

ABILITY OF TWO AIR-TIGHT CONTAINERS TO PRESERVE SOIL SAMPLES  
CONTAMINATED WITH VOLATILE ORGANIC COMPOUNDS

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**Abstract**

Preservation of soil samples for the analysis of volatile organic compounds (VOCs) contamination has two broad components with respect to sample integrity: to prevent analyte degradation and to prevent analyte vapor loss. Analyte degradation in soil is predominantly microbially mediated. Therefore, techniques that effect soil sterilization, or at least inhibit the activity of soil microbes, will foster preservation of soil VOCs, especially for the aromatic VOCs. This study was conducted to evaluate the ability of two air tight soil sampling containers (Encore™ and Core N' One™) to prevent vapor loss from a soil sample contaminated with VOCs. Standard methods require that samples taken in an airtight sampler must be refrigerated ( $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ) and then transferred as soon as possible to a analytical vial or analyzed within 48 hours of collection. The results show that for the nine VOCs spiked in three different soils, the Encore™ airtight container was able to preserve the prepared samples for 48 hours without significant [ $P(T \leq t)$  one-tail,  $\alpha = 0.05$ ] analyte loss. The data for the Encore™ airtight container, when frozen, showed a significant increase [ $P(T) < 0.5$ ] in recovery for four of the nine compounds in the Day 14 samples (27 to 66 %D) and two of the nine compounds in the Day 28 samples in the Oregon soil (benzene, 60 %D and ethylbenzene, 26 %D). Significant increases in concentrations (16 %D to 155 %D) were also seen for all of the compounds for the Day 14 and 28 time periods for the Encore™ airtight container when frozen in the Charleston soil. The increases are hypothesized to be from the slower sorption of these compounds on this soil and;

therefore, the compounds may have been lost during the transfer of the sample for analysis on the Day 0. The Core N' One™ airtight container was able to preserve the prepared samples on the Oregon and Charleston soils with only a significant loss of trichloroethene on the Hayesville soil for the 48 hour holding time. Both airtight containers did show significant losses of some compound/soil combinations for holding times greater than 48 hours. Therefore, at sites where it is necessary to store a soil sample prior to analysis, either of these airtight containers will meet the EPA method sampling requirements of a 48 hour holding time limit. Longer holding time periods did result in significant analyte losses which support the EPA method sampling requirements.

### **Introduction**

Volatile organic compounds (VOCs) can exist in four phases: pure product; aqueous; sorbed; and vapor. The object of the collection and analysis of soil at contaminated sites is to account for the total amount of VOCs present in all three phases. Degradation, sorption to container surfaces and volatilization losses of VOCs during soil sample collection, storage, and handling have long been identified as the major source of negative bias in soil VOC data (Siegrist and Jenssen, 1990). Degradation of VOCs can occur by abiotic and biotic processes. Abiotic degradation reactions on mineral surfaces are reported to be extremely slow under typical environmental temperatures (Voudrias and Reinhard, 1986). Naturally occurring compounds are more readily biotically degraded in soil than are synthetic compounds (Kobayashi and Rittmann, 1982). With respect to VOCs on EPA's target compound list, the naturally occurring aromatic compounds (e.g., benzene, toluene, ethylbenzene, and xylene [BTEX]) are more easily degraded than the halogenated solvents (e.g., trichloroethylene and carbon tetrachloride). The vapor loss problem resulting from analytical transfer steps has been

potentially resolved by the SW-846 Method 5035 (USEPA, 2002), in which 1 to 5 g of soil is sealed in the analytical vial, either in the field or by extrusion from an air-tight container (e.g., the EnCore™ airtight container). Therefore, successful storage/preservation techniques must halt or slow volatilization and biological degradation of VOCs in soil.

Freezing samples to inhibit microbial degradation and retard vapor loss has been tried with mixed success. King (1993) reported that gasoline concentrations (analyzed and reported as total petroleum hydrocarbons) in soil (fine sand) remained constant for 13 days when stored in a cooler with dry ice (author estimated at -70 °C). Maskarinec et al. (1988) looked at VOC losses after freezing (-20 and -70 °C) three types of soils for up to 56 days. No specific descriptions of the soil types or composition of the soils were provided but they found essentially no loss during 56 days at -20 °C for a reference soil. A second soil, designated as “Tennessee soil”, showed a loss of some of the analytes over the 56-day storage, but data were erratic with respect to time (e.g., TCE concentrations on days 0, 3, 7, 14, 28, and 56 were 75, 81, 66, 72, 54, and 40ug/g, respectively). The third soil, “Mississippi topsoil,” exhibited a steady loss of 17 out of 19 analytes over the 28-day storage period.

In both on-site and laboratory studies, methanol has been shown to be the most effective preservative by many researchers. Perhaps the largest methanol preservation study reported to date compared 50 soil sample pairs collected from two fuel-contaminated U.S. Air Force sites in the Pacific Northwest (Liikala et al., 1996). The collocated samples were collected as (1) bulk samples sent to the laboratory for subsampling and analysis by EPA SW-846 Proposed Method 8211 and (2) preserved in methanol in the field, analyzed by EPA Method 502.2. Concentrations of benzene and toluene observed in methanol-preserved samples were one to three orders of magnitude greater than concentrations observed in bulk samples that were subsampled at the

laboratory. Lesser differences were observed for xylenes and ethylbenzene, analytes for which higher concentrations were occasionally observed by the bulk method. The authors noted that the magnitude of compound losses was related to the compound's vapor pressures. Compounds with higher vapor pressures showed higher losses than those with lower vapor pressures.

Additionally, Liikala et al. (1996) investigated the preservation of soil samples which were spiked with six chlorinated solvents at 100 and 200 ppb, immersed in 10 mL methanol, sealed in VOA vials, and stored at 4 °C for 82 days. Recoveries of all compounds after 82 days were greater than 80% with an average recovery of 84%.

Another technique used to preserve VOCs in the field is to cover and tape the ends of core liners as retrieved from a split-spoon sampler and transport these to the laboratory for subsampling. The use of a sealed core liner is believed to prevent the volatilization loss and that by opening and sampling the core only once, the losses would be comparable to vapor losses that occur during field sampling. However, Hewitt and Lukash (1996) demonstrated that soil containing VOCs cannot be sealed in core liners for transport to a laboratory without loss of analyte. They also observed losses regardless of whether Teflon<sup>®</sup> or aluminum foil was inserted before capping the cores in hopes of enhancing the sealing of the cores. Small brass cores, 3.6-cm inner diameter x 5.1-cm long, were removed from a prepared area of contaminated field soil and the ends were capped with Teflon<sup>®</sup> or aluminum. After 5 and 10 days in cold storage, the soil was sampled and analyzed. VOC concentrations were at least 90% less on day 5 than observed on day 0. Thus, according to these research findings, vapor-tight seals can not be achieved on the ends of the core-liners with current technology.

