
3. Scrape away the surface material from the area to be sampled to expose fresh soil.
4. Rapidly insert the sampler into the soil to obtain the first sample. Quickly wipe the contact areas to remove any soil particles, close and seal the device. Place device in resealable pouch
5. Repeat steps 3 and 4 for the second En Core[®]-type device.
6. Place both devices in resealable pouches, place in cooler on ice.
7. Collect separate sample for percent solids, if necessary.
8. Samples must be frozen, preserved or analyzed within 48 hours of collection.

4.5 Collection of Soil Samples for TCLP or SPLP Volatile Organic Analysis

The holding time for soil samples to begin the leaching procedure for TCLP or SPLP extraction for VOC analysis is 14 days from collection. If the environmental professional requests the laboratory to hold the samples until the results of the total (i.e., mass) analysis for VOCs is available, the total analysis must be available within a time-frame that will permit the environmental professional to give the laboratory sufficient notice to be able to start the SPLP/TCLP leaching within the 14-day holding time.

Supplies:

25-gram En Core[®]-Type Sampler

1. Label the sampler as appropriate.
2. Select the area to be sampled as soon as possible after the soil is exposed.
3. Scrape away the surface material from the area to be sampled to expose fresh soil.
4. Rapidly insert the sampler into the soil to obtain the sample. Wipe off the threads and cap; seal the sampler.
5. Place sampler in resealable pouch and place in cooler with ice.
6. Samples must be frozen or leached within 48 hours of collection.

4.6 Collection of Soil Samples for Percent Solids Determination

A laboratory typically can use any container submitted for analysis to determine the percent solids of a soil, **except a container submitted for VOC analysis**. If the other laboratory analyses, besides volatile organic compounds (either total or TCLP/SPLP volatiles), are to be performed on soil for a given sampling interval and location, a separate container(s) will be needed for the other tests. The percent solids determination can then be performed using the soil in the container(s) for the other tests. In the event that only VOCs are to be determined for a given soil sample, the environmental professional must collect additional sample (no more than 20 grams would be needed) in a separate container for submittal to the laboratory. Typically, a small plastic container would suffice, although any container would do.

5.0 QUESTIONS AND ANSWERS

Do sites that were previously verified using the “old” sampling methods need reevaluation using this guidance?

Previously verified sites or closed sites do not need to be re-assessed. However, the Department may require additional site characterization on a site-specific basis, particularly if there is a potential significant risk to human health from direct exposure to VOCs in shallow soils (i.e., exposed soils less than two feet below ground surface).

If a previously verified establishment is transferred after March 1, 2006, do I need to re-assess former Areas of Concern (“AOCs”) that were characterized without VOC sample preservation?

AOCs that were previously characterized do not need to be reassessed, provided that no hazardous substances have been used, stored, or released at the AOC since the date of the previous assessment. Any AOCs where hazardous substances have been used, stored, or released subsequent to the previous assessment would need to be characterized in accordance with current methods. However, environmental professionals should evaluate the old data in the context of their site-specific conceptual model to determine whether potentially low-biased results for VOCs that may be present in soil at the site would create a significant data gap or whether the potential low bias might result in a potential significant risk to human health. Based on this evaluation, additional investigation and/or remediation may be warranted.

Can I use VOC data from samples that were collected and submitted for laboratory analysis prior to March 1, 2006 without preservation as described in this guidance?

Yes, data from samples collected prior to the March 1, 2006 implementation date may be used. However, environmental professionals should evaluate the old data in the context of their site-specific conceptual model to determine whether potentially low-biased results for VOCs that may be present in soil at the site would create a significant data gap or whether the potential low bias might result in a potential significant risk to human health. Based on this evaluation, additional investigation and/or remediation may be warranted. Samples collected on or after March 1, 2006 should be obtained in accordance with the procedures of this guidance.

How can old data, which may be biased low, be used in my conceptual site model to determine if the investigation and remediation of the site is adequate and complete?

The process for using old data is no different than evaluating the representativeness of any analytical data. It is best to evaluate such data in light of the data quality objectives for the site using the site-specific conceptual site model. Pertinent questions that should be considered and addressed as appropriate and include the following:

- Do I have additional lines of evidence to support the soil sample results (e.g., groundwater or soil gas data)?
- If soil samples were preserved by another method, how likely is it that the procedures used minimized the loss of VOCs?
- Is the uncertainty of the potentially low-biased result a significant data gap, which could affect your conceptual site model conclusions?
- What are the potential implications of low-biased results?
- Were the results close to the regulatory standard?
- Are there sensitive receptors that could be affected?
- Is there evidence of a continuing source of groundwater contamination?
- Have site conditions changed that may have affected the VOC concentrations? (e.g. addition or removal of buildings, parking lots, or other structures)?
- Has remediation taken place since original sample collection?

What should be done in a case where the most likely potential point of release or the area of presumed worst-case pollution is not accessible for sampling methods that would result in an “undisturbed” sample for determination of VOCs?

Depending on the data quality objectives, it may be necessary to collect a sample from this area. Any sample collected in this area would be considered a “disturbed” sample (e.g., hand auger, samples collected from subsurface after using an air knife, or hand sampling that would not result in a bulk, undisturbed sample) and thus, would underestimate the concentration of VOCs in the subsurface. Even when “disturbed” samples are collected, it is still critical to preserve the sample as quickly as possible, in accordance with this guidance. In relying on this data for decision-making purposes (e.g., determining if a release has occurred), it would be necessary to consider this potential underestimation of VOCs. Further, it is important to evaluate the data in accordance with the conceptual site modeling process. Other lines of evidence, such as soil vapor sampling or groundwater sampling, may be necessary to determine if the data collected from the “disturbed” sample is useable given the specific data quality objective for such a sample.

What are considered “low” and “high” concentration samples?

“Low” and “high” refer to concentration levels. The specific concentration can vary between laboratories, but generally “low” refers to a concentration below the detection limit to approximately 200 ug/kg and “high” would refer to any concentration greater than this value.

Can we freeze the samples in the office or field prior to delivery to the laboratory?

