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ERTP Presents...

Soil Sampling and Analysis for Volatile Organic Compounds (VOCs)

Sponsored by: EPA Office of Superfund Remediation and Technology Innovation (OSRTI)

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Instructors:

- Eric Watt, Geologist, Tetra Tech eric.watt@tetratech.com

Moderator:

- Jean Balent, Environmental Scientist, USEPA Office of Superfund Remediation and Technology Innovation balent.jean@epa.gov

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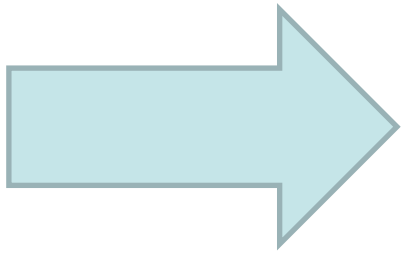
Soil Sampling and Analysis for Volatile Organic Compounds (VOCs)

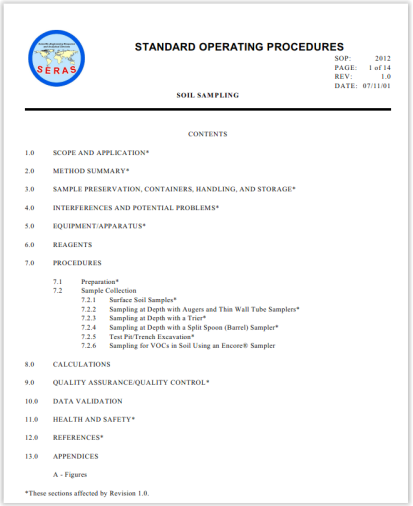
- Why?
  - Precise characterization of volatile organic compounds (VOCs) in soil is often critical since decisions for remediation are based on analytical measurement.
  - Unfortunately, the acts of collecting and storing soil can subject soils to numerous variables that can alter VOC concentrations.
    - Volatilization,
    - Biodegradation,
    - Other loss of VOCs in the sample.

Today's Webinar Topics

- The collection of high-quality soil samples for VOCs;
- Best practices for sampling techniques to minimize the loss of VOCs;
- The advantages and disadvantages of soil sampling devices, such as Encore and TerraCore samplers.

EPA ERT SOP #2012
"Soil Sampling"





STANDARD OPERATING PROCEDURES

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SOIL SAMPLING

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*These sections affected by Revision 1.0.

Soil Sampling and Analysis for VOCs

Good Soil VOC Data Begins with Good Sampling Practices

- As with all environmental samples, the laboratory data are only as good as the samples collected in the field
- When it comes to sampling soils for VOCs, everything we do from the time the soil cores are retrieved to the preparation of the sample for shipment can affect VOC recovery and sample quality

If samples are to be analyzed for volatile organic compounds, they should be collected in a manner that minimizes disturbance of the sample. For example, when sampling with a bucket auger, the sample for VOC analysis should be collected directly from the auger bucket (preferred) or from minimally disturbed

Samples for VOC analysis are not homogenized.

5035. Consult the method or the principal analytical chemist to determine if preservatives are necessary.

Good Soil VOC Data Begins with Good Sampling Practices (continued)

- Soil samples for VOCs can show significant losses within seconds to minutes of opening soil cores
- Handling cores and sample preparation methods can contribute to additional losses
- Using a sampling method that does not maintain VOCs during sample collection, shipment, and during the hold before analysis adds to the problem
- Do not collect composite or ISM samples for VOCs

Handling of Samples When a Soil Core Liner is Opened

Managing sampling from a soil core can be a complex situation - stability of VOCs in the core depends on factors such as:

1. Whether the soil is consolidated (e.g., clay, till) or unconsolidated (e.g., sand, gravel)
2. Time that the core is open or exposed before sampling (off-gassing)
3. Moisture content of the soil
4. Disturbance of integrity of the core
5. Ambient temperature
6. Heat generated by the soil coring method (e.g., sonic drilling)

VOC Loss…

Analyte compound list

- Heavier VOCs are less likely to be lost whereas lighter VOCs will likely be lost.