Hewitt (1997) compared the trichloroethene (TCE) concentrations of soil collected by a truncated syringe and placed into methanol with soil collected in an EnCore™ airtight container

(En Chem, Inc., Green Bay, WI), stored cold ( $4\text{ }^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ) for 2 and 7 days, and then transferred into methanol. Two early designs of the Encore™ airtight container showed TCE losses, but the third-generation EnCore™ airtight container demonstrated no measurable loss of TCE even after samples were stored for 7 days before transferring to methanol. TCE is not likely to degrade and, therefore, the study demonstrated that volatile losses during the storage and transfer were not measurable in the EnCore™ airtight container. The main disadvantage of the EnCore™ airtight container is that it has not been designed for, nor tested in, soils that contain a significant portion of gravel or rock fragments. Sample replicates in the Hewitt (1997) study were collected in a silty clay soil with few coarse fragments.

This paper presents the data from a research study evaluating the ability of two air-tight containers to preserve soil samples contaminated with VOCs.

## **Materials and Methods**

### Experimental Methods

For each of the three different soils used in this experiment, thirty five, approximately 5g samples were prepared for each of the Encore™ and Core N' One™ airtight containers. The Encore™ and Core N' One™ airtight containers are disposable soil sampling devices developed to improve the collection of soils contaminated with VOCs and retain the contaminants until the soil sample is analyzed; thereby, improving the accuracy of the sample data. The soils used in these experiments are described in Table 1. For each airtight container, the thirty five samples were divided into the following seven preservation treatments: five refrigerated ( $4\text{ }^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), one for each of the holding time periods of 0, 2, 7, 14, and 28 days and; two frozen ( $-20\text{ }^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ), one for each of the holding time periods of 14 and 28 days. Five replicates were prepared for each of the holding time periods for each of the preservation treatments. The samples for each

soil/airtight container combination were prepared on separate days. Each empty airtight container, with cap, was tared on a balance. The soil was then added and lightly packed into the container until the level of soil was even with the top of the airtight container. The exterior of the airtight container was then wiped clean with a Kim-wipe and the container's cap was installed. Once soil had been placed in all containers, one by one the tops of the containers were removed and the soil within the container was spiked below the soil surface with 1  $\mu\text{L}$  of a methanolic standard containing benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, cis-1,2-dichloroethene (DCE), 1,1,1-trichloroethene (TCA), TCE, and perchloroethene (PCE), each at 400 ng/ $\mu\text{L}$ , then the container's cap was re-installed. To minimize potential bias caused by volatilization during the addition of the VOC standard, containers were spiked in a random order. All of the containers were then transferred to the refrigerator ( $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ) or freezer ( $-20\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ) approximately 30 minutes after preparation. For each soil/airtight container/treatment combination on each of the days of analysis, five replicates were randomly chosen and analyzed. Analysis of the Day 0 samples was initiated approximately 2 hr after sample preparation was completed. In preparation for analysis, the samples were allowed to equilibrate to room temperature, then one at a time, the containers were opened and the contents were transferred to a VOA vial which was sealed and placed in the Varian Archon Purge & Trap autosampler.

### Analytical Methods

Samples were analyzed by closed-system purge-and trap introduction (SW-846 Method 5035 [USEPA, 2002]) into a gas chromatograph with mass spectrometer detector (SW-846 Method 8260C [USEPA, 1997], calibrated only for the analytes of interest). The closed-system purge-and-trap system was a Varian Archon Purge & Trap autosampler connected to a Tekmar

3000 Sample Concentrator containing a Vocarb 3000 trap. An Agilent 6890 gas chromatograph with a Agilent Model 5973 MSD was used for separation and detection of the compounds of interest in the samples. The GC system was equipped with a 60-m x 0.25-mm x 1.4-um, RTX-VMS fused silica capillary column. Agilent ChemStation<sup>®</sup> software was used to analyze the chromatography. The analytical instrumental operating conditions are specified in Table 2. Soil particle-size analyses were determined using the hydrometer method as specified in Gee and Bauder (1986). TOC determinations were performed following the high-temperature induction furnace method of Nelson and Sommers (1996).

## **Results and Discussions**

### Data Analysis Methods

The Shapiro-Wilks normality test (Origin Pro 8, Origin Lab Corporation) was used to evaluate the distribution of each day/soil/treatment set of data. For a majority of the data, no significant departure from normality was found but some of the data does not fit the normal distribution for an  $\alpha = 0.05$ . For the data shown to be normally distributed, a two sample F-test for variance [P(F<=f) one-tail, f = 0.05 (Origin Pro 8, Origin Lab Corporation)] was then used to evaluate the equality of variances of the Day 0 data versus the Day 2, 7, 14, and 28 data for each analyte/soil/treatment combination. The results of the F-test were used to determine if the T-test [P(T<=t) one-tail,  $\alpha = 0.05$ ] (Origin Pro 8, Origin Lab Corporation) to be used was for equal or unequal variances. If the F-test result were greater than or equal to 0.05 then it was assumed that the variance between the data sets was equal and if the F-test results were less than 0.05 then the variance between the data sets was assumed to unequal. Utilizing the F-test results, the appropriate T-test was applied to the data to evaluate the significance of change in concentration

for each container and treatment over time. For the data that was shown to be non-normally distributed by the Shapiro-Wilks normality test, the Mann-Whitney Test (Origin Pro 8, Origin Lab Corporation) was used to evaluate if there was a statistical difference between the data sets. The bar graphs in Figures 1 through 12 present the mean of each day's replicates with error bars of one standard error about the mean with the associated T or U value above each compounds data over time.

#### Encore™, Hayesville Soil

No significant VOC losses [defined by the Standard or Welch's T-test [ $P(T \leq t)$ ,  $\alpha = 0.05$ ] or the Mann-Whitney test [ $P(U > u)$ ,  $\alpha = 0.05$ ]] occurred when the Encore™ airtight container was stored refrigerated for the 48 hour storage period (Figure 1). The 7 day refrigerated storage period results show a statistically significant loss of benzene (25 percent difference from Day 0 [%D D<sub>0</sub>]), toluene (18 %D D<sub>0</sub>), DCE (38 %D D<sub>0</sub>), 1,1,1-TCA (24 %D D<sub>0</sub>), and TCE (27 %D D<sub>0</sub>). Statistically significant losses were seen in the data from the 14 day period for these same analytes (Figure 1). The results also show that the Encore™, when refrigerated, was capable of preventing volatilization losses for five (toluene, ethylbenzene, m,p-xylene, o-xylene, and PCE) of the nine analytes in this study for up to 28 days. When the airtight container was stored frozen, the Encore™ airtight container was able to contain all nine compounds for a period of 14 days but only six (benzene, toluene, ethylbenzene, m,p-xylene, o-xylene, and PCE) of the nine analytes were preserved without statistically significant loss for a period of 28 days (Figure 2).

