WRI-07-R011

NEW SOIL VOC SAMPLER: ACCU CORE® SAMPLING/ STORAGE DEVICE FOR VOC ANALYSIS

TOPICAL REPORT

Reporting Period Start 1998 Reporting Period End 2006

By Susan S. Sorini John F. Schabron Mark M. Sanderson

April 2007

DE-FC26-98FT40323 Task 5

For U.S. Department of Energy Office of Fossil Energy National Energy Technology Laboratory Morgantown, West Virginia

And En Novative Technologies, Inc. 1241 Bellview Street, Suite 7 Green Bay, Wisconsin 54302

By Western Research Institute 365 North 9th Street Laramie, Wyoming 82072

Kamalendu Das, Task 5

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 Novative Technologies, 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

The Accu Core[®] sampler system consists of alternating cylindrical clear acrylic sections and one-inch cylindrical stainless steel sections arranged in clear shrink wrap. The set of alternating acrylic and stainless steel sections in the shrink wrap are designed to fit in a Geoprobe[®] dual-tube penetrometer for collection of continuous soil cores. The clear acrylic sections can have ½-inch access holes for easy soil headspace screening without violating the integrity of the adjacent stainless steel sections. The Accu Core sampler system can be used to store a soil sample collected in the stainless steel section by capping the ends of the section so it becomes a sample storage container. The sampler system can also be used to collect a subsurface soil sample in one of the sections that can be directly extruded from the section into a container for storage during shipment to the laboratory. In addition, the soil in a sampler section can be quickly sub-sampled using a coring tool and extruded into a storage container so the integrity of the soil is not disrupted and the potential for VOC loss during sub-sampling is greatly reduced.

A field validation study was conducted to evaluate the performance of the Accu Core sampler to store VOC soil samples during transportation to the laboratory for analysis and to compare the performance of the Accu Core with current sampling and storage techniques, all of which require sub-sampling when the soil sample is brought to the surface. During some of the validation testing, the acrylic sections having access holes for headspace screening were included in the Accu Core sampler configuration and soil in these sections was screened to show the usefulness of the sample screening capability provided by the Accu Core system. This report presents the results of the field validation study as well as recommendations for the Accu Core sampler system.

TABLE OF CONTENTS

Page 1

ACKNOWLEDGEMENTS	ii
DISCLAIMER	ii
ABSTRACT	iii
EXECUTIVE SUMMARY	viii
INTRODUCTION	1
BACKGROUND Accu Core Subsurface Soil Sampling/Storage Device Current Sampling and Storage Techniques for Subsurface Samples	2 2 2
OBJECTIVES	4
TECHNICAL APPROACH Selection of Sampling Locations Sample Collection Sample Shipment to the Laboratory Laboratory Analysis of Field Samples	4 4 6 6
RESULTS AND DISCUSSION Performance Study Screening Results for the Performance Study Samples Comparison Study	7 7 11 12
CONCLUSIONS AND RECOMMENDATIONS Accu Core Sampler Performance Screening Capability Provided by the Accu Core System Comparison Study	14 14 14 14
REFERENCES	15

LIST OF TABLES

<u>Table</u> <u>H</u>	Page
Table 1. Accu Core Validation Study Sampling Locations.	17
Table 2. Accu Core Validation Study Analytes of Interest.	18
Table 3. Accu Core Performance for Location 1: Sample Storage at 4 ± 2 °C for 48 Hours	19
Table 4. Accu Core Performance for Location 2: Sample Storage at 4 ± 2 °C for 48 Hours	20
Table 5. Accu Core Performance for Location 3: Sample Storage at 4 ± 2 °C for 48 Hours	22
Table 6. Accu Core Performance for Location 4: Sample Storage at 4 ± 2 °C for 48 Hours	24
Table 7. Accu Core Performance for Location 5: Sample Storage at 4 ± 2 °C for 48 Hours.	26
Table 8. Accu Core Performance for Location 6: Sample Storage at 4 ± 2 °C for 48 Hours	28
Table 9. Accu Core Performance for Location 7: Sample Storage at 4 ± 2 °C for 48 Hours	30
Table 10. Accu Core Performance for Location 8: Sample Storage at 4 ± 2 °C for 48 Hours.	32
Table 11. Accu Core Performance for Location 9: Sample Storage at 4 ± 2 °C for 48 Hours	34
Table 12. Accu Core Performance for Location 10: Sample Storage at 4 ± 2 °C for 48 Hours.	36
Table 13. Performance Study Sample Sets Showing Percent Recovery Values in the Range o 75 to 126% for the Group of Six Compounds (1,2,4-Trichlorobezene, 1,2,4- Trimethylbenzene, 1,3,5-Trimethylbenzene, Napthalene, p-Isopropyltoluene, and Tetrachloroethene).	
Table 14. Boiling Point Temperatures of Compounds Detected in the Accu Core Performance Study Samples	39

LIST OF TABLES (Continued)

<u>Table</u>	<u>P</u>	Page
Table 15.	Percent Recovery Values for the Compounds in the Performance Study Samples Stored in the Accu Core Samplers for the Locations/Sample Sets Listed in Table 13	40
Table 16.	. Range of Percent Recovery Values for the Group of Six Compounds (1,2,4- Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p- Isopropyltoluene, and Tetrachloroethene) and 1,1,1-Trichloroethane in Soil Store in Accu Core Samplers for Location 2/Sample Set 1, Location 4/Samples Set	
	1, Location 8/Sample Sets 1-3, and Location 9/Sample Set 2	42
Table 17.	Percent Recovery Values for the Compounds in the Performance Study Samples Stored in the Accu Core Samplers for the Locations/Sample Sets Listed in Table 16	43
Table 18.	Accu Core Comparison Study Data for Location 2	45
Table 19.	Accu Core Comparison Study Data for Location 3	46
Table 20.	Accu Core Comparison Study Data for Location 7	47
Table 21.	Accu Core Comparison Study Data for Location 8	48
Table 22.	Accu Core Comparison Study Data for Location 9	49
Table 23.	Accu Core Comparison Study Data for Location 10	50

LIST OF FIGURES

<u>Figure</u>	Page
Figure 1. Example of an Accu Core Configuration: Stainless Steel Sampler Sections Arranged in Alternating Order with Clear Acrylic Sections	51
Figure 2. Accu Core Sampler	52
Figure 3. Accu Core Sampler Sealed for Sample Shipment to the Laboratory	

LIST OF FIGURES (continued)

