

**VALIDATION OF A NEW SOIL VOC SAMPLER: PRECISION OF
THE PERFORMANCE OF THE EN CORE® SAMPLER TO STORE
LOW VOC CONCENTRATIONS**

Topical Report

By

Susan S. Sorini

John F. Schabron

Joseph F. Rovani, Jr.

June 2002

**Work Performed Under Cooperative Agreement
DE-FC26-98FT40323 Task 5**

For

En Chem, Inc.

Green Bay, Wisconsin

And

U.S. Department of Energy

Office of Fossil Energy

National Energy Technology Laboratory

Morgantown, West Virginia

By

Western Research Institute

Laramie, Wyoming

ACKNOWLEDGMENTS

Funding for this study was provided by the U.S. Department of Energy, National Energy Technology Laboratory, under Cooperative Agreement DE-FC26-98FT40323 and En Chem, Inc., Green Bay, Wisconsin.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe on privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

ABSTRACT

Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) from samples. The En Core® sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An American Society for Testing and Materials (ASTM) standard practice, D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis, describes use of the En Core sampler to collect and store a soil sample of approximately 5 grams or 25 grams for volatile organic analysis.

To support the ASTM practice, four studies have been performed to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs. The first study was conducted to evaluate the performance of the device to store soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg under various conditions. This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. A second study was conducted to answer questions on the performance of the En Core sampler for storage of soil samples containing low-level (<200 µg/Kg) concentrations of VOCs. In this study, soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The third study was performed to generate data on the performance of the 25-gram En Core sampler to store soil samples spiked with EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), volatile organic analytes under various conditions for 14 days. The low-level performance data and TCLP analyte storage data are included in appendices to the ASTM practice, and the ASTM research report describing the high-level study is referenced in the practice.

The fourth study, which is described in this report, was performed to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs. This was done so that information on the precision of the performance of the devices can be added to the ASTM practice. Data generated in the precision validation study show very good precision of the performance of the En Core samplers to store soil samples spiked with low concentrations of a variety of VOCs.

TABLE OF CONTENTS

	<u>Page</u>
LIST OF TABLES AND FIGURES	vi
EXECUTIVE SUMMARY	vii
INTRODUCTION	1
OBJECTIVES	3
TECHNICAL APPROACH	3
Testing to Estimate the Precision of the Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs	3
Proposed Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis	6
Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds	6
Development of a Subsurface Sampling/Storage Device for VOC Analysis	7
RESULTS AND DISCUSSION	8
Testing to Estimate the Precision of the Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs	8
Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs	10
Proposed Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis	11

TABLE OF CONTENTS (continued)

	<u>Page</u>
Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds	11
Development of a Subsurface Sampling/Storage Device for VOC Analysis	11
CONCLUSIONS	12
REFERENCES	13
APPENDIX A	25
APPENDIX B	54

LIST OF TABLES AND FIGURES

<u>Table</u>	<u>Page</u>
1. Storage Conditions for Testing the Precision of the Performance of the Disposable En Core Samplers to Store Spiked Soil Samples	15
2. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations Due to Storage of Spiked River Bank Soil Samples in 5-Gram En Core Samplers	16
3. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations Due to Storage of Spiked River Bank Soil Samples in 25-Gram En Core Samplers	17
4. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations Due to Storage of Spiked Prairie Soil Samples in 5-Gram En Core Samplers	18
5. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations Due to Storage of Spiked Prairie Soil Samples in 25-Gram En Core Samplers	19
6. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations Due to Storage of Spiked Soil in En Core Samplers	20
7. Summary of Average Percent Recoveries of Low-Level VOCs from River Bank and Prairie Soil Samples Stored in the 5-Gram and 25-Gram En Core Samplers	21

<u>Figure</u>	<u>Page</u>
1. Components of the En Core Sampler	23
2. Reusable Attachments to the En Core Sampler	24

EXECUTIVE SUMMARY

Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) from samples. The En Core® sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An American Society for Testing and Materials (ASTM) standard practice, D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis, describes use of the En Core sampler to collect and store a soil sample of approximately 5 grams or 25 grams for volatile organic analysis. The 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. After the sample is collected in the En Core sampler, the coring body is sealed with a slide-on cap and immediately becomes a sample storage chamber. To minimize loss of volatile compounds due to volatilization and/or biodegradation from the time of collection until analysis or preservation in the laboratory, sample storage in the device is specified at 4 ± 2 °C for up to 48 hours. When holding times beyond 48 hours are necessary, storage of samples in the En Core sampler at 4 ± 2 °C or -12 ± 2 °C for longer than 48 hours is an option, provided it can be shown that the longer storage time does not influence the concentrations of VOCs of interest in the samples or that the data generated by analysis of the samples meet the data quality objectives (DQOs).

To support the ASTM practice, four studies have been performed to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs. The first study was conducted to evaluate the performance of the device to store soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg under various conditions. This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. A second study was conducted to answer questions on the performance of the En Core sampler for storage of soil samples containing low-level (<200 µg/Kg) concentrations of VOCs. In this study, soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The third study was performed to generate data on the performance of the 25-gram En Core sampler to store soil samples spiked with EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP), volatile organic analytes under various conditions for 14 days. The low-level performance data and TCLP analyte storage data are included in appendices to the ASTM practice, and the ASTM research report describing the high-level study is referenced in the practice.

The fourth study involving the En Core sampler was performed to estimate the precision of the performance of the 5-gram and 25-gram devices to store soil samples spiked with low concentrations of VOCs. This was done so that information on the precision of the devices can be added to the ASTM practice. A proposal to include a statement in D 6418 on the estimated precision of the performance of the 5-gram and 25-gram En Core samplers to store soils containing low concentrations of VOCs under various conditions and to reference an ASTM research report describing the precision validation study is to be balloted within ASTM Subcommittee D 34.01 on

Sampling, Monitoring, and Characterization and ASTM Committee D 34 on Waste Management. If this proposal passes, information on the precision of the performance of the En Core sampler to store VOC-spiked soil will be provided in the ASTM practice.

In August 1998, a revision of ASTM D 4547, Standard Practice for Sampling Waste and Soils for Volatile Organics, was approved. This revision is titled “D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds.” Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that use of the En Core device is included (and not excluded) as an option for sampling and storing soil for volatile organic analysis and to help ensure that the revision is technically sound. Work continues on achieving ASTM approval of a second revision of this standard.

The En Core sampler is designed to collect soil samples for VOC analysis at the soil surface. To date, a sampling tool for collecting and storing subsurface soil samples for VOC analysis does not exist. Development of a subsurface VOC sampling/storage device was initiated in 1999. This device is designed so that a soil sample can be collected below the surface and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been produced and tested for use in the field.

This report describes the study that was conducted to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low-level concentrations of VOCs. The proposed revision of ASTM Practice D 6418 to include information from this study is discussed, as well as the proposed revision of ASTM Guide D 4547 and progress towards development of the subsurface soil sampling/storage device for VOC analysis.

INTRODUCTION

A major problem in sampling soil for volatile organic analysis is preservation of sample

integrity during storage and shipment of soil samples to the laboratory. Soil sampling and storage practices for volatile organic analysis must be designed to minimize loss of volatile organic compounds (VOCs) due to volatilization and/or biodegradation. Laboratory data can grossly underestimate the actual VOC concentrations in a soil if great attention is not paid to sampling and handling techniques (Turriff and Klopp 1995).

The En Core® sampling/storage device provides a simple means for sampling soil and holding a soil sample during shipment to the laboratory for VOC analysis (Vitale et al. 1999). This device has three components: (1) the coring body/storage chamber, which is volumetrically designed to collect and store either a soil sample of approximately 5 grams or 25 grams, (2) an O-ring sealed plunger for nondisruptive extrusion of the sample into an appropriate container for analysis or preservation, and (3) a slide-on cap having an O-ring seal and locking arm mechanism. A diagram of the En Core sampling/storage device is shown in Figure 1. The seals of the device are provided by three Viton™ O-rings (Figure 1). The coring body/storage chamber, plunger, and cap of the En Core sampler are constructed of a glass-filled inert composite polymer, polyphthalamide (RTP), making the device chemically compatible with soil matrices and contaminants. The En Core sampler is disposable. It is certified as clean when received from the manufacturer and is not to be reused. The En Core sampler has two reusable stainless steel attachments. 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. These are shown in Figure 2. Each En Core sampler is supplied with a protective moisture-proof bag for shipment to the laboratory.

Western Research Institute developed an American Society for Testing and Materials (ASTM) standard practice for using the En Core device. This practice is D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis (ASTM 2001a). To support the ASTM practice, four studies have been performed to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs. The first study was conducted to evaluate the performance of the device to store soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg under various conditions (Sorini and Schabron 1999a). This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. A second study was conducted to answer questions on the performance of the En Core sampler for storage of soil samples containing low-level (<200 µg/Kg) concentrations of VOCs (Sorini et al. 2001 and 2002). In this study, soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The third study was performed to generate data on the performance of the 25-gram En Core sampler to store soil samples spiked with EPA Method 1311, Toxicity Characteristic Leaching Procedure (TCLP) (U.S. EPA 1992), volatile organic analytes under various conditions for 14 days (Sorini et al. 2001 and 2002). The low-level performance data and TCLP analyte storage data are included in appendices to the ASTM practice (ASTM 2001b), and the ASTM research report describing the high-level study (Sorini and Schabron 1999b) is referenced in the practice.

The fourth study involving the En Core sampler was performed to estimate the precision of the performance of the 5-gram and 25-gram devices to store soil samples spiked with low concentrations of VOCs. This was done so that information on the precision of these devices can be added to the ASTM practice. A proposal to include a statement in D 6418 on the estimated precision of the performance of the 5-gram and 25-gram En Core samplers to store soils containing low concentrations of VOCs under various conditions and to reference an ASTM research report describing the precision validation study is to be balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Committee D 34 on Waste Management. If this proposal passes, information on the precision of the performance of the En Core sampler to store VOC-spiked soil will be provided in the ASTM practice.

In August 1998, a revision of ASTM D 4547, Standard Practice for Sampling Waste and Soils for Volatile Organics, was approved. This revision is titled “D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds” (ASTM 2001c). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that the En Core sampler is fairly represented and to help ensure that the revision is technically sound. Work continues on achieving ASTM approval of a second revision of this standard.

The En Core sampler is designed to collect soil samples for VOC analysis at the soil surface. To date, a sampling tool for collecting and storing subsurface soil samples for VOC analysis does not exist. Development of a subsurface VOC sampling/storage device was initiated in 1999. This device is designed so that a soil sample can be collected below the surface and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been produced and tested for use in the field.

This report describes the study that was conducted to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low-level concentrations of VOCs. The proposed revision of ASTM Practice D 6418 to include information from this study is discussed, as well as the proposed revision of ASTM Guide D 4547 and progress towards development of the subsurface soil sampling/storage device for VOC analysis.

