

**VALIDATION OF A NEW SOIL VOC SAMPLER:  
PERFORMANCE OF THE EN CORE<sup>®</sup> SAMPLER AT -7 °C  
AND -2°C AND DEVELOPMENT OF THE ACCU CORE<sup>™</sup>  
SUBSURFACE SAMPLING/STORAGE DEVICE FOR VOC  
ANALYSIS**

**Topical Report**

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## 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<sup>®</sup> sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An ASTM International (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 five grams or 25 grams for volatile organic analysis.

Prior to the study described in this report, D 6418 specified sample storage in the En Core sampler at  $4 \pm 2$  °C for up to 48 hours; at  $-12 \pm 2$  °C for up to 14 days; or at  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-12 \pm 2$  °C for up to five days to minimize loss of volatile compounds due to volatilization and/or biodegradation. The study described in this report was conducted to evaluate the performance of the disposable En Core sampler to store low concentrations of VOCs in soil at  $-7 \pm 1$  °C and  $-21 \pm 2$  °C. In the study, data on the performance of the En Core sampler to store soils spiked with low-level concentrations of VOCs at  $4 \pm 2$  °C for 48 hours followed by storage at  $-7 \pm 1$  °C for five days, at  $-7 \pm 1$  °C for 14 days, at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for five days, and at  $-21 \pm 2$  °C for 14 days were generated. Based on these data, a new revision of D 6418 was prepared and balloted in ASTM. The new revision, which was approved on February 1, 2004, now specifies sample storage in the En Core sampler at  $4 \pm 2$  °C for up to 48 hours;  $-7$  to  $-21$  °C for up to 14 days; or  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days.

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, which is called the Accu Core sampler, is designed so that a soil sample can be collected below the surface using a dual-tube penetrometer and transported to the laboratory for analysis in the same container. During the past year, prototype devices have been tested for their performance in storing soil samples containing low concentrations of VOCs. Evaluation of the various Accu Core prototypes and the design selected for additional validation testing are described in this report.

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## 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<sup>®</sup> sampler is designed to collect and store soil samples in a manner that minimizes loss of contaminants due to volatilization and/or biodegradation. An ASTM International (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. Prior to the study described in this report, sample storage in the device was specified at  $4 \pm 2$  °C for up to 48 hours; at  $-12 \pm 2$  °C for up to 14 days; or at  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-12 \pm 2$  °C for up to five days to minimize loss of volatile compounds due to volatilization and/or biodegradation. Storage of samples in the En Core sampler at  $4 \pm 2$  °C or  $-12 \pm 2$  °C for longer holding times than those listed above was an option, provided it could be shown that the longer storage time did not influence the concentrations of VOCs of interest in the samples or that the data generated by analysis of the samples met the data quality objectives (DQOs).

During development of the ASTM practice, a study was conducted to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg. This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. Results from this study were originally presented in Appendix X1 to D 6418. Since it is believed that information on the performance of the sampler at lower concentrations is more critical to the use of the device, a second study was performed in which soil samples were spiked with concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis. The original D 6418 Appendix X1 was replaced with a new appendix describing the second study on the performance of the En Core sampler to store samples spiked with VOCs at low-level concentrations. As a result, the low-level performance data are included with the revised ASTM practice in Appendix X1, and the ASTM research report describing the high-level study is referenced in the practice. In this way, information from both studies is available to the user.

The storage temperatures used in the high and low-level studies discussed above were  $4 \pm 2$  °C and  $-12 \pm 2$  °C. A study, which is described in this report, has now been conducted to evaluate the performance of the disposable En Core sampler to store low concentrations of VOCs in soil at  $-7 \pm 1$  °C and  $-21 \pm 2$  °C. This study generated data on the performance of the En Core sampler to store soils spiked with low-level concentrations of VOCs at  $4 \pm 2$  °C for 48 hours

followed by storage at  $-7 \pm 1$  °C for five days, at  $-7 \pm 1$  °C for 14 days, at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for five days, and at  $-21 \pm 2$  °C for 14 days. Based on these data, a new revision of D 6418 was prepared and balloted in ASTM. In the new revision, the storage data for  $-7$  and  $-21$  °C have been added to Appendix X1, and storage specifications given in the practice have been changed to  $4 \pm 2$  °C for up to 48 hours;  $-7$  to  $-21$  °C for up to 14 days; or  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days. The new revision of D 6418 was approved on February 1, 2004.

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, which is called the Accu Core sampler, is designed so that a soil sample can be collected below the surface using a dual-tube penetrometer and transported to the laboratory for analysis in the same container. Sampling with the Accu Core can begin either at the ground surface or at a predetermined depth to approximately 60 meters below ground.

During the past year, prototype Accu Core devices have been tested for their performance in storing soil samples containing low concentrations of VOCs. Features of the Accu Core design that were evaluated include length of the Accu Core sampler section, type of septum in the sampler cap, addition of an O-ring to the sampler cap, and use of canisters to house the sampler sections. Testing to evaluate the various Accu Core prototypes and the prototype design selected for additional validation tests are described in this report.

## 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 (Turrieff and Klopp 1995).

### **En Core<sup>®</sup> Sampler**

The En Core<sup>®</sup> 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<sup>™</sup> 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 (WRI) developed an ASTM International (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 2000a). During development of the ASTM practice, a study was conducted to evaluate the performance of the En Core sampler for storage of soil samples spiked with VOCs at high-level concentrations of approximately 2,500 µg/Kg (Sorini and Schabron 1999). This analyte concentration in the soil was selected to limit the influence of the analytical method on the data. Results from this study were originally presented in Appendix X1 of D 6418. However, there were questions on the performance of the En Core sampler for storage of soil samples containing low-level concentrations of VOCs. In response to these questions, a second study was performed in which soil samples were spiked with low-level concentrations of VOCs at approximately 100 µg/Kg and stored under various conditions prior to analysis (Sorini et al. 2001). A proposal to

replace the original D 6418 Appendix X1 with a new appendix describing the second study on the performance of the En Core sampler to store soil samples spiked with VOCs at low-level concentrations was balloted within ASTM and was approved. In this revision of D 6418 (ASTM 2002), the low-level performance data are included with the ASTM practice in Appendix X1, and the ASTM research report describing the high-level study is referenced in the practice. In this way, information from both studies is available to the user. The low-level data were selected for inclusion with the practice, because it is believed that information on the performance of the sampler at lower concentrations is more critical to the use of the device.

A third revision of D 6418 to include  $-12 \pm 2$  °C, along with  $4 \pm 2$  °C, as a specified storage temperature and to provide information on the precision of the performance of the En Core sampler was balloted in 2002. In this revision of D 6418 (ASTM 2003a), sample storage in the device is specified at  $4 \pm 2$  °C for up to 48 hours; at  $-12 \pm 2$  °C for up to 14 days; or at  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-12 \pm 2$  °C for up to five days. Storage of samples in the En Core sampler at  $4 \pm 2$  °C or  $-12 \pm 2$  °C for longer holding times than those listed above 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).

This report describes a study that was performed to provide data for a fourth revision of D 6418. This study was conducted to generate data on the performance of the En Core sampler to store soils spiked with low-level concentrations of VOCs at  $-7 \pm 1$  °C and  $-21 \pm 2$  °C. These data were added to Appendix X1 of ASTM Practice D 6418, and storage specifications given in the practice were changed to  $4 \pm 2$  °C for up to 48 hours,  $-7$  to  $-21$  °C for up to 14 days, or  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days, with an option for longer holding times 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 DQOs. The new revision of D 6418 was balloted within ASTM and was approved on February 1, 2004 (ASTM 2004a).

### **ASTM Standard D 4547**

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 2003b). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 was proposed and approved by ASTM on June 10, 2003 (ASTM 2004b). Prior to approval of this second revision of D 4547, several draft revisions of the guide were reviewed to ensure that the En Core sampler is fairly represented and to help ensure that the revision is technically sound.