Figure	Page
Figure 4a.	An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core
Figure 4b.	An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core
Figure 4c.	An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core
Figure 5.	Demonstration of a Soil Sample Being Extruded into an Empty VOA Vial55
-	A Soil Sample That Has Been Extruded from a Coring Tool into a 40 mL VOA Vial Containing Methanol
•	Shown Left to Right, an En Core Sampler T-Handle, a 5-Gram En Core Sampler, a 25-Gram En Core Sampler, and an En Core Sampler Extrusion Tool
-	Locations SB1, SB2, and SB3 in Sludge Beds 9 and 10 Sampled in the Preliminary Study
Figure 9.	Geoprobe Equipment at the Accu Core Validation Study Sampling Site
Figure 10.	Geoprobe Equipment Being Used to Collect a Soil Boring for the Accu Core Validation Study
Figure 11.	Accu Core Liner Configuration for the Soil Borings Collected for the Performance Study
Figure 12.	A Soil Boring Collected for the Accu Core Performance Study
Figure 13.	Accu Core Liner Configuration for the Soil Borings Collected for the Comparison Study
Figure 14.	A Soil Boring Collected for the Accu Core Comparison Study

EXECUTIVE SUMMARY

To date there is no commercially available sampling tool for collecting subsurface soil samples for volatile organic compound (VOC) analysis with minimal loss of VOCs. Current subsurface sampling devices require samples that are collected below the surface be sub-sampled and transferred to a second container in a manner that disrupts sample integrity once the samples are brought to the surface. This can result in significant VOC loss. The Accu Core[®] sampler system consists of alternating cylindrical clear acrylic sections and one-inch cylindrical stainless steel sections arranged in clear shrink wrap. The set of alternating acrylic and stainless steel sections in the shrink wrap are designed to fit in a Geoprobe® dual-tube penetrometer for collection of continuous soil cores. The clear acrylic sections can have 1/2-inch access holes for easy soil headspace screening without violating the integrity of the adjacent stainless steel sections. The Accu Core sampler system can be used to store a soil sample collected in the stainless steel section by capping the ends of the section so it becomes a sample storage container. The sampler system can also be used to collect a subsurface soil sample in one of the sections that can be directly extruded from the section into a container for storage during shipment to the laboratory. In addition, the soil in a sampler section can be quickly sub-sampled using a coring tool and extruded into a storage container so the integrity of the soil is not disrupted and the potential for VOC loss during sub-sampling is greatly reduced.

A field validation study was conducted to evaluate the performance of the Accu Core sampler to store VOC soil samples during transportation to the laboratory for analysis and to compare the performance of the Accu Core with current sampling and storage techniques, all of which require sub-sampling when the soil sample is brought to the surface. During some of the validation testing, the acrylic sections having access holes for headspace screening were included in the Accu Core sampler configuration and soil in these sections was screened using a photoionization detector (PID), and for some samples an X-Wand[®] device, to show the usefulness of the sample screening capability provided by the Accu Core system. This report describes the Accu Core field validation study and provides the data that were generated by the study. Conclusions concerning performance of the Accu Core sampling/storage system based on the validation study results are presented.

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).

To date there is no sampling tool for collecting subsurface soil samples for VOC analysis with minimal loss of VOCs. The new Accu Core[®] sampler system consists of alternating cylindrical clear acrylic sections and one-inch cylindrical stainless steel sections arranged in clear shrink wrap. The set of alternating acrylic and stainless steel sections in the shrink wrap are designed to fit in a Geoprobe[®] dual-tube penetrometer for collection of continuous soil cores. The clear acrylic sections can have ¹/₂-inch access holes for easy soil headspace screening without violating the integrity of the adjacent stainless steel sections. The Accu Core sampler system can be used to store a soil sample collected in the stainless steel section by capping the ends of the section so it becomes a sample storage container. The sampler system can also be used to collect a subsurface soil sample in one of the sections that can be directly extruded from the section into a container for storage during shipment to the laboratory. In addition, the soil in a sampler section can be quickly sub-sampled using a coring tool and extruded into a storage container so the integrity of the soil is not disrupted and the potential for VOC loss during sub-sampling is greatly reduced.

Laboratory testing performed by Western Research Institute (WRI) to evaluate the performance of the capped Accu Core stainless steel sections to store soil spiked with VOCs shows acceptable performance of the Accu Core sampler to hold VOCs during sample storage (Sorini et al. 2004). As the next step in evaluation of the sampler as a storage device, the field testing described in this report was conducted to provide quantitative data on the performance of the device to store subsurface soil samples during transportation to the laboratory and to compare Accu Core performance with current sampling and storage practices for subsurface samples, all of which require sub-sampling when the soil sample is brought to the surface. During some of the validation testing, the acrylic sections having access holes for headspace screening were included in the Accu Core sampler configuration and soil in these sections was screened using a photoionization detector (PID), and for some samples an X-Wand[®] device, to show the usefulness of the sample screening capability provided by the Accu Core system.

This report describes the Accu Core field validation study and provides the data that were generated by the study. Conclusions concerning performance of the Accu Core sampling/storage system based on the validation study results are presented.

BACKGROUND

Accu Core Subsurface Soil Sampling/Storage Device

The Accu Core sampler system typically consists of six alternating three-inch clear acrylic cylindrical sections and one-inch cylindrical stainless steel sections arranged in clear shrink wrap. However, the sampler sections can be arranged in many different configurations, depending on sampling requirements. An example of Accu Core stainless steel sampler sections arranged in alternating order with one-inch clear acrylic sections is shown in Figure 1. The sampler sections in the shrink wrap are designed to fit in a Geoprobe dual-tube penetrometer soil sampling system for subsurface soil sample collection. Sample collection may begin either from ground surface or a predetermined depth down to approximately 60 meters below ground. The Accu Core sampler system can be used to store a soil sample collected in the stainless steel section by capping the ends of the section so it becomes a sample storage container. The sampler system can also be used to collect a subsurface soil sample in one of the sections that can be directly extruded from the section into a container for storage during shipment to the laboratory. In addition, the soil in a sampler section can be quickly sub-sampled using a coring tool and extruded into a storage container so the integrity of the soil is not disrupted and the potential for VOC loss during sub-sampling is greatly reduced.

For use of the system to collect and store soil samples, the Accu Core sampler is designed to collect approximately 25-gram soil samples. After sample collection, the sections are removed from the Geoprobe liner and the shrink wrap is cut to expose the sections. If sample screening is desired, each of the clear acrylic sections can have a ¹/₂-inch access hole for easy soil headspace screening without violating the integrity of the soil in the adjacent stainless steel section. The stainless steel sample sections selected for laboratory analysis are removed from the shrink wrap, and the ends of the sections are thoroughly cleaned and immediately capped. For each stainless steel section, there are two end caps having locking arms. Each cap has a septum port and contains a polytetrafluoroethylene (PTFE)-lined natural rubber septum. The components of the Accu Core sampler are shown in Figure 2. When a stainless steel section is capped, the PTFE side of the septum in the cap contacts the soil. The caps on the ends of the section lock together for shipment to the laboratory for analysis. When the Accu Core stainless steel sample sections are sealed, they immediately become sample storage containers. A sealed Accu Core sampler is shown in Figure 3. To minimize loss of volatile compounds due to volatilization, biodegradation, or both, from the time of collection until analysis or chemical preservation in the laboratory, samples are stored at 4 ± 2 °C for up to 48 hours.