OBJECTIVES

The overall objective of this task is to facilitate national acceptance of the En Core device for sampling and storing soil for VOC analysis. The activities that were performed over the last twelve months to help achieve this objective are described below.

- Testing to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low-concentrations of VOCs was performed.
- Activities involving reviewing and commenting on proposed draft revisions of D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds, were performed.

A second objective for this twelve-month period was to continue work on development of a subsurface soil sampling/storage device for VOC analysis. As part of this, prototype devices have been produced and are currently being tested.

TECHNICAL APPROACH

Testing to Estimate the Precision of the Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs

This study was designed to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store two different soil types spiked with an aqueous solution containing eleven VOCs and stored under various conditions. Five replicate samples were prepared for each storage condition and each set of time-zero samples (no storage). One En Core sampler from each of five different lot numbers was used to prepare each set of five replicates. In this way, precision across different lot numbers could be estimated. The lot numbers for the 5-gram samplers were K111124, K111128, K111129, K111130, and K111143. The lot numbers for the 25-gram samplers were K111088, K111094, K111117, K111137, and K111138.

The soils used in the study represented two different environments and contained native microbial populations. They were 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 hr 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 hr. Characterization of the soils was performed by the University of Wyoming Soils Laboratory using standard soil analytical methods. The soils were homogenized prior to their characterization and use in the testing.

The VOCs used in the study were vinyl chloride, methylene chloride (MeCl_2), methyl tert-butyl ether (MTBE), 1,1-dichloroethane, *cis*-1,2-dichloroethylene (CDCE), chloroform, benzene, trichloroethylene (TCE), toluene, tetrachloroethylene (PCE), ethyl benzene, and o-xylene. These compounds were selected as the analytes of interest because they are representative of halogenated and aromatic compounds that are of environmental concern and were used in the low-level study for which data are presented in one of the appendices of D 6418.

In the study, soil samples were collected in the En Core samplers from a large container of loose soil. After soil was collected in a sampler, a spatula was scraped across the bottom of the coring body/storage chamber so the surface of the soil in the sampler was flush with the opening. Then the external surface of the device was wiped with a clean tissue. The open end of the sampler was wrapped with aluminum foil, and the sampler was placed in the protective bag with its cap. After all of the samples of a particular soil were collected, the soil samples were stored in a walk-in cooler at a temperature of approximately 4 °C, so they would be cold when the low-level spiking was performed. This was done to help minimize loss of the low concentrations of the volatile analytes during spiking.

The spiking solution used in the study was prepared by adding 250 µL of gasoline to approximately 80 mL of VOC-free water in a 100-mL volumetric flask and diluting to volume. The mixture was stirred for 24 hours, and then the gasoline was separated from the water using a separatory funnel. A portion of the resulting gasoline-saturated water was added to a 40-mL volatile organic analysis (VOA) vial, and a methanol solution containing the compounds of interest, except for benzene and toluene, was injected into the gasoline-saturated water to give the spiking solution. The benzene and toluene present in the spiking solution came from the gasoline-saturated water. All of the solutions, including the spiking solutions, were kept on ice to prevent loss of the volatile analytes.

The soil samples were spiked in the walk-in cooler at a temperature of approximately 4 °C. This was also done to minimize loss of the low concentrations of the volatile analytes during spiking. The spiking solution was injected into the middle of the soil plug in the sampler, and the sampler was immediately capped. No pooling of spiking solution was observed on the surface of any of the samples. The spiking solution was absorbed by the soil in the middle of the soil plug. The 5-gram samples were spiked with 100 µL of spiking solution, and the 25-gram samples were spiked with 0.5 mL of spiking solution to give an approximate concentration of 100 µg/Kg of each analyte of interest in the samples, with the exception of benzene, toluene, and o-xylene. Because of their presence in the gasoline-saturated water, the concentrations of benzene were approximately 150 µg/Kg, and the concentrations of toluene ranged from approximately 700 to 1,400 µg/Kg in the spiked soil samples. Additional o-xylene was also added to the spiking solution by the gasoline-saturated water. This resulted in o-xylene concentrations in the spiked soil samples of approximately 200 µg/Kg. After all samples were spiked and capped, five random samples for each soil type, one representing each of the different lots, were extruded from each size of En Core sampler into methanol for analysis to give time-zero concentrations of the analytes of interest. The 5-gram samples were extruded into 5 mL of methanol, and the 25-gram samples were extruded into 25 mL of methanol. The remaining samples were stored in sets of five, one from each of the different lots, under the conditions shown in Table 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 guidance given in EPA Methods 8260B (U.S. EPA 1996a) and 5030B (U.S. EPA 1996b).

For each soil type and sample size, an analytical standard was analyzed to determine the standard deviation of the analytical method for analysis of each analyte. For analysis of the extracts of the 5-gram samples, each of the analytical standards was a 100- μ L sample of spiking solution injected into 5 mL of methanol that was analyzed five times prior to and five times after analysis of the corresponding set of time-zero samples. For analysis of the extracts of the 25-gram samples, each of the analytical standards was a 0.5-mL sample of spiking solution injected into 25 mL of methanol that was analyzed five times prior to and five times after analysis of the corresponding set of time-zero samples. The analyte concentrations in the analytical standards ranged between 105 ng/mL and 145 ng/mL for all of the analytes of interest, except for benzene, toluene, and o-xylene. Once again, because of their presence in the gasoline-saturated water that was used to make the spiking solutions, the concentrations of benzene were approximately 150 ng/mL, the concentrations of toluene ranged from approximately 700 ng/mL to 1,400 ng/mL, and the concentrations of o-xylene were approximately 200 ng/mL in the analytical standards.

The analyte concentrations in the analytical standards are expressed as nanogram of VOC per milliliter of methanol. These concentrations can be related to the analyte concentrations in the soil samples because for analysis there is one gram of soil per one milliliter of methanol. Therefore, the analyte concentrations in the analytical standards can be expressed as one nanogram of VOC per gram of soil or one microgram of VOC per kilogram of soil.

The data sets from analysis of the time-zero and stored samples were evaluated for outlier data points as described in ASTM Practice D 2777, Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D 19 on Water (ASTM 1997). After this evaluation was completed, data from analysis of the stored samples were used to calculate the total standard deviation for each analyte, s , which includes the analytical standard deviation, s_a , the standard deviation due to spiking and extrusion, $s_{s/e}$, and the standard deviation due to storage of the VOC-spiked soil in the En Core sampler, s_{st} . Data from analysis of the analytical standard were used to calculate the analytical standard deviation for analysis of each analyte. The standard deviation determined using data from analysis of the time-zero samples, $s_{t=0}$, represents the combined standard deviation for each analyte due to spiking, extrusion, and analysis. Using the total standard deviation, analytical standard deviation, and time-zero standard deviation, the estimated precision of the performance of the 5-gram and 25-gram En Core samplers to store each of the VOCs of interest in the river bank and prairie soil samples under the various conditions was calculated using the equations shown below.

Estimated standard deviation due to spiking and extrusion:

$$s_{s/e} = [s_{t=0}^2 - s_a^2]^{1/2} \quad (1)$$

Estimated standard deviation due to storage in the En Core sampler:

$$s_{st} = [s^2 - s_{s/e}^2 - s_a^2]^{1/2} \quad (2)$$

Proposed Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis

As discussed in the introduction of this report, the study to estimate the precision of the performance of the 5-gram and 25-gram En Core samplers to store soil samples spiked with low concentrations of VOCs was performed so that information on the precision of these devices can be added to ASTM Practice D 6418. A proposal to include a statement in D 6418 on the estimated precision of the performance of the 5-gram and 25-gram En Core samplers to store soils containing low concentrations of VOCs under various conditions and to reference an ASTM research report describing the precision validation study will be balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Committee D 34 on Waste Management. If this proposal passes, information on the precision of the performance of the En Core sampler to store VOC-spiked soil will be provided in the ASTM practice.

Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

In August 1998, a revision of ASTM D 4547, ASTM's guidance document on sampling waste and soils for VOCs, was approved by ASTM. This revision is titled "D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds" (ASTM 2001c). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 has been proposed. Several draft revisions of the guide have been reviewed to ensure that the En Core sampler is fairly represented and to help ensure that the revision is technically sound. Changes to the guide that have been proposed and accepted by ASTM Subcommittee D 34.01 and Main Committee D 34 for the second revision include discussing freezing as a preservation technique and referencing D 6418 in the section of the guide that discusses sample storage in a coring device.

In the proposed revision of D 4547, freezing is discussed as an acceptable sample preservation method for VOC samples. Recently, data have been generated showing that loss of VOCs can be prevented by storing samples for VOC analysis at $-12 \pm 2^\circ\text{C}$, either immediately after collection in the field or after storage at $4 \pm 2^\circ\text{C}$ for 48 hours to allow sample transportation to the laboratory (Hewitt 1999; Sorini and Schabron 1999a; and Sorini et al. 2001 and 2002). Freezing as a method of preservation has several advantages over chemical preservation in the field in that no prior knowledge of VOC concentrations in the sample is required, field personnel do not have to handle chemicals or weigh samples in the field, concern about acidifying carbonaceous soils can be avoided, and Department of Transportation regulations concerning transport of hazardous chemicals

do not apply. The En Core sampler has been shown to perform well for storage of soil samples for VOC analysis under freezing conditions (Sorini and Schabron 1999a and Sorini et al. 2001 and 2002). As a result, inclusion of freezing as an accepted preservation technique for VOC samples in D 4547 will increase the usefulness of the En Core device.

A revision of D 4547 will reference D 6418, the ASTM practice for using the En Core sampler, so the reader will know that a separate standard on using the En Core device exists for their use. This will be included in the section of the guide that discusses sample storage in a coring device. D 6418 will also be mentioned in one or two other sections of the revised guide.

It has also been proposed that a revision of D 4547 include reference to a technique for high-level analysis involving sample collection using a modified disposable plastic syringe from which the lower end with the needle attachment and the plunger cap has been removed. The sample is collected in the modified syringe and then immediately extruded into an empty VOA vial for storage of the sample during transportation to the laboratory. This technique is referred to as the “empty VOA vial method.”

Several drafts of a proposed second revision of D 4547 have been reviewed, and comments have been provided. A number of proposed revisions have been balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Main Committee D 34 on Waste Management; however, none of these have passed balloting. As a result, work continues on achieving ASTM approval of a second revision of this standard.