## **Accu Core™ Subsurface Sampler**

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, which is called the Accu Core™ sampler, is designed so that a soil sample can be collected below the surface using a dual-tube penetrometer and transported to the laboratory for analysis in the same container. Sampling with the Accu Core can begin either at ground surface or at a predetermined depth down to approximately 60 meters below ground.

During the past year, prototype Accu Core devices have been tested for their performance in storing soil samples containing low concentrations of VOCs. Features of the Accu Core design that were evaluated include length of the Accu Core sampler section, type of septum in the sampler cap, addition of an O-ring to the sampler cap, and use of canisters to house the sampler sections. Testing to evaluate the various Accu Core prototypes and the prototype design selected for additional validation tests are described in this report.

### **Current Work**

This report describes the three activities discussed above. These are (1) testing that was conducted to evaluate the performance of the En Core sampler to store soil containing low concentrations of VOCs at  $-7 \pm 1$  °C and  $-21 \pm 2$  °C and revision of D 6418 to include new data and specifications for storing samples at freezing temperatures; (2) activities related to revision of ASTM Guide D 4547; and (3) Accu Core sampler prototype testing and design selection.

### **OBJECTIVES**

The overall objectives of this task are to facilitate national acceptance of the En Core device for sampling and storing soil for VOC analysis and to validate the performance of the Accu Core subsurface sampler for sampling and storing soil for VOC analysis. The activities that were performed over the last 12 months to help achieve these objectives are described below.

- The performance of the 5-gram and 25-gram En Core samplers to store a soil spiked with low-level concentrations of VOCs at temperatures of  $-7 \pm 1$  °C and  $-21 \pm 2$  °C was evaluated. In this way, the performance of the En Core samplers for a range of freezing temperatures from  $-7$  to  $-21$  °C could be determined.

- ASTM Practice D 6418 was revised to specify sample storage in the En Core devices at  $4 \pm 2^\circ\text{C}$  for up to 48 hours,  $-7$  to  $-21^\circ\text{C}$  for up to 14 days, or  $4 \pm 2^\circ\text{C}$  for up to 48 hours followed by storage at  $-7$  to  $-21^\circ\text{C}$  for up to five days.
- The revision of D 6418 was balloted within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management and the ASTM Society, and was approved.
- 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.
- Prototype Accu Core samplers were tested for their performance in storing soil samples containing low concentrations of VOCs.

## **TECHNICAL APPROACH**

### **Evaluation of the Performance of the En Core Sampler for Sample Storage at $-7$ to $-21^\circ\text{C}$**

This study was designed to evaluate the performance of the 5-gram and 25-gram En Core samplers to store soil spiked with an aqueous solution containing 11 VOCs at temperatures of  $-7 \pm 1^\circ\text{C}$  and  $-21 \pm 2^\circ\text{C}$ . The En Core samplers used in the study were manufactured in 2003. The 5-gram devices that were used represent lot number K113224, and the 25-gram devices that were used represent lot number K113206.

The soil used in the study is a mountain soil that was collected in the mountains east of Laramie, Wyoming. The mountain soil contains 75% sand, 13% silt, 12% clay, 4.3% organic material, and approximately 12% moisture. It is the same mountain soil that was used in previous evaluations of the performance of the En Core samplers.

The VOCs used in the study are methylene chloride ( $\text{MeCl}_2$ ), 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. They have been used in previous evaluations of the performance of the En Core samplers.

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 mountain 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. 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 prepare 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 solution, 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 done to help 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. The 5-gram samples were spiked with 100 µL of spiking solution, and the 25-gram samples were spiked with 500 µL 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, ethyl benzene, and o-xylene. Because of their presence in the gasoline-saturated water, the concentrations of benzene were approximately 200 µg/Kg to approximately 400 µg/Kg, and the concentrations of toluene were approximately 650 µg/Kg to approximately 900 µg/Kg in the spiked soil samples. Additional ethyl benzene and o-xylene were also added to the spiking solution by the gasoline-saturated water. This resulted in an ethyl benzene concentration in the spiked soil samples of approximately 200 µg/Kg and an o-xylene concentration in the spiked soil samples of approximately 300 µg/Kg. After all samples were spiked and capped, five random samples were extruded from each size of En Core sampler into methanol for analysis to give time-zero concentrations of the analytes of interest. The remaining samples were stored under the conditions shown in Table 1. Storage temperatures were monitored to make sure they were at the specified temperature. The temperatures in the coolers used to store the samples at 4 °C were monitored using minimum/maximum thermometers. The temperatures in the freezers used to store the samples at -7 and -21 °C were monitored using mini temperature data loggers.

After the samples were held for the appropriate times, they were extruded into methanol for extraction and analysis. All samples were contacted with methanol for  $24 \pm 4$  hours prior to analysis. The methanol extracts of the samples were analyzed using EPA Methods 8260B (U.S. EPA 1996a) and 5030B (U.S. EPA 1996b). To evaluate the data, 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. Before average percent recovery was calculated, the data sets 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 2000b) and Grubbs (1950). For this study, an average percent recovery of 80% or greater was used to indicate acceptable performance of the En Core samplers to store soil containing low levels of VOCs.

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

The data that were generated in the study to evaluate the performance of the En Core sampler to store soils spiked with low-level VOC concentrations at  $-7 \pm 1$  °C and  $-21 \pm 2$  °C were added to Appendix X1 of ASTM Practice D 6418 as a revision to the practice. In addition, the sample storage conditions specified in the practice were changed to  $4 \pm 2$  °C for up to 48 hours,  $-7$  to  $-21$  °C for up to 14 days, or  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days, with an option for longer holding times 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 DQOs. The new revision of D 6418 was balloted for approval within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society in December 2003.

### **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 2003b). Because of continuing developments in the area of sampling and handling techniques for VOC analysis, a second revision of D 4547 was proposed. Several draft revisions of the guide were reviewed over a number of years 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 were proposed 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.

It was also 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.” Testing has been performed showing that extrusion of high-level samples into empty VOA vials can result in significantly greater VOC losses from samples as compared to losses resulting from sample extrusion directly into methanol (Sorini et al. 2002). As a result, a statement was included in the proposed revision of D 4547 that states “having methanol present in the collection vessel reduces the possibility of VOCs being lost during the transfer step (that is, extrusion of a plug of soil from the sampling tool into the empty vessel).” This revision of D 4547 was balloted for approval within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society.

### **Preliminary Tests to Evaluate the Performance of the Accu Core Sampler Prototypes**

The Accu Core sampler consists of a cylindrical stainless steel sampler section that is designed to fit in a Geoprobe<sup>®</sup> dual-tube penetrometer soil sampling system and two end caps having locking arms. The end caps are constructed of the RTP inert composite polymer. Each end cap has a septum port and contains a Teflon<sup>®</sup> (PTFE) -lined natural rubber septum. When the soil-filled stainless steel sampler section is removed from the Geoprobe liner, the ends of the section are thoroughly cleaned and immediately capped with the Teflon side of the septum in the cap contacting the soil. The caps on the ends of the section lock together for shipment to the laboratory for analysis (see Figure 3). Each Accu Core sampler will be supplied with a protective moisture-proof bag for shipment. The Accu Core sampler is designed to collect a soil sample of approximately 25 grams, based on an assumed soil density of 1.7 g/cm<sup>3</sup>.

During the past year, prototype Accu Core devices were tested for their performance in storing soil samples containing low concentrations of VOCs. Features of the Accu Core design that were evaluated include length of the Accu Core sampler section, type of septum in the sampler cap, addition of an O-ring to the sampler cap, and use of canisters to house the sampler sections. Testing the performance of the Accu Core samplers in the laboratory is challenging because the conditions vary greatly from those encountered in the field. The four sets of tests that were performed during the past year to evaluate the performance of prototype Accu Core devices are described below.