Current Sampling and Storage Techniques for Subsurface Samples

There are often several steps to soil sampling, particularly if it involves obtaining a sample from bulk material collected from the subsurface. Most of the equipment used to obtain samples from the subsurface was originally developed for the geotechnical industry. These subsurface bulk sample retrieval systems are designed to obtain intact cylindrical cores of soil,

ranging anywhere from one to four inches in diameter, and one to several feet in length (ASTM 2006a).

Subsurface soil cores for VOC characterization are to be collected as quickly as possible and are to remain intact and undisturbed until they are sub-sampled. Sub-sampling a bulk soil sample should occur within two minutes of the sample being brought to the surface. Before sampling, several centimeters of the soil core should be removed to expose a fresh surface for sampling. A hand-operated coring tool is used to collect samples of the appropriate size for analysis from the soil core. If one of these coring devices is used to store the sample for more than a few seconds, the main body of the device should be constructed of materials that are nonreactive and have airtight seals that show limited sorption and penetration of VOCs. When the coring device is used only to rapidly transfer the sample to another container, requirements for the coring device material are less stringent (ASTM 2006a). Examples of various coring tools that are used to collect a soil sample from a soil core and transfer the sample to another container are shown in Figures 4a, 4b, and 4c.

If a coring tool is used to collect and transfer a sample of soil from a soil core, the sample can be extruded from the device into an empty volatile organic analysis (VOA) vial for storage during transportation to the laboratory (see Figure 5), or the sample can be extruded into a VOA vial or other glass container having an airtight seal to which methanol has been added for storage during transportation to the laboratory (see Figure 6) (ASTM 2006a).

Another option for collection and storage of a sample from a soil core is to use the En Core[®] sampler, which is designed to collect and store a soil sample for volatile organic analysis. In past years under this task and the task that preceded it, WRI developed an ASTM International (ASTM) standard practice for using the En Core device and performed numerous studies to validate the performance of the device. The ASTM practice for using the En Core sampler is D 6418, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis (ASTM 2006b). The En Core 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. The seals of the device are provided by three Viton[™] O-rings. The coring body/storage chamber, plunger, and cap of the En Core sampler are constructed of a glass-filled inert composite polymer, polyphthalamide (PPA), making the device chemically compatible with soil matrices and contaminants. The device 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. A 5 and 25-gram En Core sampler, along with a T-handle and extrusion tool, are shown in Figure 7.

During the initial 48-hour sample storage and transportation period, the current sampling and storage techniques described above require samples be cooled to at least 4 ± 2 °C (ASTM

2006a). As previously mentioned, all of the current sampling and storage practices for subsurface samples require sub-sampling when the soil sample is brought to the surface.

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 objective of the work performed over the last 12 months was to conduct field evaluation of the Accu Core subsurface soil sampling/storage device to show performance of the device in the field and to compare Accu Core performance with current sampling and storage techniques for subsurface soil samples. Performance data obtained from field validation are very important because these data represent actual use of the device. The validation study was designed so the performance of the laboratory could be evaluated and so the performance of the device could be compared with current sampling and storage techniques for subsurface sampling and storage techniques for subsurface of the sumpling and storage techniques for subsurface of the sampling and storage techniques is brought to the surface.

TECHNICAL APPROACH

Selection of Sampling Locations

The Accu Core field validation study was performed at a VOC-contaminated site located within the North Island U.S. Naval Air Station in San Diego, California. No current information was available on the location or distribution of the VOC contamination at the field site. As a result, a preliminary sampling activity was performed to collect soil samples for analysis prior to the validation study so sampling locations for the study could be selected. This activity involved collection of 18 soil samples from areas designated as Sludge Beds 9 and 10. Older data from the site indicated that the sludge bed areas have high VOC concentrations. The preliminary sampling was performed to determine which VOCs are still present in the soil and to determine their current concentrations at various locations and depths. Figure 8 shows Sludge Beds 9 and 10 and the locations that were sampled during the preliminary sampling activity, which are designated as SB1, SB2, and SB3. At each location, six samples were collected at the following depths: 16, 18, 20, 22, 24, and 26 feet. The soil samples were extruded into methanol and shipped overnight to a commercial analytical laboratory for VOC analysis. Data from analysis of the samples were used to select 10 sampling locations within Sludge Beds 9 and 10 for the Accu Core validation study.

Sample Collection

The Accu Core validation study was divided into two parts. One part of the study involved performance testing to evaluate the performance of the Accu Core sampler to store soil samples for 48 hours at 4 ± 2 °C. The second part of the study involved comparison testing, in

which performance of the Accu Core sampler was compared to performance of current sampling/storage techniques used for subsurface soil samples. For both parts of the study, soil borings were collected using a Geoprobe dual-tube DT-21 penetrometer soil sampling system fitted with equipment to adapt the unit to the Accu Core sampler system. Geoprobe subsurface sampling was performed by personnel from a company that professionally performs direct push sample collection using Geoprobe equipment. Figures 9 and 10 show the Geoprobe equipment being used at the site for the Accu Core validation study.

To evaluate the performance of the Accu Core sampler, 10 soil borings at various locations in Sludge Beds 9 and 10 were collected. The Accu Core liner configuration that was used to collect the soil borings for the performance study is shown in Figure 11. Each soil boring contained soil in three pairs of Accu Core stainless steel sampler sections. The Accu Core sections in the pairs were adjacent to each other. One Accu Core sampler section in each pair was cleaned, capped, and shipped in a cooler at 4 ± 2 °C to the analytical laboratory where the soil in the sampler was extruded into methanol and analyzed. The soil in the second Accu Core sampler section was extruded into a 2-oz glass jar containing 25 mL of high purity methanol at the field site and shipped in a cooler at 4 ± 2 °C to the same analytical laboratory for analysis. Each pair of Accu Core stainless steel sections made up a sample set for which data could be compared. For each of the sample sets, the VOC concentrations determined in the soil shipped in the sealed Accu Core sampler was compared to the VOC concentrations determined in the soil extruded into methanol at the field site. In this way, it can be determined if VOCs were lost from the soil samples stored in the Accu Core samplers for 48 hours at 4 ± 2 °C. As shown in Figure 11, four three-inch acrylic sections having ¹/₂-inch access holes for headspace screening were placed adjacent to the three pairs of Accu Core stainless steel sections to evaluate the screening capability offered by the Accu Core system. The soil in the three-inch acrylic sections having 1/2-inch access holes was screened using a PID, and for some samples an X-Wand device was also used. A PID selectively responds to VOCs having a double bond, and an X-Wand selectively responds to halogenated VOC and does not respond to hydrocarbon fuels or water vapor. Figure 12 shows a soil boring that was collected as part of the performance study. In this photograph, one of the stainless steel sections has been removed for sample preparation. The screening holes in the acrylic sections adjacent to the pairs of stainless steel sections can be seen.