Yes, as long as the conditions specified in this guidance are met and documented. This includes using a temperature-measuring device that is National Institute of Standards and Technology traceable and a daily log of freezer temperatures is kept. A standard operating procedure for storing samples is needed. Typically, household freezers do not maintain the required temperature range.

Will preserving soil samples in accordance with this guidance significantly change the cost of VOC sample collection, preservation, and analysis?

The cost for collection and preservation will increase compared to samples not collected in accordance with this guidance. The amount of the cost increase will vary depending on the collection and preservation options selected. The cost of the laboratory analysis should not increase.

Will the use of this guidance result in an increase of potential PMC issues?

Yes – preserved soil samples will result in an increase in potential PMC issues based on the mass results. It is anticipated, that the SPLP/TCLP analysis may be more common to evaluate PMC issues than results based solely on mass analysis. Section

4.5 provides information regarding the collection of soil samples for SPLP/TCLP analysis.

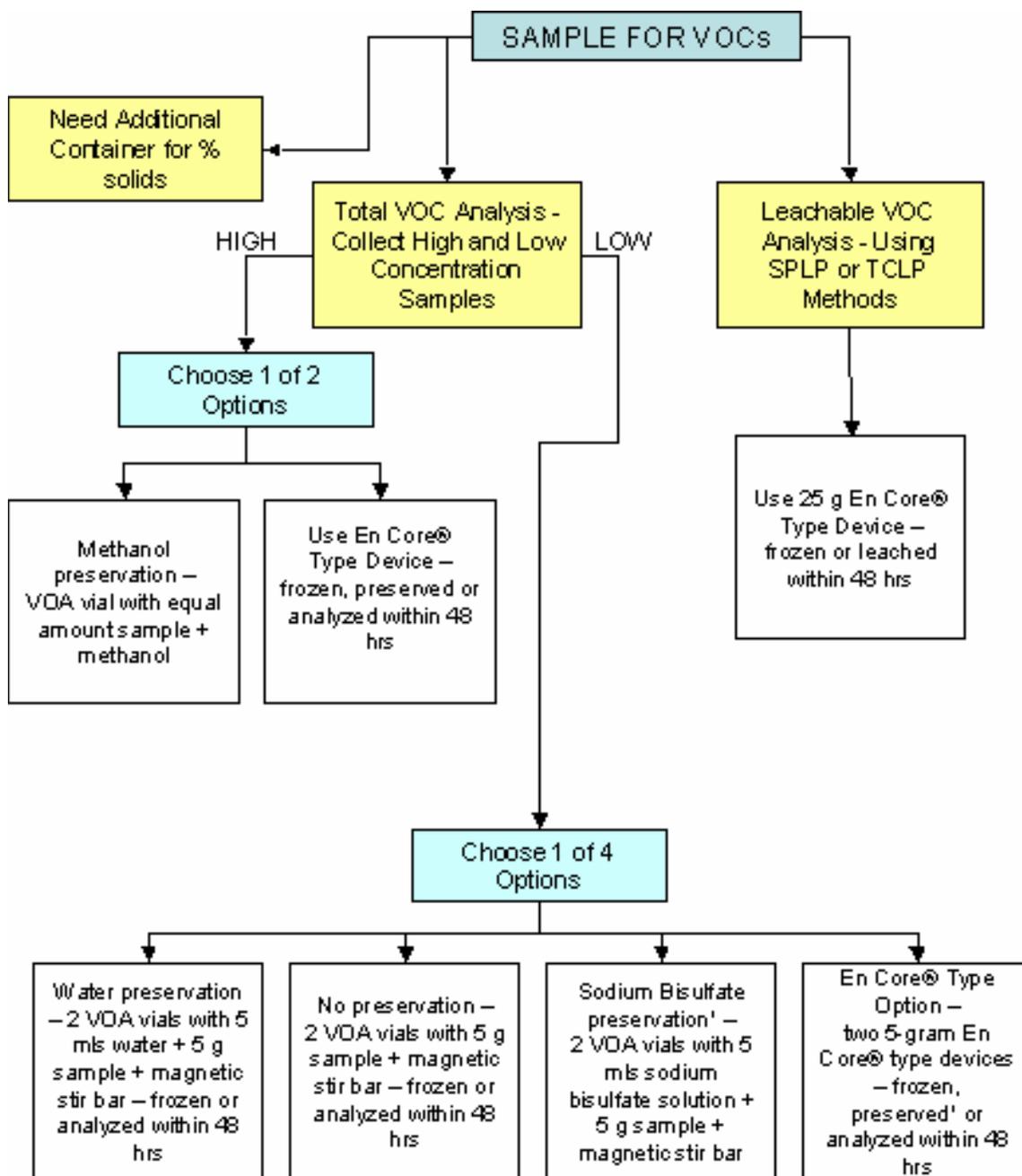
How can I get further guidance?

If you have questions regarding this guidance or the application of this guidance, please contact the DEP project manager for the site, the DEP District Supervisor for the Remediation Section, or DEP Supervisor for any other applicable program.

6.0 REFERENCES

1. *Behavior and Determination of Volatile Organic Compounds in Soil*, U.S. Environmental Protection Agency (EPA), EPA/600/R-93/140, May 1993.
2. Chemical Preservation of Volatile Organic Compounds in Soil Subsamples, Hewitt, A.D., U.S. Army Corps of Engineers (USACE), USACE Special Report 95-5, February 1995.
3. Obtaining and Transferring Soils for In-Vial Analysis of Volatile Organic Compounds, Hewitt, A.D. and Lukash, N., U.S. Army Corps of Engineers, USACE Special Report 96-5, February 1996.
4. U.S. Army Corps of Engineers Sample Collection and Preparation Strategies for Volatile Organic Compounds in Solids (October 1998).
<http://clu-in.org/download/stats/sampling.pdf>.
5. Preparing Soil Samples for Volatile Organic Compound Analysis, Hewitt, A.D., U.S. Army Corps of Engineers, USACE Special Report 97-11, April 1997.
6. Storage and Preservation of Soil Samples for Volatile Organic Compound Analysis, Hewitt, A.D., U.S. Army Corps of Engineers, USACE Special Report 99-5, May 1999.
7. Rationale for Preservation of Soil and Sediment Samples for Determination of Volatile Organic Compounds, Connecticut Department of Environmental Protection Laboratory Quality Assurance and Quality Control Workgroup, October 2005,
<http://www.epoc.org>.