Development of a Subsurface Sampling/Storage Device for VOC Analysis

With participation by WRI, the project cosponsor, En Chem, Inc., entered into an agreement with Geoprobe, Inc. to develop and test a new subsurface VOC sampler. A prototype design for the device was selected, and a mold for the design was to be made. WRI was scheduled to start testing the performance of the subsurface sampler in the fall of 2001. However, a reevaluation of the product market by Geoprobe showed that the market is moving away from use of the size of sampling tube for which the sampler was designed. As a result, the design of the sampler was modified so it can be used with the sampling tube identified as now being one of the most used in the industry. Tests using the redesigned subsurface sampler have been performed. Currently, a mold for the new design is being made. Future work by WRI will include evaluation of the sampler for its performance to store VOC-spiked soils and work towards inserting descriptions of the device and its use in appropriate ASTM standards and/or other methods used by the soil sampling industry.

RESULTS AND DISCUSSION

Testing to Estimate the Precision of the Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs

The data generated in this study are specific to the experimental design of the study. They give information on the performance of the En Core sampler for storing soil samples collected from loose soil, spiked with a gasoline-saturated water solution containing selected analytes of interest, and then stored under specific storage conditions. The data generated by this testing are also 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.

Precision data for the low-level concentration values of the analytes of interest determined in the VOC-spiked river bank soil stored in the 5-gram En Core samplers are given in Tables A1-A7 in Appendix A. These tables show the analytical standard deviation that was calculated using the data from analysis of the analytical standard, s_a ; the standard deviation data from analysis of the time-zero samples, $s_{t=0}$; the estimated standard deviation due to sample spiking and extrusion, $s_{s/e}$; the standard deviation data from analysis of the stored samples, s ; the estimated standard deviation due to storage of the spiked soil in the En Core samplers, s_{st} ; and the estimated percent relative standard deviation due to storage of the 5-gram soil samples in the En Core samplers. The data presented in Tables A1-A7 are summarized in Table 2. This table shows the estimated percent relative standard deviations of the concentration values of the analytes of interest due to storage of the spiked 5-gram river bank soil samples in the En Core samplers. As shown in Table 2, the estimated percent relative standard deviations are 7% or less for all of the storage conditions, except for $4\pm2^{\circ}\text{C}$ for 14 days. For this storage condition, the estimated percent relative standard deviations of the VOC concentrations due to storage in the En Core samplers range from 5% to 13%.

Precision data for the low-level concentration values of the analytes of interest determined in the VOC-spiked river bank soil stored in the 25-gram En Core samplers are given in Tables A8-A14 in Appendix A. The data presented in these tables are summarized in Table 3. This table shows the estimated percent relative standard deviations of the concentration values of the analytes of interest due to storage of the spiked 25-gram river bank soil samples in the En Core samplers. As shown in Table 3, the estimated percent relative standard deviations are 7% or less for all of the storage conditions, except for storage at $4\pm2^{\circ}\text{C}$ for 48 hours followed by storage at $-12\pm2^{\circ}\text{C}$ for 5 days. For this storage condition, the estimated percent relative standard deviations of the VOC concentrations due to storage in the En Core samplers are 5% or less, except for the estimated value for vinyl chloride, which is 14%.

Precision data for the low-level concentration values of the analytes of interest determined in the VOC-spiked prairie soil stored in the 5-gram En Core samplers are given in Tables A15-A21 in Appendix A. The data presented in these tables are summarized in Table 4. This table shows the estimated percent relative standard deviations of the concentration values of the analytes of interest due to storage of the spiked 5-gram prairie soil samples in the En Core samplers. As shown in Table 4, the estimated percent relative standard deviations are 8% or less for all of the compounds for all but two of the storage conditions. For storage at $4\pm2^{\circ}\text{C}$ for 48 hours followed by storage at $-12\pm2^{\circ}\text{C}$ for 5 days, the estimated percent relative standard deviations are 10% and 11% for the two compounds.

/C for 5 days, the estimated percent relative standard deviations of the VOC concentrations are 0%, except for the estimated value for vinyl chloride, which is 10%, and the estimated value for methylene chloride, which is 5%. For storage at 4 ± 2 /C for 14 days, the estimated percent relative standard deviations of the VOC concentrations due to storage in the En Core samplers are 5% or less, except for the estimated value for o-xylene, which is 11%.

Precision data for the low-level concentration values for the analytes of interest determined in the VOC-spiked prairie soil stored in the 25-gram En Core samplers are given in Tables A22-A28 in Appendix A. The data presented in these tables are summarized in Table 5. This table shows the estimated percent relative standard deviations of the concentration values of the analytes of interest due to storage of the spiked 25-gram prairie soil samples in the En Core samplers. As shown in Table 5, the estimated percent relative standard deviations of the concentrations range from 0% to 6% for storage at -12 ± 2 /C for 7 days, -12 ± 2 /C for 14 days, 4 ± 2 /C for 48 hours followed by storage at -12 ± 2 /C for 5 days, and 4 ± 2 /C for 48 hours followed by storage at -12 ± 2 /C for 12 days. For storage at 4 ± 2 /C for 48 hours, the estimated percent relative standard deviations of the VOC concentrations due to storage also range from 0% to 6%, except for the value for vinyl chloride at 13%, for TCE at 10%, and for o-xylene at 8%. For storage at 4 ± 2 /C for 7 days, the values range from 0% to 7%, except for the value for vinyl chloride at 12%; and for storage at 4 ± 2 /C for 14 days, the values range from 0% to 14%.

The estimated percent relative standard deviations of the low-level concentration values for the analytes of interest due to storage of the spiked soil samples in the En Core samplers can be summarized in terms of storage conditions as follows and as shown in Table 6.

- For storage at -12 ± 2 /C for 7 days, -12 ± 2 /C for 14 days, and 4 ± 2 /C for 48 hours followed by storage at -12 ± 2 /C for 12 days, the estimated percent relative standard deviation values range from 0% to 8%.
- For storage at 4 ± 2 /C for 48 hours, the estimated percent relative standard deviation values range from 0% to 8%, except for the values for vinyl chloride and TCE for the 25-gram prairie soil samples, which are 13% and 10%, respectively.
- For storage at 4 ± 2 /C for 7 days, the estimated percent relative standard deviation values range from 0% to 7%, except for the value for vinyl chloride for the 25-gram prairie soil samples, which is 12%.
- For storage at 4 ± 2 /C for 14 days, the estimated percent relative standard deviation values range from 0% to 14%.
- For storage at 4 ± 2 /C for 48 hours followed by storage at -12 ± 2 /C for 5 days, the estimated percent relative standard deviation values range from 0% to 5%, except for the value for

vinyl chloride for the 25-gram river bank soil samples, which is 14%, and the value for vinyl chloride in the 5-gram prairie soil samples, which is 10%.

Performance of the En Core Sampler to Store Soils Spiked with Low Concentrations of VOCs

In generating the precision data in this study, concentration values of the analytes of interest in the time-zero and stored samples were determined. Since these data were available, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the time-zero samples by calculating average percent recovery values. These values show the performance of the En Core samplers to store the river bank and prairie soil samples spiked with low concentrations of VOCs.

The average percent recovery values for the VOCs of interest from the spiked river bank and prairie soil samples are shown in Tables B1 through B4 in Appendix B. These values are summarized in Table 7, which shows the storage conditions for which all or most of the analytes of interest have 90% or greater or 80% or greater average percent recovery for storage of the spiked river bank and prairie soil samples in the 5-gram and 25-gram En Core devices. Data from this study show that the river bank and prairie soil samples containing VOCs at concentrations of approximately 100 $\mu\text{g}/\text{Kg}$ were stored in the 5-gram and 25-gram En Core samplers at $4\pm2\ ^\circ\text{C}$ for 48 hours, $-12\pm2\ ^\circ\text{C}$ for 7 days, $-12\pm2\ ^\circ\text{C}$ for 14 days, and $4\pm2\ ^\circ\text{C}$ for 48 hours followed by $-12\pm2\ ^\circ\text{C}$ for 5 days with 80% or greater of the original analyte concentrations recovered after storage. In many cases, 90% or greater of the analyte concentrations were recovered for these storage conditions. These findings are very similar to those previously reported for the soils, storage conditions, analytes of interest, and analyte concentrations used in this study (Sorini et al. 2001 and 2002). A table showing similar results to those presented in Table 7 is given in Appendix X1 of ASTM Practice D 6418 (ASTM 2001b).

Proposed Revision of ASTM Practice D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis

Based on the results from the precision validation study, information will be added to ASTM Practice D 6418 on the estimated percent relative standard deviation of low-level VOC concentrations due to storage of spiked soil samples in the En Core samplers. A proposal to include a statement in D 6418 on the estimated precision of the performance of the En Core samplers to store soils containing low concentrations of VOCs under various conditions and to reference an ASTM research report describing the precision validation study will be balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization and ASTM Committee D 34 on Waste Management. If this proposal passes, information on the precision of the performance of the En Core sampler to store VOC-spiked soil will be provided in the ASTM practice.

Proposed Revision of ASTM Guide D 4547, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

As mentioned, it has been proposed that a revision of D 4547 include reference to a technique for high-level analysis involving sample collection using a modified disposable plastic syringe. The sample is collected in the modified syringe and then immediately extruded into an empty VOA vial for storage of the sample during transportation to the laboratory. After the sample is received in the laboratory, methanol is injected through the septum of the VOA vial for methanol extraction and subsequent analysis of the extract. This technique is referred to as the “empty VOA vial method.”

The section in the most recently proposed revision of ASTM Guide D 4547 that describes activities related to methanol injection into VOA vials containing samples received one negative comment during balloting. The negative comment was found to be persuasive, and as a result, changes will be made to the section. The new version of the section will then be reviewed and voted on by subcommittee and main committee members in a future balloting activity.

Development of a Subsurface Sampling/Storage Device for VOC Analysis

Reevaluation of the market for subsurface soil sampling devices by Geoprobe showed that the market is moving away from use of the size of sampling tube for which the subsurface sampling/storage device was designed. As a result, the design of the sampler was modified so it can be used with the sampling tube identified as now being one of the most used in the industry. Tests using the redesigned subsurface sampler have been performed. Currently, a mold for the new design is being made. Future work by WRI will include evaluation of the sampler for its performance to store VOC-spiked soils and work towards inserting descriptions of the device and its use in appropriate ASTM standards and/or other methods used by the soil sampling industry.

CONCLUSIONS

Progress was made towards facilitating national acceptance of the En Core device through the activities performed during the last twelve months. The precision validation study on the performance of the En Core samplers to store soil samples spiked with low concentrations of VOCs was completed, and involvement in the activities related to revising D 4547 continued to help ensure that the En Core sampler is included in the guide and fairly represented.

Data generated in the precision validation study show that the estimated percent relative standard deviations of the low-level VOC concentration values for the analytes of interest due to storage of the spiked river bank and prairie soil samples in the 5-gram and 25-gram En Core samplers, for the most part, range from 0% to 10%, with the exception of slightly higher values of approximately 10% to 14% for vinyl chloride for some of the storage conditions and sample types.