#### **Evaluation of Various Types of Septa in the Accu Core Sampler Cap**

These tests were conducted to evaluate Accu Core sampler seals using four different types of septa: (1) DuPont FEP with foil base on silicone rubber, (2) DuPont Tefzel-lined silicone rubber, (3) standard PTFE on silicone rubber, and (4) the current Accu Core septum consisting of PTFE-lined natural rubber. For the testing, the samplers contained mountain soil spiked with an aqueous solution containing twelve VOCs at low-level concentrations of <200 µg/Kg. The mountain soil is 75% sand, 13% silt, 12% clay, 4.3% organic material, and approximately 7% moisture.

The VOCs used in the study were vinyl chloride, methylene chloride (MeCl<sub>2</sub>), 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.

Using a spatula, soil was added to the open end of a sampler section fitted on the other end with a cap. The soil was compacted in the section using a stainless steel extrusion tool by patting the soil with the piston head of the tool. The end of the section to which soil was added was then capped, and the sampler section was turned over. The cap on the upper end of the sampler section was removed, and the above steps were repeated until a compacted soil plug completely filled the sampler section. After a sampler section was filled with soil, both ends of the section were wiped clean and wrapped with aluminum foil. After soil was added to all of the sampler sections, the soil samples were stored in a walk-in cooler at a temperature of approximately 4 °C, so they were cold when the low-level spiking was performed.

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 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 solution, 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 minimized loss of the low concentrations of the volatile analytes during spiking. For spiking, the aluminum foil was removed from the soil-filled sampler section, the surface of the section was wiped clean, a sampler cap was placed on one end of the sampler section, the spiking solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped so that the two caps on the ends of the section locked together. The samples were spiked with 200 µL 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, ethyl benzene, and o-xylene. Because of their presence in the gasoline-saturated water, the concentrations of these analytes in the soil samples were expected to be greater than 100 µg/Kg.

In performing the testing, a set of 15 samples was spiked on one day, and a set of 15 samples was spiked on a second day. Each set of 15 samples included 5 time-zero samples in samplers having PTFE-lined natural rubber septa and two sets of 5 samples in samplers having seals made with two of the septum types for storage at  $4 \pm 2$  °C for 48 hours. Every third sample that was spiked was a time-zero sample. After all of the samples were spiked, the time-zero samples were immediately extruded into methanol. The stored samples were extruded into methanol after being stored for 48 hours at  $4 \pm 2$  °C. After extrusion, all of the samples were contacted with methanol for  $24 \pm 4$  hours prior to analysis.

The methanol extracts of the samples were analyzed using guidance given in EPA Methods 8260B and 5030B. To evaluate the data, the mean concentrations of the analytes of interest in the stored samples were compared to their mean concentrations in the appropriate time-zero samples by calculating average percent recovery.

#### Evaluation of Accu Core Sampler Caps Having O-rings

These tests were conducted to evaluate Accu Core sampler seals using caps fitted with an inner viton O-ring. Accu Core sampler caps were modified at the University of Wyoming (UW) machine shop by having a groove made inside the caps so an O-ring could be inserted in the groove and provide additional sealing for the caps on the sampler sections. In addition, new sampler sections were cut by the machine shop using higher quality stainless steel than used for the previous sections. The ends of the new sections were also highly polished by the machine shop.

For this testing, the samplers contained the mountain soil that was used in the previously described septa study. In addition, the soil was spiked with an aqueous solution containing the 12 VOCs used in the previous testing at low-level concentrations of <200 µg/Kg. The mountain soil was added to the Accu Core sampler sections for spiking using the same procedure described for the septa study. The spiking solution used for this testing was also prepared as described for the septa testing; samples were spiked in the same manner as the samples in that study.

In performing the O-ring testing, a set of five time-zero samples in the old sampler sections sealed with caps containing the original septa was spiked. In addition, two sets of samples for storage at  $4 \pm 2$  °C for 48 hours were spiked. One of these sets contained six samples in the new polished sampler sections that were capped with caps having O-rings, as well

as septa. The second set contained five samples that were in the new polished sampler sections sealed with caps containing the original septa. Every third sample type was spiked. After all of the samples were spiked, the time-zero samples were immediately extruded into methanol. The stored samples were extruded into methanol after being stored for 48 hours at  $4 \pm 2$  °C. After extrusion, all of the samples were contacted with methanol for  $24 \pm 4$  hours prior to analysis.

The methanol extracts of the samples were analyzed using guidance given in EPA Methods 8260B and 5030B. To evaluate the data, 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.

### Evaluation of Use of Canisters for Accu Core Sample Storage

These tests were conducted to evaluate the use of canisters for storing Accu Core sampler sections during transportation of subsurface samples to the laboratory for analysis. To test the concept, aluminum 35-mm film canisters were used, along with longer stainless steel sections that fill the length of the canisters. The longer sections were prepared by the UW machine shop. The canisters have steel screw-on caps. Samples were not extruded. Instead, when a sample was stored in the canister for the appropriate time, the cap of the canister was unscrewed until it was loose enough to come off when the canister was placed in methanol and swirled. As a result, the sampler section containing the sample, canister, and cap were placed in methanol for extraction prior to analysis.

For this testing, the sampler sections contained the mountain soil that was used in the two previously described studies, and the soil was spiked with an aqueous solution containing the 12 VOCs used in the previous testing at low-level concentrations of  $<200$  µg/Kg. The spiking solution was prepared as previously described.

Five time-zero samples were prepared using the shorter Accu Core sampler sections. To fill the sampler sections for the time-zero samples, soil was added to the open end of a sampler section fitted on the other end with a cap. The soil was compacted in the section using a stainless steel extrusion tool by patting the soil with the piston head of the tool. After a sampler section was filled with soil, it was wiped clean and wrapped with aluminum foil. Four soil samples were prepared for storage in the canisters at  $4 \pm 2$  °C for 48 hours. To fill the sampler sections for the stored samples, soil was added to both ends of the longer stainless steel section with compacting until the section was filled. Each section was wrapped with aluminum foil after it was filled. After soil was added to all of the sampler sections, 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.

The soil samples were spiked in the walk-in cooler at a temperature of approximately 4 C. For spiking the time-zero samples, the aluminum foil was removed from the soil-filled sampler sections, the surface of the sections was wiped clean, and the spiking solution was injected into the middle of the soil plug. The time-zero samples were spiked with 200  $\mu$ L of spiking solution to give an approximate concentration of 100  $\mu$ g/Kg of each analyte of interest, except for benzene, toluene, ethyl benzene, and o-xylene. The soil in the longer sampler sections was spiked after the sections were placed in the canisters. Prior to being placed in the canisters, the sections were removed from the aluminum foil and wiped clean. The soil samples for storage in the canisters were spiked with 800  $\mu$ L of spiking solution to give an approximate concentration of 100  $\mu$ g/Kg of each analyte of interest, except for benzene, toluene, ethyl benzene, and o-xylene.

After the time-zero soil samples were spiked, they were sealed with caps containing the PTFE-lined natural rubber septa. The time zero samples were not extruded. When they were added to methanol for extraction, the caps were removed, and the sampler sections with the spiked soil were added to the methanol. The sampler sections and soil were contacted with the methanol for  $24 \pm 4$  hours prior to analysis.

After a soil sample in the canister was spiked, the canister was sealed with the screw-on lid, which was filled with aluminum foil to take up void space in the cap, and stored in a cooler with cold packs. After storage, the cap of the canister was unscrewed until it was loose enough to come off when the canister was placed in methanol and swirled. The sampler section with the sample, canister, aluminum foil, and cap were contacted with the methanol for  $24 \pm 4$  hours prior to analysis.

The methanol extracts of the samples were analyzed using guidance given in EPA Methods 8260B and 5030B. To evaluate the data, 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.

