



**En Novative  
Technologies**

## Best Practices for Collecting Soil Samples for VOC Analysis

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### Today's Webinar Topics



- The science behind collecting high-quality soil samples for VOCs – why it matters
- Collecting and handling soil cores to minimize losses
- Standards for collecting soil cores and soil samples for VOC analysis
- Overview and comparison of soil sampling methods
- Collecting better samples for field preservation using Terra Core™ and EasyDraw® samplers
- The En Core® sampler – how it works, how it compares to field-preserved samples
- Q&A session

## Good soil VOC data begins with good sampling practices

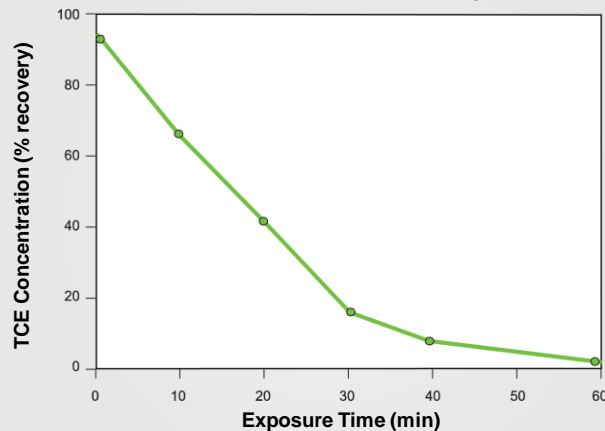


- As with any environmental sample, the laboratory data are only as good as the samples collected in the field
- When it comes to sampling soils for VOCs, everything from the time the soil cores are retrieved from the subsurface to the preparation of the sample for analysis can affect VOC recovery and sample quality
  - Soil cores can show significant VOC losses within seconds to minutes of opening core liners
  - Handling cores and sample collection methods can contribute to additional losses
  - Sampling methods that don't maintain VOCs during collection, shipment and the hold time before sample prep and analysis can add to the problem

## Volatilization losses occur within minutes of exposure



Uncontrollable volatilization for samples with a large surface to mass ratio



*Losses in trichloroethylene from soil during sample collection, storage and laboratory handling;*  
Hewitt, A. D. , U.S. Army Cold Regions Research and Engineering Lab, Hanover, NH; 1994; SR94-8

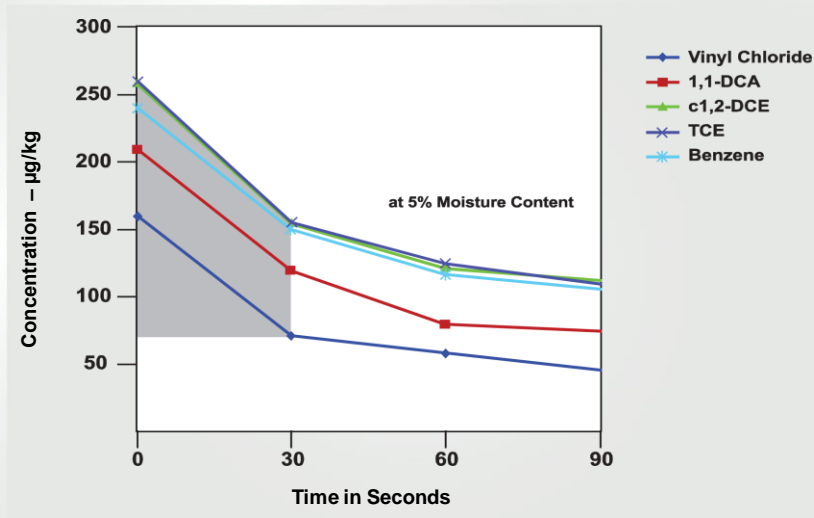
## Factors that affect VOC concentrations in soil cores



The stability of VOC concentrations in soil cores depends on factors such as:

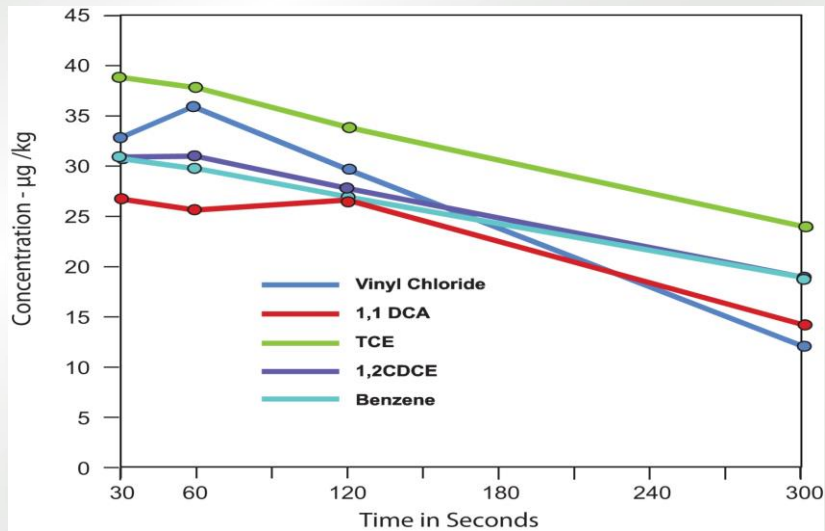
- soil type - consolidated (e.g., clay, till) or unconsolidated (e.g., sand, gravel)
- time that the core is open or exposed before sampling (off-gassing)
- moisture content of the soil
- disturbance of integrity of the core
- ambient temperature
- heat generated by the soil coring method (e.g., sonic drilling)

## VOC stability at 5% soil moisture content



Source: En Novative Technologies, Inc. internal research; Dr. David Turriff

## VOC stability at 10% soil moisture content

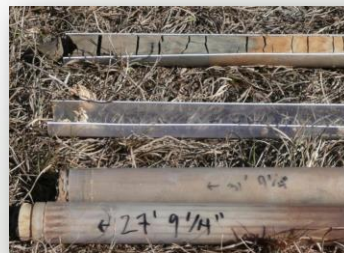


Source: En Novative Technologies, Inc. internal research; Dr. David Turriff

## Best practices when sampling with acrylic soil core liners



- Cap the acrylic liner when collected to preserve VOCs before sampling
- Sample as soon as possible – every minute counts!
- Screen the core for VOCs to select areas to sample
- Sample immediately with En Core or field-preserved vials upon opening liner
- Keep liner on ice if extended hold times are required
- USEPA SW 846 Method 3815 is the current method for screening, although its use is not mandatory



## 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



## Things to avoid when sampling from soil cores



- Sampling from the container used for screening – samples should come from undisturbed core section
- Taking a second soil core for samples after screening the initial boring – there's no guarantee that samples will match from different locations
- Using bulk sampling rather than sealed or field-preserved sampling methods

## ASTM Standards and EPA methods for soil VOC sample collection



- Standards of practice for obtaining, handling and screening of soil cores for environmental sampling
  - ASTM Standard D6282 (2014) – Direct Push Soil Sampling
  - ASTM Standard D6640 (2015) – Collection and Handling of Soils Obtained in Core Barrel Samplers
- Sample collection, preservation, shipment, and analysis
  - US EPA Method 5035A, various state guidance documents
  - ASTM Standard D4547 (2015) – Sampling Waste & Soils for VOCs
  - ASTM Standard D6418 (2009) – Using the Disposable En Core Sampling & Storing Soil for VOC Analysis

## 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 – 25 grams) collected in field and placed in pre-preserved VOA vials for analysis
- En Core® Sampler – sealed chamber, no field preservation required – samplers are shipped to lab for analysis



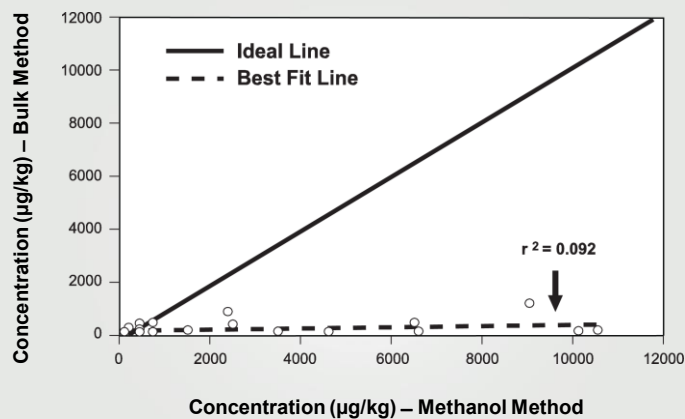
## Factors that affect accuracy using bulk sampling method



- Type of soil
- Microbe content of soil
- Grit on threads of jar
- Time to collect sample
- Air space in jar
- Ambient temperature
- Excessive hold time before sample prep & analysis
- Time spent in lab subsampling for 5.0 g sample
- Purge in water (where 5035A is not used)
- **RANGE OF ACCURACY: 0.01 - 95% true value!**

