



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 and methods for collecting soil VOC samples
- Overview and comparison of soil sampling methods
- The En Core[®] sampler – how it works, how it compares to field preserved samples
- Collecting better field preserved samples using Terra Core[™] and EasyDraw[®] samplers
- Q&A session

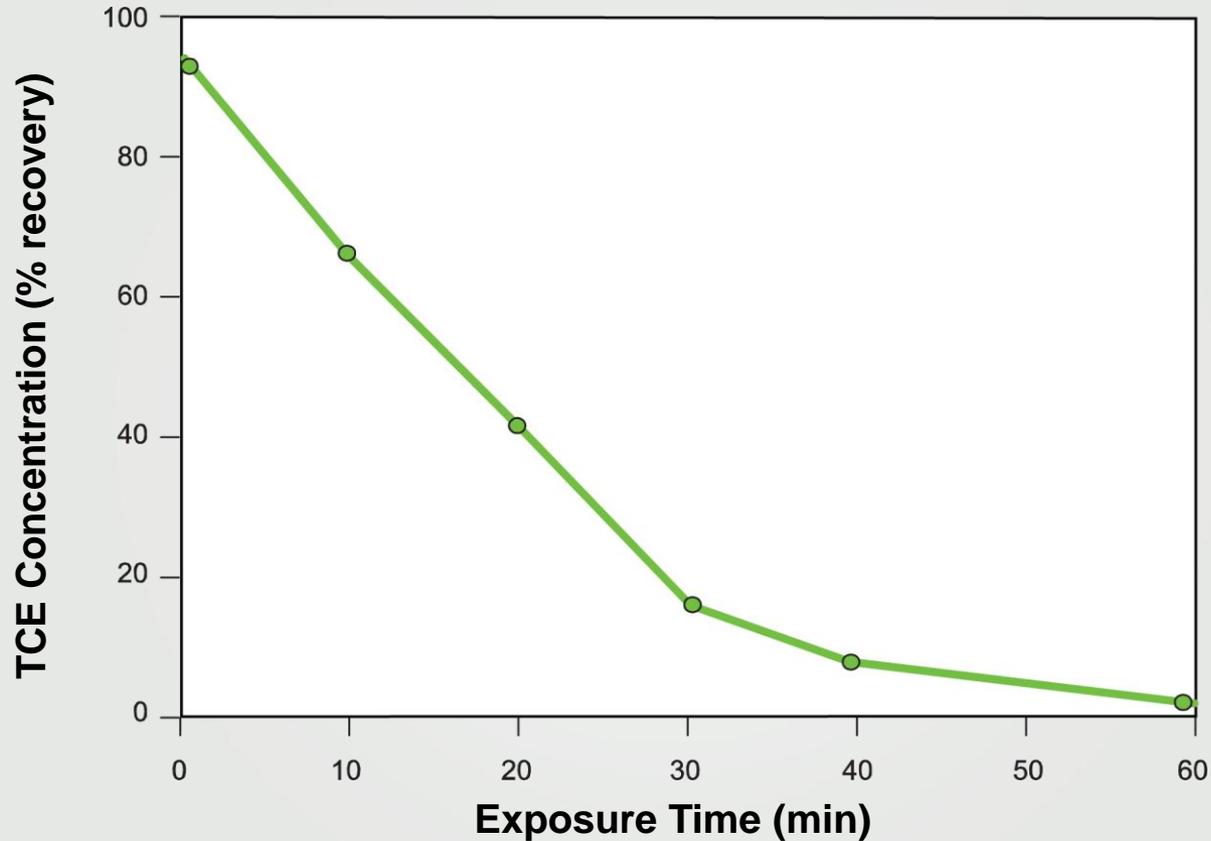
Good soil VOC data begins with good sampling practices



- 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
 - 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 doesn't maintain VOCs during sample collection, shipment and during the hold before analysis adds to the problem

Volatilization losses occur within seconds 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