### Evaluation of Accu Core Performance Using Modified Experimental Design and Longer Sampler Sections

Testing the performance of the Accu Core samplers in the laboratory is challenging because the conditions vary greatly from those encountered in the field. When samples are collected in the field using Accu Core samplers, the soil is highly compacted in the sampler sections, no spiking is required, and the sections are simply removed from the protective sleeve, cut apart, cleaned, and capped. At this point in the testing, it was concluded that the

experimental design of the laboratory testing was affecting the results that were being generated. It was decided that preparation of the soil in the sampler sections should be modified to more closely simulate sections that are filled in the field, and that the soil used in the testing should be more moist than the mountain soil that had been used to more closely simulate the moist soil that is typically collected from the subsurface. Another modification that was made to the experimental design was to use a damp cloth to clean the edges of the sampler sections prior to capping. To make spiking the samples less of a challenge because of the Accu Core sampler section being unsealed at both ends during spiking, the volume of spiking solution used was reduced from 200  $\mu\text{L}$  to 5  $\mu\text{L}$ , and the spiking solution was prepared in methanol, instead of gasoline-saturated water.

In addition to modifying the experimental design for this testing, the Accu Core sampler sections were also modified based on testing that had been performed by En Chem, Inc. The standard length of the Accu Core sampler sections that had been used in previous tests was nominally one inch, except when longer sections were used in the canister testing described above. Tests performed by En Chem showed higher VOC percent recovery values when sampler sections of 1.010 inch were used. As a result, 1.010-inch sections having lathe-turned ends were used in this set of tests.

The VOCs used in the study were vinyl chloride, methylene chloride ( $\text{MeCl}_2$ ), methyl tert-butyl ether (MTBE), 1,1-dichloroethane, *cis*-1,2-dichloroethylene (CDCE), chloroform, trichloroethylene (TCE), tetrachloroethylene (PCE), ethyl benzene, and *o*-xylene. No benzene or toluene was present in the spiking solution because the analytical standard used to prepare the spiking solution was part of the set used previously to prepare spiking solutions made with gasoline-saturated water, for which benzene and toluene are provided by the gasoline.

The Accu Core sampler sections were stacked in sets of seven with shrink wrap holding them together. A moist river bank soil, which is approximately 49 % sand, 26% silt, 24% clay, and contains approximately 20% moisture, was added to the stacked sections. The soil was compacted in the sections using a wooden mallet to strike a dowel that is just smaller in diameter than the inner diameter of the sampler sections. After the sets of the stacked sections were filled with soil, the sections were cut apart and wiped clean. The two end sections from each stack were discarded. To cut the sections apart, they were placed in a section holder provided by En Chem. The shrink wrap was cut using a utility knife, and the sections were separated using a putty knife. The edges of the sections were cleaned using a damp cloth. An Accu Core sampler cap was placed on one end of each of the soil-filled sampler sections, and the sections were placed in a plastic bag inside an air-tight plastic container. The plastic container with the samples was stored in a walk-in cooler at a temperature of approximately 4 °C.

The spiking solution used in the study was prepared by adding approximately 7 mL of high purity methanol to a 10-mL glass vial having a screw-on septum cap. A standard solution in methanol containing the compounds listed above was pipeted into the methanol in the vial, and the volume of the solution in the vial was brought up to 10 mL. The solution was mixed and kept on ice to prevent loss of the volatile analytes.

The samples were spiked in the walk-in cooler at a temperature of approximately 4 °C. Ten samples were spiked. The spiking solution was injected into the middle of the soil plug through the open end of the section, and the section was immediately capped so the two caps on the ends of the section locked together. The samples were spiked with 5 µL of spiking solution to give an approximate concentration of 100 µg/Kg of each analyte of interest in the samples. After all samples were spiked and capped, five random samples were extruded into 25 mL of methanol for analysis to give time-zero concentrations of the analytes of interest. The remaining five samples were stored at  $4 \pm 2$  °C for 48 hours. Storage temperatures were monitored to make sure they were at the specified temperature. After the samples were held for the appropriate time, they were extruded into methanol for extraction and analysis. All samples were contacted with methanol for  $24 \pm 4$  hours prior to analysis. The methanol extracts of the samples were analyzed using guidance given in EPA Methods 8260B and 5030B.

To evaluate the data, 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. Before average percent recovery was calculated, the data sets were evaluated for outlier data points.

## **RESULTS AND DISCUSSION**

### **Evaluation of the Performance of the En Core Sampler for Sample Storage at -7 to -21 °C**

The average concentrations of the VOCs of interest in the spiked 5-gram mountain soil samples stored for 14 days at  $-7 \pm 1$  °C and in the corresponding time-zero samples are shown in Table 2. The average concentrations of the VOCs of interest in the spiked 5-gram mountain soil samples stored for 48 hours at  $4 \pm 2$  °C followed by storage for five days at  $-7 \pm 1$  °C and in the corresponding time-zero samples are shown in Table 3. The average concentrations of the VOCs of interest in the spiked 5-gram mountain soil samples stored for 14 days at  $-21 \pm 2$  °C and in the corresponding time-zero samples are shown in Table 4. The average concentrations of the VOCs of interest in the spiked 5-gram mountain soil samples stored for 48 hours at  $4 \pm 2$  °C followed by storage for five days at  $-21 \pm 2$  °C and in the corresponding time-zero samples are shown in Table 5.

The average percent recovery values for the VOCs of interest from the spiked 5-gram samples of mountain soil for each of the four storage conditions are shown in Tables 2-5. These

values are also listed in Table 6. As shown in Table 6, the VOCs of interest have average percent recovery values greater than 90% for storage of the spiked 5-gram mountain soil samples at  $4 \pm 2$  °C for 48 hours followed by storage at  $-7 \pm 1$  °C for five days and for storage at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for 5 days. In addition, the values listed in Table 6 show that the VOCs of interest have average percent recovery values greater than 80% for storage of the spiked 5-gram mountain soil samples at  $-7 \pm 1$  °C for 14 days and for storage at  $-21 \pm 2$  °C for 14 days.

The average concentrations of the VOCs of interest in the spiked 25-gram mountain soil samples stored for 14 days at  $-7 \pm 1$  °C and in the corresponding time-zero samples are shown in Table 7. The average concentrations of the VOCs of interest in the spiked 25-gram mountain soil samples stored for 48 hours at  $4 \pm 2$  °C followed by storage for five days at  $-7 \pm 1$  °C and in the corresponding time-zero samples are shown in Table 8. The average concentrations of the VOCs of interest in the spiked 25-gram mountain soil samples stored for 14 days at  $-21 \pm 2$  °C and in the corresponding time-zero samples are shown in Table 9. The average concentrations of the VOCs of interest in the spiked 25-gram mountain soil samples stored for 48 hours at  $4 \pm 2$  °C followed by storage for five days at  $-21 \pm 2$  °C and in the corresponding time-zero samples are shown in Table 10.

The average percent recovery values for the VOCs of interest from the spiked 25-gram samples of mountain soil for each of the four storage conditions are shown in Tables 7-10. These values are also listed in Table 11. As shown in Table 11, the VOCs of interest have average percent recovery values greater than 90% for storage of the spiked 25-gram mountain soil samples at  $-7 \pm 1$  °C for 14 days, for storage at  $4 \pm 2$  °C for 48 hours followed by storage at  $-7 \pm 1$  °C for 5 days, for storage at  $-21 \pm 2$  °C for 14 days, and for storage at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for five days.

A summary of the average percent recoveries of the VOCs of interest from the mountain soil stored in the disposable 5-gram and 25-gram En Core samplers determined in the previous low-level study (Sorini et al. 2001) and in this study is shown in Table 12. Based on these average percent recoveries, a decision was made to revise D 6418 to specify storage of soil samples in the En Core sampler at  $4 \pm 2$  °C for up to 48 hours, at  $-7$  to  $-21$  °C for up to 14 days, or at  $4 \pm 2$  °C for up to 48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days.