For storage at $4\pm2^{\circ}\text{C}$ for 14 days, these values are also slightly higher for some of the sample types, ranging from 0% to 14%. These estimated percent relative standard deviation values show very good precision of the performance of the En Core devices to store soil samples spiked with low concentrations of a variety of VOCs. Data generated in this study also support previous findings showing average percent recovery values for low-level VOC concentrations in soil samples stored in En Core samplers at 80% or greater for a number of storage conditions.

REFERENCES

American Society for Testing and Materials, 2001a, ASTM Practice D 6418-99, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis. *Annual Book of Standards*, 11.04, 576-584.

American Society for Testing and Materials, 2001b, ASTM Practice D 6418-01, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis. *Annual Book of Standards*, 11.04, in print.

American Society for Testing and Materials, 2001c, ASTM Guide D 4547-98, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds. *Annual Book of Standards*, 11.04, 28-37.

American Society for Testing and Materials, 1997, ASTM Practice D2777-96, Standard Practice for Determination of Precision and Bias of Applicable Methods of Committee D-19 on Water. *Annual Book of Standards*, 11.01, 223-235.

Hewitt, A.D., 1999, Frozen Storage of Soil Samples for VOC Analysis. *Environmental Testing & Analysis*, Vol. 8, No. 5, 18-25.

Sorini, S.S., J.F. Schabron, and J.F. Rovani, Jr., 2002, En Core Sampler Performance: Storing Soil for VOC Analysis. *Soil and Sediment Contamination*, 11(1):19-40.

Sorini, S.S., J.F. Schabron, and J.F. Rovani, Jr., 2001, Validation of a New Soil VOC Sampler: Performance of the En Core Sampler for Storage of Low VOC Concentrations and EPA Method 1311 Volatile Organic Analytes. American Society for Testing and Materials, West Conshohocken, PA. ASTM Research Report D 34-1013.

Sorini, S.S., and J.F. Schabron, 1999a, Performance of the Disposable En Core Sampler for Storing Soil for Volatile Organic Analysis. *Proceedings of the 15th Annual Waste Testing and Quality Assurance Symposium*, Arlington, VA, July 18-22, pp. 129-134.

Sorini, S.S., and J.F. Schabron, 1999b, Development of a Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis. American Society for Testing and Materials, West Conshohocken, PA. ASTM Research Report RR:D34-1012.

REFERENCES (continued)

Turriff, D.E., and C. Klopp, 1995, Comparison of Soil Preservation and Analysis Methods for VOC Analytes. *Proceedings of the Field Screening Methods for Hazardous Wastes and Toxic Chemical International Symposium*, Air & Waste Management Association, pp. 1236-1237.

U.S. EPA, 1996a, Method 8260B: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS). *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol. 1B, Final Update III.

U.S. EPA, 1996b, Method 5030B: Purge-and-Trap for Aqueous Samples. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol. 1B, Final Update III.

U.S. EPA, 1992, Method 1311: Toxicity Characteristic Leaching Procedure. *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846)*, Vol. 1C.

Vitale, R.J., R. Forman, and L. Dupes, 1999, Comparison of VOC Results Between Methods 5030 and 5035 on a Large Multi-State Hydrocarbon Investigation. *Environmental Testing & Analysis*, January/February, pp. 18, 20, 21, 36.

Table 1. Storage Conditions for Testing the Precision of the Performance of the Disposable En Core Samplers to Store Spiked Soil Samples

Storage Temperatures and Times

No Storage (Time-zero)

$4\pm2 ^\circ\text{C}$ in a cooler for 48 hours

$4\pm2 ^\circ\text{C}$ in a cooler for 48 hours then $4\pm2 ^\circ\text{C}$ in a refrigerator for 5 days ($4\pm2 ^\circ\text{C}$ for 7 days)

$4\pm2 ^\circ\text{C}$ in a cooler for 48 hours then $4\pm2 ^\circ\text{C}$ in a refrigerator for 12 days ($4\pm2 ^\circ\text{C}$ for 14 days)

$4\pm2 ^\circ\text{C}$ in a cooler for 48 hours then $-12\pm2 ^\circ\text{C}$ in a freezer for 5 days

$4\pm2 ^\circ\text{C}$ in a cooler for 48 hours then $-12\pm2 ^\circ\text{C}$ in a freezer for 12 days

$-12\pm2 ^\circ\text{C}$ in a freezer for 7 days

$-12\pm2 ^\circ\text{C}$ in a freezer for 14 days

Table 2. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations^a Due to Storage of Spiked River Bank Soil^b Samples in 5-Gram En Core Samplers

Storage Conditions:	$4\pm2 ^\circ\text{C}$ / 48 hr	$4\pm2 ^\circ\text{C}$ / 7 Days	$4\pm2 ^\circ\text{C}$ / 14 Days	$-12\pm2 ^\circ\text{C}$ / 7 Days	$-12\pm2 ^\circ\text{C}$ / 14 Days	$4\pm2 ^\circ\text{C}$ 48 hr/ -12±2 °C 5 Days	$4\pm2 ^\circ\text{C}$ 48 hr/ -12±2 °C 12 Days
---------------------	--------------------------------	---------------------------------	----------------------------------	-----------------------------------	------------------------------------	---	--

VOCs

Vinyl Chloride	4	4	11	0	3	2	3
----------------	---	---	----	---	---	---	---

MeCl ₂	0	5	9	0	5	0	7
MTBE	2	0	8	0	0	0	0
1,1-Di-chloro-ethane	5	1	9	0	6	3	5
CDCE	0	0	11	0	3	0	0
Chloro-form	4	5	5	0	0	0	0
Benzene	0	0	11	0	0	0	0
TCE	0	0	7	0	0	0	0
Toluene	0	0	13	0	0	0	4
PCE	0	2	7	0	0	0	0
Ethyl benzene	0	0	12	2	0	0	4
o-Xylene	0	0	10	3	0	0	0

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^b The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

Table 3. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations^a Due to Storage of Spiked River Bank Soil^b Samples in 25-Gram En Core Samplers

Storage Conditions:	4±2 %/C/ 48 hr	4±2 %/C/ 7 Days	4±2 %/C/ 14 Days	-12±2 %/C/ 7 Days	-12±2 %/C/ 14 Days	4±2 %/C 48 hr/ -12±2 %/C 5 Days	4±2 %/C 48 hr/ -12±2 %/C 12 Days
<u>VOCs</u>							
Vinyl Chloride	0	0	0	0	3	14	3
MeCl ₂	0	0	0	0	0	0	0

MTBE	0	0	4	0	0	3	6
1,1-Di-chloro-ethane	0	0	0	0	4	5	1
CDCE	0	0	4	0	0	0	0
Chloro-form	4	0	0	0	4	4	0
Benzene	3	0	0	0	0	0	0
TCE	5	0	0	0	2	0	0
Toluene	5	0	3	0	0	0	0
PCE	4	0	0	0	4	0	0
Ethyl benzene	5	0	0	0	0	0	0
o-Xylene	7	0	0	0	0	0	0

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^b The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

Table 4. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations^a Due to Storage of Spiked Prairie Soil^b Samples in 5-Gram En Core Samplers

Storage Conditions:	4±2 /C/ 48 hr	4±2 /C/ 7 Days	4±2 /C/ 14 Days	-12±2 /C/ 7 Days	-12±2 /C/ 14 Days	4±2 /C/ 48 hr/ -12±2 /C/ 5 Days	4±2 /C/ 48 hr/ -12±2 /C/ 12 Days
<u>VOCs</u>							
Vinyl Chloride	0	0	0	0	0	10	0
MeCl ₂	0	2	0	0	0	5	0

MTBE	0	4	1	2	0	0	0
1,1-Di-chloro-ethane	0	0	5	1	0	0	6
CDCE	0	0	0	3	0	0	5
Chloro-form	0	0	0	0	0	0	0
Benzene	0	0	0	3	0	0	3
TCE	0	6	0	2	0	0	8
Toluene	0	0	0	0	0	0	0
PCE	0	0	0	0	0	0	5
Ethyl benzene	0	0	0	0	0	0	5
o-Xylene	0	0	11	0	0	0	0

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^b The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

Table 5. Estimated Percent Relative Standard Deviation of Low-Level VOC Concentrations^a Due to Storage of Spiked Prairie Soil^b Samples in 25-Gram En Core Samplers

Storage Conditions:	4±2 %/C/ 48 hr	4±2 %/C/ 7 Days	4±2 %/C/ 14 Days	-12±2 %/C/ 7 Days	-12±2 %/C/ 14 Days	4±2 %/C 48 hr/ -12±2 %/C 5 Days	4±2 %/C 48 hr/ -12±2 %/C 12 Days
<u>VOCs</u>							
Vinyl Chloride	13	12	8	3	3	5	6
MeCl ₂	5	0	10	4	0	0	3
MTBE	6	5	0	4	0	0	4

1,1-Dichloroethane	6	1	10	4	4	0	3
CDCE	5	0	11	3	0	0	1
Chloroform	6	0	0	1	0	0	0
Benzene	0	0	8	0	0	0	0
TCE	10	7	11	0	0	4	3
Toluene	0	0	14	0	0	0	0
PCE	0	7	0	5	0	0	2
Ethyl benzene	0	0	6	0	0	0	0
o-Xylene	8	0	0	4	0	0	0

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^b The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

Table 6. Estimated Percent Relative Standard Deviation of Low-Level VOC^a Concentrations^b Due to Storage of Spiked Soil in En Core Samplers

Storage Condition	Estimated Percent Relative Standard Deviations of Concentration Values Due to Storage in the En Core Samplers			
	<u>5-g RBS^c</u>	<u>25-g RBS</u>	<u>5-g PS^d</u>	<u>25-g PS</u>
4±2 °C for 48 hours	0% - 5%	0% - 7%	0%	0% - 6%, except vinyl chloride at 13%; TCE at 10%; o-xylene at 8%

4±2 °C for 7 days	0% - 5%	0%	0% - 6%	0% - 7%, except vinyl chloride at 12%
4±2 °C for 14 days	5% - 13%	0% - 4%	0% - 5%, except o-xylene at 11%	0% - 14%
-12±2 °C for 7 days	0% - 3%	0%	0% - 3%	0% - 5%
-12±2 °C for 14 days	0% - 6%	0% - 4%	0%	0% - 4%
4±2 °C for 48 hours then -12±2 °C for 5 days	0% - 3%	0% - 5%, except vinyl chloride at 14%	0%, except vinyl chloride at 10%; MeCl ₂ at 5%	0% - 5%
4±2 °C for 48 hours then -12±2 °C for 12 days	0% - 7%	0% - 6%	0% - 8%	0% - 6%

^a Analytes of interest are vinyl chloride, MeCl₂, MTBE, 1,1-dichloroethane, CDCE, chloroform, benzene, TCE, toluene, PCE, ethyl benzene, and o-xylene.

