Designation: D 6418 – 99$^{\epsilon 1}$

Standard Practice for
Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis[1]

This standard is issued under the fixed designation D 6418; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—The title was corrected editorially in August 1999.

1. Scope

1.1 This practice is intended for application to soils that may contain volatile waste constituents.

1.2 This practice provides a procedure for using the disposable En Core[2] sampler to collect and store a soil sample for volatile organic analysis.

1.3 It is recommended that this standard be used in conjunction with Practice D 4547 and Guide D 4687.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Note 1—ASTM takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

2. Referenced Documents

2.1 *ASTM Standards:*

D 2777 Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water[3]

D 4547 Practice for Sampling Waste and Soils for Volatile Organics[4]

D 4687 Guide for General Planning of Waste Sampling[4]

D 5792 Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives[4]

3. Summary of Practice

3.1 This practice provides a procedure for using the disposable En Core sampler to collect and store a soil sample of approximately 5 or 25 g for volatile organic analysis. The En Core sampler, which consists of a coring body/storage chamber, O-ring sealed plunger, and O-ring sealed cap, is designed to collect and hold a soil sample during shipment to the laboratory for analysis.

3.2 After the sample is collected in the En Core sampler, the coring body/storage chamber is sealed with a slide-on cap and immediately becomes a sample storage chamber. To minimize loss of volatile compounds due to volatilization, or biodegradation, or both, from the time of collection until analysis or preservation in the laboratory (see Practice D 4547), sample storage in the device currently is specified at 4 ±2°C for up to 48 h. For cases where holding times beyond 48 h are necessary, storage of samples in the En Core sampler at 4 ±2°C or –12 ± 2°C for longer than 48 h is an option, provided it can be shown that the longer storage time used does not influence the concentrations of the volatile organic compounds (VOCs) of interest in the samples, or that the data generated by analysis of the samples meet the data quality objectives (DQOs), (see Practice D 5792). This practice does not use methanol preservation or other chemical preservatives in the field.

4. Significance and Use

4.1 This practice is for use in collecting and storing 5- or 25-g soil samples for volatile organic analysis in a manner that minimizes loss of contaminants due to volatilization, or biodegradation, or both. The En Core sampler serves as both the sampling device and sample storage chamber.

4.2 In performing the practice, the integrity of the soil sample structure is maintained during sample collection, storage, and transfer in the laboratory for analysis or preservation.

4.3 During sample collection, storage, and transfer, there is very limited exposure of the sample to the atmosphere.

4.4 Laboratory subsampling is not required when performing this practice. The sample is expelled directly from the coring body/storage chamber into the appropriate container for analysis or preservation without disrupting the integrity of the sample. Subsampling from the En Core device should not be performed to obtain smaller sample sizes for analysis.

4.5 This practice specifies sample storage in the En Core sampler at 4 ±2°C for up to 48 h. For cases where holding

---

[1] This practice is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.03 on Sampling Equipment.

Current edition approved June 10, 1999. Published August 1999.

[2] En Core is a registered trademark of En Novative Technologies, Inc., Green Bay, WI. The En Core sampler is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.04.

times beyond 48 h are necessary, storage of samples in the En Core sampler at 4 ±2°C or –12 ±2°C for longer than 48 h is an option, provided it can be shown that the longer storage time used does not influence the concentrations of the VOCs of interest in the samples, or that the data generated by analysis of the samples meet the DQOs (see Practice D 5792).

4.6 This practice does not use methanol preservation or other chemical preservatives in the field. As a result, there are no problems associated with flammability hazards, shipping restrictions, or dilution of samples containing low volatile concentrations due to solvents being added to samples in the field.

4.7 The En Core sampler is a single-use device. It should not be cleaned, or reused, or both.

4.8 This practice cannot be used for sampling cemented material, consolidated material, or material having fragments coarse enough to interfere with proper coring techniques.

5. Apparatus

5.1 *En Core Sampler*—The En Core sampler has three components: the coring body/storage chamber, which is volumetrically designed to collect and store a soil sample of approximately 5 or 25 g; an O-ring sealed plunger for nondisruptive extrusion of the sample into an appropriate container for analysis or preservation; and, a slide-on cap having an O-ring seal and locking arm mechanism (see Fig. 1). The coring body/storage chamber of the 5-g sampler has a volume of 3 cm^3 to give a sample size of approximately 5 g, assuming a soil density of 1.7 g/cm^3. The coring body/storage chamber of the 25-g sampler has a volume of 14.5 cm^3 to give a sample size of approximately 25 g, assuming a soil density of 1.7 g/cm^3. The seals of the sampler are provided by three polytetrafluoroethylene-coated fluoroelastomer O-rings (see Fig. 1). The coring body/storage chamber, plunger, and cap of the En Core sampler are constructed of an inert composite polymer. The En Core sampler is certified as clean and should not be reused. There are two stainless steel reusable attachments that are used with the En Core sampler. These are a T-handle, which is used to push the sampler into the soil for sample collection; and an extrusion tool, which attaches to the plunger for extrusion of the sample from the coring body/storage chamber (see Fig. 2). Each En Core sampler is supplied with a protective moisture-proof bag for shipment to the laboratory.