Day 0, refrigerated treatment replicate data for toluene, ethyl benzene, m,p-xylene, and o-xylene had large percent relative standard deviations (%RSD) of 16, 35, 32, and 39, respectively



(Table 7). The %RSD for these compounds would affect the statistical evaluation of the data which could result in underestimating the significance between the storage period and treatment data. A statistically significant difference might have been determined for the Day 7 (refrigerated) data for ethylbenzene and m,p-xylene and the Day 28 (frozen) data for ethylbenzene, m,p-xylene, and o-xylene if the Day 0 replicates %RSD had been equal to or less than 15%.

#### Core N' One™, Hayesville Soil

For the refrigerated 48 hour storage period, the Core N' One™ airtight container was able to maintain the nine compounds without statistically significant ( $P(T \text{ or } U) < 0.05$ ) loss of VOCs, but a statistically significant increase was seen in recovery of toluene (16 %D D<sub>0</sub>), ethyl benzene (32 %D D<sub>0</sub>), m,p-xylene (21 %D D<sub>0</sub>), and o-xylene (35 %D D<sub>0</sub>) [Figure 3]. The airtight container was able to retain toluene, ethyl benzene, m,p-xylene, and o-xylene for the 7 and 14 day periods. Only m,p-xylene and o-xylene were retained with no statistically significant concentration changes after being stored refrigerated for 28 days. Freezing of the Core N' One™ airtight container resulted in preservation of ethylbenzene for 14 day period. The 28 day period data resulted in preservation of only toluene, ethylbenzene, m,p-xylene, and o-xylene without statistically significant loss of VOCs (Figure 4).

The Day 0, refrigerated treatment replicate data for m,p-xylene had an %RSD of 21 (Table 8). The large %RSD for this compound would affect the statistical evaluation of the data which could have resulted in underestimating the significance between the Day 0 data and storage period/treatment data but for four of the six storage period/treatment data sets for m,p-xylene the %RSDs were equal to or greater than the Day 0 data.

## Encore™, Oregon Soil

The Encore™ airtight container was able to preserve all of the compounds spiked into this soil for the 48 hour and 7 day storage periods when refrigerated (Figure 5). The Day 14 data had statistically significant [ $P(T \text{ or } U) < 0.05$ ] losses for ethylbenzene (36 %D D<sub>0</sub>), m,p-xylene (28 %D D<sub>0</sub>), and o-xylene (28 %D D<sub>0</sub>). All of the analytes had no statistically significant loss for the 28 day storage period. Benzene data for the Days 14 and 28 had a significant increase in concentration of 37 and 57 %D D<sub>0</sub>, respectively. The apparent increases in analyte concentration over the holding times are hypothesized to be the result of these compounds remaining in the gas phase in the early storage periods on this soil and were; therefore, lost due to volatilization during the transfer of the soil from the airtight Encore™ container to the VOA vial for analysis. With the longer holding times, the compounds might diffuse throughout the sample more evenly and away from exposed surfaces or penetrate into “microsites” in the soil and; therefore, are better retained during the transfer step. The data for the Encore™ airtight container, when frozen, showed a statistically significant increase in recovery for benzene (66 %D D<sub>0</sub>), DCE (27 %D D<sub>0</sub>), 1,1,1-TCA (30 %D D<sub>0</sub>), and TCE (28 %D D<sub>0</sub>, in the Day 14 samples (Figure 6). A statistically significant increase in concentration was also seen for benzene (60 %D D<sub>0</sub>) and PCE (31 %D D<sub>0</sub>) for the 28 day storage period. The increases are hypothesized to result from the slower sorption of these compounds on this soil and; therefore, the compounds may have been lost during the transfer of the sample for analysis on the Day 0. The increase in analyte concentration with storage time would probably not be seen in the data for soils collected from an aged contaminated site because the contaminant would have been in contact with the soil for a sufficient amount of time to be retained during sampling or transfer.

The Day 0, refrigerated treatment replicate data, %RSDs for all analytes were between 19 and 31% (Table 9). The large %RSD for these compounds in the Day 0 data would affect the statistical evaluation of the data resulting in underestimating the significance between the Day 0 data and storage period/treatment data. The only analyte data that might have shown a statistically significant difference was the Day 7 and 14 (refrigerated) for ethylbenzene, Day 28 (refrigerated) for o-xylene, and Day 28 (frozen) for ethylbenzene, if the Day 0 replicates had a %RSD of 15% or less.

#### Core N' One™, Oregon Soil

For this soil, the Core N' One™ airtight container was able to preserve all of the compounds for the 48 hour period when refrigerated without a statistically significant ( $P(T \text{ or } U) < 0.05$ ) change in concentration (Figure 7). Statistically significant increases in concentration were seen for o-xylene (50 %D  $D_0$ ), DCE (15 %D  $D_0$ ), and PCE (40 %D  $D_0$ ) for the 7 day period when refrigerated. At Day 14, data had no statistically significant losses for all nine of the analytes were identified but a statistically significant increase was seen for PCE (47 %D  $D_0$ ). The day 28 data show a statistically significant loss of DCE (17 %D  $D_0$ ), 1,1,1-TCA (38 %D  $D_0$ ), and TCE (29 %D  $D_0$ ) with a statistically significant increase in PCE (110 %D  $D_0$ ). The data for the Core N' One™ airtight container, when frozen, showed a statistically significant increase in recovery for benzene (38 %D  $D_0$ ) in the 14 day period and benzene (49 %D  $D_0$ ), toluene (36 %D  $D_0$ ), and PCE (19 %D  $D_0$ ) in the 28 day storage period samples (Figure 8). The increase is hypothesized to be from a slower sorption mechanism for these compounds and; therefore, these compounds were lost during the transfer step for analysis. The only compound to show a statistically significant loss was 1,1,1-TCA (17 %D  $D_0$ ) after 28 days (Figure 8).

The Day 0, refrigerated treatment replicate data, %RSDs for toluene, ethylbenzene, m,p-xylene, o-xylene, and TCE were greater than 15% (Table 10). The large %RSD for these compounds in the Day 0 data would affect the statistical evaluation of the data resulting in underestimating the significance between the Day 0 data and storage period/treatment data. If the Day 0 replicates %RSD had been equal to or less than 15%, a statistically significant difference might have occurred for the Day 2 (refrigerated) data for m,p-xylene, Day 7 (refrigerated) data for o-xylene, and Day 14 (frozen) data for TCE.