Figure 1 – Sample Collection Flow Chart



1. Not appropriate for all circumstances – see Section 4.4 of this document.

CT DEP Soil Preservation Workgroup responses to comments regarding “GUIDANCE FOR COLLECTING AND PRESERVING SOIL AND SEDIMENT SAMPLES FOR LABORATORY DETERMINATION OF VOLATILE ORGANIC COMPOUNDS” Version 1.0, DRAFT FOR PUBLIC COMMENT,” dated January 2006.

The Workgroup’s responses to comments on the above document received from the public are presented in ***bold italics***.

Dr Schulthess, UCONN

C-1 I read the report. It seems to be a smart improvement from the old procedure.

We concur.

David E. Costolnick
Weston Solutions, Inc.

C-2 After reviewing the draft guidance document on the above referenced subject I don't see much that varies from what many other states use. My question relates primarily to how data collected on sites over the past 6 years using the "old" method will be affected. When the UST group started recommending the 5035 Method we asked if it needed to be used on other sties and were told that if we were not then using the method we should not switch. I have serious concerns about where this is going to end up.

This comment was received for the document titled “Rationale for Preservation of Soil and Sediment Samples for Determination of Volatile Organic Compounds (October 2005, Connecticut Department of Environmental Protection Laboratory Quality Assurance and Quality Control Workgroup),” (the “White Paper”) and was subsequently addressed by the guidance.

Dr. Gail Batchelder and Dr. Nick Skoularikis
Loureiro Engineering Associates, Inc.

C-3 Investigation/Remediation
Collection of Undisturbed Surface Sediment Samples: An efficient and effective way to collect undisturbed surface sediment samples has become the focus of monitoring and assessment of contaminated sediments. In response, EPA developed a new and innovative sediment sampler was designed and fabricated by the EPA. The new undisturbed surface sediment (USS) sampler consists of a core tube housed within a stand that provides isolated, mechanical support in a sediment bed. The tension on the deployment line is slowly released once the USS sampler has touched the bottom so that the core tube gently descends into the

sediment. A weight spindle then descends and pushes the core tube farther into the sediment, collecting the sample. Upon retrieval of the sampler, the sample is maintained undisturbed inside the tube until it is removed for subsampling. For more information, click here. <http://www.epa.gov/nerl/research/2005/g3-1.html>

This is beyond the scope of this guidance.

Mary Anne Stanley Magnuson
Consulting Environmental Sciences, Inc.

C-4 I have no major problems with the new procedure except it may be impossible to use during tank removals. Unless the soil is extremely stable, I am reluctant to climb down into a tank grave to get side wall and bottom samples. Usually I have the excavator take the bucket and scrape the sides of the walls and bring it to the surface where I collect the sample in a jar as tight as possible, put the cap on and put it on ice. If I can get into the tank grave I can certainly extend a sampling tube into the side wall and collect as procedure. If we are putting in shoring it gets even more complicated with really loose soil falling into the excavation before the shoring is pounded and then the side walls really can't be accessed.

Please see the guidance regarding the collection of bulk samples from a backhoe bucket. DEP does not endorse unsafe work practices.

C-5 One other issue that I have encountered is when soil samples are collected below the water table and the ground water is already known or soon to be discovered to have VOC's dissolved in the system. The soil sample will be wet and the VOC's reported are the result of the water in the sample and not from the soil particularly when its coming in from an off site source and the soils above the water table are clean.

This is beyond the scope of this guidance.

C-6 My experience has been (since 1979) that all analytical results are only a probability that certain compounds are present at 95% certainty unless you ask the lab to lower the probability to 90% in the case of highly degraded releases. Highly degraded compounds can be problematic as say TCE for example may be partially acted on biologically and will be present but not recognized against the standard at 95% certainty so is reported as ND. Also samples that are extremely high and have multiple components present a problem as most of the minor components will be below the detection limit if the lab has to dilute the sample for one major component so as not to mess up the column.

This is beyond the scope of this guidance.

The accuracy you are seeking is admirable, but I honestly don't think that it will improve on my ability to interpret the data I obtain or to evaluate the natural resources of the sites with any greater degree of accuracy.

Robert K. Kennedy
AMRO Environmental Laboratories

C-7 The assumption on page 7 that the VOA reporting limits for methanol preserved samples are greater than 250 ug/kg is incorrect. Most newer GC/MS systems for VOC analysis can be calibrated down to 1ug/L for analytes that purge well, which covers most of the 8260 analyte list. If the soil to methanol ratio is 1:1 and the aliquot of MeOH added to each 5mL purged is 200uL, then the nominal reporting limit for most analytes will be 25ug/kg.

This is sufficiently sensitive to meet all the GB mobility criteria and most all of the GA/GAA criteria without the use of more problematic and less efficient "low-level" analysis of 5g soil per VOA vial without MeOH. In our experience QA/QC and technical problems are much more common with the "low-level" analysis than the MeOH preserved/extracted, moreover the methanol extraction is so much more efficient than direct heated sparging of the soils that the MeOH preserved sample results frequently have higher VOA concentrations than the same samples analyzed by the "low-level" technique.

The laboratory instrumentation must be calibrated at its reporting limit. Newer instrumentation and technologies may allow for lower reporting limits than listed in Table 1.0 of the RCP Method 8260. The appropriate reporting limit will depend on the data quality objectives of the project. For example 25 ug/Kg may be adequate for delineating the extent of a known release, but a lower reporting limit (5 ug/kg) is generally needed for determining whether or not a release has occurred.

Kathy Shaw
Conestoga-Rovers & Associates

C-8 I have recently moved to Connecticut from Michigan where they have adopted the use of TerraCore samplers (MeOH preservation in the field), which requires the submission of both trip and field blanks with all soils collected for VOC analysis. In section 4.2 of GUIDANCE FOR COLLECTING AND PRESERVING SOIL AND SEDIMENT SAMPLES FOR LABORATORY DETERMINATION OF VOLATILE ORGANIC COMPOUNDS (below), the collection of both TB/FBs are mentioned; would these be vials of DI or MeOH which would accompany the samples, with the FB being exposed to the environment? I look forward to your comments.