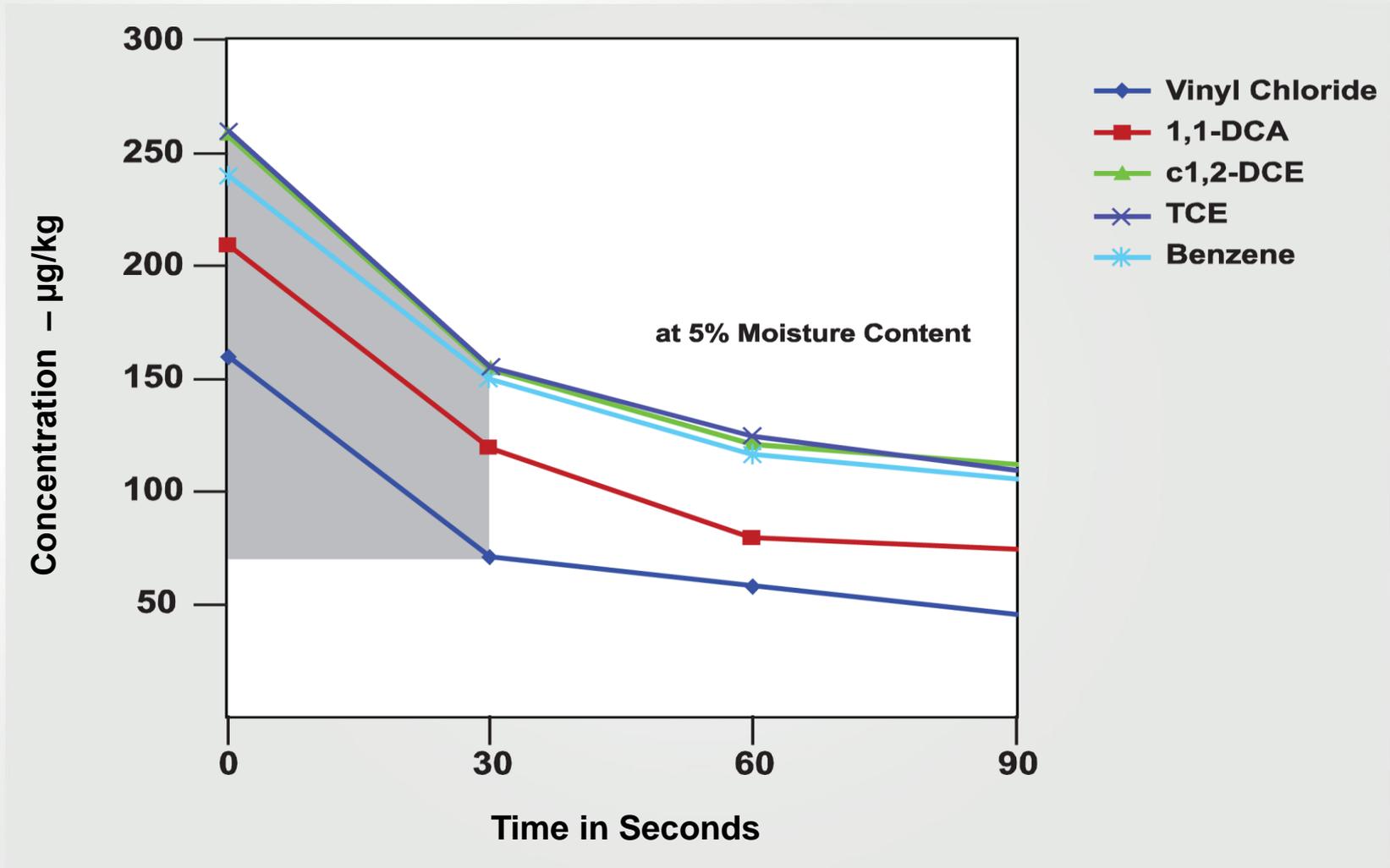
Handling of samples when a soil core liner is opened



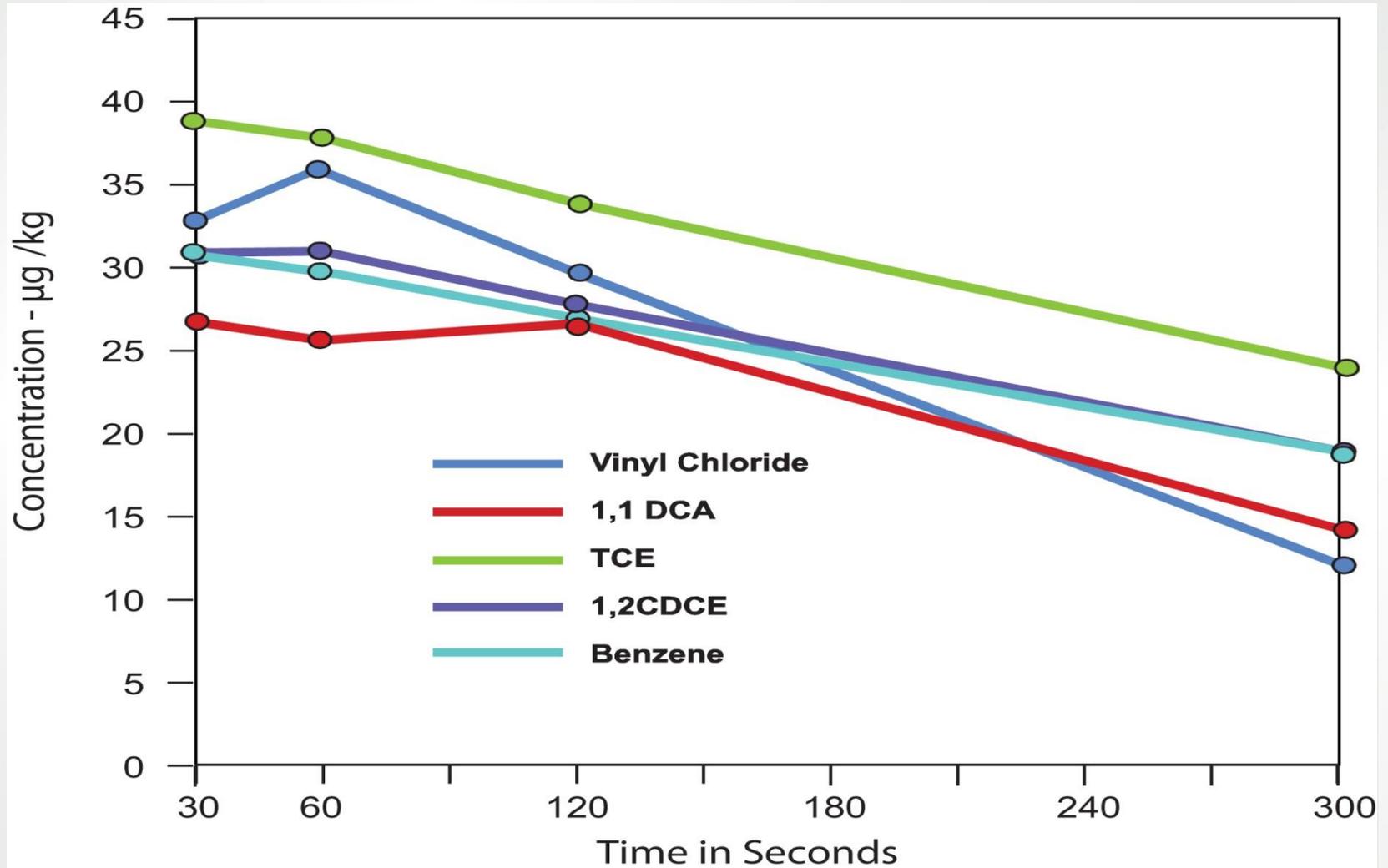
Managing sampling from a soil core can be a complex situation - stability of VOCs in the core depends on factors such as:

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 stability at 5% soil moisture content



VOC stability at 10% soil moisture content

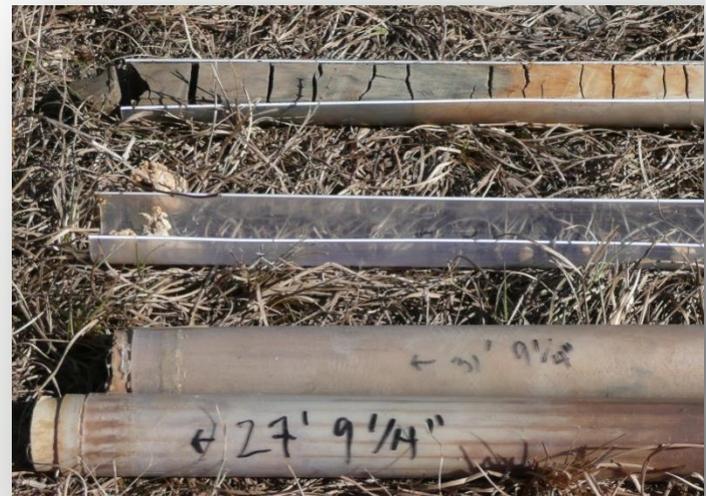


Source: En Novative Technologies, Inc. internal research; Dr. David Turriff

Best practices when sampling with acrylic soil core liners



- Cap the acrylic liner to preserve VOCs, don't 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 En Core or field-preserved vials upon opening liner
- Screen the core and select samples for analysis; USEPA SW 846 Method 3815 is the current method for screening, although its use is not mandatory



Things to avoid when sampling with acrylic soil core liners



- Leaving the liner exposed while screening
- Sampling from the container used for screening
- Taking a second boring for samples after screening the initial boring - no guarantee that 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.



A quick poll...

How long does it take you to sample from the time you open a soil core?

- a) Less than a minute
- b) 1 – 3 minutes
- c) 3 – 5 minutes
- d) 5 – 10 minutes
- e) More than 10 minutes

ASTM Standards and EPA methods for soil VOC sample collection



- 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 – En Core Sampling for VOCs in Soils
- The Nielsen Environmental Field School offers an online training module for soil sampling practices (www.envirofieldschool.com)