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

ASTM Practice D 6418 was revised to specify storage of soil samples in the En Core sampler at  $4 \pm 2$  °C for up to 48 hours, at  $-7$  to  $-21$  °C for up to 14 days, or at  $4 \pm 2$  °C for up to

48 hours followed by storage at  $-7$  to  $-21$  °C for up to five days. The practice was also revised to include data on the performance of the En Core sampler to store soils spiked with low-level concentrations of VOCs at  $4 \pm 2$  °C for 48 hours followed by storage at  $-7 \pm 1$  °C for five days, at  $-7 \pm 1$  °C for 14 days, at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for five days, and at  $-21 \pm 2$  °C for 14 days in Appendix X1. The revision was approved by ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society on February 1, 2004. In the concurrent subcommittee/main committee balloting, the revision received 77 affirmative votes, no negative votes, and 77 abstentions. There was a 100% ballot return in the voting. In the concurrent subcommittee/main committee balloting, three comments were received. These were discussed with the voters who submitted them and also with the ASTM task group during a D 34 Main Committee meeting. No changes to the standard were required based on these comments. As a result, the new revision is currently available from ASTM as a single standard and will be published in ASTM Volume 11.04 later in 2004 (ASTM 2004a).

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

As discussed under the technical approach section of this report, a revision of D 4547 was balloted for approval within ASTM Subcommittee D 34.01 on Sampling, Monitoring, and Characterization, ASTM Main Committee D 34 on Waste Management, and the ASTM Society. The revision was approved on June 10, 2003 (ASTM 2004b). Currently, further revision of this guide is listed as an activity on the ASTM Subcommittee D 34.01 activities list; however, no additional revisions have been reviewed or balloted at this time.

### **Preliminary Tests to Evaluate the Performance of the Accu Core Sampler Prototypes**

#### Evaluation of Various Types of Septa in the Accu Core Sampler Cap

Average percent recovery values for the VOCs of interest from the soil samples stored in the Accu Core samplers were all well below 80% when the following four septa types were used in the caps: (1) DuPont FEP with foil base on silicone rubber, (2) DuPont Tefzel-lined silicone rubber, (3) standard PTFE on silicone rubber, and (4) the current Accu Core septum consisting of PTFE-lined natural rubber. As for the previously discussed En Core sampler testing, an average percent recovery of 80% or greater was used to indicate acceptable performance of the Accu Core samplers to store soil containing low levels of VOCs. As a result, under the testing conditions used in this study, the Accu Core samplers with the various septum types did not meet the performance criterion.

#### Evaluation of Accu Core Sampler Caps Having O-rings

Because of the results described above for the septa study, testing was done to evaluate Accu Core sampler performance using caps fitted with an inner viton O-ring. In addition, new sampler sections were prepared for the testing using higher quality stainless steel than was used for the previous sampler sections. The ends of the new sections were also highly polished.

In testing these devices, it was found that the caps having O-rings were difficult to lock together on the Accu Core sampler sections, and in some cases, they were difficult to remove from the sampler sections for sample extrusion. The delay in sealing and extrusion may have caused VOCs to be lost from the samples.

In this testing, average percent recovery values for the VOCs of interest from the samples stored in the Accu Core samplers sealed with only septa in their caps were all below 80%, except for the value for o-xylene, which was at 85% recovery. Average percent recovery values for the VOCs of interest from the samples stored in the Accu Core samplers having septa and O-rings in their caps were also below 80%, except for the values for o-xylene at 87% and ethyl benzene at 80%. Once again, under the testing conditions used in this study, the performance of the Accu Core samplers was determined to be unacceptable.

#### Evaluation of Use of Canisters for Accu Core Sample Storage

These tests were conducted to evaluate the use of canisters for storing Accu Core sampler sections during transportation of subsurface samples to the laboratory for analysis. This approach varies significantly from those tested previously. In addition to storage of the sampler sections in the canisters, samples were not extruded. Instead, when samples were stored in the canisters for the appropriate time, the caps of the canisters were unscrewed until they were loose enough to come off when the canister was placed in a container with methanol and swirled. As a result, the sampler section containing the sample, canister, and cap were placed in methanol for extraction prior to analysis.

Average percent recovery values for the analytes of interest in the canister testing were greater than 80% for MTBE, 1,1-dichloroethane, chloroform, TCE, toluene, PCE, ethyl benzene, and o-xylene. However, for vinyl chloride, MeCl<sub>2</sub>, CDCE, and benzene, average percent recovery values were less than 80%. Because the average percent recovery values for these compounds were below the acceptable level and because this approach differed significantly from the original concept, it was decided that use of canisters for Accu Core sample storage would not be further evaluated.

#### Evaluation of Accu Core Performance Using Modified Experimental Design and Longer Sampler Sections

As stated in the technical approach, at this point in the testing, it was concluded that the experimental design of the laboratory studies was affecting the results that were being generated. It was decided that preparation of the soil in the sampler sections should be modified to more closely simulate sections that are filled in the field, and that the soil used in the testing should be more moist than the mountain soil that had been used to more closely simulate the moist soil that is typically collected from the subsurface. Another modification that was made to the experimental design was to use a damp cloth to clean the edges of the sampler sections prior to capping. To make spiking the samples less of a challenge because of the Accu Core sampler section being unsealed at both ends during spiking, the volume of spiking solution used was reduced to 5  $\mu$ L, and the spiking solution was prepared in methanol, instead of gasoline-saturated water. In addition to modifying the experimental design for this testing, the length of the Accu Core sampler sections was increased from nominally one inch to 1.010 inch, and the edges of the sections were lathe-turned.

The results from analysis of the time-zero and stored samples for the Accu Core samplers having sections of 1.010 inch are shown in Table 13. These data show average percent recoveries of the analytes of interest from the stored samples ranging from 80% for vinyl chloride to 93% for MeCl<sub>2</sub>, 1,1-dichloroethane, and o-xylene. Average percent recovery values for the other compounds of interest are at approximately 90%. These average percent recovery values show that the Accu Core sampler, with the longer sampler section having lathe-turned edges, performed well for storing the moist river bank soil containing low concentrations of VOCs at 4  $\pm$  2 C for 48 hours. It appears that using the longer sampler sections with lathe-turned edges and wiping the edges of the sections with a damp cloth prior to capping resulted in a VOC-tight seal between the edges of the sampler sections and the Teflon-lined natural rubber septa. The 80% recovery of vinyl chloride from the stored samples shows that a VOC-tight seal was achieved. If this was not the case, vinyl chloride, because of its high volatility, would have shown significant loss from the samples during storage.

Previous experiments performed by En Chem, in which 5 $\mu$ L of methanol-based spiking solution was used, showed significant VOC loss from samples when a VOC-tight seal was not achieved. These results show that the methanol in the spiking solution does not serve as an artificial preservative and does not bias results towards the retention of VOCs.

Based on the results from the laboratory tests described above, a decision was made that the Accu Core sampler consisting of a 1.010-inch long, cylindrical, high-quality stainless steel sampler section having lathe-turned edges and two end caps, each having a septum port, locking arms, and a Teflon-lined natural rubber septum, should be tested in field evaluations to generate additional performance data. As a result, the next objective in development and validation of the sampler will be to validate its performance to store contaminated soil samples collected in the field using the Geoprobe dual-tube penetrometer. Information obtained from this activity will be

very important, because it will represent actual use of the device and will eliminate difficulties encountered in performing laboratory testing.