Density of sample material

- More porous samples (e.g. sand) will be prone to loss of VOCs whereas dense sample material (e.g. clay) would be less prone to loss.

Best Practices When Sampling with Acrylic Soil Core Liners

- Cap the acrylic liner to preserve VOCs, do not split until ready to sample
- Split liner as soon as possible – every minute counts!
- Keep liner on ice if extended times are required
- Sample immediately with EnCore or field-preserved vials upon opening
- Screen the core and select samples for analysis; USEPA SW 846 Method 3815 is the current method for screening, although use is not mandatory

Best Practices When Sampling with Acrylic Soil Core Liners (continued)

- Once a core barrel has been opened, the collection of samples is the first activity performed (i.e., pre-empting even the logging of sub-surface core sample recovery and other soil characteristics).
- Drill rig personnel should adjust their pace accordingly to allow collection of samples.

Things to Avoid When Sampling with Acrylic Soil Core Liners

- Leaving the liner exposed while screening
- Sampling from the container used for screening
  - Use of intermediate storage containers (e.g., core barrel liners, plastic bags, large glass jars) to hold soil for extended periods prior to sub-sampling defeats intent and should be avoided.
- Taking a second boring for samples after screening initial boring - no guarantee samples will match from different locations
- Using bulk sampling rather than sealed or field-preserved sampling methods

Avoid Using Soil Samplers Without Core Barrel Liners

- Soil sampling tools used without core barrel liners, such as split spoon samplers, make it nearly impossible to retain VOCs before samples are collected, even using proper sampling methods and procedures.

ASTM Standards and EPA Methods for Soil VOC Sample Collection and Analysis

- Standards of practice for obtaining, handling, and screening of soil cores:
  - ASTM Standards D6282 and D6640 cover collection and handling of soil cores for environmental sampling
- Sample collection, preservation, transportation, and analysis:
  - US EPA Method 5035, various state guidance documents
  - ASTM Standard D4547 – Sampling Waste & Soils for VOCs
  - ASTM Standard D6418 – EnCore Sampling for VOCs in Soils

Methods for Sampling Soils for VOC Analysis

- Bulk sampling method
  - No core liner or brass sleeve liner
  - Sample is scooped or sliced from soil cores and packed in wide-mouth jar without preservation
  - Lab prepares a weighed sub-sample prior to analysis
- Sub-core samples (~5) collected in field and placed in pre-preserved VOA vials for analysis
- EnCore® Sampler – sealed chamber, no field preservation required – samplers are shipped to lab for analysis

US EPA Method 5035

- Implemented in 1997 to standardize sampling and field preservation and avoid random error and low bias from bulk sampling
- Incorporates two approved methods of sample collection and preservation:
  - Field preservation of soil core sub-samples using methanol or sodium bisulfate
  - EnCore sealed samplers, with no preservation required

High Level and Low Level Methods in 5035

- Samples are preserved in methanol for “high level” analysis
  - Calibration range increases to 200 µg/kg with only a 5 gram sample.
- Samples are preserved in sodium bisulfate for “low level” analysis
  - Method Detection Limits down to 0.5 µg/Kg and concentrations < 200 µg/Kg
- Field preservation is typically done at a 1:1 ratio (weight/volume) of soil and preservative
- Pre-preserved vials supplied by the laboratory are weighed to allow calculation of soil sample weight – careful not to add additional label!
- Soil samples are based on volume and weigh approximately 5, 10, or 25 grams based on sample type
- Hold time for samples is 14 days when chilled @ 4 ^{o}C

Why are there two preservatives used in Method 5035 samples?