4.2 Sub-Soil Sample Collection Procedures

When collecting soil sub-samples for determination of volatile organic compounds, up to four types of samples may be required:

- A high-concentration-level sample
- A low-concentration-level sample
- An SPLP/TCLP sample
- A sample for percent solids determination

Additional samples may be necessary for matrix spikes and matrix spike duplicates. Field and trip blanks also may be required. An overview of the various options for sample collection procedures is attached as Figure 1.

Trip blanks and field blanks should be matched with respect to matrix and volume of the preservatives used. If no preservatives are used, a vial of water can be used as a trip blank. The use of trip and field blanks should be based on the project specific work plan, quality assurance project plan, or the like. Therefore, the use of such samples is beyond the scope of this guidance.

Dave Scott

Radiation Safety and Control Services, Inc.

C-9 I have reviewed the subject Draft Guidance on Soil Sampling for VOC Analysis. It is not clear to me why, under Sections 4.3.1, 4.4.1, 4.4.2, and 4.4.3, it is necessary to weigh the sample vial before and after injecting the soil sample into the vial, (and to provide the weight to the analyzing laboratory) if one has already conducted a site-specific test to determine what volume of a given matrix results in 5 grams of sample. Could you please provide some clarification of the need for the sample weighing?

Weighing is needed to determine if there is leakage of the liquid preservative and to ensure that the appropriate proportion of sample and preservative is present.

Andrew J. Danzig
Malcolm Pirnie, Inc.

- C-10 1. The Rationale document stated that sodium bisulfate preservation may result in low bias of styrene, while the Guidance document stated styrene, TCE, and vinyl chloride results may be biased low. Please clarify this inconsistency. What is the degree of low bias for each compound?

The loss of styrene, TCE, and vinyl chloride is documented in Method 5035A. The degree of low bias is unknown.

- C-11 2. The Rationale document recommends that the laboratory reject any sample that arrives without a good seal. Since determination of the quality of the seal on a VOC vial is subjective, rather than reject the sample and lose potentially irreplaceable data, I suggest the sample be analyzed and the results flagged that there may a low bias due to suspected poor seal.

The sample may be analyzed and flagged and may be used depending on the data quality objectives for the project. The guidance document has been revised to clarify this issue.

- C-12 3. The Guidance document makes repeated reference to En Core-type samplers. A neutral term that can be used is "syringe-type" sampler. Despite the disclaimer that DEP doesn't endorse or recommend any product, repeated use of one product name may give the reader the impression that it is the preferred product. (It is also one of the most expensive samplers available.)

The En Core-type samplers are storage devices as well as core type samplers. The syringe is a sampling device only. The DEP does not recommend syringe type samplers for storage. DEP does not endorse the use of any brand product over another.

- C-13 3a. Why is the En Core the only sampler that may be used for sampling, storage, and transportation (Section 3.1)? Other syringe-type samplers can be handled the same way.

See item C-12. This is covered in the guidance.

- C-14 4. The recommended requirement in the Guidance to weigh the samplers and the sample to achieve 5 gram +/- 1 gr is cumbersome and unnecessary. The Guidance already states that 3 cc is the approximate volume for a 5-gram sample and VOC vials supplied by most labs these days include fill to lines. The benefit gained by having +/- 1 gram precision in

sample weight must be weighed against the loss of volatilization from the sample during the extra time necessary to complete the repeated weighings of the sampler. Also, since the sample weight will be measured based on the tare weight of the vial (usually in the lab), the pre-sample weighing is redundant. If the lab finds a problem with sample quantity, good labs will report that problem to the sampler so steps can be taken to avoid them in the future. We haven't encountered any problems using the 3 cc measurement or fill lines on the vials as guides for sample quantity. The goal is to improve the sample collection process to produce good, reliable samples, not create more work for field crews.

See item C-9.

Gary Trombly, CT DEP

C-15 The flow chart depicts all preservation options concisely however, problems using sodium bisulfate preservation have been documented.

It has been recommended that the Department remove this preservation option because the data could be impacted by the sodium bisulfate preservation. Please consider removing this option.

The use of sodium bisulfate can be an acceptable option that has limitations, which must be considered on a site-specific basis. Clarification has been added to section 4.4 of the guidance.

Amy L. Doupé
Lancaster Laboratories

C-16 One of our clients would like to implement the use of encore samplers for TCLP/SPLP analysis. Putting this system in place is going to require some changes in our sample handling procedures and I would like to clarify the requirement of freezing if samples cannot be leached within 28 hours of receipt. Must the samples be frozen, or are we permitted to store the samples in the manner described in the TCLP/SPLP methods (refrigerated at 4 C for 14 days).

The samples must be frozen or leached within 48 hours from collection.

Robert R. Kovach, II
Fuss & O'Neill, Inc.

C-17 I have a comment regarding Section 4.5 of the draft VOC sample collection and preservation guidance document. In the guidance, the only acceptable sample collection/preservation option given for leachability testing of soil for VOCs is the 25 gram EnCore sampler. Although the number of leachability samples is anticipated to increase somewhat with implementation of the new guidance, the number of these analyses will still be low compared to the total number of VOC samples collected. As written, the draft guidance forces the sampler to incur the expense of sample collection in the 25-gram EnCore sampler for every sample, even though most will never be analyzed, or risk the need for an additional mobilization to collect the sample.

I submit that an alternative collection/preservation method should be added that minimizes the VOC loss while avoiding the need for the enCore sampler. One possibility is collection and subsequent freezing of a zero-headspace vial of soil for contingent SPLP or TCLP analysis. During the February 2 meeting, Mr. Curran indicated that opening and manual extraction of the vial using this method would be a concern relative to VOC loss. This concern could be largely addressed if the laboratory extracted an approximately 25 gram "plug" of soil directly from the vial by the lab using a syringe or similar device instead of scooping the soil out of the vial when initiating the extraction procedure. As during sample collection, this method can be done relatively quickly and minimizes disturbance, while avoiding the explicit need for an EnCore sampler.