## CONCLUSIONS

### **Evaluation of the Performance of the En Core Sampler for Sample Storage at -7 to -21 °C**

The data given in this report provide information on the performance of the En Core sampler for storage of soil samples containing low concentrations of VOCs at freezing temperatures of  $-7 \pm 1$  °C and  $-21 \pm 2$  °C. Based on the data generated in this study and in the previous low-level study (Sorini et al. 2001), which evaluated sample storage at  $-12 \pm 2$  °C in the En Core samplers, it can be concluded that the En Core device performs well for storing soil containing low concentrations of VOCs over the temperature range of -7 to -21 °C.

In the study described in this report, soil samples containing VOCs at concentrations of approximately 100 µg/Kg were stored in the 5-gram and 25-gram En Core samplers at  $-7 \pm 1$  °C for 14 days, at  $-21 \pm 2$  °C for 14 days; at  $4 \pm 2$  °C for 48 hours followed by storage at  $-7 \pm 1$  °C for five days, and at  $4 \pm 2$  °C for 48 hours followed by storage at  $-21 \pm 2$  °C for up to five days with 80% or greater of the original analyte concentrations recovered after storage. In the previous low-level study (Sorini et al. 2001), soil samples containing VOCs at concentrations of approximately 100 µg/Kg were stored in the 5-gram and 25-gram En Core samplers at  $-12 \pm 2$  °C for 14 days and at  $4 \pm 2$  °C for 48 hours followed by storage at  $-12 \pm 2$  °C for 5 days with 80% or greater of the original analyte concentrations recovered after storage. As a result, it can be concluded that the En Core sampler performs well in storing soil samples for VOC analysis at temperatures ranging from -7 to -21 °C. Based on the results of the En Core evaluations, storage of soil samples in the En Core sampler at  $4 \pm 2$  °C for up to 48 hours, at -7 to -21 °C for up to 14 days, or at  $4 \pm 2$  °C for up to 48 hours followed by storage at -7 to -21 °C for up to five days is specified in D 6418 (ASTM 2004a).

### **Preliminary Tests to Evaluate the Performance of the Accu Core Sampler Prototypes**

The laboratory tests that were conducted to evaluate the performance of the Accu Core sampler to store soil samples containing low levels of VOCs demonstrated that testing the devices by performing spiking experiments in the laboratory is challenging. Based on the results of the laboratory testing, it can be concluded that the Accu Core sampler sections should be 1.010 inches in length with lathe-turned edges to facilitate a VOC-tight seal, and that wiping the edges of the sampler sections with a damp cloth prior to capping the sections is critical to achieving a VOC-tight seal. Based on the laboratory data generated using the 1.010-inch sampler sections in the Accu Core samplers, it can be concluded that the devices hold low-level concentrations of VOCs in soil samples during 48-hour storage at  $4 \pm 2$  °C and that the devices

are ready for field evaluations to generate additional performance data. The Accu Core sampler used in these evaluations will consist of a 1.010-inch long, cylindrical, high-quality stainless steel sampler section having lathe-turned edges and two end caps, each having a septum port, locking arms, and a Teflon-lined natural rubber septum. Information obtained from field evaluations will be very important, because it will represent actual use of the device and will eliminate difficulties encountered in performing laboratory testing.

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**Table 1. Storage Conditions for Testing to Evaluate the Performance of the Disposable En Core Samplers to Store VOC-Spiked Soil at -7 and -21 °C**

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Storage Times and Conditions	Number of Samples
No Storage (Time-zero)	5 samples
14 days at $-7 \pm 1$ °C in a freezer	5 samples
14 days at $-21 \pm 2$ °C in a freezer	5 samples
48 hrs. in a cooler at $4 \pm 2$ °C then 5 days at $-7 \pm 1$ °C in a freezer	5 samples
48 hrs. in a cooler at $4 \pm 2$ °C then 5 days at $-21 \pm 2$ °C in a freezer	5 samples

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**Table 2. Performance of the 5-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at  $-7 \pm 1$  °C for 14 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	%Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	110	2.6	2	97.4	3.4	3	88
MTBE	134	2.4	2	125	3.4	3	93
1,1-Di-chloro-ethane	106	1.6	2	104	3.1	3	98
CDCE	100	4.4	4	96.7	3.4	4	97
Chloro-form	112	2.7	2	108	3.0	3	96
Benzene	313	6.5	2	287	19	7	92
TCE	97.8	4.9	5	93.4	5.7	6	95
Toluene	799	17	2	794	42	5	99
PCE	97.9	3.0	3	95.8	4.5	5	98
Ethyl benzene	200	5.0	3	192	9.7	5	96
O-Xylene	291	8.1	3	298	15	5	102

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 3. Performance of the 5-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at 4 ± 2 °C for 48 Hours Followed by Storage at -7 ± 1 °C for 5 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	% Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	110	2.6	2	106	7.2	7	96
MTBE	134	2.4	2	125	10	8	93
1,1-Di-chloro-ethane	106	1.6	2	107	7.7	7	101
CDCE	100	4.4	4	95.2	6.2	7	95
Chloro-form	112	2.7	2	116	6.1	5	104
Benzene	313	6.5	2	293	21	7	94
TCE	97.8	4.9	5	94.9	5.2	5	97
Toluene	799	17	2	798	39	5	100
PCE	97.9	3.0	3	100	3.9	4	102
Ethyl benzene	200	5.0	3	205	8.9	4	102
O-Xylene	291	8.1	3	302	14	5	104

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 4. Performance of the 5-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at  $-21 \pm 2$  C for 14 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	%Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	121	10	8	108	4.3	4	89
MTBE	138	12	9	140	3.3	2	101
1,1-Di-chloro-ethane	119	11	9	106	4.4	4	89
CDCE	113	8.0	7	101	6.0	6	89
Chloro-form	122	11	9	112	1.8	2	92
Benzene	352	36	10	298	4.6	2	85
TCE	111	10	9	98.0	2.6	3	88
Toluene	857	63	7	758	10	1	88
PCE	111	10	9	100	4.6	5	90
Ethyl benzene	222	15	7	201	3.4	2	91
O-Xylene	324	23	7	287	1.3	0.5	89

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 5. Performance of the 5-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at 4 ± 2 C for 48 Hours Followed by Storage at -21 ± 2 C for 5 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	%Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	121	10	8	115	10	9	95
MTBE	138	12	9	135	7.3	5	98
1,1-Di-chloro-ethane	119	11	9	117	8.3	7	98
CDCE	113	8.0	7	110	13	12	97
Chloro-form	122	11	9	120	9.7	8	98
Benzene	352	36	10	323	26	8	92
TCE	111	10	9	105	8.4	8	95
Toluene	857	63	7	834	53	6	97
PCE	111	10	9	108	6.8	6	97
Ethyl benzene	222	15	7	212	15	7	96
O-Xylene	324	23	7	310	18	6	96

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 6. Average Percent Recoveries of Low-Level<sup>a</sup> VOCs from Samples of Mountain Soil<sup>b</sup> Stored in 5-Gram En Core Samplers**

Storage Conditions:	-7 ± 1 C 14 Days	48Hrs./4 ± 2 C 5 days/-7 ± 1 C	-21 ± 2 C 14 Days	48Hrs./4 ± 2 C 5 days/-21 ± 2 C
<u>VOCs</u>				
MeCl <sub>2</sub>	88 <sup>c</sup> (3) <sup>d</sup>	96(7)	89(4)	95(9)
MTBE	93(3)	93(8)	101(2)	98(5)
1,1-Dichloroethane	98(3)	101(7)	89(4)	98(7)
CDCE	97(4)	95(7)	89(6)	97(12)
Chloroform	96(3)	104(5)	92(2)	98(8)
Benzene	92(7)	94(7)	85(2)	92(8)
TCE	95(6)	97(5)	88(3)	95(8)
Toluene	99(5)	100(5)	88(1)	97(6)
PCE	98(5)	102(4)	90(5)	97(6)
Ethyl benzene	96(5)	102(4)	91(2)	96(7)
O-Xylene	102(5)	104(5)	89(0.5)	96(6)

<sup>a</sup> Concentrations of the VOCs in the samples were ~100 µg/Kg, except for benzene concentrations at ~200 µg/Kg to ~400 µg/Kg, toluene concentrations at ~650 µg/Kg to ~900 µg/Kg, ethyl benzene concentrations at ~200 µg/Kg, and o-xylene concentrations at ~300 µg/Kg.