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

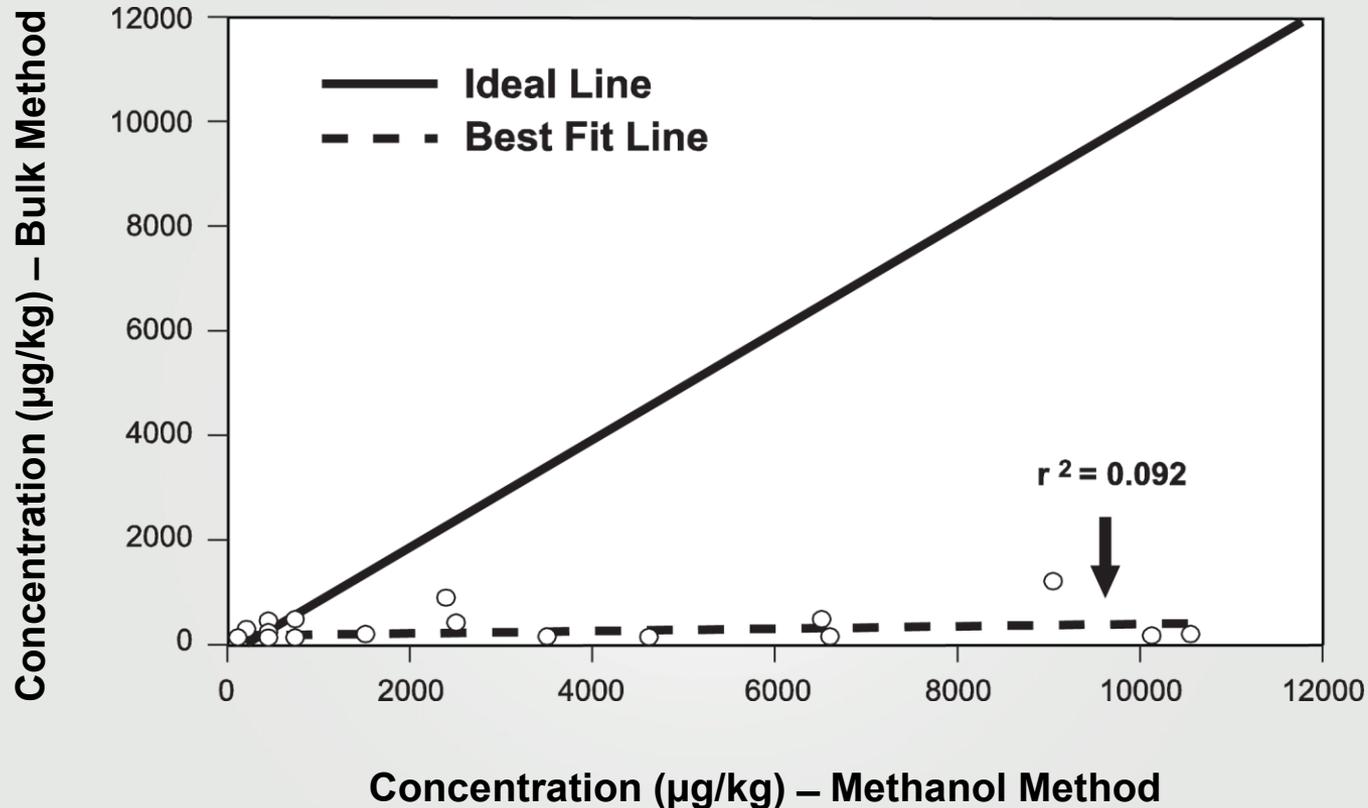


Factors that affect accuracy using bulk sampling method

- 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 5035 is not used)
- **RANGE OF ACCURACY: 0.01 - 95% true value!**

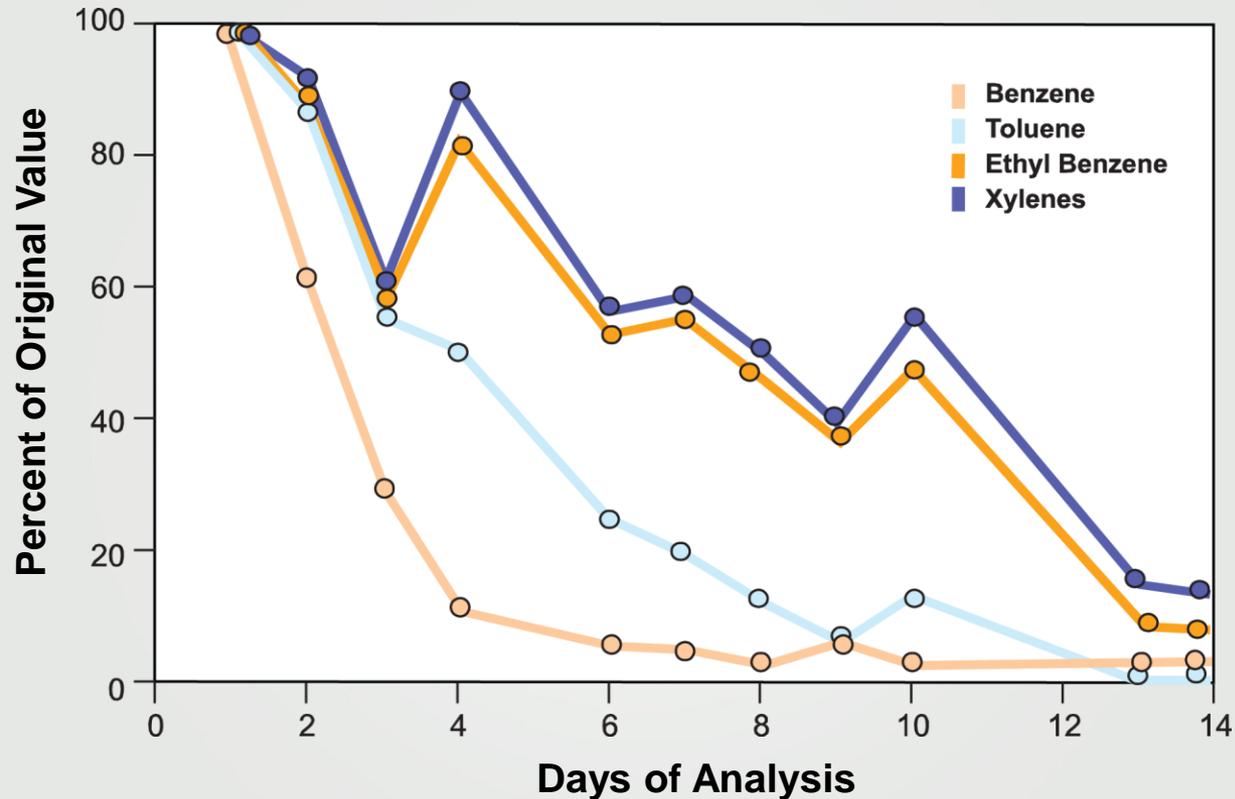


Significant bias between bulk sampling and field preservation



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 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
 - En Core sealed samplers with no preservation required



High Level and Low Level Methods in 5035



- Samples are preserved in methanol for “high level” analysis – concentration $> 200 \mu\text{g/Kg}$
- Samples are preserved in sodium bisulfate for “low level” analysis – Method Detection Limits down to $0.5 \mu\text{g/Kg}$ and concentrations $< 200 \mu\text{g/Kg}$
- 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 5035 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.