5.2 *Minimum/Maximum Thermometer*—This is any thermometer that registers the minimum and maximum temperatures reached during any given period of time in °C and has a range that includes the specified storage temperature in divisions of 1°C.

6. Procedure

6.1 The size of the En Core sampler used is determined by the size of the sample required by the laboratory procedure that will be used to analyze the sample. If a sample size of approximately 5 g is required for analysis, the 5-g En Core sampler, and not the 25-g device, should be used to collect and

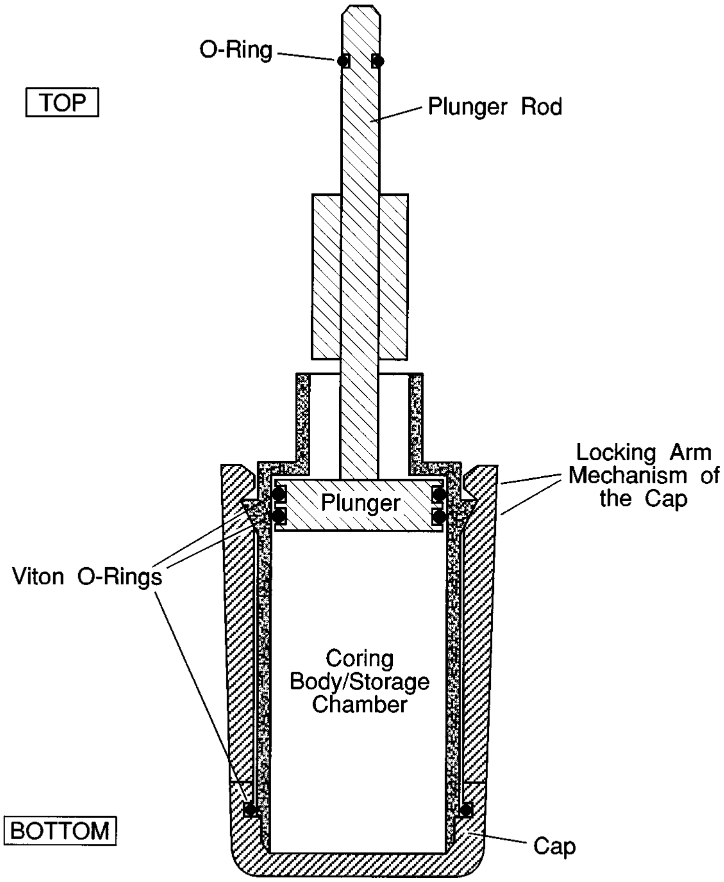

FIG. 1 Components of the En Core Sampler

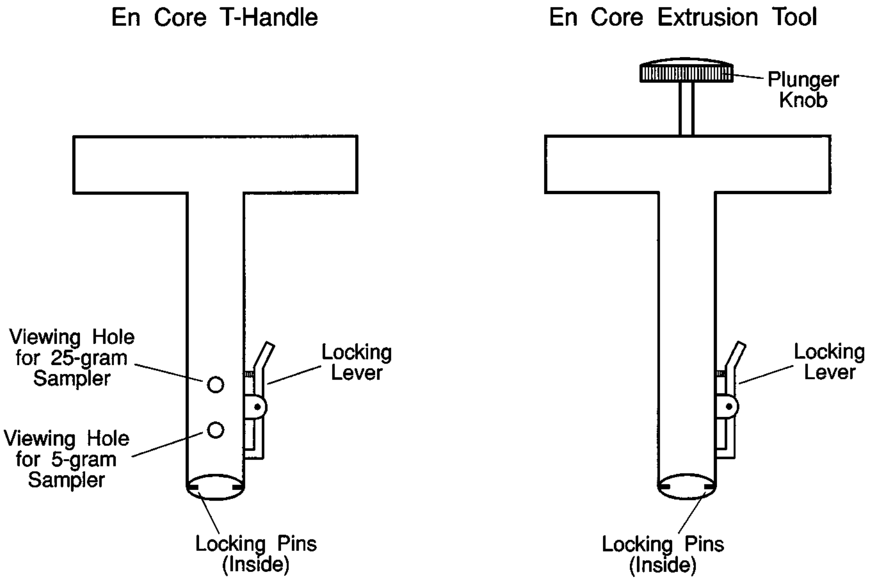

FIG. 2 Reusable Attachments to the En Core Sampler

store the sample. Subsampling from the En Core devices should not be performed to obtain smaller sample sizes for analysis.

6.2 If volatile contaminant levels in the soil being sampled are not known, it is recommended that three samples be collected at each sampling location using three En Core samplers. If determination of moisture content is required for reporting sample results on a dry weight basis, a fourth sample should be collected from each sampling location using a suitable container. The proper sample preparation method for low-level or high-level volatile analysis[5,6] can be determined by screening one of the three samples collected in the En Core samplers for high or low concentrations of VOCs. If a high concentration is present, one of the remaining samples in the En Core devices can be extruded into methanol for high-level analysis; if a low concentration is present, the two remaining samples in the En Core devices can be extruded into two appropriate vials for low-level analysis giving an additional low-level sample for analysis, if needed. For guidance on sample collection, sample handling, and sample preparation methods for volatile organic analysis, see Practice D 4547. For quality assurance considerations related to field sampling, see Guide D 4687.