To compare the performance of the Accu Core with current subsurface sampling and storage techniques, 10 additional soil borings at various locations in Sludge Beds 9 and 10 were collected. The Accu Core liner configuration that was used to collect the soil borings for the comparison study is shown in Figure 13. Figure 14 shows one of the soil borings that was collected as part of the comparison study. Each boring contained soil in two sets of the following sections, which were adjacent to each other, a four-inch clear acrylic section for sub-sampling and a one-inch stainless steel Accu Core section to be cleaned and capped for sample storage during shipment of the soil sample to the laboratory. The soil in the four-inch acrylic section was sub-sampled using 1) a five-gram Terra Core device (Figure 4c) to collect a soil sample for extrusion into a 40-mL empty VOA vial (Figure 5) in the field prior to shipment to

the laboratory; 2) a five-gram En Core sampler (Figure 7) for sample collection and storage during shipment to the laboratory; and 3) a five-gram Terra Core device (Figure 4c) to collect a sample for extrusion into high purity methanol (Figure 6) in the field prior to shipment to the laboratory. All samples were shipped in coolers at 4 ± 2 °C for next-day delivery to the laboratory. All samples were stored at 4 ± 2 °C for 48 hours prior to preparation for analysis.

The locations that were sampled in the Accu Core validation study are listed in Table 1. Samples for the performance study were collected from areas 1 through 5 in Sludge Beds 9 and 10 at a depth of 22 to 24 feet. Samples for the comparison study were collected from areas 1 through 4 in Sludge Bed 9 at a depth of 24 to 26 feet, from area 1 in Sludge Bed 10 at depths of 20 to 22 feet and 24 to 26 feet, and from areas 2 through 5 in Sludge Bed 10 at a depth of 24 to 26 feet. The soil collected from these locations was wet fine-grained sand. Samples were collected from Locations 1 through 7 listed in Table 1 on the first day of sampling and from Locations 8 through 10 on the second day of sampling.

Sample Shipment to the Laboratory

At each of the locations, 1 through 10 shown in Table 1, five soil samples in Accu Core samplers, three soil samples in 2-oz jars, four soil samples in 40-mL VOA vials, and 2 soil samples in 5-gram En Core samplers were prepared. The 14 samples from each location were packed in an Igloo MAX COLD 32 cooler. This cooler has ultratherm[®] insulation in the body and lid to keep contents cold. The samples were wrapped in pre-cut sheets of bubble wrap to prevent breakage. Each cooler contained either frozen ice packs or bags of ice. The coolers were packed with two ice packs or bags of ice on the bottom and alternating layers of samples and ice packs or bags of ice, with at least two ice packs or bags of ice at the top. Each cooler contained a portable digital temperature data logger so cooler temperatures during sample storage and shipment could be monitored. Samples were shipped to the analytical laboratory by Federal Express at the end of each sampling day for next-day delivery. As the samples were collected at a location, they were packed in a cooler for shipment to the laboratory. After the last location was sampled for the day, coolers were immediately transported to a nearby Federal Express office for shipment for next-day delivery.

Laboratory Analysis of Field Samples

An independent commercial analytical laboratory located in Green Bay, WI analyzed all of the samples collected in the field validation study. The samples were analyzed using gas chromatography/mass spectrometry (GC/MS). EPA Method 5030B was used to prepare the samples for analysis (U.S. EPA 1996a) and EPA Method 8260 B was used to analyze the samples (U.S. EPA 1996b). The samples were analyzed for the compounds listed in Table 2. This list is the Wisconsin Method 8260 analyte list. The analytical laboratory was notified about when to expect the sample coolers and the estimated number of samples that would be in each cooler. Sample container tare weights and volumes of methanol added to the soil samples were reported to the laboratory. The laboratory was requested to ensure that all samples in a cooler be

contacted with methanol for approximately the same amount of time (± 4 hours) so analytical data could be compared. Samples shipped in the Accu Core samplers, En Core samplers, and empty VOA vials were to be stored at 4 ± 2 °C for a total of 48 hours from the time of sample collection to extrusion to show performance under this storage condition. The analytical laboratory received the sample coolers within approximately 24 hours after sample collection. All samples stored in the Accu Core samplers, En Core samplers, and empty VOA vials were stored by the lab at 4 ± 2 °C to give a total storage time of 48 hours from the time of sample collection to extrusion into methanol. All samples were contacted with methanol for 5 days ± 4 hours prior to having methanol aliquots withdrawn for analysis.

RESULTS AND DISCUSSION

Performance Study

Four to seven of the compounds listed in Table 2 were determined to be present in the performance study samples. For the ten sampling locations, the concentrations of the compounds detected in the samples stored in the Accu Core samplers and the concentrations of the compounds detected in the corresponding samples extruded into methanol in the field are shown in Tables 3 through 12. Each table contains data for the three sample sets that were collected at each location, except for Table 3. Table 3 shows the data for Location 1 and includes information for two sample sets. For Location 1, the stainless steel sections making up the third sample set were not completely filled with soil. As a result, samples were not prepared for analysis. The third column of data in Tables 3 through 12 shows the percent recovery for each compound in the sample stored in the Accu Core sampler as compared to the concentration of the compound in the corresponding methanol-extruded sample. In this way, the effect of sample storage in the Accu Core sampler on the analyte concentrations can be evaluated. This is based on the assumption that when the soil was collected in the two adjacent Accu Core stainless steel sections, the contamination in the soil was homogeneous and the samples contained similar analyte concentrations. Unfortunately, no current information is available on the homogeneity of the contamination or the distribution of the contaminants in the sludge beds that were sampled in the study.

When the performance of hand-operated coring devices to store soil samples for VOC analysis is evaluated in the laboratory, average percent recovery, which is calculated using five percent recovery values for each analyte of interest, must be 80% or greater in order for performance of the device to be considered acceptable (ASTM 2006a). In the laboratory evaluation, soil samples are spiked with known concentrations of analytes so all samples contain approximately the same analyte concentrations prior to extrusion into methanol or sample storage. In the Accu Core performance study, homogeneity of the contamination in the soil was not known. There was no way to control or to know the analyte concentrations in the soil samples at the time they were collected. Therefore, an 80% or greater recovery value for the analytes in the samples stored in the Accu Core samplers or an expected correlation within 20% of the analytical data for the corresponding samples collected in the Accu Core samplers and

those extruded into methanol in the field are most likely not appropriate for the data generated in this field study.