^b Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations ranged from ~700 µg/Kg to ~1,400 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^c River bank soil

^d Prairie soil

Table 7. Summary of Average Percent Recoveries of Low-Level VOCs from River Bank and Prairie Soil Samples Stored in the 5-Gram and 25-Gram En Core Samplers

Soil Type/Sample Size	Average Percent Recovery	Storage Condition	Analytes of Interest
River Bank, 5-g	90% or Greater	4±2 °C for 48 hours 4±2 °C for 7 days -12±2 °C for 7 days -12±2 °C for 14 days 4±2 °C for 48 hours/-12±2 °C for 5 days 4±2 °C for 48 hours/-12±2 °C for 12 days	All ^a All All All All All except MeCl ₂ at 89%

	80% or Greater	4±2 °C for 14 days	All except toluene at 76%
River Bank, 25-g	90% or Greater	4±2 °C for 48 hours 4±2 °C for 7 days	All All except benzene and toluene at 87%
		-12±2 °C for 7 days -12±2 °C for 14 days	All All
		4±2 °C for 48 hours/-12±2 °C for 5 days	All
		4±2 °C for 48 hours/-12±2 °C for 12 days	All
	80% or Greater	4±2 °C for 14 days	All

^aThe analytes of interest are vinyl chloride, MeCl₂, MTBE, 1,1-dichloroethane, CDCE, chloroform, benzene, TCE, toluene, PCE, ethyl benzene, and o-xylene.

Table 7. Summary of Average Percent Recoveries of Low-Level VOCs from River Bank and Prairie Soil Samples Stored in the 5-Gram and 25-Gram En Core Samplers (continued)

Soil Type/Sample Size	Average Percent Recovery	Storage Condition	Analytes of Interest
Prairie, 5-g	90% or Greater	4±2 °C for 48 hours -12±2 °C for 7 days -12±2 °C for 14 days	All ^a except for MeCl ₂ at 86% & o-xylene at 84% All except CDCE & toluene at 87% All except CDCE at 88%

			& toluene at 83%
80% or Greater		4±2 °C for 48 hours/-12±2 °C All for 5 days	
		4±2 °C for 48 hours/-12±2 °C All except for for 12 days	CDCE at 76%, toluene at 69%, & o-xylene at 73%
Prairie, 25-g	90% or Greater	-12±2 °C for 7 days -12±2 °C for 14 days 4±2 °C for 48 hours/-12±2 °C All for 5 days	All All except vinyl chloride at 88% All
80% or Greater		4±2 °C for 48 hours 4±2 °C for 48 hours/-12±2 °C All for 12 days	All

^aThe analytes of interest are vinyl chloride, MeCl₂, MTBE, 1,1-dichloroethane, CDCE, chloroform, benzene, TCE, toluene, PCE, ethyl benzene, and o-xylene.

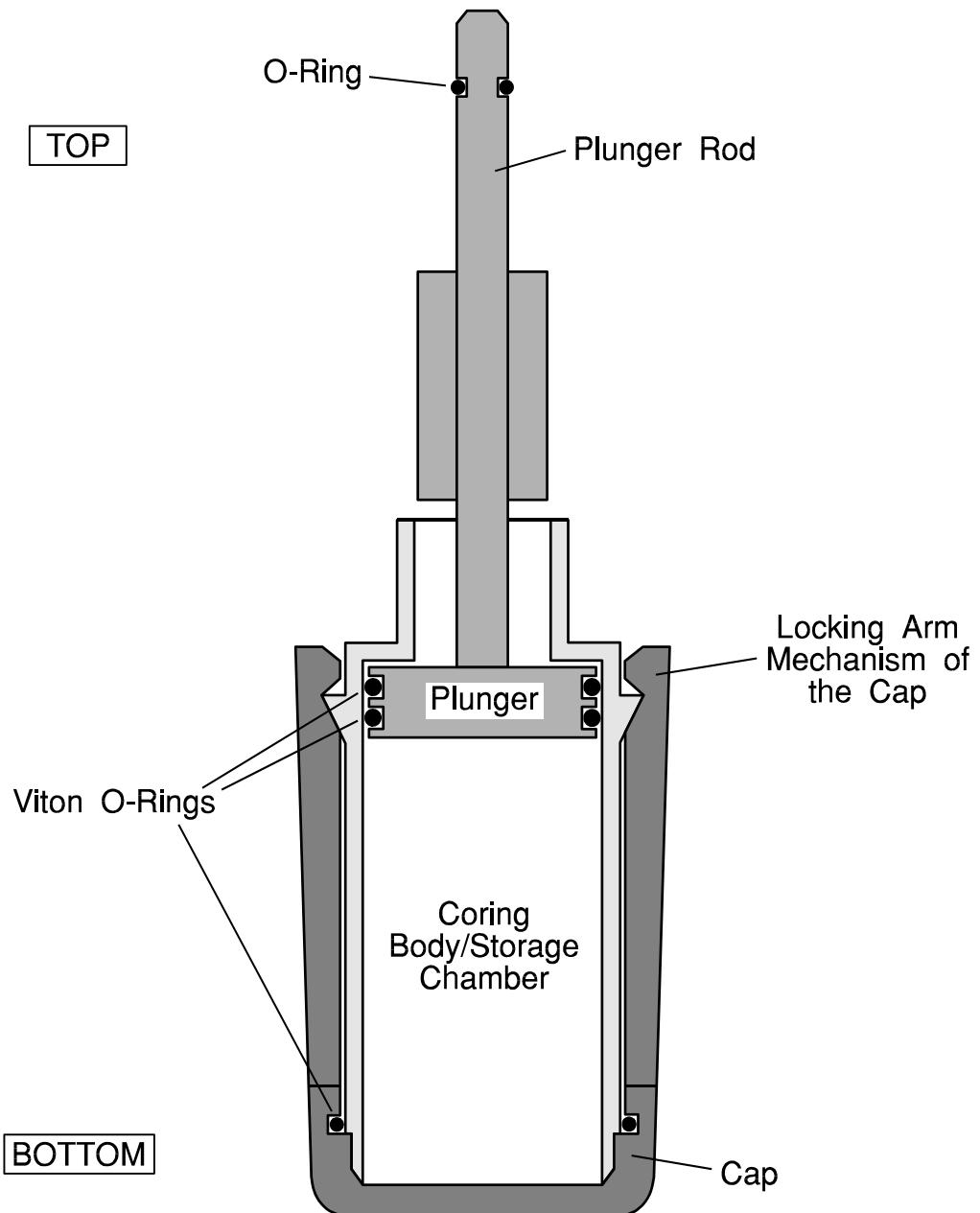


Figure 1.
Components

Com
of the

En Core Sampler

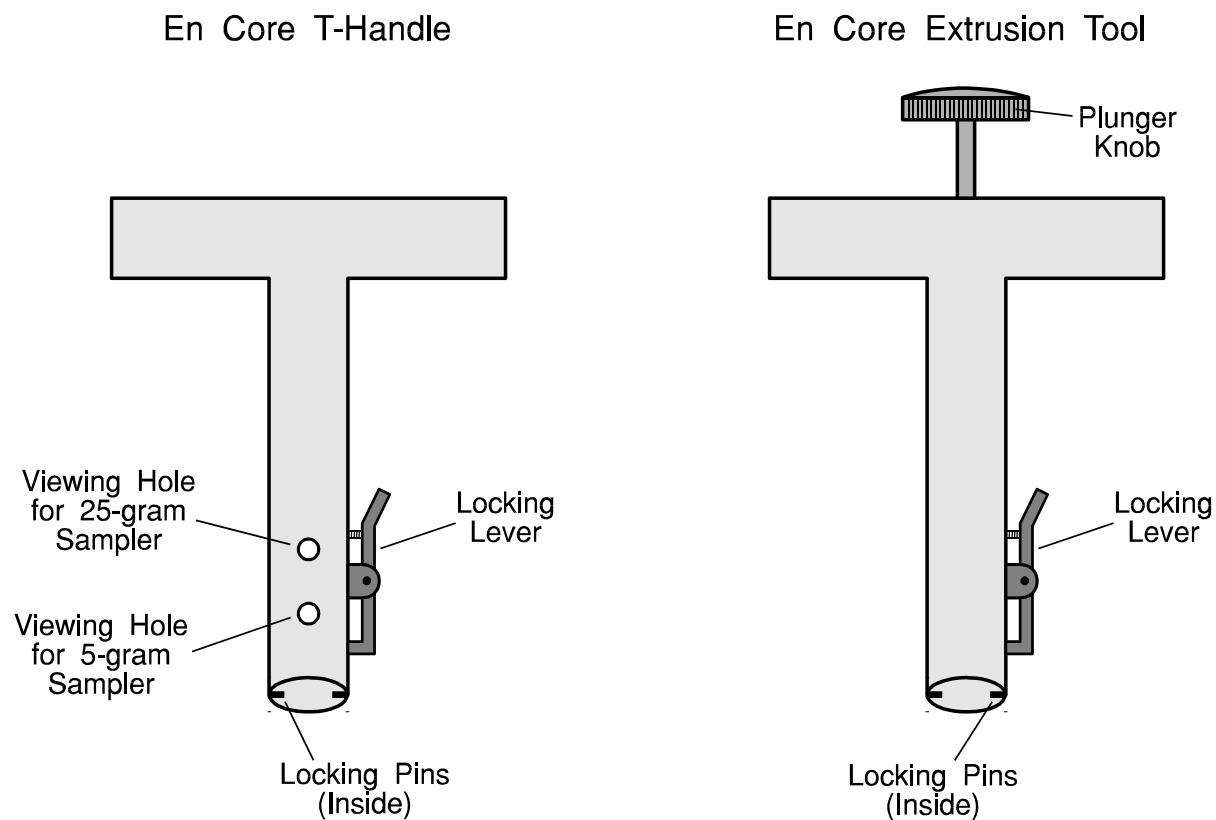


Figure 2. Reusable Attachments to the En Core Sampler

APPENDIX A

Precision Data for the Low-Level Concentration Values of the Analytes of Interest Determined in the VOC-Spiked River Bank and Prairie Soil Samples Stored in the En Core Samplers

Table A1. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C

VOC	s_a^b	$s_{t=0}^c$	$s_{s/e}^d$	s^e	s_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	4.0	3.6	0	5.4	3.6	4
MeCl ₂	4.4	5.7	3.6	5.7	0.4	0.3
MTBE	3.6	8.7	7.9	9.1	2.7	2
1,1-Dichloro-ethane	4.4	4.2	0	7.1	5.6	5
CDCE	5.1	6.7	4.3	4.5	0	0
Chloroform	4.3	6.4	4.7	8.2	5.2	4
Benzene	5.8	8.2	5.8	6.1	0	0
TCE	6.1	6.7	2.8	5.3	0	0
Toluene	51	110	97	74	0	0
PCE	5.9	7.8	5.1	7.4	0	0
Ethyl benzene	3.3	8.0	7.3	4.3	0	0
o-Xylene	7.0	12	9.7	10	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A2. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at 4±2 °C in a Refrigerator