The Soil Preservation Workgroup (Workgroup) received several comments related to the analysis of soil samples by TCLP/SPLP methods. The Workgroup has explored potential options for collection and storage of samples for analysis for volatile organic compounds following TCLP/SPLP extraction. The Workgroup considered three options for such samples.

Option 1: Collection using a new procedure – The Workgroup has not encountered a new procedure that was found acceptable during the writing of the guidance or responding to comments on the draft guidance. However, the Workgroup agrees that alternative sample collection procedures should be studied and evaluated further. The Workgroup intends to determine if freezing of a sample in some other type of container than an En Core® -type device is feasible, while providing acceptable recoveries of volatile organic compounds and minimizing sample handling by the laboratory.

Option 2: Collection of Samples in 2-ounce Soil Jars with Septum tops – This procedure is essentially the current practice. The literature (see the “White Paper”) has shown that significant losses of volatile organics occur if samples are stored in such containers at 4° C. Also losses can and do occur during sample handling by the laboratory, such as using a spatula to remove 25 grams of soil from such a container. This will result in the breakup of the “intact” soil structure and lead to significantly low biased results. Freezing of the containers will result in adequate protection of the volatile organics during storage, but significant losses are apt to occur during sample handling after the sample is thawed and processed for the leaching procedure.

Option 3: Using 25 gram En Core® -type devices – ASTM Method D-6418-04, Standard Practice for Using the Disposable En Core® Sampler for Sampling and Storing for Volatile Organic Analysis describes how to collect and store soil samples using such devices. The document also includes studies, which show that volatile organic compounds are not significantly lost when stored in such devices at freezing temperatures over a 14-day period. The Workgroup is unaware of any other studies, which show that other devices are suitable for storage of samples over the 14-day holding time and will allow the laboratory to quickly transfer the soil sample from the storage container to the zero headspace extractor without disturbing the “intact” soil structure.

For these reasons the Workgroup, at this time, does not recommend any procedure other than Option 3 for the collection and storage of soil samples for later determination of volatile organic compounds following SPLP/TCLP extraction. The Workgroup notes that other states, such as the NJDEP, have mandated sample collection and storage using such devices. The Workgroup will continue to research potential alternative procedures and solicit interested parties to submit data about or ideas for potential alternative procedures. Acceptable alternative procedures should be documented to adequately preserve the volatile organic compounds while allowing for minimal sample handling in the laboratory.

The Workgroup intends to revise this guidance to include other acceptable alternative procedures for collection and storage of samples for analysis for volatile organic compounds following TCLP/SPLP extraction. As soon as possible, the Workgroup intends to research this issue and revise this guidance, as appropriate. Interested parties should submit information to Peter G. Hill, CT DEP, 79 Elm Street, Hartford, CT 06106 or by e-mail at peter.hill@po.state.ct.us

Paul Jobmann
LEGGETTE, BRASHEARS & GRAHAM, INC.

C-18 Presently there is only one option for collecting a sample for SPLP/TCLP analysis. Is it possible to collect 5 - 5 gram samples using the no preservation method in 40 ml VOA vials? I don't know if it will be easier but it will cost less.

Your suggested method may result in low bias results and is not acceptable. See item C-17.

C-19 In the past when using the 5035 extraction method, the lab provided preserved bottles with a line drawn on it. The sampler was directed to add soil to the vial until the liquid level rose to meet the line. Is this method an approved method of collecting a sample?

Continuing to add soil until soil and preservative reaches a pre-marked line is contrary to this document. One plug of soil must be added quickly to the VOAs. Since adding one plug is critical, the test samples are very important to ensure this plug is the correct weight.

C-20 If a geoprobe acetate liner is the method of collecting soil cores, does the clock start as soon as the liner is removed from the tools or when the liner is cut? I am just trying to determine if the geoprobe contractor gets ahead of the field staff if the intact cores can still be sampled within a reasonable amount of time.

The clock starts when the core sampler is removed from the ground. The field consultant needs to manage the driller so that the driller doesn't get ahead of the field staff.

C-21 My company is looking into the use of our field room freezers for freezing samples. If we do freeze samples and use a traceable thermometer, is the temperature data more for internal QA/QC during data evaluation or do the DEP staff expect to see the data in reports?

This temperature information should be part of the evaluation of the usability of the data for the intended purpose. It should be documented and available upon request.

Phyllis Shiller
Phoenix Environmental Laboratories

C-22 First, many of the low level sampling options that were discussed come from the draft procedure 5035A. The promulgated method 5035 discusses the use of sodium bisulfate for low level collection and only discusses alternatives if problems arise (ie when the samples are strongly alkaline or highly calcareous).

I think that some of the concerns for the sodium bisulfate preservative can be addressed by the analysis of site specific MS/MSD samples taken in sodium bisulfate. If the recoveries of compounds of interest are below acceptable criteria, more samples can be taken.

I am a little concerned about forcing my clients to use procedures that the EPA considers draft, especially when they contradict the established method which states that field preservation with sodium bisulfate is the primary.

See item C-15 regarding sodium bisulfate preservation.

Dr. Nick D. Skoularikis
Loureiro Engineering Associates, Inc.

C-23 Quite often, due to the potential presence of utilities and long history of the site the client requests that we advance the borings by hand auger to the first 8 feet, and then use the Geoprobe(r). This technique appears to be widespread in industrial sites. I feel that the guidance document should allow the collection of these samples, even if considered "disturbed".

Section 2.3 of the draft guidance recognizes that disturbed samples may be collected, if necessary. If disturbed soil samples must be collected, the rationale for collecting such disturbed samples must be provided, and attempts should be made to limit the disruption of the soil being collected for analysis.

C-24 Page 6 of 21, 2nd paragraph from the bottom: "It is better to use a slightly lower weight than a higher weight..." Explain; lower weight of soil or liquid?

Soil. This has been clarified in the guidance document.

Sheri Hardman
Sovereign Consulting Inc.

C-25 There was some question as to whether or not some of the laboratories would even run volatile analysis using the method currently in use. Would this strictly be a lab call or has there been some discussion with the DEP concerning this? I am thinking more from the standpoint of collecting samples in other states and sending them to a CT lab for analysis. Have any of the labs you talked to given you the same impression?