As shown in Tables 3 through 12, the percent recovery values vary with compound and location. Data presented in Table 8 for Location 6 show that despite positive PID screening results, none of the analytes detected in the other performance study samples were determined to be present in concentrations above the analytical detection limits in the samples from this location. The concentrations for all of the analytes listed in Table 2 were reported as below detection limit for the samples from Location 6. These results show either the presence of VOCs in the soil that are not on the analyte list in Table 2 or that the soil adjacent to the stainless steel Accu Core sections for this location contained high enough levels of contamination to be detected by the PID, but that the soil in the adjacent Accu Core sections contained very low levels or no contamination, which would indicate significant non-homogeneity of the contamination in the soil at this location.

The data listed in Tables 3 through 7 and Tables 9 and 10 show that percent recovery 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethyl-benzene, values for Naphthalene, p-Isopropyltoluene, and Tetrachloroethene are very high for Sample Set 1 from Location 3 (Table 5) and Sample Set 2 from Location 10 (Table 12). These values range from 120 to 188%, indicating that the contamination in the soil in the adjacent Accu Core stainless steel sections for these sample sets was not homogeneous. It appears that the analyte concentrations in the soil stored in the Accu Core sampler were much higher than those in the soil extruded into methanol in the field. Based on this conclusion, it can be assumed that the percent recovery values for 1,1,1-Trichloroethane in these sample sets (120 and 100%, respectively) are also high due to non-homogeneity of the contamination in the soil. Therefore, data from these sample sets can not be used to evaluate the performance of the Accu Core sampler.

There are, however, several sample sets from the various locations (Tables 4, 5, 6, 7, 11, and 12) for which the percent recoveries of 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene all fall within the range of 75 to 126%. For the same sample sets in most cases, the percent recovery values for 1,1,1-Trichloroethane are less than 70%. Data for these sample sets are summarized in Table 13. Two of the sample sets listed in Table 13 have percent recovery values for 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethyl-benzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene ranging from 104 to 126% and 108 to 115%. Four of the sample sets listed in the table have percent recovery values for the six compounds ranging from 78 to 90%, 79 to 89%, 75 to 89%, and 79 to 91%. The remaining eight sample sets listed in Table 13 have percent recovery values for the six compounds that are approximately 80 to 100%. The variability in percent recovery for the sample sets would be expected because of analytical variability and differences in analyte concentrations in the soil when the samples were collected. For field evaluation, the data for the 14 sample sets listed in Table 13 show good performance of the Accu Core sampler to store soil containing 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene,

1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene at 4 ± 2 °C for 48 hours. The lower percent recovery values listed in Table 13 for the six compounds are 75, 78, and 79%, falling just below the 80% average recovery specified for controlled laboratory testing.

The data for the 14 sample sets listed in Table 13 show some loss of 1,1,1-Trichloroethane from the samples stored in the Accu Core samplers at 4 ± 2 °C for 48 hours. The percent recovery of 1,1,1-Trichloroethane for the first sample listed in Table 13 (Location 2/Sample Set 3) is 81%. However, for all of the other sample sets listed, except Location 7/Sample Set 3, which did not contain 1,1,1-Trichloroethane, the percent recovery values for 1,1,1-Trichloroethane range from 41 to 70%.

1,1,1-Trichloroethane is the most volatile of the seven compounds detected in the performance study samples. The boiling point temperatures for the compounds are listed in Table 14. As shown in Table 14, the boiling point of 1,1,1-Trichloroethane, 74 °C, is significantly lower than the boiling points of the other compounds, which are approximately 120 to 220 °C. Table 15 shows a listing of the percent recovery values for 1,1,1-Trichloroethane, 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene for the samples stored in the Accu Core samplers for the locations/sample sets listed in Table 13. The compounds are listed in the table in order of increasing boiling point temperature, and the percent recovery values for each compound are listed in increasing order. The average percent recovery value for 1,1,1-Trichloroethane is 61%, while the average percent recovery values for the other six compounds are 90 to 96%.

The percent recovery values listed in Table 15 show good performance of the Accu Core sampler for storing the soil containing 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene at 4 ± 2 °C for 48 hours. As stated above, the average percent recovery values for the six compounds range from 90 to 96%, well above the 80% average recovery specified for laboratory testing.

The performance of the Accu Core sampler to store soil containing 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene at 4 ± 2 °C for 48 hours shown by the data for the 14 samples listed in Table 13 can not be attributed to homogeneity differences in the soil contamination when the samples were collected. The data shown in Table 15 for the six compounds indicate that there were minimal homogeneity differences in the soil sample pairs. The average percent recovery values (90 to 96%) and the low percent relative standard deviation values (10 to 13%) listed in Table 15 for the six compounds show consistency in the data and suggest that the analyte concentrations in the samples being compared did not vary significantly when the samples were collected.

Review of the data for samples collected from some of the other locations shows slightly lower percent recovery values than those discussed above. Data for Location 2/Sample Set 1 (Table 4), Location 4/Sample Set 1 (Table 6), Location 8/Sample Sets 1 through 3 (Table 10), and Location 9/Sample Set 2 (Table 11) show that some of the percent recovery values for the seven compounds for these sample sets are slightly lower than those for the 14 sample sets listed in Table 13. Table 16 shows the range of percent recovery values for 1,1,1-Trichloroethane, 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethyl-benzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene for the samples stored in the Accu Core samplers for the six sample sets listed above. Table 17 shows a listing of the percent recovery values for the seven compounds in the samples for the locations/sample sets listed in Table 16. The compounds are listed in the table in order of increasing boiling point temperature, and the percent recovery values for each compound are listed in increasing order. For the sample sets listed in Table 16, the percent recovery values for 1,1,1-Trichloroethane range from 44 to 59% with an average percent recovery of 52% as shown in Table 17. Once again, these data show loss of this more volatile compound from the samples stored in the Accu Core samplers. The lower values in the percent recovery ranges shown in Table 16 for the other six compounds (67, 68, 68, 71, 73, and 74%) and some of the percent recovery values listed for the six compounds in Table 17 are slightly lower than would be expected if the study was performed in the laboratory where the contaminant concentrations in the soil samples can be controlled. However, this field study involves comparing contaminant concentrations in soil samples for which the homogeneity and contaminant distribution are unknown. The percent recovery ranges listed in Table 16 and percent recovery values and average percent recovery values listed in Table 17 for 1,2,4-1,2,4-Trimethylbenzene, Trichlorobenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene show acceptable performance of the Accu Core sampler to store soil containing these compounds at 4 ± 2 °C for 48 hours. The lowest percent recovery value listed in Tables 16 and 17 for the six compounds, 67%, is only 13% lower than the 80% average recovery specified for laboratory testing. The average percent recovery values (75-87%) and low percent relative standard deviation values (6 to 13%) listed in Table 17 show consistency in the data and suggest that the analyte concentrations in the samples being compared did not vary significantly when they were collected.