<sup>b</sup> The mountain soil is 75% sand, 13% silt, 12% clay, 4.3% organic material, and ~12% moisture.

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

<sup>d</sup> 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 10%.

**Table 7. Performance of the 25-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at  $-7 \pm 1$  °C for 14 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	% Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	106	4.6	4	100	3.1	3	94
MTBE	113	4.9	4	119	5.0	4	105
1,1-Di-chloro-ethane	106	3.8	4	102	2.5	2	96
CDCE	97.1	5.1	5	93.2	2.2	2	96
Chloro-form	106	4.2	4	106	2.9	3	100
Benzene	326	16	5	303	11	4	93
TCE	95.8	5.3	6	94.3	3.2	3	98
Toluene	731	27	4	715	11	2	98
PCE	103	4.3	4	100	4.4	4	97
Ethyl benzene	189	7.8	4	183	4.8	3	97
O-Xylene	271	11	4	256	8.2	3	94

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 8. Performance of the 25-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at  $4 \pm 2$  °C for 48 Hours Followed by Storage at  $-7 \pm 1$  °C for 5 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	% Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	106	4.6	4	104	1.7	2	98
MTBE	113	4.9	4	118	4.0	3	104
1,1-Di-chloro-ethane	106	3.8	4	105	3.4	3	99
CDCE	97.1	5.1	5	94.6	1.6	2	97
Chloro-form	106	4.2	4	105	2.2	2	99
Benzene	326	16	5	312	2.4	1	96
TCE	95.8	5.3	6	95.2	4.1	4	99
Toluene	731	27	4	733	16	2	100
PCE	103	4.3	4	104	1.6	2	101
Ethyl benzene	189	7.8	4	176	3.0	2	93
O-Xylene	271	11	4	259	7.5	3	96

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 9. Performance of the 25-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at  $-21 \pm 2$  C for 14 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	%Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	115	12	10	113	6.2	5	98
MTBE	131	11	8	123	1.3	1	94
1,1-Di-chloro-ethane	112	9.6	9	110	5.5	5	98
CDCE	103	12	12	102	5.5	5	99
Chloro-form	116	11	9	115	5.7	5	99
Benzene	216	16	7	215	7.2	3	99
TCE	99.0	6.4	6	95.5	2.8	3	96
Toluene	689	37	5	693	3.5	0.5	101
PCE	100	4.5	5	103	4.6	4	103
Ethyl benzene	211	13	6	206	0.8	0.4	98
O-Xylene	295	14	5	296	1.5	0.5	100

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 10. Performance of the 25-Gram En Core Sampler for Storage of VOC-Spiked Mountain Soil<sup>a</sup> Samples at 4 ± 2 °C for 48 Hours Followed by Storage at -21 ± 2 °C for 5 Days**

	<u>Time-Zero Data<sup>b</sup></u>			<u>Stored Sample Data<sup>b</sup></u>			
	Mean Value	Standard Deviation	% Rel.Std. Deviation	Mean Value	Standard Deviation	% Rel.Std. Deviation	Average % Recovery
MeCl <sub>2</sub>	115	12	10	110	3.8	3	96
MTBE	131	11	8	131	9.7	7	100
1,1-Di-chloro-ethane	112	9.6	9	106	3.0	3	95
CDCE	103	12	12	99.3	3.3	3	96
Chloro-form	116	11	9	113	2.3	2	97
Benzene	216	16	7	213	9.7	5	99
TCE	99.0	6.4	6	97.4	4.2	4	98
Toluene	689	37	5	671	17	3	97
PCE	100	4.5	5	102	3.5	3	102
Ethyl benzene	211	13	6	212	5.7	3	100
O-Xylene	295	14	5	295	5.9	2	100

<sup>a</sup> 75% sand, 13 % silt, 12% clay, 4.3% organic material, and ~12% moisture

<sup>b</sup> Units are µg/Kg soil unless otherwise stated.

**Table 11. Average Percent Recoveries of Low-Level<sup>a</sup> VOCs from Samples of Mountain Soil<sup>b</sup> Stored in 25-Gram En Core Samplers**

Storage Conditions:	-7 ± 1 C 14 Days	48Hrs./4 ± 2 C 5 days/-7 ± 1 C	-21 ± 2 C 14 Days	48Hrs./4 ± 2 C 5 days/-21 ± 2 C
<u>VOCs</u>				
MeCl <sub>2</sub>	94 <sup>c</sup> (3) <sup>d</sup>	98(2)	98(5)	96(3)
MTBE	105(4)	104(3)	94(1)	100(7)
1,1-Dichloroethane	96(2)	99(3)	98(5)	95(3)
CDCE	96(2)	97(2)	99(5)	96(3)
Chloroform	100(3)	99(2)	99(5)	97(2)
Benzene	93(4)	96(1)	99(3)	99(5)
TCE	98(3)	99(4)	96(3)	98(4)
Toluene	98(2)	100(2)	101(0.5)	97(3)
PCE	97(4)	101(2)	103(4)	102(3)
Ethyl benzene	97(3)	93(2)	98(0.4)	100(3)
O-Xylene	94(3)	96(3)	100(0.5)	100(2)

<sup>a</sup> Concentrations of the VOCs in the samples were ~100 µg/Kg, except for benzene concentrations at ~200 µg/Kg to ~400 µg/Kg, toluene concentrations at ~650 µg/Kg to ~900 µg/Kg, ethyl benzene concentrations at ~200 µg/Kg, and o-xylene concentrations at ~300 µg/Kg.

<sup>b</sup> The mountain soil is 75% sand, 13% silt, 12% clay, 4.3% organic material, and ~12% moisture.

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

<sup>d</sup> 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 4% to 6%.

**Table 12. Summary of Average Percent Recoveries of Low-Level VOCs<sup>a</sup> from Mountain Soil<sup>b</sup> Stored in the Disposable En Core Samplers**

Sample Size	Average % Recovery	Storage Condition
5-Gram	90% or Greater	$4 \pm 2$ C for 48 hrs/ $-7 \pm 1$ C for 5 days $4 \pm 2$ C for 48 hrs/ $-12 \pm 2$ C for 5 days $4 \pm 2$ C for 48 hrs/ $-21 \pm 2$ C for 5 days $-12 \pm 2$ C for 7 days
5-Gram	80% or Greater	$4 \pm 2$ C for 48 hrs $-7 \pm 1$ C for 14 days $-12 \pm 2$ C for 14 days $-21 \pm 2$ C for 14 days
25-Gram	90% or Greater	$4 \pm 2$ C for 48 hrs $4 \pm 2$ C for 48 hrs/ $-7 \pm 1$ C for 5 days $4 \pm 2$ C for 48 hrs/ $-12 \pm 2$ C for 5 days $4 \pm 2$ C for 48 hrs/ $-21 \pm 2$ C for 5 days $4 \pm 2$ C for 48 hrs/ $-12 \pm 2$ C for 12 days $-12 \pm 2$ C for 7 days $-7 \pm 1$ C for 14 days $-12 \pm 2$ C for 14 days $-21 \pm 2$ C for 14 days
25-Gram	80% or Greater	$4 \pm 2$ C for 7 days $4 \pm 2$ C for 14 days

<sup>a</sup> VOCs are MeCl<sub>2</sub>, MTBE, 1,1-dichloroethane, CDCE, chloroform, benzene, TCE, toluene, PCE, ethyl benzene, and o-xylene.