A quick poll...



Do you screen soil core concentrations before sampling?

- a) Yes, with a PID, FID or other vapor analyzer
- b) Yes, by visual inspection
- c) No, we don't screen the cores, just collect samples

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

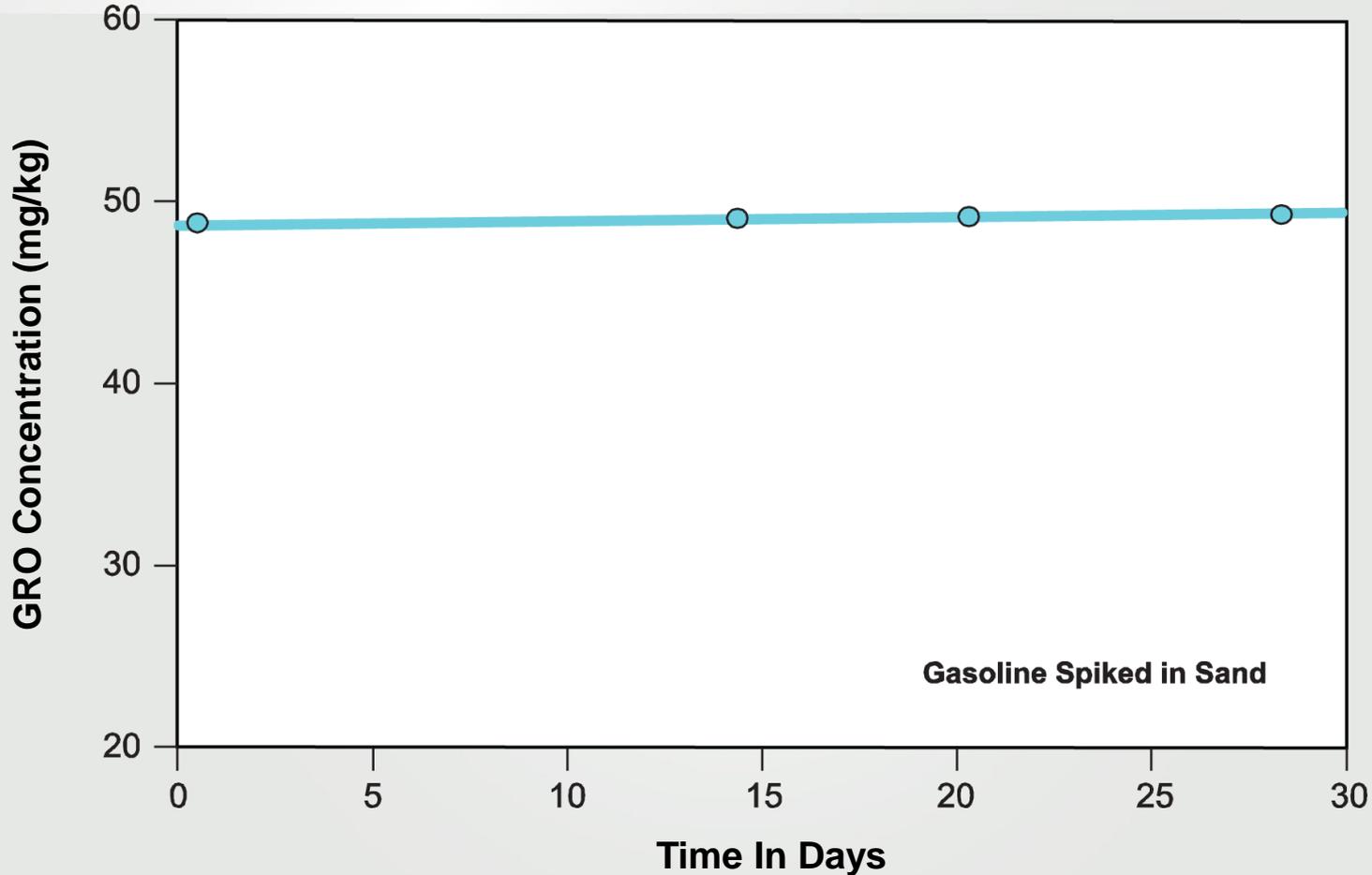


EasyDraw® Syringe



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 5035



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

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
- 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 weight is determined in lab
- Zero headspace design – minimal air trapped in the sample



**En Core[®] Sampler
and sampling handle**

The En Core[®] sampler: How it works



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En Core[®] sampler components



**Sample Collection
Handle (field)**

5g sampler

25g sampler

**Sample Extraction
Handle (lab)**

A quick poll...



What method do you use most often to sample soils for VOCs?