6.3 As stated in 4.8, the En Core device cannot be used for sampling cemented material, consolidated material, or material having fragments coarse enough to interfere with proper coring techniques.

6.4 *Sample Collection*—The manufacturer's specific instructions for operating the En Core sampler and T-handle to collect a soil sample should be followed. The steps involved in sample collection using the En Core sampler are given below.

6.4.1 Before collecting a sample in the En Core sampler, the plunger rod (see Fig. 1) should be positioned so that the plunger can be moved freely from the top to the bottom of the coring body/storage chamber. For sample collection, the T-handle (see Fig. 2) should be attached to the sampler, and the plunger should be positioned so that the bottom of the plunger is flush with the bottom of the coring body/storage chamber. This prevents air from being trapped in the device during sample collection. When inserting a coring device into a surface for sample collection, air should not be trapped behind the sample. If this occurs, air can pass through the soil causing VOCs to be lost, or it can cause the sample to be prematurely pushed from the coring device.

6.4.2 Sample collection should be performed as quickly as possible. Using the T-handle, push the En Core sampler into the soil until the coring body/storage chamber is completely full. It can be verified that the coring body/storage chamber is full by looking into the appropriate viewing hole (either 5 or 25-g) in the T-handle (see Fig. 2). The coring body/storage chamber is completely full if the small O-ring on the plunger rod (see Fig. 1) is centered in the T-handle viewing hole. If possible, look at the viewing hole while the device is in the soil to check if the coring body/storage chamber is completely full. If it is not possible to view the hole when the device is in the soil, remove the sampler from the soil to view the hole. If the coring body/storage chamber is not completely full, quickly insert the device back into the same sampling spot and push harder to fill the coring body/storage chamber. When the coring body/storage chamber is completely full, scrape a spatula across the bottom of the coring body/storage chamber so the surface of the soil in the sampler is flush with the opening of the coring body/storage chamber (see Note 2). Quickly wipe the external surface of the coring body/storage chamber with a clean tissue or cloth. After ensuring that the sealing surfaces are clean, cap the coring body/storage chamber while it is still on the T-handle. This is done by gently sliding the cap onto the coring body/storage chamber with a twisting motion. The cap is

---

[5] U.S. EPA, 1996, Method 5035: Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol 1B, Final Update III.

[6] U.S. EPA, 1996, Method 5021: Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol 1B, Final Update III.

locked into position when the grooves on the locking arms seat over the ridge on the coring body/storage chamber (see Fig. 1). If the cap of the En Core sampler is difficult to lock into position, the O-ring in the cap (see Fig. 1) may be bent out of position, and a new cap should be used to seal the device. A bent O-ring in the cap may result in loss of VOCs from the stored sample (see X1.1.8.2 and X1.1.9.2). After the cap is locked into position, the T-handle is removed from the sampler, and the plunger is locked into position by rotating the plunger rod (see manufacturer's specific operating instructions).

NOTE 2—For drier soils that are difficult to compact in the sampler, scraping a spatula across the surface of the soil to make it flush with the opening of the coring body/storage chamber may loosen particles of the soil in the coring body/storage chamber. These particles may scatter when the device is capped and may affect the seal between the cap and coring body/storage chamber (see X1.1.9.3). Caution should be used when working with these types of soils to prevent scattering from happening.

6.4.3 A sample label showing the sample number is attached to the cap of the En Core sampler, and the sampler is placed in its protective moisture-proof shipping bag (provided with each En Core sampler). This bag has a label attached for recording sample name, date collected, comments, and sample number.

6.5 *Sample Storage*—The capped coring body immediately becomes a sealed sample storage chamber. A sample can be stored in the sealed coring body/storage chamber at 4 ±2°C for up to 48 h. For cases where holding times beyond 48 h are necessary, storage of samples in the En Core sampler at 4 ±2°C or –12 ±2°C for longer than 48 h is an option, provided it can be shown that the longer storage time used does not influence the concentrations of the VOCs of interest in the samples, or that the data generated by analysis of the samples meet the DQOs (see Practice D 5792). Data showing performance of the En Core sampler as a storage chamber for samples of three different soil types stored at 4 ±2°C for 48 h; 4 ±2°C for four days; 4 ±2°C for 48 h followed by storage for 5 days at –12± 2°C; and, –12 ±2°C for seven days are given in Appendix X1.

6.5.1 Storage of samples at 4 ±2°C can be achieved by placing the protective shipping bag, in which the En Core sampler has been placed, in a cooler with ice, cold packs, or in a refrigerated compartment regulated at 4 ±2°C. If the optional storage temperature of –12± 2°C is used as discussed in 6.5, the En Core sampler should be stored in a freezer compartment regulated at –12 ±2°C. A minimum/maximum thermometer should be placed with the samples during storage to verify that the temperature requirement is maintained.

6.6 Arrangements with the receiving laboratory for sample log in, sample handling, required storage conditions, and analysis should be made.

6.7 *Extrusion of the Soil Sample from the En Core Sampler*—The manufacturer's specific instructions for operating the En Core sampler and extrusion tool to extrude the soil sample from the coring body/storage chamber should be followed. The steps involved in sample extrusion from the En Core sampler are outlined in 6.7.1.