#### Encore™, Charleston Soil

The Encore™ airtight container showed no statistically significant ( $P(T \text{ or } U) < 0.05$ ) loss in VOC concentrations for the refrigerated or frozen treatments over all of the storage periods (Figure 9). All of the compounds showed a statistically significant increase in concentration for the 48 hour refrigerated treatment: benzene, 134 %D D<sub>0</sub>; toluene, 116 %D D<sub>0</sub>; ethylbenzene, 65 %D D<sub>0</sub>; m,p-xylene, 131 %D D<sub>0</sub>; o-xylene, 123 %D D<sub>0</sub>; DCE, 221 %D D<sub>0</sub>; 1,1,1-TCA, 224 %D D<sub>0</sub>; TCE, 134 %D D<sub>0</sub>; and PCE, 145 %D D<sub>0</sub>. A statistically significant increase in concentration was observed for m,p-xylene (17 %D D<sub>0</sub>) in the 7 day refrigerated treatment. The day 14 refrigerated treatment results had a statistically significant increase in concentration for benzene (17 %D D<sub>0</sub>), toluene (17 %D D<sub>0</sub>), m,p-xylene (17 %D D<sub>0</sub>), o-xylene (17 %D D<sub>0</sub>), DCE (17 %D D<sub>0</sub>), 1,1,1-TCA (17 %D D<sub>0</sub>), TCE (17 %D D<sub>0</sub>), and PCE (17 %D D<sub>0</sub>). For the 28 day results, a statistically significant increase in concentration was seen for all compounds with the exception of 1,1,1-TCA and TCE. The 1,1,1-TCA and TCE concentrations did show an increase but it was determined to not be statistically significant due to the large variance in the replicate concentrations for these analytes.

All of the analytes had a statistically significant increase in concentration for the 14 and 28 day frozen treatments (Figure 10). The increase seen in the recovery of some compounds may be due to a slow sorption mechanism on this soil for these compounds and their subsequent loss during the transfer step for analysis of the Day 0 replicate samples. The Day 0, refrigerated treatment replicate data, %RSDs all analytes were less than 15% (Table 11).

#### Core N' One™, Charleston Soil

The 48 hour refrigerated storage period had no statistically significant ( $P[T \text{ or } U] < 0.05$ ) loss of seven of the nine VOCs but does show a statistically significant increase in concentration for benzene (41 %D D<sub>0</sub>) and TCE (57 %D D<sub>0</sub>) [Figure 11]. In Figure 11, the column graph for toluene shows the same pattern as benzene but because of the large variance in the Day 0 data for toluene, the statistical test results for all data comparisons show no difference between each day's data. No statistically significant losses were seen in the data for the 7 day period. Statistically significant losses for the 14 day refrigerated treatment were seen for benzene, DCE, 1,1,1-TCA, and TCE. The day 28 results show statistically significant losses for 1,1,1-TCA and TCE. The only statistically significant loss of VOCs for the frozen treatments were on Day 28 for benzene, o-xylene, DCE, 1,1,1-TCA and TCE (Figure 12).

The Day 0, refrigerated treatment replicate data, %RSDs for all analytes except TCE were greater than 15% (Table 10). The large %RSD for these compounds in the Day 0 data would affect the statistical evaluation of the data resulting in underestimating the significance between the Day 0 data and storage period/treatment data. A statistically significant difference might have been determined for the Day 7 (refrigerated) data for DCE, 1,1,1-TCA, and PCE; Day 14 (refrigerated) data for toluene, ethylbenzene, m,p-xylene, o-xylene, and PCE; Day 28

(refrigerated) data for toluene, ethylbenzene, m,p-xylene, o-xylene, and PCE; Day 14 (frozen) data for o-xylene, DCE, 1,1,1-TCA, and PCE; Day 28 (frozen) data for DCE and PCE if the Day 0 replicates %RSD had been equal to or less than 15%.

### **Summary and Conclusions**

The current version of US EPA SW-846 Method 5035 (US EPA, 2002) requires that samples taken in an airtight container must be refrigerated ( $4\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ ) and then transferred to an analytical vial as soon as possible or analyzed within 48 hours of collection. This study demonstrated the ability of the Encore™ and Core N' One™ airtight containers to meet the EPA Method 5035 required 48 hour holding time for soils contaminated with VOCs where it is necessary to store the soil sample prior to analysis. In support of the EPA specified maximum holding time of 48 hours for airtight containers, some VOCs showed statistically significant losses for some soil/contaminant with either airtight container. Statistically significant increases in some compounds concentrations were seen on the Oregon and Charleston Soils with both airtight containers. The increases in analyte concentration over the holding times are hypothesized to be the result of these compounds remaining in the gas phase in the early storage periods on this soil and volatilizing during the transfer from airtight Encore™ and Core N' One™ containers to the VOA vial for analysis. These two soils have an approximate 20% higher sand content than the Hayesville soil which would result in an increase the available macro-pore space and pathways for diffusion throughout the sample. This increase in macro-pore space could provide more large void space for these compounds to remain in the gas phase in these soils resulting in less surface exposure and; therefore, decrease the sorption speed of these compounds. It is recommended that the sand content of site specific soils be taken into

account when soil sample collection techniques are determined during the planning phase at a site.

Table 1. Soil Characteristics.

Soil Number	Soil Designation	Soil Horizon	Sand (%)	Silt (%)	Clay (%)	Organic Carbon (%)
1	Hayesville	Bt2	46	22	32	0.2
2	Oregon	B	65	8	27	0.8
3	Charleston	A	61	31	8	3.8

Table 2. Analytical Instrumentation Operating Conditions.

<b>Purge-and-Trap Operating Conditions</b>	
Purge time: 10 min.	Helium purge gas flow: 40mL/min.
Desorb time: 4 min.	Purge temperature: 40 °C
Bake time: 10 min.	Desorb temperature: 250 °C
Transfer line temperature: 125 °C	Bake temperature: 260 °C
Valve temperature: 125 °C	External line temperature: 125 °C
<b>GC Operating Conditions</b>	
Split ratio: 40:1	Column flow rate: 20 cm/sec
Initial hold time: 0	Initial temperature: 35 °C
Final temperature: 150 °C	Ramp rate-1: 13 °C/min.
Final temperature: 250 °C	Ramp rate-2: 20 °C/min.
Injection port temperature: 200 °C	Final hold time: 2 min.
<b>MSD Operating Conditions</b>	
Solvent delay: 3.00 min.	Low mass: 35
High mass: 300	Plot type: total ion

Table 7. Percent Relative Standard Deviations of the Hayesville soil replicates for each time period and treatment for Encore™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	11	5	11	19	16	21	8
Toluene	16	7	7	7	17	13	8
Ethylbenzene	35	11	9	15	14	20	12
m,p-Xylene	32	13	8	18	20	19	15
o-Xylene	39	10	7	17	18	17	15
c-1,2-DCE	7	4	15	20	18	31	21
1,1,1-TCA	8	4	11	14	15	25	12
TCE	11	5	14	36	17	28	28
PCE	12	4	8	6	15	21	11