<sup>b</sup> The mountain soil is 75% sand, 13% silt, 12% clay, 4.3% organic material, and ~12% moisture.

**Table 13. Accu Core Sampler Testing Using 1.010-Inch Sampler Sections**

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Analyte	Time-Zero Ave. Conc. <sup>a</sup>	48 hrs./4 ± 2 C Ave. Conc. <sup>c</sup>	Average Percent Recovery
Vinyl Chloride	93 (22%) <sup>b</sup>	74 (18%) <sup>d</sup>	80%
MeCl <sub>2</sub>	115 (7%)	107 (7%)	93%
MTBE	118 (6%)	102 (6%)	86%
1,1-Dichloro- ethane	108 (8%)	100 (6%)	93%
CDCE	112 (7%)	97 (3%)	87%
Chloroform	115 (7%)	106 (4%)	92%
TCE	107 (2%)	93 (9%)	87%
PCE	102 (7%)	88 (10%)	86%
Ethyl benzene	109 (9%)	98(5%)	90%
O-xylene	112 (9%)	104 (3%)	93%

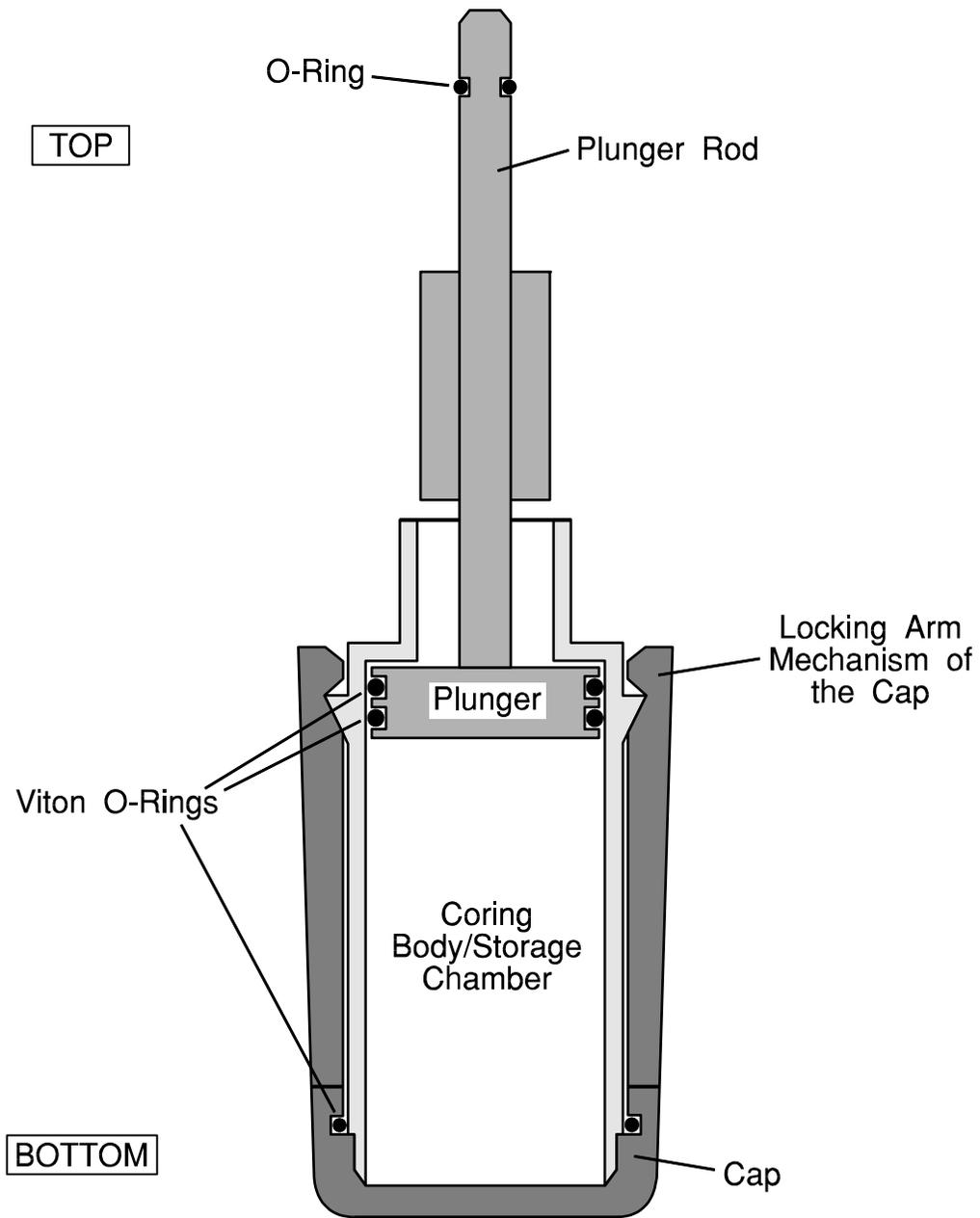
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<sup>a</sup> Average of analyte concentrations in five time-zero samples minus outlier data points, µg/Kg

<sup>b</sup> Percent relative standard deviation of the time-zero concentration values used to calculate the average concentration value

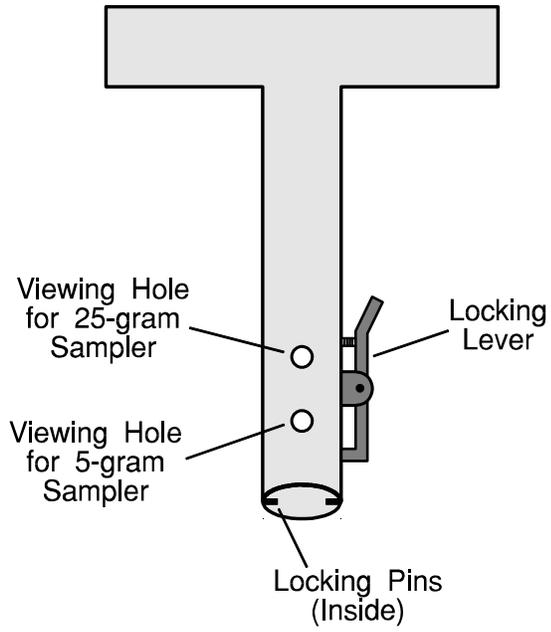
<sup>c</sup> Average of the analyte concentrations in five stored samples minus outlier data points, µg/Kg

<sup>d</sup> Percent relative standard deviation of the stored sample concentration values used to calculate the average concentration value

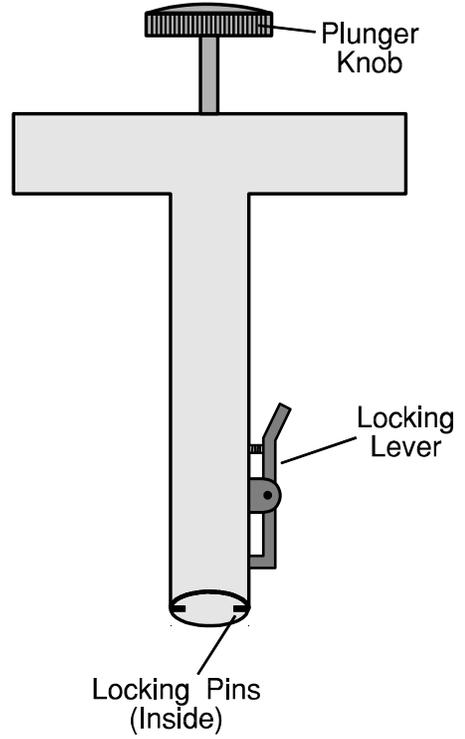


**Figure 1. Components of the En Core Sampler**

En Core T-Handle



En Core Extrusion Tool



**Figure 2. Reusable Attachments to the En Core Sampler**



**Figure 3. Accu Core Sampler Being Sealed**