6.7.1 At the laboratory, the soil is transferred into the appropriate container without disturbing the integrity of the sample by removing the cap from the coring body/storage chamber and using the plunger to expel the soil into the receiving container (see Note 3). This is done by performing the following steps according to the manufacturer's specific operating instructions: attach the extrusion tool (see Fig. 2) to the En Core sampler; rotate the extrusion tool plunger knob into position for sample extrusion; unlock the locking arms of the cap; carefully remove the cap from the sampler; and, push down on the plunger knob of the extrusion tool (see Fig. 2) to expel the sample from the coring body/storage chamber directly into the appropriate container for analysis or preservation (see Practice D 4547). After the sample is expelled from the En Core sampler, the O-ring in the sampler cap should be inspected to make sure that the O-ring was not bent when the cap was placed on the coring body/storage chamber. A bent O-ring in the sampler cap can result in loss of VOCs from the stored sample and should be documented (see X1.1.8.2 and X1.1.9.2).

NOTE 3—Samples that have been stored at –12 ±2°C should be allowed to sit at room temperature for 2 to 3 min before extrusion from the En Core sampler, which will facilitate extrusion of the frozen sample from the device.

7. Report

7.1 For guidance on information that should be recorded for a sampling activity, see Guide D 4687.

8. Precision and Bias

8.1 *Precision*—Data on the precision of this practice are to be generated in the near future.

8.2 *Bias*—No information can be given on the bias of the sampling/storage procedure described in this practice because there is no standard reference material for sampling soil for VOCs in the field; however, data have been generated to provide information on the performance of the En Core sampler for storage of soil samples at 4± 2°C for 48 h, at 4 ±2°C for four days, at 4 ±2°C for 48 hours followed by storage for five days at –12 ±2°C, and at –12 ±2°C for seven days[7]. These data are shown in Appendix X1.

9. Keywords

9.1 En Core sampler; soil sampling; storage of soil samples; volatile organic compounds (VOCs); VOC analysis

---

[7] A copy of the research report on the study described in Appendix X1 is available from ASTM Headquarters. Request RR:D34-1012.

APPENDIX

(Nonmandatory Information)

X1. PERFORMANCE OF THE EN CORE SAMPLER TO STORE VOC-SPIKED SOIL SAMPLES

X1.1 A study was conducted to evaluate the performance of the 5- and 25-g En Core samplers to store three different soil types spiked with an aqueous solution containing nine volatile compounds. The En Core samplers used in this study were manufactured in 1998. The 5-g devices that were used represent lot numbers K018166, K028132, and K038132. The 25-g devices that were used represent lot numbers K218266, K018140, P018138, 018134, 018135, 018126, and 018128. The manufacturer can be contacted for information on these lot numbers.

X1.1.1 The soils used in the study are representative of different environments and contained native microbial populations. They are a river bank soil having 49 % sand, 26 % silt, 24 % clay, 5.3 % organic material, approximately 14 % moisture, and a dehydrogenase (microbial) activity of 22-mg total product formed (TPF)/g/24 h; a mountain soil having 75 % sand, 13 % silt, 12 % clay, 4.3 % organic material, approximately 12 % moisture, and a dehydrogenase activity of 11 mg TPF/g/24 h; and, a prairie soil having 67 % sand, 17 % silt, 16 % clay, 1.5 % organic material, approximately 8 % moisture, and a dehydrogenase activity of 17 mg TPF/g/24 h.

X1.1.2 The VOCs used in the study are *cis*-dichloroethylene (CDCE), benzene, trichloroethylene (TCE), toluene, perchloroethylene (PCE), ethylbenzene, m/p-xylene, o-xylene, and methylethylketone (MEK). These compounds were selected as the analytes of interest because they are representative of halogenated, aromatic, and polar VOCs typically found in contaminated soils.

X1.1.3 In the study, soil samples were collected in the En Core samplers from a large container of loose soil and then spiked with an aqueous solution containing the compounds listed in X1.1.2 at concentrations of approximately 50 mg/L. The spiking solution was injected into the middle of the soil plug in the sampler, after which, the sampler was immediately capped. The 5-g samples were spiked with 250 µL of the spiking solution, and the 25-g samples were spiked with 1250 µL of the spiking solution to give an approximate concentration of 2.5 µg/g of each analyte of interest in the samples. This analyte concentration in the soil was selected to limit the influence of the analytical method on the data.

X1.1.4 After all samples were spiked and capped, five random samples for each soil type were extruded from each size of En Core sampler into methanol for analysis to give time-zero concentrations of the analytes of interest. The remaining samples were stored under the storage conditions shown in Table X1.1. Storage temperatures were monitored to make sure they were at the specified temperature. After the samples were held for the appropriate times, they were extruded into methanol for extraction and analysis. The methanol extracts of the samples were analyzed using EPA Method 8021B.[8]

X1.1.5 To evaluate the data, the mean values of the analytes of interest in the stored samples were compared to their mean values in the time-zero samples by calculating average percent recovery. Before average percent recovery was calculated, the data sets were evaluated for outlier data points as described in Practice D 2777.

X1.1.6 The data generated by this testing are specific to the experimental design of the study. The data give information on the performance of the En Core samplers for storing soil samples collected from loose soil, spiked with an aqueous solution of selected analytes of interest, and then stored under specific storage conditions. The data generated by this testing also are specific to the soils used in the study, the analytes of interest, the analyte concentrations, and the storage conditions that were evaluated. For other soil types, analytes, analyte concentrations, and storage conditions, these data may not apply.