Table 8. Percent Relative Standard Deviations of the Hayesville soil replicates for each time period and treatment for Core N'One™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	6	7	18	27	37	9	30
Toluene	12	7	12	17	24	8	18
Ethylbenzene	17	15	20	22	9	25	22
m,p-Xylene	22	20	21	26	11	13	25
o-Xylene	14	23	25	18	9	12	23
c-1,2-DCE	12	16	22	39	26	15	27
1,1,1-TCA	7	9	24	37	15	11	25
TCE	5	22	32	25	37	23	46
PCE	12	8	18	29	11	12	35

Table 9. Percent Relative Standard Deviations of the Oregon soil replicates for each time period and treatment for Encore™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	19	11	6	9	9	11	7
Toluene	21	17	9	15	6	13	3
Ethylbenzene	32	18	7	8	12	15	3
m,p-Xylene	25	13	9	16	14	19	7
o-Xylene	19	22	5	10	13	22	10
c-1,2-DCE	20	8	5	13	17	15	8
1,1,1-TCA	21	9	8	7	11	13	12
TCE	26	7	10	11	9	7	9
PCE	25	11	10	15	14	9	5



Table 10. Percent Relative Standard Deviations of the Oregon soil replicates for each time period and treatment for Core N'One™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	15	6	12	41	13	11	6
Toluene	16	8	12	30	18	10	13
Ethylbenzene	25	6	11	37	20	16	14
m,p-Xylene	28	6	18	40	24	14	15
o-Xylene	24	13	14	35	19	14	13
c-1,2-DCE	7	4	9	32	20	19	8
1,1,1-TCA	7	8	12	30	10	13	8
TCE	18	11	16	43	14	11	10
PCE	11	4	8	26	19	10	7

Table 11. Percent Relative Standard Deviations of the Charleston soil replicates for each time period and treatment for Encore™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	13	3	13	19	17	11	20
Toluene	7	1	11	15	6	14	11
Ethylbenzene	1	0	5	14	5	11	11
m,p-Xylene	2	1	6	17	4	12	9
o-Xylene	12	4	8	14	3	12	12
c-1,2-DCE	9	10	15	5	20	20	20
1,1,1-TCA	8	4	21	8	31	13	22
TCE	4	5	15	18	19	10	24
PCE	6	5	14	14	10	13	17

Table 12. Percent Relative Standard Deviations of the Charleston soil replicates for each time period and treatment for Core N'One™.

<b>Treatment</b>	<b>Refrigerated</b>					<b>Frozen</b>	
<b>Day</b>	<b>0</b>	<b>2</b>	<b>7</b>	<b>14</b>	<b>28</b>	<b>14</b>	<b>28</b>
Benzene	16	9	28	33	32	27	26
Toluene	36	7	9	21	21	14	23
Ethylbenzene	36	9	7	20	20	9	20
m,p-Xylene	44	11	7	19	22	8	18
o-Xylene	25	14	6	16	20	9	23
c-1,2-DCE	35	12	25	37	46	23	22
1,1,1-TCA	33	17	26	19	15	27	13
TCE	2	9	22	21	25	20	33
PCE	36	8	10	17	11	22	32

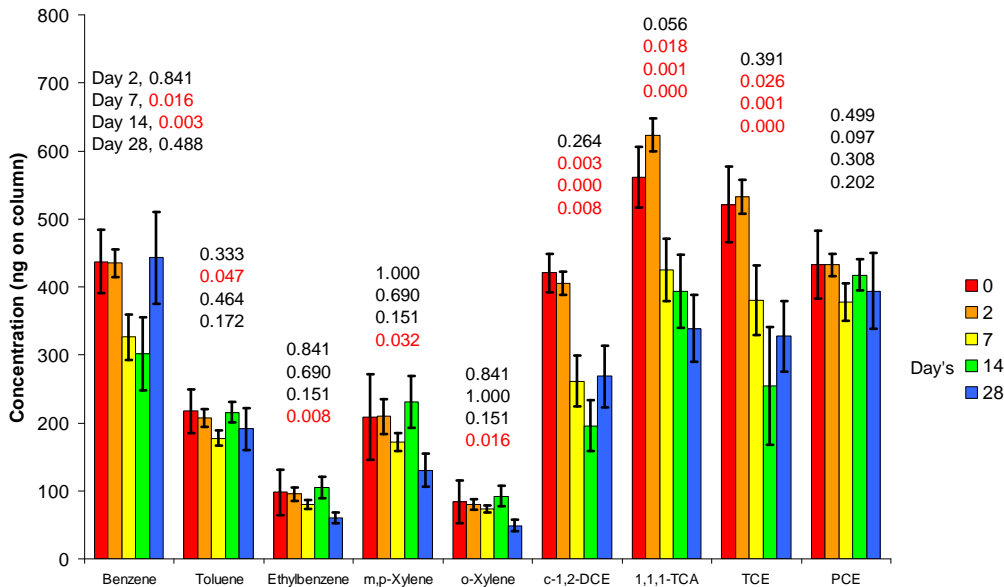


Figure 1. Hayesville Soil – Encore™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

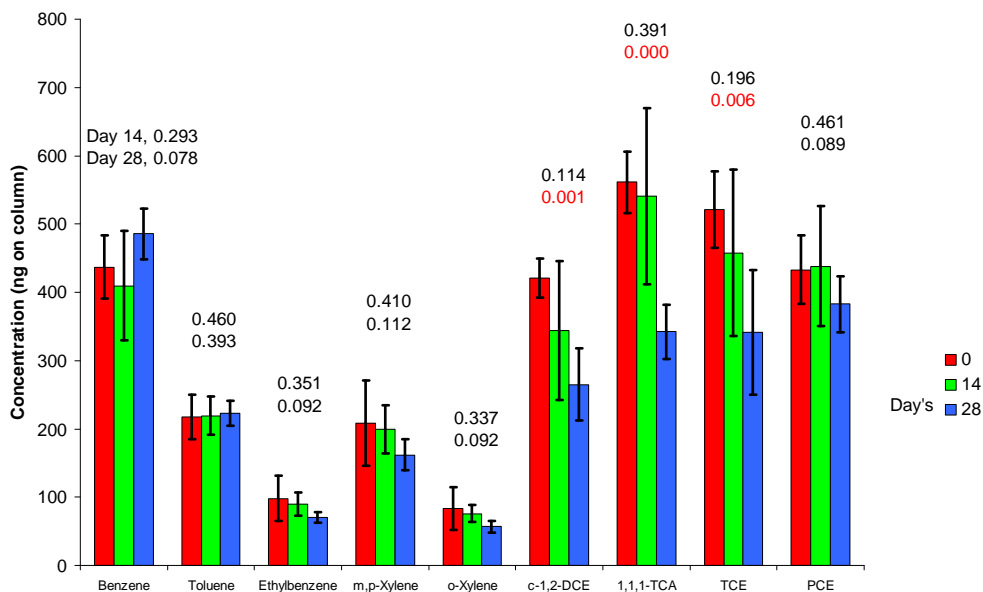


Figure 2. Hayesville Soil – Encore™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