The data listed in Table 3 for Location 1/Sample Set 1 and in Table 7 for Location 5/Sample Set 1 show very low percent recovery values for the compounds detected in the samples. Recoveries range from 24 to 57%. These data suggest that either the soil in the adjacent stainless steel Accu Core sections was not homogeneous, resulting in consistently low recovery values for all compounds in the samples, or that the Accu Core sampler failed in storing the contaminated soil. If this was the case, it would be expected that all of the 1,1,1-Trichloroethane would have been lost from the samples, which was not the case. Due to the uncertainty about the contaminant concentrations in the samples when they were collected, no conclusion about the performance of the Accu Core sampler can be made based on the data from these sample sets.

The data listed in Table 4 for Location 2/Sample Set 2 show moderately low percent recovery values for the compounds detected in the samples. Recoveries range from 59 and <64% to 77%. The percent recovery of 1,1,1-Trichloroethane is 60%. These data suggest that the percent recovery values are due to non-homogeneity of the contaminants in the soil. If the

lower percent recovery values were due to a problem with the Accu Core sampler performance, a greater loss of 1,1,1-Trichloroethane would be expected.

The data presented in Table 3 for Sample Set 2 from Location 1 show percent recovery values for only four compounds. These are <17% and <34% for 1,1,1-Trichloroethane and Tetrachloroethene, respectively; and 112% and 94% for 1,2,4-Trichlorobenzene and Naphthalene, respectively. It appears that there may have been loss of the more volatile compounds (see Table 14); however, if this loss was due to poor Accu Core performance, some loss of 1,2,4-Trichlorobenzene and Naphthalene from the sample would be expected.

The following summarizes the Accu Core performance study results for the 29 sample sets that were collected and compared.

- 20 sample sets show acceptable performance of the Accu Core samplers to store soil contaminated with 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and Tetrachloroethene at 4 ± 2 °C for 48 hours.
- 20 sample sets show some loss of 1,1,1-Trichloroethane from soil stored in the Accu Core samplers at 4 ± 2 °C for 48 hours.
- Three sample sets contained no contamination above the analytical detection limits.
- Results from six of the sample sets are non-conclusive because the data suggest the two samples in the sample sets may not have contained similar analyte concentrations when the samples were collected.

Screening Results for the Performance Study Samples

For each sample set presented in Tables 3 through 12, screening results are listed. The soil in the three-inch acrylic sections adjacent to the stainless steel Accu Core sections for each sample set (Figures 11 and 12) was screened using a PID. In addition, along with the PID, an X-Wand was used to screen soil adjacent to the sample sets collected from Locations 1 and 8. All screening results were positive. However, data presented in Table 8 for Location 6 show that despite positive PID screening results, none of the analytes detected in the other performance study samples were determined to be present in concentrations above the analytical detection limits in the samples from this location. In addition, the concentrations for all of the analytes listed in Table 2 were reported as below detection limit for the samples from Location 6. These results show either the presence of VOCs in the soil that are not on the analyte list in Table 2 or that the soil adjacent to the stainless steel Accu Core sections for this location contained high enough levels of contamination to be detected by the PID, but that the soil in the adjacent Accu Core sections contained very low levels or no contamination, which suggests that the contamination in the soil at this location was not homogeneous. The screening results generated

during the performance study show the usefulness of the sample screening capability provided by the Accu Core system.

Comparison Study

All but one of the comparison study samples were collected at a depth of 24 to 26 feet (Table 1). Unfortunately, there was much less contamination in the soil collected at this depth than in the soil collected at 22 to 24 feet for the performance study. Once again, this shows non-homogeneity of the contamination in the sludge bed soil. The comparison study samples were analyzed for the analytes listed in Table 2. Analyte concentrations in the comparison study samples collected from Locations 1, 4, 5, and 6 were all reported to be less than the analytical detection limits.

The comparison study data for Locations 2, 3, 7, 8, 9, and 10 are shown in Tables 18 through 23, respectively. As shown in these tables, analyte concentrations in the Sample Set 2 samples collected from Locations 2 (Table 18), 3 (Table 19), and 7 (Table 20) were also all reported to be less than the analytical detection limits. This gives very limited data for review in the study.

In addition to limited data due the absence of contamination in the soil samples that were collected, there also appears to be significant non-homogeneity of the contamination that was present in some of the samples. This is shown by the data given in Tables 21 and 22.

The data presented in Table 21 for Location 8/Sample Set 1 show that the contamination in the soil in the four-inch acrylic section that was sampled to give the empty VOA vial, En Core, and methanol-extruded samples and the contamination in the soil in the adjacent Accu Core stainless steel section (see Figures 13 and 14) was not homogeneous. Several compounds were detected in the Accu Core sample at concentrations that are in most cases significantly higher than in the other sample types. These significant differences in the analyte concentrations can not be attributed to volatile loss during sub-sampling.

The data presented in Table 21 for Location 8/Sample Set 2 and Table 22 for Location 9/Sample Set 1 show that not only was the contamination in the soil in the four-inch acrylic sections and the contamination in the soil in the adjacent Accu Core stainless steel sections not homogeneous, but the contamination in the soil in the four-inch acrylic sections was also not homogeneous. For these sample sets, the concentrations of the analytes are significantly higher in the En Core samples than in the methanol-extruded samples, the empty VOA vial samples, and the Accu Core samples. These significant differences in the analyte concentrations also can not be attributed to volatile loss during sub-sampling.

The remaining data presented in Table 18 for Location 2/Sample Set 1, Table 19 for Location 3/Sample Set 1, Table 20 for Location 7/Sample Set 1, Table 22 for Location 9/Sample Set 2, and Table 23 for Location 10/Sample Sets 1 and 2 show a number of cases for the one or

two compounds detected in the six sample sets where the analyte concentrations are higher in the Accu Core samples. Unfortunately in most cases, the analyte concentrations vary only slightly between the sample types within a set and are near the analytical detection limit. The data given in Table 18 for Location 2/Sample Set 1 show a higher concentration of Trichloroethene, which is well above the analytical detection limit, in the Accu Core sample than in the other sample types. The concentrations of 1,1,1-Trichloroethane listed in Table 20 for Location 7/Sample Set 1 and in Table 23 for Location 10/Sample Set 1 and the concentrations of cis-1,2-Dichloroethane listed in Table 23 for Location 10/Sample Set 2 show that these compounds were detected at concentrations above the analytical detection limits in the Accu Core samples but not in the other sample types. The concentrations of 1,1,1-Trichloroethane given in Table 19 for Location 3/Sample Set 1 show a lower concentration of this compound in the Accu Core sample as compared to the methanol-extruded sample; however, the concentrations vary by only 9 µg/Kg. Due to analytical variably, these concentrations can be considered to be comparable. The concentration data listed for cis-1,2-Dichloroethene in Table 19 for Location 3/Sample Set 1 show that this compound was only detected in the methanol-extruded sample. However, the concentration of cis-1,2-Dichloroethene reported in the methanol-extruded sample is only 6 µg/Kg above the analytical detection limit. The data given in Table 22 for Location 9/Sample Set 2 shows concentrations of cis-1,2-Dichloroethene in the empty VOA vial sample at <50 μ g/Kg; in the En Core sample at 56 μ g/Kg; in the methanol-extruded sample at 60 μ g/Kg; and in the Accu Core sample at 64 µg/Kg. The concentrations of this compound in the En Core, methanol-extruded, and Accu Core samples vary by such a small amount that they can be considered to be the same. The data listed for this compound in Table 23 for Location 10/Sample Set 1 can also be concluded to be comparable for the methanol-extruded and Accu Core samples.