X1.1.7 The average percent recoveries of the VOCs of interest from samples of the river bank soil stored in the 5-g En Core samplers are shown in Table X1.2; and, the average percent recoveries of the VOCs of interest from samples of the river bank soil stored in the 25-g En Core samplers are shown in Table X1.3. In each of these tables, the percent relative standard deviation of the concentration values in the stored samples is given in parentheses next to the corresponding average percent recovery value. The percent relative standard deviation of the concentration values in the time-zero samples is given in a footnote to each of the tables.

X1.1.7.1 As shown in Table X1.2, the VOCs of interest in this study have average percent recovery values ranging from

TABLE X1.1 Storage Conditions for Testing the Disposable En Core Samplers

Storage Times	Storage Conditions	No Storage	4 ±2°C on Ice	4 ±2°C on Ice for 48 h then Refrigerated at 4 ±2°C	4 ±2°C on Ice for 48 h then Frozen at –12 ±2°C	Frozen at –12 ±2°C
Time-zero		5 samples				
48 h			5 samples			
4 days				5 samples		
7 days					5 samples	5 samples

---

[8] U.S. EPA, 1996, Method 8021B: Aromatic and Halogenated Volatiles by Gas Chromatography Photoionization and/or Electrolytic Conductivity Detectors. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol 1B, Final Update III.

TABLE X1.2 Average Percent Recoveries of VOCs from Samples of Soil 1[A] Stored in 5-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then –12± 2°C/5 Days	–12± 2°C/7 Days
CDCE		91 %[B] (15 %)[C]	96 %[B] (4 %)[C]	89 %[B] (3 %)[C]	98 %[B] (7 %)[C]
Benzene		93 % (3 %)	94 % (4 %)	87 % (5 %)	93 % (10 %)
TCE		97 % (1 %)	100 % (3 %)	95 % (4 %)	101 % (8 %)
Toluene		99 % (1 %)	96 % (4 %)	96 % (3 %)	99 % (6 %)
PCE		100 % (1 %)	100 % (1 %)	99 % (3 %)	99 % (7 %)
Ethylbenzene		101 % (3 %)	100 % (4 %)	101 % (3 %)	101 % (6 %)
m\p-Xylene		102 % (2 %)	100 % (3 %)	96 % (9 %)	99 % (6 %)
o-Xylene		99 % (1 %)	99 % (4 %)	91 % (7 %)	100 % (7 %)
MEK		100 % (0 %)	100 % (0 %)	92 % (6 %)	91 % (6 %)

[A] Soil 1 is a river bank soil (49 % sand, 26 % silt, 24 % clay, 5.3 % organic material, and approximately 14 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the five stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 0 to 6 % for all tests using soil 1 in the 5-g device.

TABLE X1.3 Average Percent Recoveries of VOCs from Samples of Soil 1[A] Stored in 25-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then –12± 2°C/5 Days	–12± 2°C/7 Days
CDCE		91 %[B] (1 %)[C]	94 %[B] (3 %)[C]	81 %[B] (8 %)[C]	90 %[B] (8 %)[C]
Benzene		90 % (4 %)	94 % (4 %)	79 % (11 %)	87 % (12 %)
TCE		92 % (3 %)	96 % (8 %)	87 % (6 %)	100 % (6 %)
Toluene		94 % (3 %)	94 % (6 %)	91 % (4 %)	98 % (6 %)
PCE		96 % (3 %)	100 % (5 %)	92 % (5 %)	102 % (6 %)
Ethylbenzene		98 % (3 %)	100 % (1 %)	98 % (3 %)	102 % (4 %)
m\p-Xylene		98 % (1 %)	98 % (2 %)	95 % (6 %)	100 % (3 %)
o-Xylene		98 % (3 %)	100 % (2 %)	97 % (5 %)	102 % (4 %)
MEK		96 % (1 %)	96 % (3 %)	90 % (4 %)	93 % (4 %)

[A] Soil 1 is a river bank soil (49 % sand, 26 % silt, 24 % clay, 5.3 % organic material, and approximately 14 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the five stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 1 to 4 % for all tests using soil 1 in the 25-g device.

87–102 % for storage of the river bank soil samples in the 5-g En Core samplers for all of the storage conditions used in this study. Taking into account the error introduced in preparation and analysis of the samples, these data show little loss of the VOCs of interest from the river bank soil during storage in the 5-g En Core samplers. The mean of the nine average percent recovery values listed in Table X1.2 for the analytes of interest in the samples of river bank soil stored at 4 ±2°C for 48 h is 98 % recovery with a standard deviation of 4 % and a percent relative standard deviation of 4 %. The overall mean of the 36 average percent recovery values listed in Table X1.2 for the analytes of interest and various storage conditions is 97 % recovery with a standard deviation of 4 % and a percent relative standard deviation of 4 %.