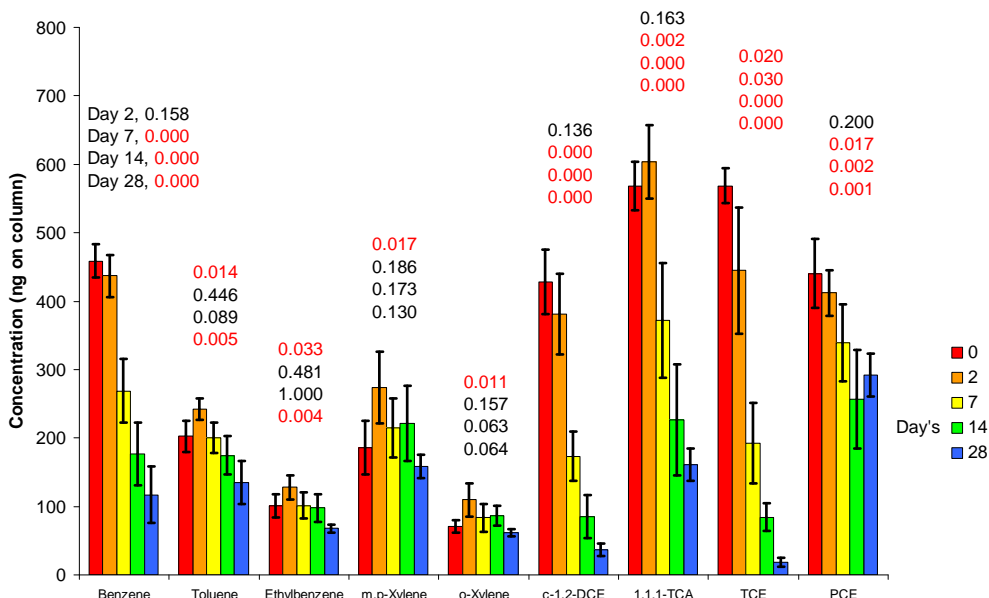


Figure 3. Hayesville Soil – Core N' One™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

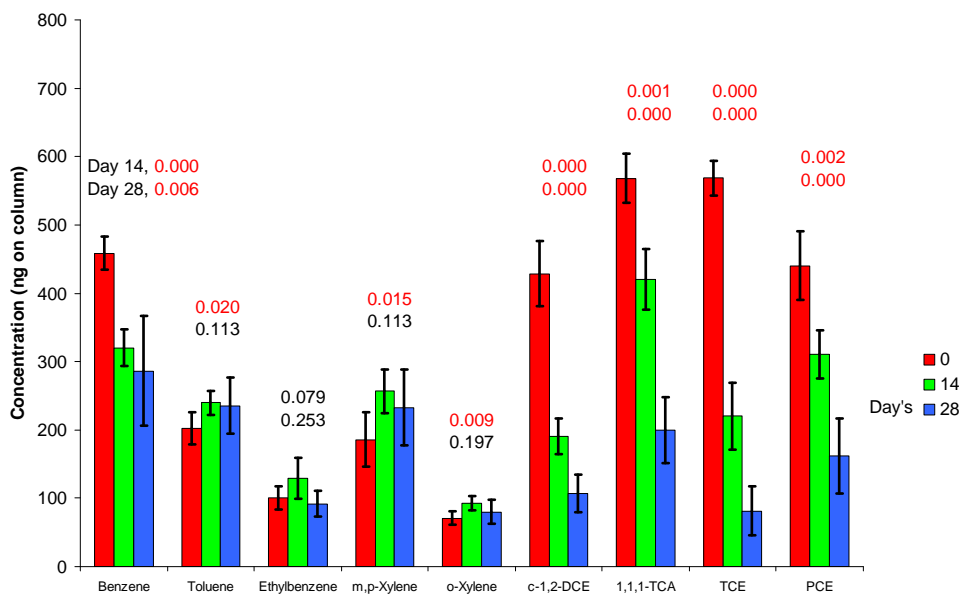


Figure 4. Hayesville Soil – Core N' One™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

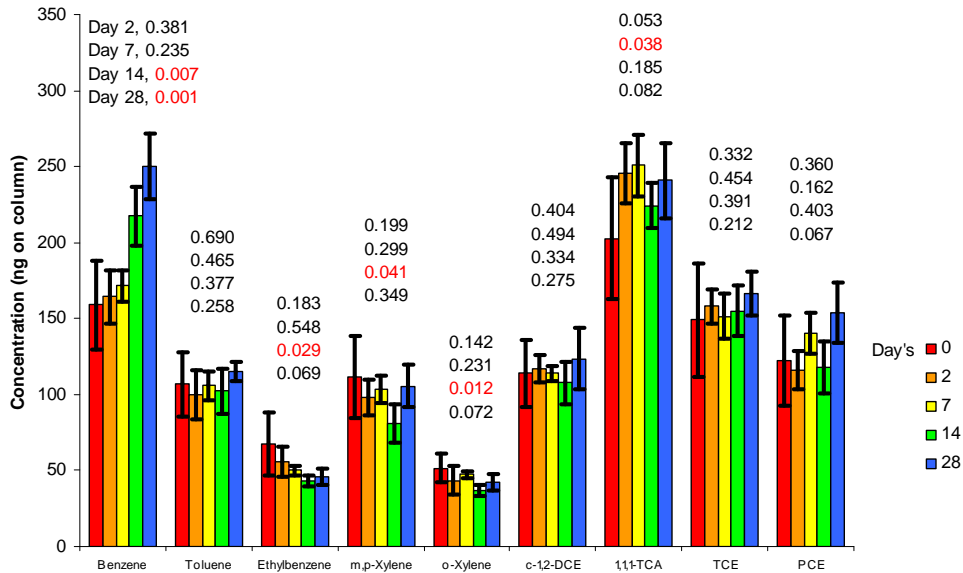


Figure 5. Oregon Soil – Encore™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