- a) Bulk sampling in jars, no preservation
- b) Field preserved sampling (methanol & sodium bisulfate)
- c) En Core sampling (no preservation required)

Advantages of the En Core[®] Sampler



- 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 – no expiration
- Ships in a proprietary VOC-proof bag that is also used to ship En Core samples to the laboratory



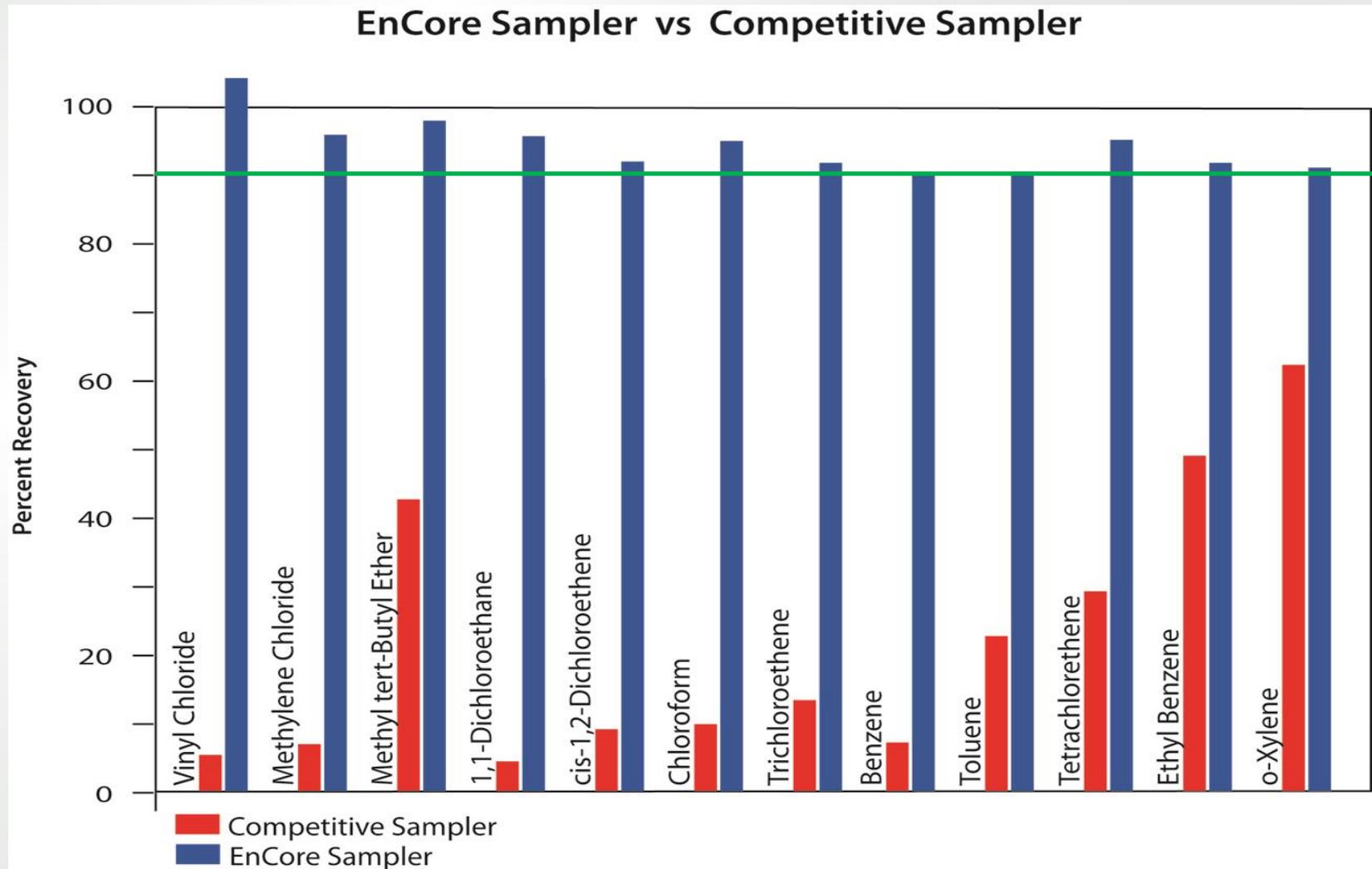
Advantages of the En Core[®] Sampler



- 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



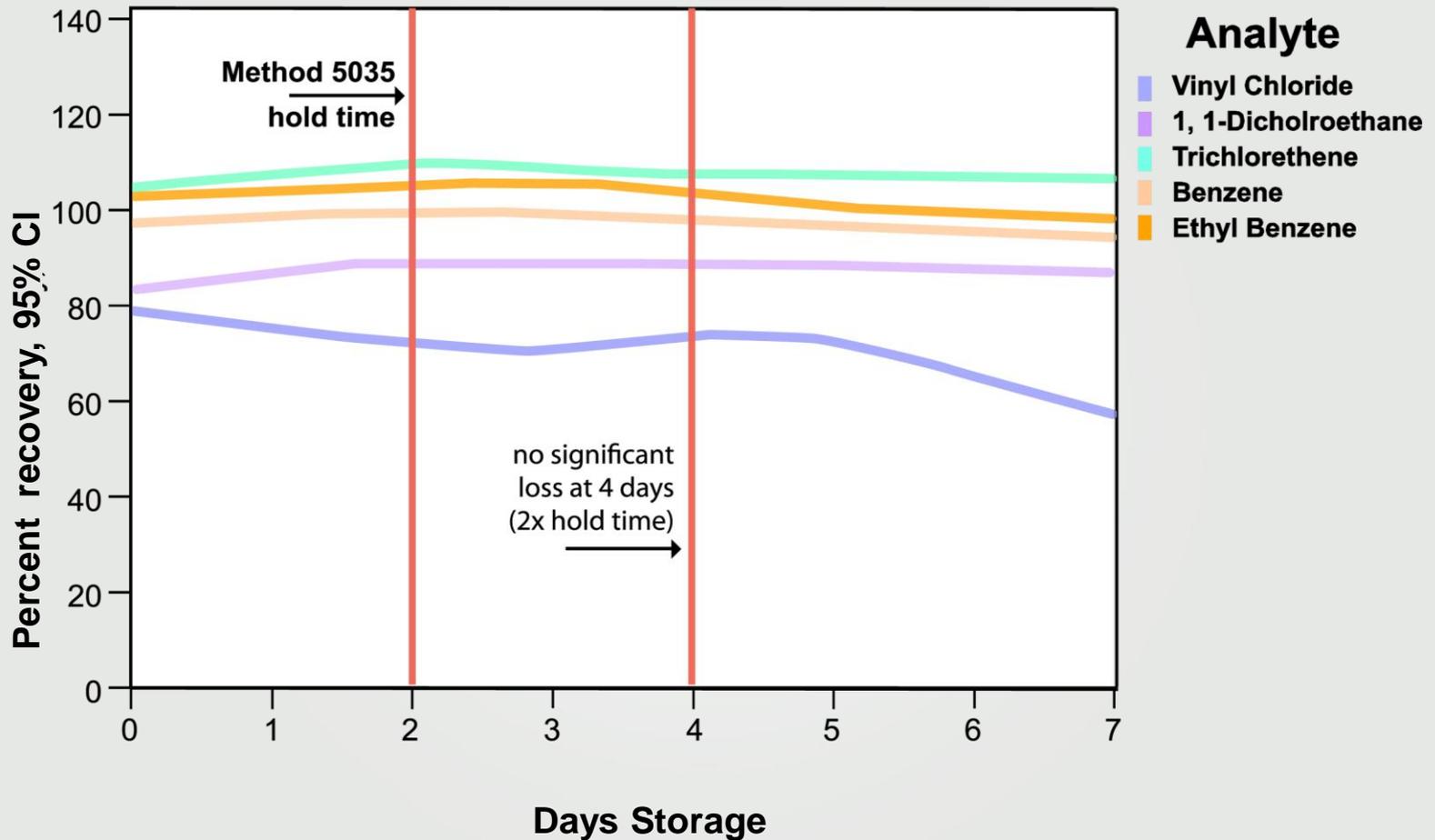
Source: En Novative Technologies, Inc. internal research; ASTM D4547 methodology