X1.1.7.2 As shown in Table X1.3, the VOCs of interest in this study have average percent recovery values ranging from 79–102 % for storage of the river bank soil samples in the 25-g En Core samplers for all of the storage conditions used in this study. Taking into account the error introduced in preparation and analysis of the samples, these data show little loss of the VOCs of interest from the river bank soil during storage in the 25-g En Core samplers. The mean of the nine average percent recovery values listed in Table X1.3 for the analytes of interest in the samples of river bank soil stored at 4 ±2°C for 48 h is 95 % recovery with a standard deviation of 3 % and a percent relative standard deviation of 3 %. The overall mean of the 36 average percent recovery values listed in Table X1.3 for the analytes of interest and various storage conditions is 95 % recovery with a standard deviation of 6 % and a percent relative standard deviation of 6 %.

X1.1.8 The average percent recoveries of the VOCs of interest from samples of the mountain soil stored in the 5-g En Core samplers are shown in Table X1.4; and, the average percent recoveries of the VOCs of interest from samples of the mountain soil stored in the 25-g En Core samplers are shown in Table X1.5. In each of these tables, the percent relative standard deviation of the concentration values in the stored samples is given in parentheses next to the corresponding average percent recovery value. The percent relative standard deviation of the concentration values in the time-zero samples is given in a footnote to each of the tables.

X1.1.8.1 As shown in Table X1.4, TCE, toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene have average percent recovery values for storage of the mountain soil at 4 ±2°C for 48 h in the 5-g En Core samplers that range from 90–97 %. Average percent recovery values of these compounds from the mountain soil samples stored for 7 days at –12 ±2°C are similar, ranging from 91–101 %. Taking into account the error introduced in preparation and analysis of the samples, these data show very little or no loss of the six VOCs from the mountain soil during storage in the 5-g samplers under the two different storage conditions. The average percent recovery values for TCE, toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene from the mountain soil samples stored for four days at 4 ±2°C and for 48 h at 4 ±2°C followed by five-day storage at –12± 2°C in the 5-g En Core samplers are slightly lower, ranging from 80–92 %. The same trend is shown in Table X1.4 for CDCE and benzene. These compounds have average

TABLE X1.4 Average Percent Recoveries of VOCs from Samples of Soil 2[A] Stored in 5-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then –12± 2°C/5 Days	–12± 2°C/7 Days
CDCE		87 %[B] (10 %)[C]	77 %[B] (10 %)[C]	74 %[B] (7 %)[C]	84 %[B] (11 %)[C]
Benzene		86 % (11 %)	74 % (12 %)	69 % (9 %)	84 % (12 %)
TCE		91 % (8 %)	83 % (9 %)	80 % (5 %)	91 % (9 %)
Toluene		90 % (5 %)	83 % (7 %)	86 % (2 %)	97 % (8 %)
PCE		96 % (4 %)	89 % (6 %)	90 % (5 %)	99 % (6 %)
Ethylbenzene		92 % (7 %)	90 % (6 %)	90 % (10 %)	96 % (16 %)
m\p-Xylene		92 % (2 %)	86 % (6 %)	89 % (2 %)	101 % (5 %)
o-Xylene		97 % (2 %)	92 % (6 %)	92 % (5 %)	97 % (10 %)
MEK		83 % (0 %)	76 % (7 %)	74 % (4 %)	71 % (9 %)

[A] Soil 2 is a mountain soil (75 % sand, 13 % silt, 12 % clay, 4.3 % organic material, and approximately 12 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the five stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 3 to 8 % for all tests using soil 2 in the 5-g device.

TABLE X1.5 Average Percent Recoveries of VOCs from Samples of Soil 2[A] Stored in 25-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then – 12 ±2°C/5 Days	–12 ±2°C/7 Days
CDCE		87 %[B] (8 %)[C]	82 %[B] (11 %)[C]	59 %[D] (17 %)[C]	71 %[B] (25 %)[C]
Benzene		90 % (11 %)	81 % (14 %)	56 % (18 %)	71 % (30 %)
TCE		94 % (6 %)	88 % (9 %)	69 % (9 %)	82 % (17 %)
Toluene		93 % (6 %)	87 % (5 %)	78 % (5 %)	89 % (12 %)
PCE		98 % (6 %)	92 % (4 %)	85 % (4 %)	92 % (11 %)
Ethylbenzene		96 % (4 %)	94 % (4 %)	90 % (2 %)	98 % (7 %)
m\p-Xylene		93 % (4 %)	91 % (3 %)	92 % (2 %)	98 % (7 %)
o-Xylene		96 % (4 %)	96 % (5 %)	93 % (2 %)	96 % (6 %)
MEK		86 % (2 %)	83 % (6 %)	75 % (2 %)	73 % (2 %)

[A] Soil 2 is a mountain soil (75 % sand, 13 % silt, 12 % clay, 4.3 % organic material, and approximately 12 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 1 to 8 % for all tests using soil 2 in the 25-g device.

[D] Average percent recovery based on mean values determined for five time-zero samples and three stored samples.

percent recovery values ranging from 84–87 % for the samples stored at 4 ±2°C for 48 h and –12 ±2°C for seven days. While the percent recovery values of CDCE and benzene from the mountain soil samples stored for four days at 4 ±2°C and for 48 h at 4 ±2°C followed by five-day storage at –12 ±2°C are slightly lower, ranging from 69 to 77 %. The average percent recovery values of MEK from the mountain soil samples stored under the four storage conditions in the 5-g En Core samplers range from 71–83 %. These values are lower than would be expected for this less volatile compound, especially when compared to the average percent recovery values of the other VOCs listed in Table X1.4. The reason for the lower average percent recovery values for MEK from the mountain soil may be that the ketone reacts with this soil causing a decrease in MEK concentrations with time.