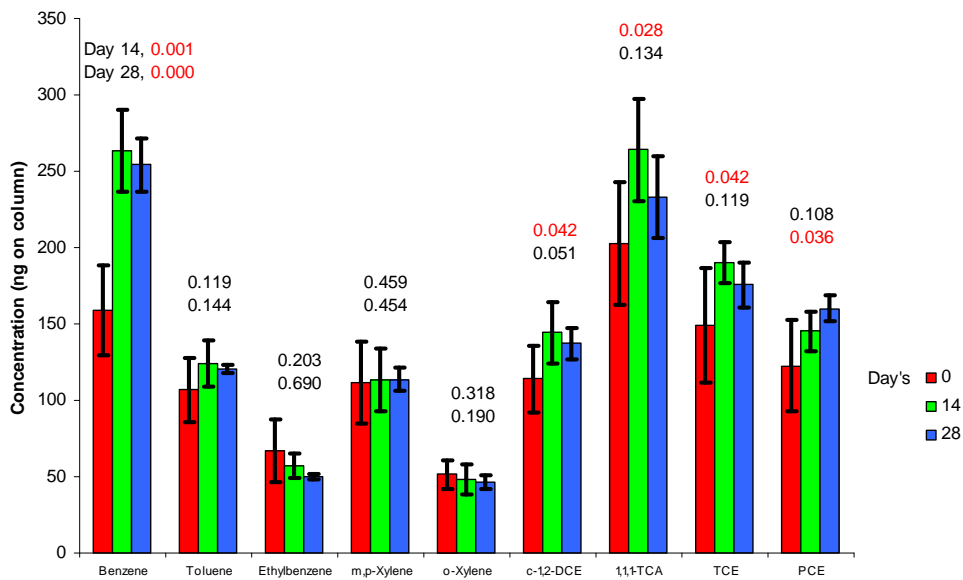


Figure 6. Oregon Soil – Encore™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

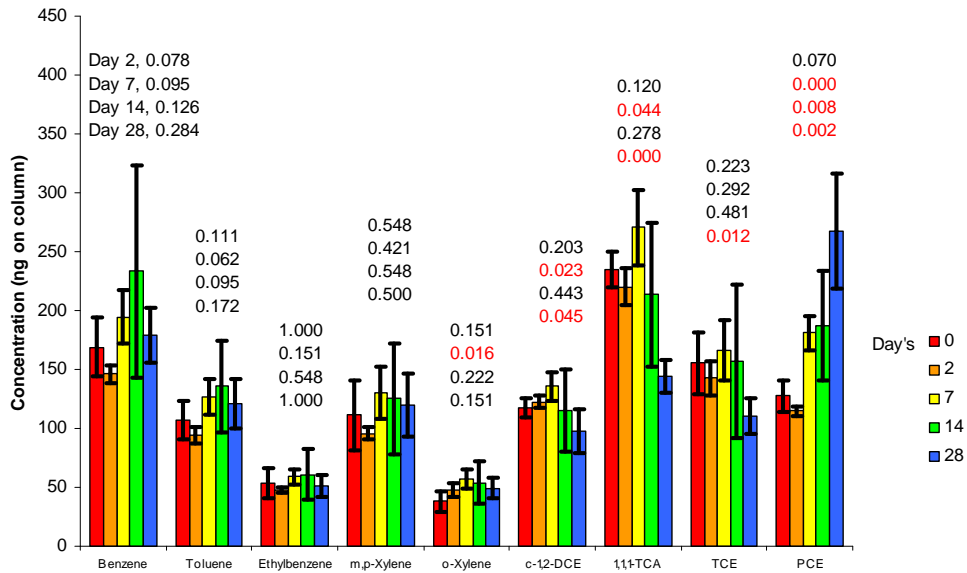


Figure 7. Oregon Soil – Core N' One™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

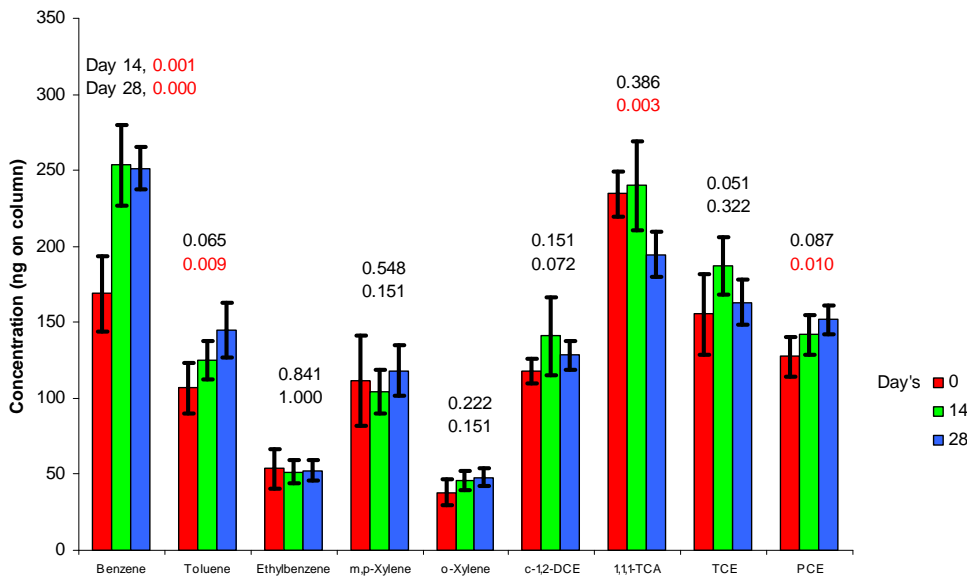


Figure 8. Oregon Soil – Core N' One™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

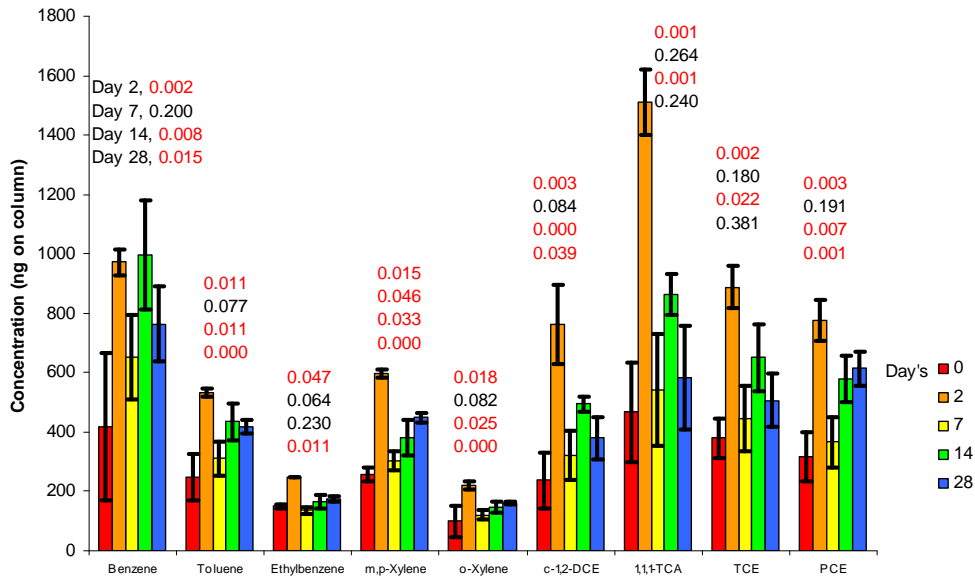


Figure 9. Charleston Soil – Encore™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