VOC	s_a^b	$s_{t=0}^c$	$s_{s/e}^d$	s^e	s_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	4.0	3.6	0	5.1	3.2	4
MeCl ₂	4.4	5.7	3.6	8.2	5.9	5
MTBE	3.6	8.7	7.9	6.7	0	0
1,1-Dichloro-ethane	4.4	4.2	0	4.7	1.6	1
CDCE	5.1	6.7	4.3	4.1	0	0
Chloroform	4.3	6.4	4.7	9.3	6.8	5
Benzene	5.8	8.2	5.8	1.7	0	0
TCE	6.1	6.7	2.8	4.4	0	0
Toluene	51	110	97	31	0	0
PCE	5.9	7.8	5.1	8.2	2.5	2
Ethyl benzene	3.3	8.0	7.3	3.2	0	0
o-Xylene	7.0	12	9.7	7.4	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A3. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 12 Days at 4±2 °C in a Refrigerator

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	4.0	6.3	4.9	9.6	7.2	11
MeCl ₂	4.4	5.3	3.0	11	9.6	9
MTBE	3.6	6.8	5.8	11	8.6	8
1,1-Dichloro-ethane	4.4	1.3	0	9.6	8.5	9
CDCE	5.1	4.3	0	12	11	11
Chloroform	4.3	8.9	7.8	11	6.4	5
Benzene	5.8	7.0	3.9	15	13	11
TCE	6.1	3.6	0	9.1	6.8	7
Toluene	51	57	25	155	144	13
PCE	5.9	6.7	3.2	10	7.4	7
Ethyl benzene	3.3	2.3	0	14	14	12
o-Xylene	7.0	5.7	0	17	15	10

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A4. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 7 Days at -12±2 °C in a Freezer

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated % Rel. Std. Dev.
-----	---------	-------------	-------------	-------	------------	-------------------------------

	Due to Storage					
Vinyl chloride	4.0	6.3	4.9	2.0	0	0
MeCl ₂	4.4	5.3	3.0	3.6	0	0
MTBE	3.6	6.8	5.8	6.0	0	0
1,1-Dichloro-ethane	4.4	1.3	0	2.0	0	0
CDCE	5.1	4.3	0	4.8	0	0
Chloroform	4.3	8.9	7.8	5.0	0	0
Benzene	5.8	7.0	3.9	2.8	0	0
TCE	6.1	3.6	0	5.4	0	0
Toluene	51	57	26	30	0	0
PCE	5.9	6.7	3.2	4.9	0	0
Ethyl benzene	3.3	2.3	0	4.6	3.2	2
o-Xylene	7.0	5.7	0	8.7	5.2	3

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A5. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 14 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

Vinyl chloride	4.0	3.6	0	4.7	2.5	3
MeCl ₂	4.4	5.7	3.6	8.3	6.0	5
MTBE	3.6	8.7	7.9	7.4	0	0
1,1-Dichloro-ethane	4.4	4.2	0	8.1	6.8	6
CDCE	5.1	6.7	4.3	7.6	3.6	3
Chloroform	4.3	6.4	4.7	4.8	0	0
Benzene	5.8	8.2	5.8	0.6	0	0
TCE	6.1	6.7	2.8	6.2	0	0
Toluene	51	110	97	71	0	0
PCE	5.9	7.8	5.1	3.9	0	0
Ethyl benzene	3.3	8.0	7.3	5.0	0	0
o-Xylene	7.0	12	9.7	7.1	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A6. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at -12 ±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev.
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	----------------------------

	Due to Storage					
Vinyl chloride	4.0	3.6	0	4.2	1.3	2
MeCl ₂	4.4	5.7	3.6	5.5	0	0
MTBE	3.6	8.7	7.9	5.1	0	0
1,1-Dichloro-ethane	4.4	4.2	0	5.3	2.9	3
CDCE	5.1	6.7	4.3	1.0	0	0
Chloroform	4.3	6.4	4.7	6.1	0	0
Benzene	5.8	8.2	5.8	3.8	0	0
TCE	6.1	6.7	2.8	3.9	0	0
Toluene	51	110	97	23	0	0
PCE	5.9	7.8	5.1	4.3	0	0
Ethyl benzene	3.3	8.0	7.3	3.3	0	0
o-Xylene	7.0	12	9.7	3.2	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A7. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C then 12 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	4.0	6.3	4.9	6.8	2.5	3
MeCl ₂	4.4	5.3	3.0	9.4	7.7	7
MTBE	3.6	6.8	5.8	5.0	0	0
1,1-Dichloro-ethane	4.4	1.3	0	7.1	5.6	5
CDCE	5.1	4.3	0	5.1	0	0
Chloroform	4.3	8.9	7.8	6.6	0	0
Benzene	5.8	7.0	3.9	6.6	0	0
TCE	6.1	3.6	0	5.6	0	0
Toluene	51	57	25	80	56	4
PCE	5.9	6.7	3.2	5.3	0	0
Ethyl benzene	3.3	2.3	0	6.7	5.8	4
o-Xylene	7.0	5.7	0	6.7	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A8. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated
Vinyl chloride	5.6	4.9	0	2.9	0	0
MeCl ₂	7.7	5.1	0	7.6	0	0
MTBE	5.4	6.2	3.1	5.5	0	0
1,1-Dichloro-ethane	4.8	3.8	0	3.0	0	0
CDCE	5.8	6.5	2.9	5.0	0	0
Chloroform	6.5	4.1	0	7.8	4.3	4
Benzene	7.4	3.8	0	8.8	4.8	3
TCE	6.2	4.1	0	8.2	5.4	5
Toluene	36	28	0	51	36	5
PCE	7.8	3.5	0	9.2	4.9	4
Ethyl benzene	5.5	4.8	0	8.3	6.2	5
o-Xylene	7.1	3.5	0	14	12	7

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A9. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at 4±2 °C in a Refrigerator

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated
-----	---------	-------------	-------------	-------	------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	4.9	0	3.0	0	0
MeCl ₂	7.7	5.1	0	3.3	0	0
MTBE	5.4	6.2	3.1	4.6	0	0
1,1-Dichloro-ethane	4.8	3.8	0	4.2	0	0
CDCE	5.8	6.5	2.9	4.8	0	0
Chloroform	6.5	4.1	0	6.1	0	0
Benzene	7.4	3.8	0	1.2	0	0
TCE	6.2	4.1	0	2.4	0	0
Toluene	36	28	0	16	0	0
PCE	7.8	3.5	0	4.5	0	0
Ethyl benzene	5.5	4.8	0	2.4	0	0
o-Xylene	7.1	3.5	0	4.9	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A10. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 12 Days at 4±2 °C in a Refrigerator

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	5.9	1.9	3.7	0	0
MeCl ₂	7.7	6.2	0	5.4	0	0
MTBE	5.4	4.3	0	6.7	4.0	4
1,1-Dichloro-ethane	4.8	5.3	2.3	4.8	0	0
CDCE	5.8	4.2	0	6.9	3.7	4
Chloroform	6.5	5.9	0	4.3	0	0
Benzene	7.4	6.8	0	6.4	0	0
TCE	6.2	4.2	0	3.4	0	0
Toluene	36	33	0	40	17	3
PCE	7.8	2.0	0	5.2	0	0
Ethyl benzene	5.5	8.3	6.2	7.4	0	0
o-Xylene	7.1	12	9.7	9.9	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis, and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A11. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 7 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	5.1	0	4.0	0	0
MeCl ₂	7.7	3.6	0	3.9	0	0
MTBE	5.4	5.3	0	4.6	0	0
1,1-Dichloro-ethane	4.8	4.0	0	4.7	0	0
CDCE	5.8	5.4	0	4.1	0	0
Chloroform	6.5	6.3	0	5.2	0	0
Benzene	7.4	5.6	0	4.0	0	0
TCE	6.2	5.1	0	3.8	0	0
Toluene	36	33	0	19	0	0
PCE	7.8	5.9	0	0.5	0	0
Ethyl benzene	5.5	5.6	0.7	2.9	0	0
o-Xylene	7.1	8.4	4.5	4.2	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A12. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 14 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	5.1	0	6.8	3.9	3
MeCl ₂	7.7	3.6	0	7.3	0	0
MTBE	5.4	5.3	0	4.7	0	0
1,1-Dichloro-ethane	4.8	4.0	0	7.1	5.2	4
CDCE	5.8	5.5	0	1.5	0	0
Chloroform	6.5	6.3	0	8.4	5.3	4
Benzene	7.4	5.6	0	6.1	0	0
TCE	6.2	5.1	0	6.6	2.3	2
Toluene	36	32	0	13	0	0
PCE	7.8	5.9	0	9.0	4.5	4
Ethyl benzene	5.5	5.6	1.0	4.3	0	0
o-Xylene	7.1	8.4	4.5	2.5	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A13. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at -12±2 °C in a Freezer

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	4.9	0	14	13	14
MeCl ₂	7.7	5.1	0	4.9	0	0
MTBE	5.4	6.2	3.1	7.1	3.4	3
1,1-Dichloro-ethane	4.8	3.8	0	7.0	5.1	5
CDCE	5.8	6.5	2.9	5.1	0	0
Chloroform	6.5	4.1	0	7.9	4.5	4
Benzene	7.4	3.8	0	5.8	0	0
TCE	6.2	4.1	0	3.1	0	0
Toluene	36	28	0	29	0	0
PCE	7.8	3.5	0	5.8	0	0
Ethyl benzene	5.5	4.8	0	3.2	0	0
o-Xylene	7.1	3.5	0	2.6	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A14. Precision Data for Storage of Low-Level VOC-Spiked River Bank Soil^a in the 25-Gram En Core Samplers for 4±2 °C then 12 Days at -12±2 °C in a Freezer

VOC	s_a^b	$s_{t=0}^c$	$s_{s/e}^d$	s^e	s_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	5.6	5.9	1.9	6.5	2.7	3
MeCl ₂	7.7	6.2	0	4.7	0	0
MTBE	5.4	4.3	0	8.4	6.4	6
1,1-Dichloro-ethane	4.8	5.3	2.2	5.4	1.1	1
CDCE	5.8	4.2	0	4.7	0	0
Chloroform	6.5	5.9	0	5.7	0	0
Benzene	7.4	6.8	0	3.2	0	0
TCE	6.2	4.2	0	4.1	0	0
Toluene	36	33	0	15	0	0
PCE	7.8	2.0	0	5.7	0	0
Ethyl benzene	5.5	8.3	6.2	0.8	0	0
o-Xylene	7.1	12	9.7	5.0	0	0