The guidance addresses the sampling and preservation of soil samples collected in, or for use in, Connecticut. The Department does not anticipate this guidance causing problems with laboratories analyzing samples, as requested.

James J. Clark and Thomas F. Stark
GZA Geoenvironmental, Inc.

- C-26 1. We question the time periods allowed for public comment and the stated effective date of this guidance. Version 1.0 Draft for Public Comment was issued on January 5, 2006 and the public comment period expires on February 17, 2006. However, the document indicates an effective date of March 1, 2006. How can any comments be evaluated and incorporated in the final version in such a limited timeframe? Also, including an effective date on a “guidance” document does not seem procedurally appropriate. Given the severely curtailed comment period, will technical comments be given consideration or are we to assume that this guidance becomes the definition for standard of care effective March 1, 2006? If so, adopting these procedures which represents such a radical change in VOC soil and sediment sampling and testing methodologies seems unreasonable. We would request that the effective date be delayed for at least 30 days to better allow the regulated community the opportunity to provide well considered comments, the DEP and its committee the opportunity to consider them, and more time for consultants and laboratories to purchase any necessary equipment, provide staff training, etc.

The DEP and the Workgroup recognize the expedited timeframe for the development of this guidance. The White Paper that forms the basis of this guidance was published on November 9, 2005, along with the effective date of this guidance. The draft guidance document was published on January 12, 2006. In addition, a public meeting and training class was held on February 2, 2006. Because the guidance is based on EPA Method 5035, which was published in December 1996, this guidance should be easily implemented. Lastly, the supplies needed are readily available.

- C-27 2. Target reporting limits EPA Method 8260 soils should be included in the RCP document just as the aqueous reporting limits are noted. A mechanism to specify the required regulatory values also needs to be included in the laboratory planning phase of the work.

The RCP Project Communication Form allows for environmental professionals to communicate to laboratories the required reporting limits. Reporting limits should be identified by the project specific data quality objectives.

- C-28 EPA Method 5035 was developed to more accurately quantify concentrations of volatile compounds in solid samples. Methanol preservation has been documented to increase the recovery of these target compounds. The method has

been adopted by New England area laboratories and on EPA Region 1 sites. The sodium bisulfate method for low level preservation was simultaneously released with the methanol preservation method by EPA. This low level preservation technique has shortcomings documented in the CT-DEP Rationale for Soil VOA Preservation Document. These shortcomings include, but are not limited to: generation of acetone in humic samples; decomposition of styrene, vinyl chloride, and TCE by the bisulfate to cause results biased on the low side; and effervescing of carbonate rich soils leading to loss of VOCs. As a result, CTDEP recommends using one of the other preservation options whenever possible. Given these documented issues, GZA's preference would be to collect soil samples in VOA vials containing a stir bar only (consistent with Section 4.4.2 of the guidance). However, samples collected in this fashion can only be analyzed with the newest instrumentation which has been re-designed to add an aqueous aliquot prior to analysis. There is no mechanism to add organic-free water to samples prior to analysis by laboratories owning a different piece of instrumentation. This severely limits our choice of laboratories to perform the low concentration analysis. Further, New England laboratories have had six-plus years to optimize MeOH preserved VOAs since MA & ME required the preservation in the late 1990s. Reporting limits generally run between 70-150ug/kg depending on water content and appropriately collected soil sub-samples. Laboratories have the option of reporting below these values as long as the concentrations are marked as estimated. To add the multi-container system for each sample collected to gain accuracy between the low level (approximately 10ug/kg limit) and the high level (approximately 70-150ug/kg limit) puts an unnecessary burden on the sample collectors, laboratories, and project turn-around times for little net benefit. We ask that the CT-DEP reevaluate the requirement to use the low concentration method or alternatively specify when the sole use of methanol preservation would be allowed?

See items C-7 and C-15.

- C-29 3. We question the need for requiring the use of an EnCore Sampler for SPLP or TCLP testing. Requiring this specific sampling vessel, as opposed to allowing the use of a VOA vial or other vessel with no headspace, creates problems related to selection of samples for SPLP analysis. The use of the EnCore will require us to pre-determine which samples will be analyzed for SPLP or TCLP prior to receipt of total analysis data. This reduction in flexibility would outweigh any benefit gained from use of the EnCore device. Currently EPA Methods 1311/1312 Zero Headspace Extractor (ZHE) requires that leachate VOA samples be held in 2 ounce teflon lid glass jars with no headspace. As the samples must be transferred to a ZHE vessel no matter whether they are held in these 2 ounce jars or the newly required 25 gram EnCores the integrity should not vary greatly between the two. Freezing the 2 ounce soil VOAs or a 40ml unpreserved VOA for that matter would probably yield similar results to the frozen EnCore without incurring the added cost for the container. No matter how the sample is collected, transferring exactly 25grams to the 500mls in the ZHE (method requirement) will necessarily

involve the loss of some volatile content. We ask that CT DEP allow the use of non-EnCore sample containers for the collection and freezing of SPLP/TCLP samples.

See item C-17.

- C-30 4. We note that although several suppliers are listed for providing equipment for sampling of VOCs for transfer to 40 ml vials, the only option available for SPLP testing of VOCs is using an EnCore Sampler. We believe it is never in anyone's best interest to specify a single supplier for the containers for an analysis. This leaves the entire profession at the whim of the supplier for sampler costs and with no allowable alternative in the event that the company either stops making the particular device or goes out of business entirely. Therefore, until other suppliers start making a similar 25 g sampling device, SPLP testing should be allowed on the 2 ounce Teflon lid glass jar containers with no headspace.

See Item C-17.

- C-31 5. What will be considered acceptable related to documentation of time and temperature of soil samples held in freezers before submittal to the lab? Is a copy of the freezer temperature log required to accompany the COC to document samples were held at the specified temperatures?

See Item C-21.

- C-32 6. How does freezing unpreserved samples affect holding time? Can it be assumed the sample is frozen the moment it is placed in the freezer or is there some delay we should assume? Does the lab also need to include time spent thawing the sample in preparation for extraction against the 48 hour clock?