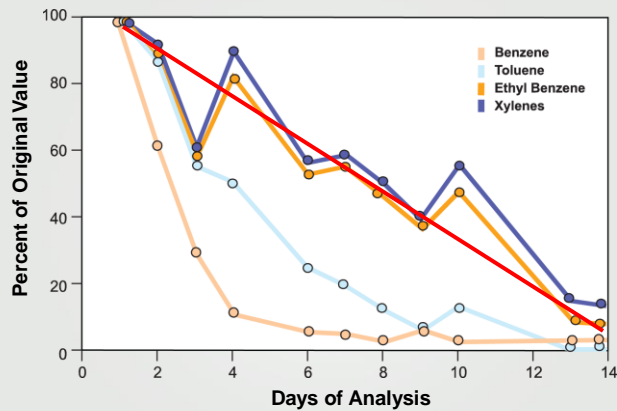


## Significant bias between bulk sampling and field preservation



*Volatile Organic Compounds: Comparison of Two Sample Collection and Preservation Methods*, Liikala, T.; Olsen, K.; Teel, S.; and Lanigan, D., November 1996.

## Biodegradation loss in bulk sample during EPA-accepted hold time



*Degradation of Hydrocarbons in Soil Samples Analyzed Within Acceptable Holding Times, Jackson, J.; Thomey, N, May 1991.*

## US EPA Method 5035A



- Implemented in 1997 (Method 5035, 1996) 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
  - En Core sealed samplers with no preservation required
- Current version is Method 5035A, 2002, (US EPA SW846)





## Adoption of EPA Method 5035A by region



Alberta AEP requires Method 5035A as of April 1, 2017



Quebec MDDELCC requires Method 5035A as of January 1, 2017

## High Level and Low Level Methods in 5035A



- Samples are preserved in methanol for “high level” analysis – concentration > 200 µg/Kg
- 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
- Soil samples are based on volume and weigh about 5, 10, or 25 grams based on sample type
- Hold time for samples is 14 days when chilled @ 4 °C

## Why are there two preservatives used in Method 5035A samples?



- Some state or federal regulatory 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  $\mu\text{g}/\text{Kg}$  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  $\mu\text{g}/\text{Kg}$  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 an EasyDraw Syringe or Terra Core sampler, place a 5 or 10 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

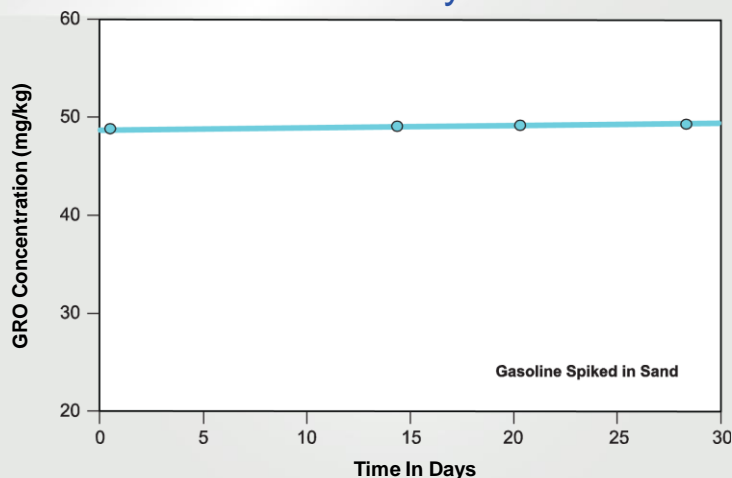


EasyDraw® Syringe



Terra Core™ Sampler

## Methanol preservation shows no VOC loss after 30 days



*Studies of Sampling, Storage and Analysis of Soils Contaminated with Gasoline and Diesel, Turriff, D. and Klopp, C., August 1994.*

## Collecting samples for field preservation with sodium bisulfate



- Using an EasyDraw Syringe or 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 (second for QC samples or replicate, since entire volume is used for analysis)
- Operation must be done quickly to prevent VOC loss
- Tared weight of pre-preserved vials should be verified before samples are collected

## Limitations of Low Level Method under 5035A



- Semi-quantitative since aqueous solution is a poor solvent for soil VOCs - results are biased low
- Can have high concentrations of acetone (formed by the preservative) as an artifact in some organic soils