The limited results discussed above suggest that the samples stored in the Accu Core samplers contained higher or comparable volatile analyte concentrations when they were analyzed compared with the methanol-extruded, En Core, and empty VOA vial samples from the same sample set. Because there are so few data sets and analyte concentrations to compare, and because in most cases, the analyte concentrations vary only slightly between the sample types and are near the analytical detection limit, a definite conclusion about the performance of the Accu Core sampler versus the performance of current subsurface sampling techniques can not be made. However, the limited data suggest higher or comparable analyte concentrations in the Accu Core samples.

The difference between the methanol extruded samples in the performance and comparison studies is that in the performance study, the soil in the Accu Core stainless steel section was extruded directly into methanol, while in the comparison study, the soil was sub-sampled from the four-inch acrylic section using a Terra Core device and extruded into methanol. It should be noted that the data presented in Tables 20 and 23 for Sample Set 1 for 1,1,1-Trichloroethane, the compound that was lost from the Accu Core samplers in the performance study, show higher concentrations of this compound in the samples stored in the Accu Core samples than in the samples extruded into methanol. Also in Table 19, Sample Set

1, the concentration of 1,1,1-Trichloroethane is just 10% more in the methanol extruded sample than in the sample stored in the Accu Core sampler. Based on the results of the performance study, these data suggest a significant loss of 1,1,1-Trichloroethane from the soil during the sub-sampling activity.

CONCLUSIONS AND RECOMMENDATIONS

Accu Core Sampler Performance

The capped Accu Core sampler sections show acceptable performance for storing sandy soil samples containing VOCs having boiling point temperatures ranging from approximately 120 to 220 °C for 48 hours at 4 ± 2 °C. However, partial losses of a more volatile compound, 1,1,1-Trichloroethane, having a boiling point temperature of 74 °C occurred. The Accu Core sampler section and caps are constructed of non-reactive materials so the loss of 1,1,1-Trichloroethane from the soil stored in the samplers most likely occurred through the seals between the stainless steel sections and the end caps of the samplers. Fine grains of soil on the edges of the stainless steel section during capping, which can compromise the sampler seals, are very difficult to prevent, especially in the field environment. Based on the difficulty of preventing fine soil grains from interfering with the cap seals of the Accu Core sampler in the field, the concept of capping the Accu Core sampler sections and shipping samples to the laboratory in the sections should no longer be pursued.

Screening Capability Provided by the Accu Core System

The usefulness of the sample screening capability provided by the Accu Core system was demonstrated. Results of the Accu Core validation testing show that the Accu Core system provides an excellent screening capability for use in identifying the portion of the subsurface soil core that should be selected for analysis without disrupting the integrity of the soil sample that will be analyzed. It is recommended that the screening capability offered by the Accu Core system be further developed and evaluated.

Comparison Study

Many of the analyte concentrations in the comparison study samples were reported to be less than the analytical detection limits. As a result, there are very limited data for review in the study. For the data that are above the analytical detection limits, in most cases, the analyte concentrations vary only slightly between the sample types within a set and are near the analytical detection limit. As a result, a definite conclusion about the performance of the Accu Core sampler versus the performance of current subsurface sampling techniques can not be made. However, the limited data suggest higher or comparable analyte concentrations in the Accu Core samples. Data for 1,1,1-Trichloroethane generated in the performance and comparison studies suggest significant loss of this compound from the soil samples that were collected using the current sub-sampling techniques in the comparison study.

The Accu Core system provides a means for collecting a soil sample in the Accu Core section and transferring the sample for transportation to the laboratory without disrupting the integrity of the soil sample. Current sub-sampling techniques require the horizontal soil core within the acrylic liner of the sampling equipment to be sub-sampled by removing a horizontal section of the liner and exposing the soil to the atmosphere while a coring tool is inserted into the soil core, usually multiple times, to collect a sub-sample. Comparing data generated for 1,1,1-Trichloroethane in the performance and comparison studies shows that this technique can result in significant VOC loss from the soil being sub-sampled. Using an Accu Core section to collect the soil from the core can prevent VOC loss from the sample. It is recommended that the Accu Core system be configured so that an Accu Core section can be removed from the soil core to collect a sample for analysis. If a five-gram sample size is required, the section can be turned on one end so a coring tool can be used to collect the soil sample from the section. In this way, the integrity of the soil sample will not be disrupted by sample collection and VOC loss will be minimized. The use of Accu Core sampler sections to provide five-gram subsurface soil samples with minimal VOC loss will be the main focus of the next validation study of this technology.

REFERENCES

- ASTM International, 2006a, ASTM Guide D 4547-06, Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds. Annual Book of ASTM Standards, 11.04, 32-43.
- ASTM International, 2006b, ASTM Practice D 6418-04, Standard Practice for Using the Disposable En Core Sampler for Sampling and Storing Soil for Volatile Organic Analysis. Annual Book of ASTM Standards, 11.04, 577 590.
- CRC Press, 1978, CRC Handbook of Chemistry and Physics. 59th Edition, CRC Press, Inc.
- Sorini, S.S., J.F. Schabron, and J.F. Rovani, Jr., 2004, Validation of a New Soil VOC Sampler: Performance of the En Core[®] Sampler at -7°C and -21°C and Development of the Accu Core[™] Subsurface Sampling/Storage Device for VOC Analysis. Laramie, WY, WRI Report WRI-04-R007.
- 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 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, 1996b, 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.