The fourteen-day holding times applies to all samples. Samples must be frozen, preserved, analyzed or leached as appropriate within 48 hours of collection. It may be assumed that the clock stops and the sample is frozen when it is first placed in an appropriate freezer. The clock begins again when the sample thawing process begins.

- C-33 7. What the CT-DEP anticipate will be required for trip blanks? These aqueous samples cannot be frozen.

See Item C-8. Partially filled vials may be frozen safely if placed on their side during the freezing process.

- C-34 8. We note that soil sample VOC results can vary drastically within a given soil sample. It is not uncommon for a soil sample and its duplicate to have differences of an order of magnitude or more, depending on the specific 5 grams selected for analysis. Results are highly dependent on the individual sample. This could lead

to a situation where a sample is first tested using a low method and be found to exceed the 200 µg/kg limit. The second vial (containing a different 5 grams of soil) is then tested by the high level method and found to be significantly below the 200 µg/kg limit. Which results should be the ones used, if neither meet the required criteria?

We recognize that soils may be heterogeneous. Both results should be considered and evaluated in relation to the conceptual site model for the site.

Mary Anne Stanley Magnuson
Consulting Environmental Sciences, Inc.

C-35 One other thought is to require the soil bottles to be brown. The Chlorinated compounds are highly photosensitive and often we are collecting them from split spoons in bright sunlight. We try to get them into bottles quickly and then on ice but sometimes the field conditions are such that there is no shade when extracting the samples. Brown bottles would limit the sunlight exposure from the time the sample is in the jar.

Samples collected in accordance with this guidance will not be exposed to sunlight for an extended time period.

Dr. Gary Robbins' UCONN Graduate Field Methods Class

Because environmental regulations and guidance like this are very sparse in Connecticut by far, this document is meaningful to set a standard in soil sample collecting and preservation to ensure achievement of reliable and comparable data in future environmental works.

As a layman in environmental engineering, I don't know much about ways environmental professionals used to collect soil samples for laboratory analysis, but based on my experience in deep sea sediments sampling and analysis, I did notice big difference in geochemical analysis results based on samples retrieved and preserved by different ways. However,

C-36 I'm not sure what is the sensitivity needed to detect the VOCs in soil samples and what could be the variation range if the samples were low-biased by different ways of collecting and preservation.

See item C-7.

C-37 How many different ways in sample collecting and preservation were adopted in previous environmental works? Is there any systematic bias relationship between results generated by old ones and the ones listed in this guidance?

This guidance was created to minimize the low bias associated with previous sampling and preservation techniques.

- C-38 How to tell a soil sample has high or low concentration of VOCs before decide to use one of the sample collecting ways listed in Figure 1 and corresponding description?

Either field screening or sample analysis can determine this.

- C-39 Since using sodium bisulfate as preservative could cause several problems (refer to text 4.4), why still list it as a recommended option? A paragraph to compare each option with their pros and cons listed might be helpful to clarify this question.

See item C-15.

- C-40 Grammatical suggestion:

- 1) Some redundancies: such as on the 5th page, the DEP QA/QC work group prepared document has been mentioned twice in previous paragraph (part 2.1);
- 2) Reorganize sentences to make it clearer when describe procedures: such as in 3.2, when describe the procedure for soil sub-sample collecting, the last sentence in step 8th can be put as the first in that step description;
- 3) Consistency: Title 4.4.1 needs underline; The sentence in last paragraph on page 10, "causing damage to the glass, septum, seals, o-rings, and cap" should be as "causing damage to the glass, septum, seals, o-rings, and caps" (?)

Thank you for your suggestions. The guidance document has been revised to incorporate changes, as appropriate.

Since I have little to no level of expertise in soil sampling, most of my comments were geared towards clarifying points and definitions that I did not understand. I do understand that this document is geared towards professionals with a certain level of technical knowledge and proficiency.

- C-41 On pg. 7, continuation of sect. 3.1: I can see the importance in minimizing, the handling, transference, and disturbing the sample. Is there a single use, disposable sampler available on the market for this purpose?

There are many samplers available. Please refer to the guidance and EPA Method 5035A.

C-42 On pg 8, sect. 3.2, item 1: the text discusses decontaminating equipment before use. How is this done? how is any contaminated material removed from equipment handled?

Refer to EPA guidance on this subject. This is beyond the scope of this guidance.

C-43 On pg 12, sect. 4.4: in bold italics, discussing methods of preservation, what are methods other than sodium bisulfate, pluses and minuses for each, so the correct method can be used.

See the "White Paper" or EPA Method 5035A for additional information.

Dr. Gary Robbins, UCONN

Dr Robbins presented a red lined version of the guidance document to communicate his comments. The Workgroup has considered the redlined comments below. For purposes of brevity, the portions of the draft guidance document that did not contain comments were not included in this response. The guidance document has been revised to incorporate changes as appropriate. **Dr. Robbins' comments are in RED.**

C-44 **Over all very good advice. I think it could use more guidance on what material to sample. Say you take a core and screen it--should you take a sample where the screening method indicates the concentrations are lowest or highest? Also, you could provide more guidance on how to screen a core sample. If use a PID or FID or portable GC, guidance should be provided on calibration. Section 4 is too repetitious.**

This is beyond the scope of this guidance.

1.1 Background

Volatile organic compounds (VOCs) ~~are maybe~~ lost from soil and sediment samples (hereinafter referred to as soil samples) due to volatilization and biodegradation during collection, storage and analysis. This leads to low-biased results. Some commonly used ~~sampling~~ techniques are prone to relatively large losses and results that are potentially biased quite low

2.2 Overview of Sampling Approach

C-45 **The term undisturbed arises from geotechnical practice and applies to soil structure (e.g., bulk density). Samples collected by split spoon samplers and backhoes are typically not considered undisturbed. Since you don't really care in this context if the soil is physically disturbed, I suggest a different term be used. I recommend "inplace sample" or "intact sample".**

The term “undisturbed” for purposes of this guidance has been defined in the guidance document.

To the extent practicable, undisturbed samples should always be collected. However, in some cases, collection of a disturbed sample may be necessary to characterize source areas or other critical locations. If disturbed soil samples must be collected, the rationale for collecting such disturbed samples must be provided.