- Some programs have detection limit requirements for some analytes (e.g., benzene) that are lower than most labs can achieve with a 50:1 dilution of methanol in water
- On the other end of the spectrum, samples with VOC concentrations above 200 µg/kg can't be accurately quantified in a sodium bisulfate solution
- Unless you know that all of your samples will fall above or below the 200 µg/kg threshold, both samples are taken in the field.
  - The lab will screen the samples to determine which to analyze.

Collecting Samples for Field Preservation with Methanol

- Using a Terra Core sampler, place a 5 gram plug of soil into pre-preserved vial containing methanol
- 1:1 or greater weight/volume ratio of soil to methanol
- Operation must be done quickly to prevent VOC loss
- Tared weight of methanol vials should be verified before samples are collected

Safety Note!

- Methanol is a toxic and flammable liquid.
- It must be handled with all required safety precautions related to toxic and flammable liquids.
- Methanol must be handled in a ventilated area.
- Use protective gloves when handling the methanol vials.
- Store methanol away from sources of ignition such as extreme heat or open flames.

Collecting Samples for Field Preservation with Sodium Bisulfate

- Using a Terra Core sampler, place 5 gram plug of soil into pre-preserved vial containing 20% sodium bisulfate aqueous solution
- Calcareous soil types should be checked for effervescence prior to sampling or vial may explode!
- 1:1 or greater weight/volume soil/preservative ratio
- Two vials are collected (plus a MeOH preserved vial)
- Collect an additional 3 vials for an MS/MSD
- Tared weight of pre-preserved vials should be verified before samples are collected

Advantages of Field Preservation

- No further preparation of the sample is required in the laboratory prior to analysis
- Widely accepted by regulators
- VOC concentrations remain stable throughout the hold time and beyond
- Laboratory doesn't require any special tools or procedures to process sample

Disadvantages of Field Preservation

- Vial seals can leak/break
- More experienced field staff required
  - Difficult under adverse weather conditions
  - More risk of error, e.g., spillage, mixing/adding labels, etc.
  - Vehicle exhaust fumes can contaminate methanol
- Shipping restrictions may apply for methanol (not typically an issue if labeled as “laboratory samples” and volume is less than 500 mL)

Disadvantages of Field Preservation (continued)

- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

EnCore® Sampler – Alternative to Field Preservation

- The only commercially available sampler listed in Method 5035
- Meets ASTM D4547 requirements for VOC retention during permitted hold time
- 5 and 25 gram sizes
- Designed to collect an average weight – exact weight is determined in lab
- Zero headspace design – minimal air trapped in the sample
- Multiple suppliers – QEC, Forestry Suppliers, En Novative