## 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 the sample

## Disadvantages of field preservation



- Vial seals can leak
- More experienced field staff required
  - Difficult under adverse weather conditions
  - More risk of error, e.g., spillage, mixing 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)



## En Core® Sampler – the best alternative to field preservation



- The only commercially available sampler listed in Method 5035/5035A
- Meets ASTM D4547 requirements for VOC retention during permitted hold time
- Both a sampler and sample container
- 5 and 25 gram sizes
  - Designed to collect an average weight; exact sample weight is determined in the lab
- Zero headspace design – minimal air trapped in the sample



En Core® Sampler and sampling T-handle

## En Core<sup>®</sup> sampler components



## The En Core<sup>®</sup> sampler: How it works



1. Insert the En Core sampler into the T-handle and lock into place



## The En Core<sup>®</sup> sampler: How it works



2. Push the En Core sampler into a soil core. The sampler is filled when the O-ring shows in viewing hole on the handle



## The En Core<sup>®</sup> sampler: How it works



3. Retract the filled sampler from the core



## The En Core<sup>®</sup> sampler: How it works



4. Scrape excess soil off end if necessary



## The En Core<sup>®</sup> sampler: How it works



5. Wipe the sampler clean before sealing





## The En Core<sup>®</sup> sampler: How it works



6. Attach the En Core cap to seal sampler



## The En Core<sup>®</sup> sampler: How it works



Place the sealed En Core sampler into the special VOC-proof bag, and then seal and label the bag for shipment to the lab. You can put three 5-gram En Core samplers into a single bag for a sample set (high and low level soil VOC analyses)



## Advantages of the En Core® Sampler



- Faster – less field time per sample
- More sample throughput per day
- 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 – no expiration
- Ships in a proprietary VOC-proof bag that is also used to ship En Core samples to the laboratory



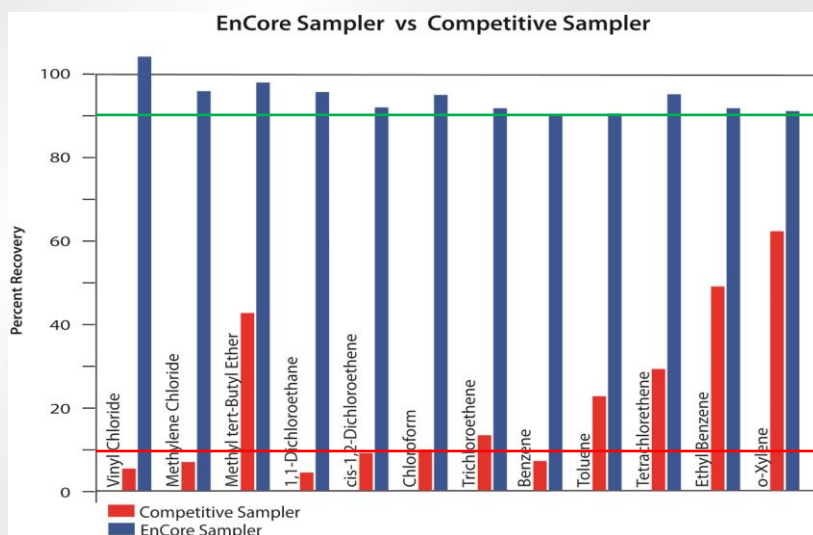
## Advantages of the En Core® Sampler



- Recognized in ASTM Standards D4547 and D6418
- The ONLY non-preserved sampler to pass ASTM specified validation method in the standard
- Allowed as an alternative to field preservation in every state that has accepted or requires Method 5035
- Often used to minimize liability – consistency of sampling despite variations in experience of field staff