X1.1.8.2 As shown in Table X1.5, TCE, toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene have average percent recovery values ranging from 93–98 % for storage of the mountain soil samples in the 25-g En Core samplers at 4 ±2°C for 48 h. Average percent recovery values of CDCE and benzene from samples of the mountain soil stored in the 25-g En Core samplers for 48 h at 4 ±2°C are 87 and 90 %, respectively. For four-day storage at 4 ±2°C in the 25-g En Core samplers, TCE, toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene have average percent recovery values from the mountain soil samples ranging from 87–96 %; and average percent recoveries of CDCE and benzene from these samples are 82 and 81 %, respectively. In testing storage of the spiked mountain soil samples in the 25-g En Core samplers for 48 h at 4 ±2°C followed by five-day storage at –12 ±2°C, two of the devices used to store the samples had O-rings in their caps that were bent out of position as discussed in 6.4.2 and 6.7.1. Because of this error in capping these devices, the seal between the cap and coring body/storage chamber was compromised. As a result, data for only three of the stored samples were used to calculate the average percent recovery values for this storage condition. As shown in Table X1.5, these values range from 56–69 % for CDCE, benzene, and TCE; and 78–93 % for toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene. For seven-day storage at –12 ±2°C in the 25-g En Core samplers, TCE, toluene, PCE, ethylbenzene, m/p-xylene, and o-xylene have average percent recovery values from the mountain soil samples ranging from 82–98 %; and average percent recovery values of CDCE and benzene from these samples are both 71 %. The average percent recovery values of MEK from the mountain soil samples stored under the four storage conditions in the 25-g En Core samplers range from 73–86 %. As discussed in X1.1.8.1, these values are lower than would be expected and may be due to a chemical reaction between MEK and the mountain soil.

X1.1.8.3 Differences in the average percent recovery values of the VOCs of interest in this study from the river bank soil (Tables X1.2 and X1.3) and mountain soil (Tables X1.4 and X1.5) may be due to the difference in the composition of these soils. As described in X1.1.1, the river bank soil is 49 % sand, 26 % silt, and 24 % clay, while the mountain soil contains

75 % sand, 13 % silt, and 12 % clay.

X1.1.9 The average percent recoveries of the VOCs of interest from samples of the prairie soil stored in the 5-g En Core samplers are shown in Table X1.6; and the average percent recoveries of the VOCs of interest from samples of the prairie soil stored in the 25-g En Core samplers are shown in Table X1.7. In each of these tables, the percent relative standard deviation of the concentration values in the stored samples is given in parentheses next to the corresponding average percent recovery value. The percent relative standard deviation of the concentration values in the time-zero samples is given in a footnote to each of the tables.

X1.1.9.1 As shown in Table X1.6, PCE, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values ranging from 90–92 % for storage of the prairie soil samples in the 5-g En Core samplers at 4 ±2°C for 48 h. Under these same storage conditions, average percent recovery values of CDCE, benzene, TCE, and toluene from samples of the prairie soil stored in the 5-g En Core samplers range from 75–82 %. For four-day storage at 4 ±2°C in the 5-g En Core samplers, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values from the prairie soil samples ranging from 85–92 %. Under these same storage conditions, average percent recovery values of CDCE, benzene, TCE, toluene, and PCE from samples of the prairie soil stored in the 5-g En Core samplers range from 42–75 %. For 48-h storage at 4 ±2°C followed by five-day storage at –12 ±2°C in the 5-g samplers, PCE, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values from the stored prairie soil samples ranging from 64–85 %. Under these same storage conditions, CDCE, benzene, TCE, and toluene have average percent recovery values ranging from 30–52 %. For seven-day storage at –12 ±2°C in the 5-g En Core samplers, PCE, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values from the prairie soil samples ranging from 83–99 %. Under these same storage conditions, average percent recovery values of CDCE, benzene, and TCE from samples of the prairie soil stored in the 5-g En Core samplers range from 36–61 %; and, the average percent recovery value of toluene is 77 %.

X1.1.9.2 As shown in Table X1.7, PCE, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values ranging from 86–97 % for storage of the prairie soil samples in the 25-g En Core samplers at 4 ±2°C for 48 h. Under these same storage conditions, average percent recovery values of CDCE, benzene, TCE, and toluene from samples of the prairie soil stored in the 25-g En Core samplers range from 69–79 %. For four-day storage at 4 ±2°C in the 25-g En Core samplers, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values from the prairie soil samples ranging from 79–93 %. Under these same storage conditions, average percent recovery values of CDCE, benzene, TCE, and toluene from samples of the prairie soil stored in the 25-g En Core samplers range from 50–64 %; and, the average percent recovery value of PCE is 73 %. In testing storage of the spiked prairie soil samples in the 25-g En Core samplers for 48-h at 4 ±2°C followed by five-day storage at –12 ±2°C, two of the devices used to store the samples had O-rings in their caps that were bent out of position as discussed in 6.4.2 and 6.7.1. Because of this error in capping these devices, the seal between the cap and coring body/storage chamber was compromised. As a result, data for only three of the stored samples were used to calculate the average percent recovery values for this storage condition. As shown in Table X1.7, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values that range from 71–85 %. Under these same storage conditions, percent recovery values for toluene and PCE are 46 and 57 %, respectively; and, average percent recovery values for CDCE, benzene, and TCE range from 22–33 %. For seven-day storage at –12 ±2°C in the 25-g En Core samplers, PCE, ethylbenzene, m/p-xylene, o-xylene, and MEK have average percent recovery values from the prairie soil samples ranging from 86–98 %. Under these same storage conditions, average percent recovery values of CDCE, benzene, and TCE range from 54–63 %; and, the average percent recovery value of toluene is 75 %.