- C-46 However, under no circumstances should a sub-sample be collected from a disturbed sample that was previously used for field-screening purposes. **Should you first screen a sample then based on field screening pick the zone to be sampled? This makes little sense.**

The purpose of this statement is to ensure that soil placed in plastic bags or jars for field screening purposes are not submitted to the laboratory for analysis.

3.2 Overview of Procedure for Collection of Sub-Samples

The goal of soil sampling for the purposes of evaluating concentrations of contaminants in soil is to obtain a representative soil sample in accordance with the data quality objectives for the project. Often, this is accomplished using an appropriate small-diameter core sampler.

For example, if the objective of sample collection is to obtain a sample from the 0 to 2-foot interval for evaluation of compliance with regulatory criteria identified in the Remediation Standard Regulations, then the sampling interval should be evaluated for homogeneity, and sub-samples should be collected to create a representative 0 to 2-foot sample.

- C-47 **Are you saying samples should be composited? Not clear. If yes, this can only result in diluting sample concentrations.**

This text has been removed from the guidance document.

3.2 Overview of Procedure for Collection of Sub-Samples

10. After sample collection, immediately return the containers to an iced cooler.

- C-48 **You might add some advice on how to keep coolers cold--say below 40C. What about holders in coolers?**

The guidance specifies that iced coolers should be used. As there are many methods for storing samples in coolers, this is beyond the scope of this guidance.

4.1 Overview of the Soil Preservation Procedure

The preservation of samples for VOC analysis can be initiated either at the time of sample collection or in the laboratory.

C-50 **In the laboratory!!! It might take hours to get there and in the meanwhile the sample is volatilizing. It should be preserved immediately after collection.**

In most cases, preservation takes place in the field, at the time of collection. In all cases samples are preserved, leached or analyzed in 48 hours. Samples are stored in hermitically sealed containers and iced in a manner to prevent loss of volatiles. Please refer to the "White Paper" or EPA Method 5035 A for further information on this topic.

Samples must be frozen, preserved or analyzed within 48 hours of collection.

C-51 **(48 hours!! Too long.)**

See item C-50.

Herbert E. Woike

Groundwater Environmental Services, Inc.

C-52 1. Will the additional costs associated with the additional bottles, sampling devices and labor be covered by the State UST reimbursement program?

This is beyond the scope of this guidance.

C-53 2. Will the Laboratory QA/QC Certification form be revised to incorporate the new soil sampling procedures?

This is beyond the scope of this guidance.

C-54 3. How critical is the 5 minute timeframe for the collection of a sub-sample? I understand the desire to collect a sub-sample from an undisturbed sample as quickly as possible, but what if it were to take up to 10 minutes, would the CTDEP consider this to be consistent with the sampling guidance?

The timeframe for the collection of the sub-sample should be minimized to the extent practicable. See item C-20.

C-55 4. In order to minimize the number of un-used sample bottles, GES often stores soil samples from each split spoon in "zip-lock" bags until all PID reading are available from the borehole. Then the selected soil samples (usually the higher PID readings) are actually weighed and put into the preserved bottles. Has the "committee" thought of ways that split spoon soil samples can be field screened with a PID and still meet the 5-minute timeframe for sub-sample collection?

The sampling techniques described in your question may result in low biased data. Please refer to the guidance.

- C-56 5. To address the concern outlined in Question #4 above, GES has spoken to Jeff Curran of the Dept. of Public Health and Reza Tand of Accutest Laboratories and developed the following plan for consideration. Soil samples will be collected from each split spoon using two EnCore sampler. Samplers will be placed in a cooler while soil is logged and screened with a PID. Upon completion of the borehole the selected soil samples, stored on ice in Encore samplers, will be placed in an 10 ml vial of methanol for preservation in the field. The samples will be analyzed for VOC by the new QA/QC Reasonable Confidence Protocols by Method 8260 and should meet the GA PMC standards. (Jeff could provide a better explanation of the lab procedures to meet these standards). Additional sodium bisulfate or water preserved samples will not be necessary. This process still meets the 5-minute timeframe and also allows for screening and selection of a limited, but representative, number of samples for lab analysis. Please let me know what the committee thinks.

Please see question C-7 regarding low level sampling. Any sampling plan should be in accordance with this guidance.

Tim O'Connor
En Novative Technologies Inc.

Our company is the manufacturer of the En Core® soil sampling system. You have a very nice method, more clean and concise than most drafts we have seen.

- C-58 Care must be taken when choosing En Core® - type devices. The ASTM has a draft practice for testing zero headspace sample storage devices but the En Core® is the only device that has passed their test. Other devices merely state En Core® equivalency and use data generated in their own labs. The ASTM wrote a practice for the En Core® based on third party testing.

[Comment refers to ASTM Method D6418-04]

We had some suggestions on the draft method:

- C-59 You may want to mention how long samples can be frozen before being analyzed.

See item C-51. This issue is discussed in the guidance.

- C-60 Page 6. you may want to note that cut-off syringes are not recommended as stated in method 5035 because they are sometimes coated with glycerin which could negatively impact the results.

The guidance document has been modified to note that glycerin coated syringes should not be used.

- C-61 Page 9. #7 it could be clarified that when using an En Core® type sampler, the soil moisture sample need not be taken in an En Core® type sampler, any container is fine. This is explained very well on page 15 in section 4.6 so it is not critical here.

This topic is discussed adequately on page 15.

- C-62 Page 10. in the second last paragraph you may want to add that En Core® samplers with soil containing >20% moisture should not be frozen. 20% is extremely moist and is easy to spot with visible pooling of liquid on the sample surface. Greater than 20% moisture is very unusual.

The guidance document has been modified to reflect this comment.

- C-63 Page 12. There is no 10 gram En Core®, only 5 and 25 Gram. For #6 it could be reiterated that an En Core® is not needed for percent solids.

There is a 10 gram EnCore®-type sampler available.

- C-64 Page 12 4.4. Collection in empty VOA vials is falling out of favor due to its very poor results. Please see the attached paper on empty vial.

Collection in an empty vial and freezing is consistent with EPA Method 5035A.

- C-65 Page 12. there is an extra or misplaced 'the' in the second last sentence on the page.

The guidance document has been modified to reflect this comment.