^a The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked river bank soil in the En Core samplers

Table A15. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C

VOC	s_a^b	$s_{t=0}^c$	$s_{s/e}^d$	s^e	s_{st}^f	Estimated % Rel. Std. Dev.
-----	---------	-------------	-------------	-------	------------	-------------------------------

	Due to Storage					
Vinyl chloride	4.1	7.7	6.5	1.8	0	0
MeCl ₂	6.5	4.4	0	5.7	0	0
MTBE	4.5	7.8	6.4	2.7	0	0
1,1-Dichloro-ethane	3.1	6.4	5.6	3.2	0	0
CDCE	4.5	7.2	5.6	4.4	0	0
Chloroform	6.3	1.5	0	3.2	0	0
Benzene	5.1	7.8	5.9	4.5	0	0
TCE	2.7	5.8	5.1	2.4	0	0
Toluene	42	81	69	68	0	0
PCE	4.8	8.7	7.3	5.6	0	0
Ethyl benzene	4.6	10	8.9	4.6	0	0
o-Xylene	8.6	13	9.7	10	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion (a calculated value)

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers (a calculated value)

Table A16. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at 4±2 °C in a Refrigerator

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	4.1	7.7	6.5	5.9	0	0
MeCl ₂	6.5	4.4	0	6.8	2.0	2
MTBE	4.5	7.8	6.4	9.2	4.8	4
1,1-Dichloro-ethane	3.1	6.4	5.6	4.1	0	0
CDCE	4.6	7.2	5.5	5.8	0	0
Chloroform	6.3	1.5	0	6.3	0	0
Benzene	5.1	7.8	5.9	5.8	0	0
TCE	2.7	5.8	5.1	6.6	3.2	6
Toluene	42	81	69	40	0	0
PCE	4.8	8.7	7.3	8.1	0	0
Ethyl benzene	4.6	10	8.9	8.4	0	0
o-Xylene	8.6	13	9.7	10	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion (a calculated value)

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers (a calculated value)

Table A17. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 12 Days at 4±2 °C in a Refrigerator

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	-----------

						% Rel. Std. Dev. Due to Storage
Vinyl chloride	4.1	4.5	1.9	3.3	0	0
MeCl ₂	6.5	3.7	0	5.6	0	0
MTBE	4.5	4.2	0	4.6	1	1
1,1-Dichloro- ethane	3.1	2.2	0	4.5	3.3	5
CDCE	4.6	1.8	0	4.3	0	0
Chloroform	6.3	2.0	0	5.7	0	0
Benzene	5.1	6.8	4.5	5.8	0	0
TCE	2.7	5.3	4.6	4.6	0	0
Toluene	42	85	74	35	0	0
PCE	4.8	10	8.8	6.1	0	0
Ethyl benzene	4.6	9.8	8.7	7.8	0	0
o-Xylene	8.6	10	5.1	12	6.6	11

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion (a calculated value)

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers (a calculated value)

Table A18. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 7 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

Vinyl chloride	4.1	4.5	1.9	2.2	0	0
MeCl ₂	6.5	3.7	0	6.3	0	0
MTBE	4.5	4.2	0	4.9	1.9	2
1,1-Dichloro-ethane	3.1	2.2	0	3.2	0.8	1
CDCE	4.6	1.8	0	5.4	2.8	3
Chloroform	6.3	2.0	0	5.7	0	0
Benzene	5.1	6.8	4.5	7.7	3.6	3
TCE	2.7	5.3	4.6	5.6	1.7	2
Toluene	42	85	74	53	0	0
PCE	4.8	10	8.8	5.9	0	0
Ethyl benzene	4.6	9.8	8.7	4.7	0	0
o-Xylene	8.6	10	5.1	6.1	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A19. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 14 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

Vinyl chloride	5.7	6.4	2.9	4.1	0	0
MeCl ₂	4.6	7.1	5.4	2.6	0	0
MTBE	6.1	8.1	5.3	5.9	0	0
1,1-Dichloro-ethane	5.7	6.8	3.7	4.1	0	0
CDCE	6.5	7.3	3.3	2.9	0	0
Chloroform	5.9	6.9	3.6	2.7	0	0
Benzene	7.5	9.3	5.5	2.7	0	0
TCE	5.7	8.1	5.8	3.9	0	0
Toluene	44	64	47	20	0	0
PCE	5.5	7.2	4.7	3.7	0	0
Ethyl benzene	5.7	8.4	6.2	3.1	0	0
o-Xylene	12	14	7.2	3.9	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A20. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

Vinyl chloride	4.1	7.7	6.5	9.3	5.2	10
MeCl ₂	6.5	4.4	0	8.2	4.9	5
MTBE	4.5	7.8	6.4	2.5	0	0
1,1-Dichloro-ethane	3.1	6.4	5.6	4.6	0	0
CDCE	4.6	7.2	5.5	4.4	0	0
Chloroform	6.3	1.5	0	5.3	0	0
Benzene	5.1	7.8	5.9	5.6	0	0
TCE	2.7	5.8	5.1	2.7	0	0
Toluene	42	81	69	48	0	0
PCE	4.8	8.7	7.3	5.7	0	0
Ethyl benzene	4.6	10	8.9	6.7	0	0
o-Xylene	8.6	13	9.7	12	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A21. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 5-Gram En Core Samplers for 48 hours at 4±2 °C then 12 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
-----	-----------------------------	-------------------------------	-------------------------------	----------------	------------------------------	---

Vinyl chloride	4.1	4.5	1.9	1.2	0	0
MeCl ₂	6.5	3.7	0	3.2	0	0
MTBE	4.5	4.2	0	2.2	0	0
1,1-Dichloro-ethane	3.1	2.2	0	5.8	4.9	6
CDCE	4.6	1.8	0	5.8	3.7	5
Chloroform	6.3	2.0	0	5.6	0	0
Benzene	5.1	6.8	4.5	7.6	3.4	3
TCE	2.7	5.3	4.6	8.0	6.0	8
Toluene	42	85	74	79	0	0
PCE	4.8	10	8.8	11	4.5	5
Ethyl benzene	4.6	9.8	8.7	11	4.9	5
o-Xylene	8.6	10	5.1	5.8	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A22. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	3.5	3.7	1.2	11	10	13

MeCl ₂	6.0	3.0	0	8.7	6.3	5
MTBE	5.4	3.2	0	8.7	6.8	6
1,1-Dichloro-ethane	4.0	2.9	0	7.1	5.9	6
CDCE	5.4	6.5	3.6	7.9	4.5	5
Chloroform	5.3	1.3	0	8.3	6.4	6
Benzene	4.3	4.4	0.9	2.2	0	0
TCE	2.8	3.5	2.1	9.0	8.3	10
Toluene	29	41	29	17	0	0
PCE	2.6	4.0	3.0	1.8	0	0
Ethyl benzene	2.6	4.6	3.8	2.2	0	0
o-Xylene	5.8	9.2	7.1	16	13	8

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A23. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at 4±2 °C in a Refrigerator

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	5.2	6.1	3.2	11	9.1	12

MeCl ₂	6.2	3.3	0	5.8	0	0
MTBE	3.0	3.1	1.0	6.3	5.4	5
1,1-Dichloro-ethane	4.9	3.2	0	5.0	1.0	1
CDCE	4.5	2.9	0	2.9	0	0
Chloroform	5.4	3.8	0	4.4	0	0
Benzene	2.4	8.4	8.1	7.6	0	0
TCE	2.6	4.4	3.5	7.2	5.7	7
Toluene	17	50	47	31	0	0
PCE	4.7	7.0	5.2	9.2	6.0	7
Ethyl benzene	1.7	6.4	6.2	1.6	0	0
o-Xylene	3.3	9.8	9.2	7.0	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A24. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 12 Days at 4±2 °C in a Refrigerator

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	3.5	4.5	2.8	6.6	4.8	8
MeCl ₂	6.0	3.2	0	11	9.2	10

MTBE	5.4	3.5	0	4.9	0	0
1,1-Dichloro-ethane	4.0	3.0	0	9.2	8.3	10
CDCE	5.4	1.8	0	9.6	7.9	11
Chloroform	5.3	3.4	0	4.7	0	0
Benzene	4.3	4.0	0	9.1	8.0	8
TCE	2.8	1.9	0	7.6	7.1	11
Toluene	29	36	21	62	51	14
PCE	2.6	3.3	2.0	2.0	0	0
Ethyl benzene	2.6	5.0	4.3	6.2	3.6	6
o-Xylene	5.8	8.4	6.1	5.0	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A25. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 7 Days at -12±2 °C in a Freezer

VOC	s_a^b	$s_{t=0}^c$	$s_{s/e}^d$	s^e	s_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	3.5	1.9	0	4.3	2.5	3
MeCl ₂	6.0	4.5	0	8.2	5.6	4
MTBE	5.4	2.4	0	6.9	4.3	4

1,1-Dichloro-ethane	4.0	3.1	0	5.5	3.8	4
CDCE	5.4	4.8	0	6.4	3.4	3
Chloroform	5.3	4.5	0	5.5	1.5	1
Benzene	4.3	7.6	6.3	4.8	0	0
TCE	2.8	6.5	5.9	1.4	0	0
Toluene	29	50	41	11	0	0
PCE	2.6	1.9	0	6.6	6.1	5
Ethyl benzene	2.6	5.1	4.4	3.0	0	0
o-Xylene	5.8	5.8	0	10	8.1	4

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A26. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 14 Days at -12±2 °C in a Freezer

VOC	S _a ^b	S _{t=0} ^c	S _{s/e} ^d	S ^e	S _{st} ^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	3.5	1.9	0	4.3	2.5	3
MeCl ₂	6.0	4.5	0	4.9	0	0
MTBE	5.4	2.4	0	3.4	0	0
1,1-Dichloro-	4.0	3.1	0	5.9	4.3	4

ethane

CDCE	5.4	4.8	0	4.9	0	0
Chloroform	5.3	4.5	0	3.9	0	0
Benzene	4.3	7.6	6.3	7.1	0	0
TCE	2.8	6.5	5.9	5.4	0	0
Toluene	29	50	41	19	0	0
PCE	2.6	1.9	0	2.1	0	0
Ethyl benzene	2.6	5.1	4.4	3.5	0	0
o-Xylene	5.8	5.8	0	3.3	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A27. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C in a Cooler then 5 Days at -12±2 °C in a Freezer

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	5.2	6.1	3.2	7.3	4.0	5
MeCl ₂	6.2	3.3	0	5.3	0	0
MTBE	3.0	3.1	1.0	0.5	0	0
1,1-Dichloro-	4.9	3.2	0	4.1	0	0

ethane

CDCE	4.5	2.9	0	4.0	0	0
Chloroform	5.4	3.8	0	4.7	0	0
Benzene	2.4	8.4	8.1	7.5	0	0
TCE	2.6	4.4	3.5	5.4	3.2	4
Toluene	17	50	47	20	0	0
PCE	4.7	7.0	5.2	5.3	0	0
Ethyl benzene	1.7	6.4	6.2	4.6	0	0
o-Xylene	3.3	9.8	9.2	9.5	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