X1.1.9.3 The average percent recovery values for the VOC-spiked prairie soil samples stored in the 5-g and 25-g En Core samplers are lower than the values reported for the river bank soil (Tables X1.2 and X1.3) and mountain soil (Tables X1.4 and X1.5). As described in section X1.1.1, the river bank and mountain soils contain approximately 12–14 % moisture, while the prairie soil contains approximately 8 % moisture. Samples of this drier soil collected from the large container of loose soil used in this study were difficult to compact in the En Core samplers. In addition, scraping the surface of the prairie soil to make it flush with the opening of the coring body/storage chamber, so the sampler could be capped, loosened particles of the dry soil. It is believed that for many of the

TABLE X1.6 Average Percent Recoveries of VOCs from Samples of Soil 3[A] Stored in 5-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then –12± 2°C/5 Days	–12± 2°C/7 Days
CDCE		82 %[B] (9 %)[C]	64 %[B] (40 %)[C]	36 %[B] (6 %)[C]	51 %[B] (15 %)[C]
Benzene		75 % (13 %)	42 % (23 %)	30 % (8 %)	36 % (58 %)
TCE		79 % (10 %)	51 % (16 %)	42 % (5 %)	61 % (17 %)
Toluene		82 % (8 %)	75 % (9 %)	52 % (3 %)	77 % (11 %)
PCE		91 % (5 %)	58 % (8 %)	64 % (3 %)	89 % (11 %)
Ethylbenzene		91 % (3 %)	85 % (16 %)	70 % (4 %)	93 % (8 %)
m\p-Xylene		90 % (1 %)	87 % (13 %)	71 % (3 %)	96 % (7 %)
o-Xylene		92 % (2 %)	86 % (4 %)	79 % (3 %)	99 % (7 %)
MEK		92 % (0 %)	92 % (6 %)	85 % (0 %)	83 % (13 %)

[A] Soil 3 is a prairie soil (67 % sand, 17 % silt, 16 % clay, 1.5 % organic material, and approximately 8 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the five stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 0 to 11 % for all tests using soil 3 in the 5-g device.

TABLE X1.7 Average Percent Recoveries of VOCs from Samples of Soil 3[A] Stored in 25-G En Core Samplers

VOCs	Storage Conditions	4 ±2°C/48 h	4 ±2°C/4 Days	4 ±2°C/48 h then −12± 2°C/5 Days	−12± 2°C/7 Days
CDCE		76 %[B] (17 %)[C]	59 %[B] (34 %)[C]	29 %[D] (42 %)[C]	59 %[B] (47 %)[C]
Benzene		69 % (25 %)	50 % (50 %)	22 % (51 %)	54 % (57 %)
TCE		72 % (22 %)	55 % (39 %)	33 % (36 %)	63 % (44 %)
Toluene		79 % (14 %)	64 % (26 %)	46 % (20 %)	75 % (29 %)
PCE		86 % (14 %)	73 % (24 %)	57 % (15 %)	86 % (21 %)
Ethylbenzene		89 % (6 %)	79 % (11 %)	71 % (7 %)	91 % (12 %)
m\p-Xylene		88 % (6 %)	81 % (9 %)	73 % (5 %)	94 % (10 %)
o-Xylene		94 % (4 %)	89 % (6 %)	79 % (3 %)	98 % (8 %)
MEK		97 % (3 %)	93 % (0 %)	85 % (2 %)	92 % (5 %)

[A] Soil 3 is a prairie soil (67 % sand, 17 % silt, 16 % clay, 1.5 % organic material, and approximately 8 % moisture).

[B] Average percent recovery based on mean values determined for five time-zero samples and five stored samples

[C] Value in parentheses is the percent relative standard deviation of the concentration values in the stored samples. The percent relative standard deviation of the concentration values in the time-zero samples ranged from 1 to 6 % for all tests using soil 3 in the 25-g device.

[D] Average percent recovery based on mean values determined for five time-zero samples and three stored samples.

prairie soil samples prepared in this study, the loose soil particles scattered when the samplers were capped affecting the seals of the samplers. Many of the percent relative standard deviation values shown in Tables X1.6 and X1.7 for the stored prairie soil samples are higher than those reported for the river bank soil samples (Tables X1.2 and X1.3) and mountain soil samples (Tables X1.4 and X1.5). These values reflect the variability of analyte concentrations in the stored samples of the spiked prairie soil. This variability is believed to be due to scattered soil particles affecting the seals of the En Core devices to varying degrees during capping and storage. A cautionary note to 6.4.2 of this practice has been included to alert the user to use caution when working with loose, dry soils, (see Note 2).