## Percent recovery comparison of two non-methanol samplers



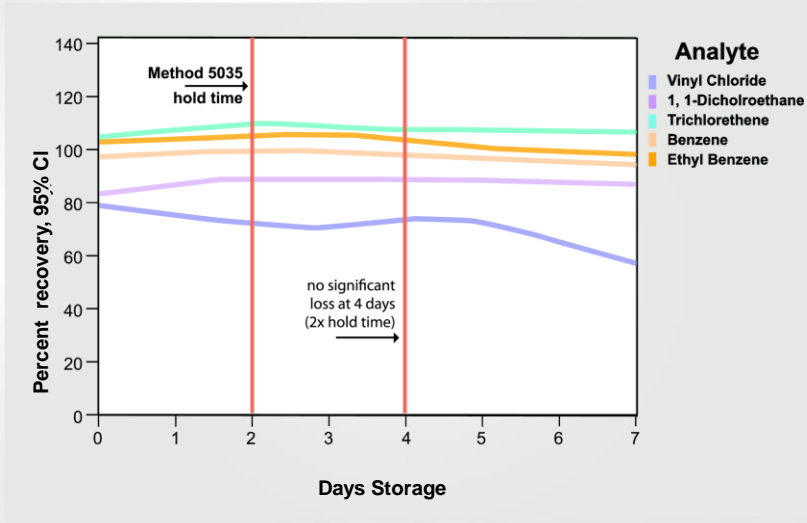
Source: En Novative Technologies, Inc. internal research; ASTM D4547 methodology

## Disadvantages of the En Core® Sampler



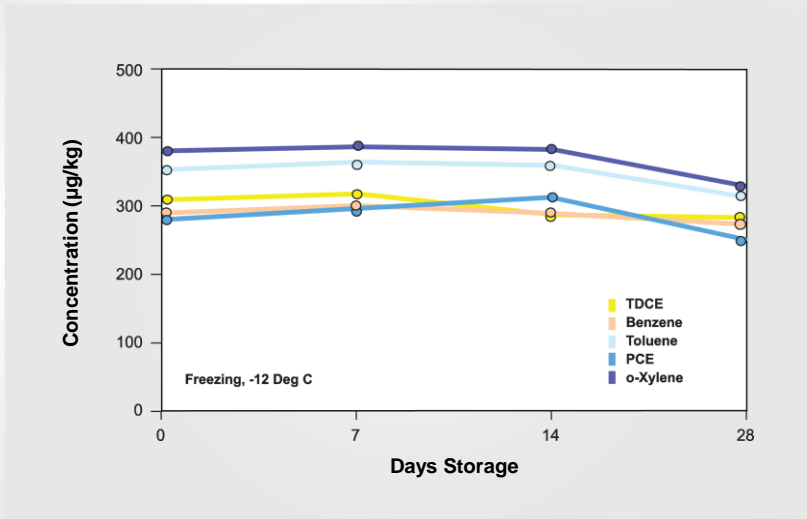
- Samples must be preserved at the lab within the **48-hour hold time** stated in Method 5035A, however, if the En Core sampler is used for other programs:
  - ASTM D4547 describe extending hold time up to 14 days by freezing samples to  $-12 \pm 5$  °C
  - ASTM D6418 describes extending hold time up to 14 days by freezing samples at  $-7$  to  $-21$  °C, or holding for 48 hours at  $4 \pm 2$  °C followed by storage at  $-7$  to  $-21$  °C for up to five days
- Samples are reported in dry weight, so a small jar of soil should be collected to measure soil moisture
- Per-sample cost is higher than field preservation
  - Some offset by time savings, especially for inexperienced sampling technicians
  - Lab may charge a sample prep fee

## En Core hold time study – samples chilled in field @ 4 °C



Source: En Novative Technologies, Inc. internal research; Dr. David Turriff

## Stability of VOCs in frozen En Core® samples



Source: En Novative Technologies, Inc. internal research; Dr. David Turriff

## En Core® sampler testing and reliability



- Quality standards are higher than for sample vials – every lot is tested and analyzed for performance and cleanliness
- Samples of each lot retained for future reference
- Tested at extreme high and low temperatures
- Samples can be stored frozen without damage
- Millions sold without one analytical incident



## For product information and to watch training videos:



En Novative Technologies  
Home of the En Core® – EPA 5035 non-methanol sampling device.

Contact Us | Call us: 1-888-211-0770

Home | Non-Methanol Sampling | Field Preservation Tools | Documents and Information | Contact Us | Multimedia

**En Core® Sampler**  
Disposable volumetric soil sampling device for minimal handling and maximum sampling accuracy

Collects, stores and delivers sample  
Eliminates need for methanol preservation  
More consistent results

**The En Core® Sampler**  
The En Core Sampler collects, stores and delivers soil samples, all within one easy-to-use device.

**The Terra Core™ Sampler**  
The Terra Core's rugged all-in-one design eliminates the need for a separate handle. No more cut-off samples!

**EasyDraw Syringe® and PowerStop Handle®**  
The Most Versatile, Disposable Soil Sampling System Available For Field Preservation

[www.ennovativetech.com](http://www.ennovativetech.com)

# Questions?



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