EnCore® Sampler and sampling handle

Collecting a Surface Soil Sample

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

Soil Sampling and Analysis for VOCs

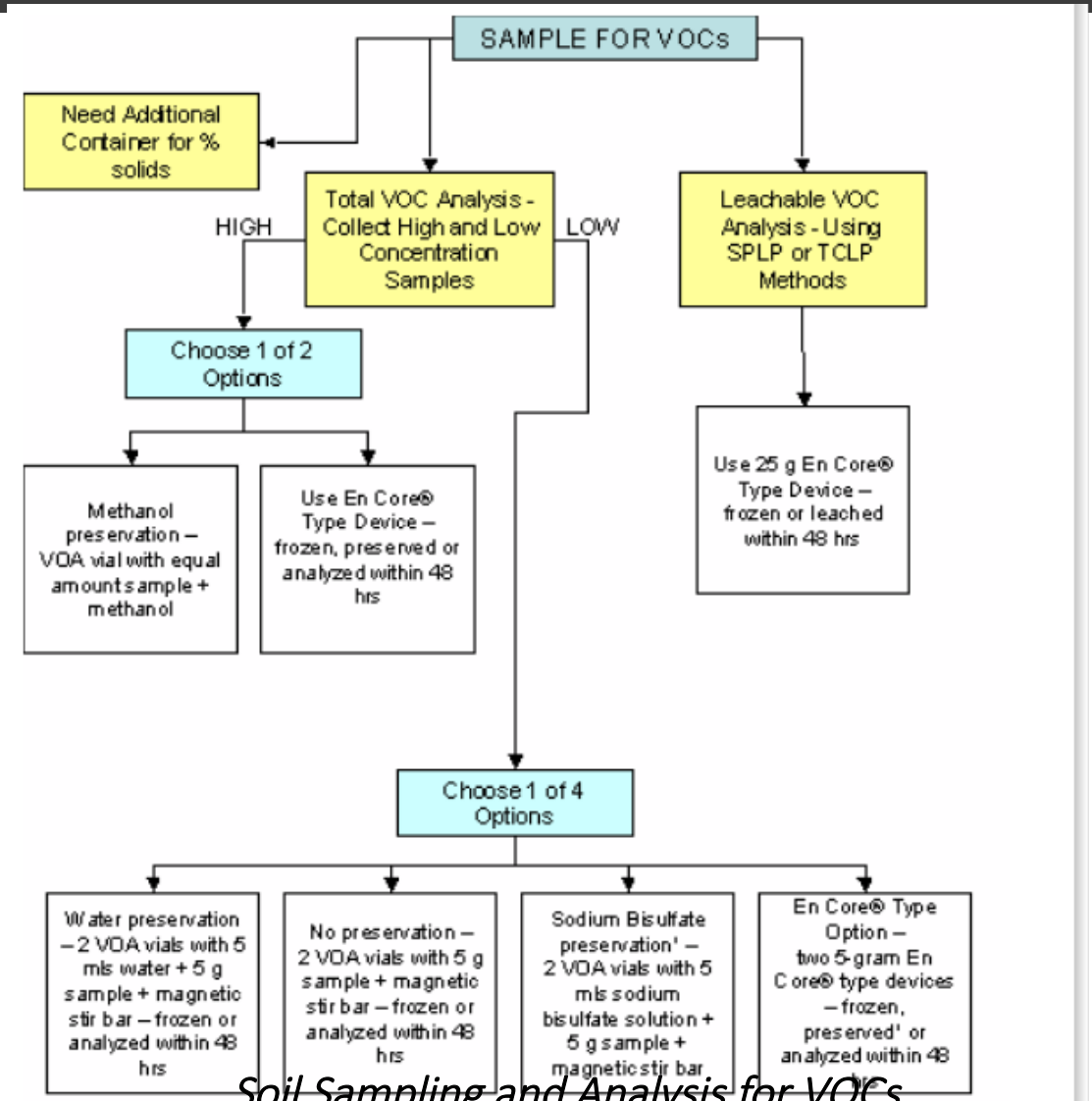
Advantages of the EnCore® Sampler

- Can be faster – less field time needed / more sample throughput
- Easy to use, minimal training
- No handling of preservative in the field
- No shipping restrictions
- Sample is preserved later in controlled laboratory
- Setting, avoids random error from field preservation
- Long shelf life – essentially no expiration
- Ships in a proprietary VOC-proof bag

Disadvantages of the EnCore® Sampler

- Samples must be preserved at lab within 48-hour hold time stated in Method 5035, however, if EnCore sampler is used for other programs:
  - ASTM D4547 describe extending hold time up to 14 days by freezing samples to -12 ± 5C
  - ASTM D6418 describes extending hold time up to 14 days by freezing samples at -7 to -21C, or holding for 48 hours at 4 ±2C followed by storage at -7 to -21C for up to five days
- Coordinate with lab if Saturday delivery expected
- Samples are reported in dry weight, so a small jar of soil should be always collected to measure soil moisture
- Most agree that per-sample cost is higher than field preservation
  - Some offset by time savings, especially for inexperienced staff
  - Lab may charge a sample prep fee

Decisions, Decisions



Decisions, Decisions, (continued)

- Sampling intervals must be decided as quickly (and safely) as possible
- If using Terra Cores®, discuss preservatives with laboratory ahead of time
- If using EnCores®, samples must be shipped to the laboratory or picked up via courier the day collected