Table A28. Precision Data for Storage of Low-Level VOC-Spiked Prairie Soil^a in the 25-Gram En Core Samplers for 48 hours at 4±2 °C then 12 Days at -12±2 °C in a Freezer

VOC	S_a^b	$S_{t=0}^c$	$S_{s/e}^d$	S^e	S_{st}^f	Estimated % Rel. Std. Dev. Due to Storage
Vinyl chloride	3.5	4.5	2.8	6.2	4.3	6
MeCl ₂	6.0	3.2	0	6.7	3.0	3
MTBE	5.4	3.5	0	7.1	4.6	4
1,1-Dichloro-ethane	4.0	3.0	0	4.8	2.7	3

CDCE	5.4	1.8	0	5.5	1.0	1
Chloroform	5.3	3.4	0	4.1	0	0
Benzene	4.3	4.0	0	3.7	0	0
TCE	2.8	1.9	0	3.6	2.3	3
Toluene	29	36	21	29	0	0
PCE	2.6	3.3	2.0	3.6	1.5	2
Ethyl benzene	2.6	5.0	4.3	3.2	0	0
o-Xylene	5.8	8.4	6.1	5.8	0	0

^a The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^b Analytical standard deviation determined from 10 analyses of the analytical standard

^c Standard deviation of the time-zero concentration values determined by analyzing the extracts of the 5 time-zero samples (includes standard deviation due to analysis and spiking/extrusion)

^d Estimated standard deviation due to spiking/extrusion

^e Standard deviation of the concentration values determined by analyzing the extracts of the 5 stored samples (includes standard deviation due to analysis, spiking/extrusion, and storage)

^f Estimated standard deviation due to storage of the spiked prairie soil in the En Core samplers

APPENDIX B

Average Percent Recovery Values for the Analytes of Interest from VOC-Spiked River Bank and Prairie Soil Samples Stored in the En Core Samplers

Table B1. Average Percent Recoveries of VOCs^a from Samples of River Bank Soil^b Stored in 5-Gram En Core Samplers

Storage Conditions:	4±2 °C/ 48 hr	4±2 °C/ 7 Days	4±2 °C/ 14 Days	-12±2 °C/ 7 Days	-12±2 °C/ 14 Days	4±2 °C 48 hr/ -12±2 °C 5 Days	4±2 °C 48 hr/ -12±2 °C 12 Days
<u>VOCs</u>							
Vinyl Chloride	109 ^c (6) ^d	106(6)	84(14)	106(2)	113(5)	104(5)	104(8)
MeCl ₂	98(5)	96(7)	80(11)	106(3)	96(7)	94(5)	89(8)
MTBE	101(8)	107(1)	87(10)	107(5)	103(6)	103(4)	97(4)
1,1-Dichloroethane	102(6)	103(4)	90(10)	107(2)	103(7)	99(5)	99(7)
CDCE	98(4)	100(4)	83(12)	103(1)	105(6)	99(1)	97(5)
Chloroform	107(6)	102(7)	97(9)	102(4)	105(4)	104(5)	102(5)

Benzene	97(4)	97(1)	81(12)	98(2)	98(0.4)	101(2)	99(5)
TCE	101(5)	104(4)	85(10)	95(5)	104(6)	101(4)	96(5)
Toluene	100(5)	99(2)	76(14)	103(2)	103(5)	100(2)	100(6)
PCE	102(6)	106(7)	92(9)	100(4)	103(3)	101(4)	102(4)
Ethyl benzene	101(3)	104(2)	85(12)	101(3)	104(4)	104(2)	101(5)
o-Xylene	103(6)	102(4)	81(12)	99(5)	104(4)	99(2)	101(4)

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations were ~1,400 µg/Kg, and o-xylene concentrations were ~175 µg/Kg.

^b The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^c Average percent recovery is based on mean concentration values determined for 5 time-zero samples and 5 stored samples minus outlier data points.

^d The 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 of the tests using the river bank soil in the 5-gram devices.

Table B2. Average Percent Recoveries of VOCs^a from Samples of River Bank Soil^b Stored in 25-Gram En Core Samplers

Storage Conditions:	4±2 °C/ 48 hr	4±2 °C/ 7 Days	4±2 °C/ 14 Days	-12±2 °C/ 7 Days	-12±2 °C/ 14 Days	4±2 °C 48 hr/ -12±2 °C 5 Days	4±2 °C 48 hr/ -12±2 °C 12 Days
VOCs							
Vinyl Chloride	104 ^c (3) ^d	101(3)	92(4)	94(4)	106(6)	99(14)	102(6)
MeCl ₂	101(6)	97(3)	88(5)	93(3)	101(5)	102(4)	96(4)
MTBE	104(5)	98(4)	93(6)	96(4)	107(4)	101(6)	92(8)
1,1-Di-chloro-ethane	104(3)	100(4)	91(5)	95(4)	105(6)	102(6)	96(5)
CDCE	101(5)	96(5)	84(7)	92(4)	102(1)	100(5)	92(4)
Chloro-form	97(6)	96(5)	87(4)	92(4)	98(6)	93(7)	91(5)

Benzene	95(6)	87(1)	81(5)	97(3)	100(4)	93(4)	90(2)
TCE	98(8)	91(2)	91(4)	97(3)	101(6)	94(3)	95(4)
Toluene	98(7)	87(2)	82(7)	92(3)	96(2)	93(4)	92(2)
PCE	97(8)	96(4)	95(5)	94(0)	97(8)	96(5)	96(5)
Ethyl benzene	98(6)	95(2)	93(6)	96(2)	100(3)	97(2)	98(1)
o-Xylene	102(8)	91(3)	89(6)	93(2)	96(1)	95(2)	95(3)

^aConcentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations were ~775 µg/Kg, and o-xylene concentrations were ~175 µg/Kg.

^b The river bank soil is 49% sand, 26% silt, 24% clay, 5.3% organic material, and ~14% moisture.

^c Average percent recovery is based on mean concentration values determined for 5 time-zero samples and 5 stored samples minus outlier data points.

^d The 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 2% to 7% for all of the tests using the river bank soil in the 25-gram devices.

Table B3. Average Percent Recoveries of VOCs^a from Samples of Prairie Soil^b Stored in 5-Gram En Core Samplers

Storage Conditions:	4±2 °C/ 48 hr	4±2 °C/ 7 Days	4±2 °C/ 14 Days	-12±2 °C/ 7 Days	-12±2 °C/ 14 Days	4±2 °C 48 hr/ -12±2 °C 5 Days	4±2 °C 48 hr/ -12±2 °C 12 Days
VOCs							
Vinyl Chloride	105 ^c (4) ^d	96(13)	63(9)	97(4)	92(8)	105(19)	83(3)
MeCl ₂	86(6)	73(8)	72(8)	94(7)	94(3)	86(8)	91(4)
MTBE	102(2)	95(8)	92(4)	100(4)	105(5)	99(2)	98(2)
1,1-Dichloroethane	101(3)	83(5)	65(7)	96(3)	102(4)	102(5)	87(7)
CDCE	93(5)	69(9)	50(8)	87(6)	88(3)	90(5)	76(7)
Chloroform	99(3)	84(7)	68(8)	91(6)	99(3)	100(5)	86(6)

Benzene	96(4)	68(8)	49(10)	93(7)	94(2)	93(5)	87(7)
TCE	95(3)	73(11)	56(10)	96(7)	97(5)	90(4)	92(10)
Toluene	94(11)	45(13)	27(16)	87(7)	83(4)	82(8)	69(14)
PCE	97(6)	75(12)	51(12)	91(7)	102(4)	97(6)	88(13)
Ethyl benzene	90(4)	56(13)	35(17)	93(4)	93(3)	90(6)	83(10)
o-Xylene	84(7)	48(13)	32(19)	90(4)	91(3)	82(9)	73(4)

^a Concentrations of the VOCs in the samples were ~100 µg/Kg, except vinyl chloride concentrations were ~50 µg/Kg, toluene concentrations were ~700 µg/Kg, and o-xylene concentrations were ~150 µg/Kg.

^b The prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^c Average percent recovery is based on mean concentration values determined for 5 time-zero samples and 5 stored samples minus outlier data points.

^d The 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 2% to 16% for all of the tests using the prairie soil in the 5-gram devices.

Table B4. Average Percent Recoveries of VOCs^a from Samples of Prairie Soil^b Stored in 25-Gram En Core Samplers

Storage Conditions:	4±2 °C/ 48 hr	4±2 °C/ 7 Days	4±2 °C/ 14 Days	-12±2 °C/ 7 Days	-12±2 °C/ 14 Days	4±2 °C 48 hr/ -12±2 °C 5 Days	4±2 °C 48 hr/ -12±2 °C 12 Days
VOCs							
Vinyl Chloride	101 ^c (15) ^d	95(14)	80(11)	91(5)	88(4)	91(10)	95(8)
MeCl ₂	97(7)	97(5)	78(12)	98(6)	98(4)	102(4)	91(6)
MTBE	101(8)	98(5)	89(5)	99(6)	101(3)	103(0.4)	96(6)
1,1-Dichloroethane	100(7)	96(5)	81(11)	95(5)	96(5)	103(4)	99(5)
CDCE	93(8)	89(3)	72(13)	92(6)	93(5)	97(4)	92(6)
Chloroform	92(7)	96(4)	82(5)	107(5)	96(4)	102(4)	92(4)
Benzene	90(2)	92(6)	67(9)	96(3)	93(5)	102(6)	88(3)

TCE	93(10)	87(9)	73(12)	91(1)	92(6)	96(6)	91(4)
Toluene	90(2)	68(7)	36(17)	94(1)	94(2)	98(3)	81(4)
PCE	87(2)	86(11)	71(2)	107(6)	97(2)	102(5)	92(4)
Ethyl benzene	84(2)	69(2)	47(10)	98(2)	97(3)	96(4)	86(3)
o-Xylene	88(10)	69(6)	41(6)	97(5)	101(2)	99(6)	85(4)

^aConcentrations of the VOCs in the samples were ~100 µg/Kg, except benzene concentrations were ~150 µg/Kg, toluene concentrations were ~1,000 µg/Kg, and o-xylene concentrations were ~200 µg/Kg.

^bThe prairie soil is 67% sand, 17% silt, 16% clay, 1.5% organic material, and ~8% moisture.

^cAverage percent recovery is based on mean concentration values determined for 5 time-zero samples and 5 stored samples minus outlier data points.

^dThe 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 12% for all of the tests using the prairie soil in the 25-gram devices.