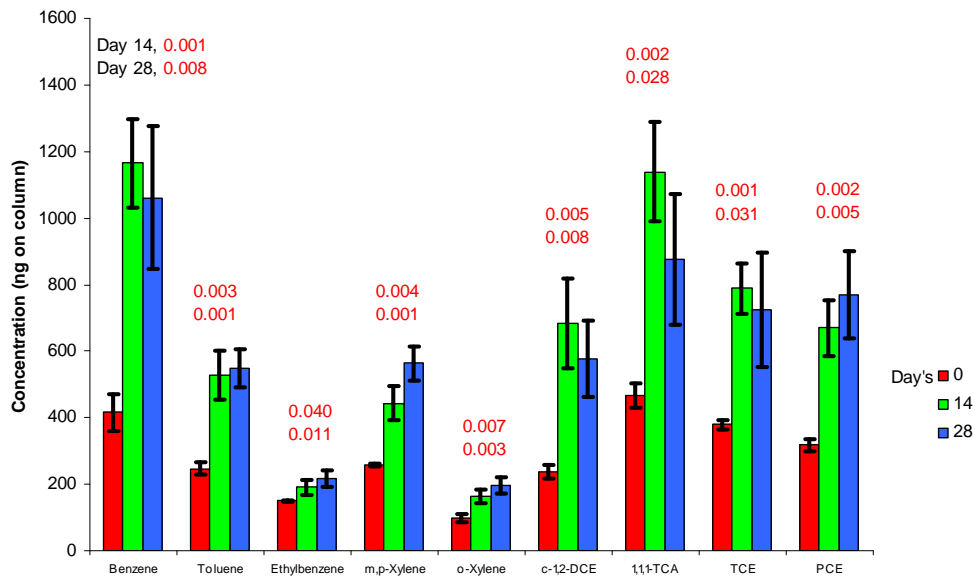


Figure 10. Charleston Soil – Encore™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

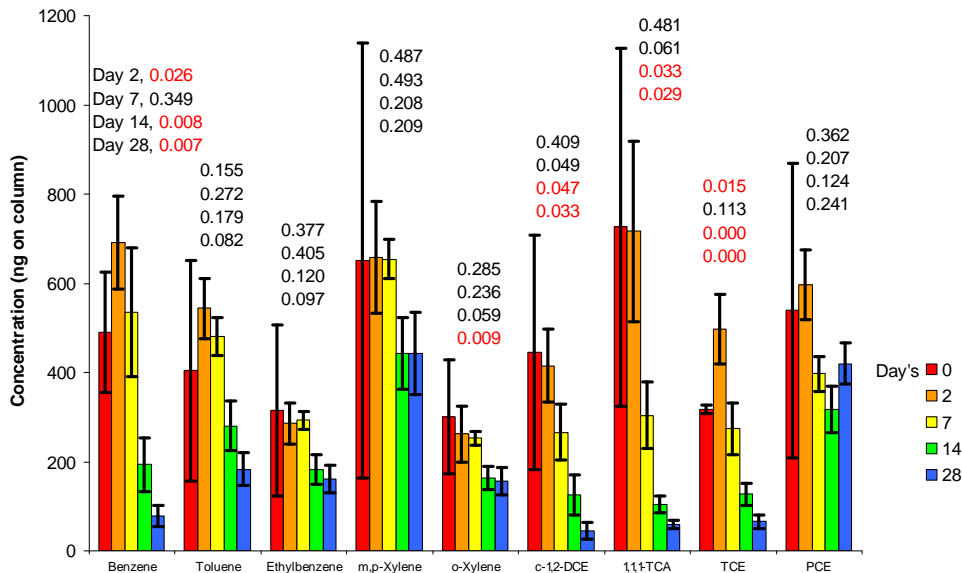


Figure 11. Charleston Soil – Core N' One™ Refrigerated Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 2 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

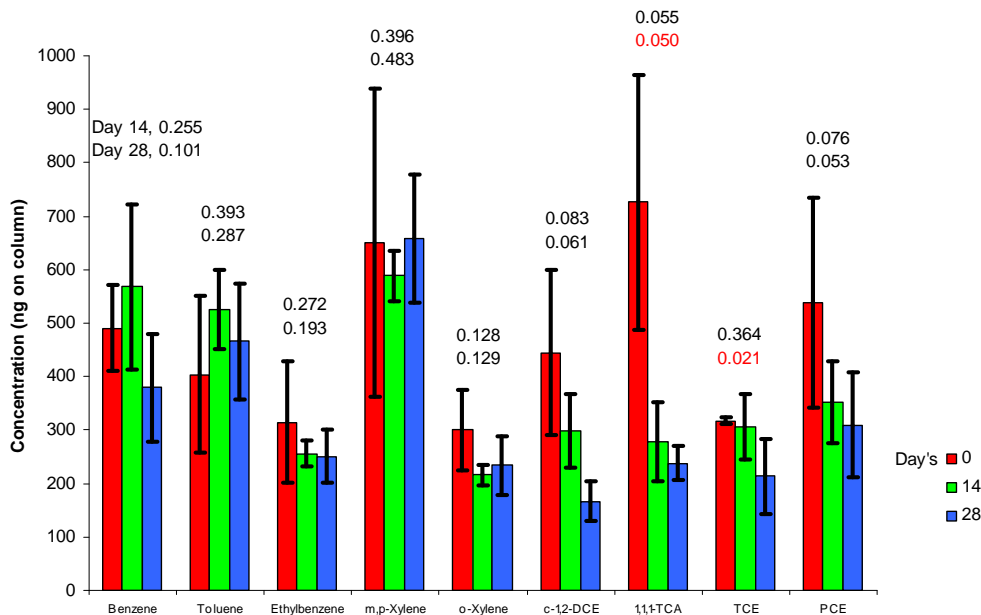


Figure 12. Charleston Soil – Core N' One™ Frozen Results. Each set of columns represent the mean concentration (ng on column) of each compound, for each of the day treatments. The data are sequentially ordered from Day 0 on the left to Day 28 on the right. The errors bars express the standard error about each mean. The values presented above each set of columns are the T-test results, expressed as probability, for the Day 0 versus that Days data. The data are sequentially ordered from Day 14 at the top to Day 28 at the bottom. Values in red are those that show a significant difference from Day 0.

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