Disadvantages of the En Core[®] Sampler

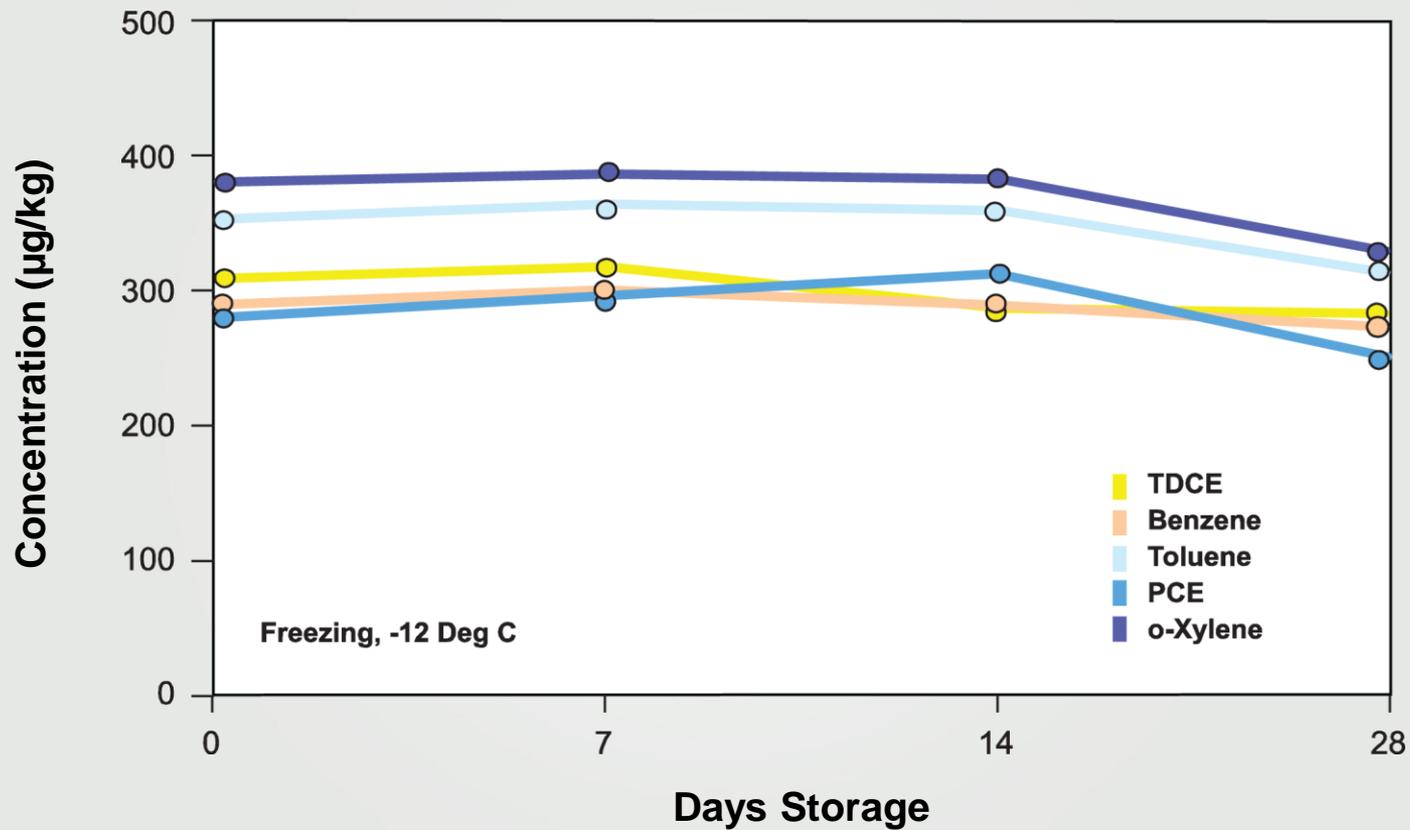


- Samples must be preserved at the lab within the **48-hour hold time** stated in Method 5035, however, if the En Core sampler is used for other programs:
 - ASTM D4547 describe extending hold time up to 14 days by freezing samples to -12 ± 5 °C
 - ASTM D6418 describes extending hold time up to 14 days by freezing samples at -7 to -21 °C, or holding for 48 hours at 4 ± 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



Stability of VOCs in frozen En Core[®] samples



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

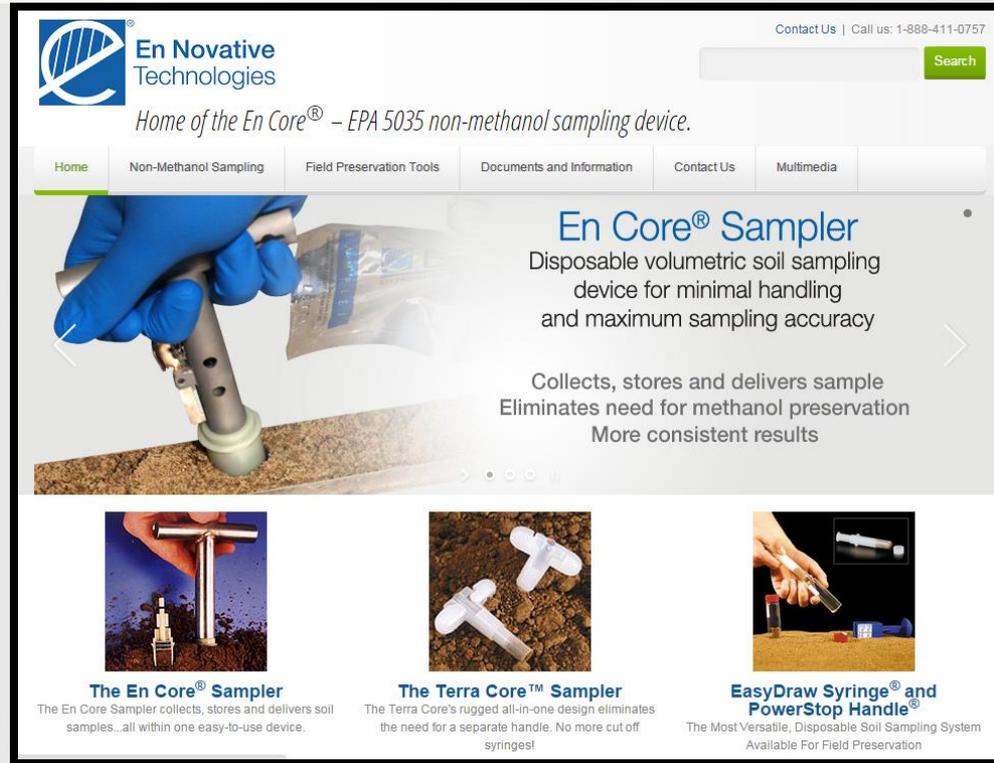
En Core[®] sampler testing and reliability



- 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:



The screenshot shows the En Novative Technologies website. At the top left is the company logo and name. To the right, it says "Contact Us | Call us: 1-888-411-0757" and has a search bar with a green "Search" button. Below the header is a navigation menu with links for "Home", "Non-Methanol Sampling", "Field Preservation Tools", "Documents and Information", "Contact Us", and "Multimedia". The main content area features a large image of a hand in a blue glove using the En Core Sampler to collect a soil sample into a clear plastic bag. To the right of this image, the text reads: "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", and "More consistent results". Below this are three smaller product images with captions: "The En Core® Sampler" (described as a device that collects, stores, and delivers soil samples), "The Terra Core™ Sampler" (described as a rugged all-in-one design that eliminates the need for a separate handle), and "EasyDraw Syringe® and PowerStop Handle®" (described as the most versatile, disposable soil sampling system available for field preservation).

www.ennovativetech.com

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Questions?



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