Location 1	Sampling Location
Performance Study	: SB 9-1; 22-24 ^{°a}
Comparison Study:	SB 9-1; 24-26 [°]
Location 2	Sampling Location
Performance Study	SB 9-2; 22-24'
Comparison Study:	SB 9-2; 24-26'
Location 3	Sampling Location
Performance Study	SB 9-3; 22-24'
Comparison Study:	SB 9-3; 24-26'
Location 4	Sampling Location
Performance Study	SB 9-4; 22-24'
Comparison Study:	SB 9-4; 24-26'
Location 5	Sampling Location
Performance Study	SB 9-5; 22-24'
Comparison Study:	SB 10-1; 20-22'
Location 6	Sampling Location
Performance Study:	SB 10-1; 22-24'
Comparison Study:	SB 10-1; 24-26'
Location 7	Sampling Location
Performance Study:	SB 10-2; 22-24'
Comparison Study:	SB 10-2; 24-26'
Location 8	Sampling Location
Performance Study:	SB 10-3; 22-24'
Comparison Study:	SB 10-3; 24-26'
Location 9	Sampling Location
Performance Study:	SB 10-4; 22-24'
Comparison Study:	SB 10-4; 24-26'
Location 10	Sampling Location
Performance Study:	SB 10-5; 22-24'
Comparison Study:	SB 10-5; 24-26'

Table 1. Accu Core Validation Study Sampling Locations

^a Explanation of Sampling Location Designation: Sludge Bed 9; Area 1; Collected at 22 to 24 feet



Table 3. Accu Core Performance for Location 1: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES; X-Wand-Yes

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	240	1,000	24
1,2,4-Trichlorobenzene	120	260	46
1,2,4-Trimethylbenzene	68	120	57
Naphthalene	190	370	51
Tetrachloroethene	160	460	35

Sample Set 2

Screening Results: PID-YES; X-Wand-Yes

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	<64	380	<17
1,2,4-Trichlorobenzene	110	98	112
Naphthalene	150	160	94
Tetrachloroethene	<64	190	<34

Table 4. Accu Core Performance for Location 2: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	320	680	47
1,2,4-Trichlorobenzene	150	170	88
1,2,4-Trimethylbenzene	180	210	86
1,3,5-Trimethylbenzene	86	90	96
Naphthalene	310	370	84
p-Isopropyltoluene	110	130	85
Tetrachloroethene	260	390	67

Sample Set 2

<u>Analyte</u>	Conc. in Accu	Conc. in MeOH	<u>% Recovery</u>
	<u>Core Sample, µg/Kg</u>	<u>Field Sample, µg/Kg</u>	
1,1,1-Trichloroethane	450	750	60
1,2,4-Trichlorobenzene	100	130	77
1,2,4-Trimethylbenzene	130	190	68
1,3,5-Trimethylbenzene	<58	91	<64
Naphthalene	220	290	76
p-Isopropyltoluene	72	120	60
Tetrachloroethene	170	290	59

Table 4 cont'd. Accu Core Performance for Location 2: Sample Storage at 4 \pm 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	420	520	81
1,2,4-Trichlorobenzene	110	110	100
1,2,4-Trimethylbenzene	180	170	106
1,3,5-Trimethylbenzene	87	85	102
Naphthalene	230	230	100
p-Isopropyltoluene	100	100	100
Tetrachloroethene	190	220	86

Table 5. Accu Core Performance for Location 3: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	1,200	1000	120
1,2,4-Trichlorobenzene	210	170	123
1,2,4-Trimethylbenzene	390	320	122
1,3,5-Trimethylbenzene	180	150	120
Naphthalene	520	420	124
p-Isopropyltoluene	220	170	129
Tetrachloroethene	620	430	144

Sample Set 2

<u>Analyte</u>	Conc. in Accu	Conc. in MeOH	<u>% Recovery</u>
	<u>Core Sample, µg/Kg</u>	Field Sample, µg/Kg	
1,1,1-Trichloroethane	660	940	70
1,2,4-Trichlorobenzene	130	150	87
1,2,4-Trimethylbenzene	240	280	86
1,3,5-Trimethylbenzene	130	130	100
Naphthalene	310	360	86
p-Isopropyltoluene	140	160	87
Tetrachloroethene	350	370	95

Table 5 cont'd. Accu Core Performance for Location 3: Sample Storage at 4 \pm 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	350	540	65
1,2,4-Trichlorobenzene	110	87	126
1,2,4-Trimethylbenzene	210	200	105
1,3,5-Trimethylbenzene	100	96	104
Naphthalene	240	230	104
p-Isopropyltoluene	120	110	109
Tetrachloroethene	210	200	105

Table 6. Accu Core Performance for Location 4: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	570	970	59
1,2,4-Trichlorobenzene	150	190	79
1,2,4-Trimethylbenzene	200	270	74
1,3,5-Trimethylbenzene	96	120	80
Naphthalene	250	280	89
p-Isopropyltoluene	110	150	73
Tetrachloroethene	340	380	89

Sample Set 2

Analyte	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	690	980	70
1,2,4-Trichlorobenzene	160	170	94
1,2,4-Trimethylbenzene	280	330	85
1,3,5-Trimethylbenzene	140	160	87
Naphthalene	290	330	88
p-Isopropyltoluene	150	180	83
Tetrachloroethene	370	390	95

Table 6 cont'd. Accu Core Performance for Location 4: Sample Storage at 4 \pm 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	890	1300	68
1,2,4-Trichlorobenzene	180	200	90
1,2,4-Trimethylbenzene	390	480	81
1,3,5-Trimethylbenzene	180	210	86
Naphthalene	340	400	85
p-Isopropyltoluene	210	270	78
Tetrachloroethene	430	480	90

Table 7. Accu Core Performance for Location 5: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	350	1200	29
1,2,4-Trichlorobenzene	150	420	36
1,2,4-Trimethylbenzene	110	280	39
1,3,5-Trimethylbenzene	55	130	42
Naphthalene	140	370	38
p-Isopropyltoluene	56	160	35
Tetrachloroethene	270	680	40

Sample Set 2

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	170	410	41
1,2,4-Trichlorobenzene	98	110	89
1,2,4-Trimethylbenzene	87	110	79
Naphthalene	100	120	83
Tetrachloroethene	150	180	83

Table 7 cont'd. Accu Core Performance for Location 5: Sample Storage at 4 \pm 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	340	580	59
1,2,4-Trichlorobenzene	120	140	86
1,2,4-Trimethylbenzene	180	220	82
1,3,5-Trimethylbenzene	88	96	92
Naphthalene	170	190	89
p-Isopropyltoluene	94	110	85
Tetrachloroethene	250	260	96

Table 8. Accu Core Performance for Location 6: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH <u>Field Sample, µg/Kg</u>	<u>% Recovery</u>
4 4 4 70 - 11 4			a
1,1,1-Trichloroethane	<58	<60	^u
1,2,4-Trichlorobenzene	<58	<60	
1,2,4-Trimethylbenzene	<58	<60	
1,3,5-Trimethylbenzene	<58	<60	
Naphthalene	<58	<60	
p-Isopropyltoluene	<58	<60	
Tetrachloroethene	<58	<60	

Sample Set 2

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH <u>Field Sample, µg/Kg</u>	<u>% Recovery</u>
1,1,1-Trichloroethane	<57	<61	a
1,2,4-Trichlorobenzene	<57	<61	
1,2,4-Trimethylbenzene	<57	<61	
1,3,5-Trimethylbenzene	<57	<61	
Naphthalene	<57	<61	
p-Isopropyltoluene	<57	<61	
Tetrachloroethene	<57	<61	

^a % Recovery can not be calculated.

Table 8 cont'd. Accu Core Performance for Location 6: Sample Storage at 4 \pm 2 °C for 48 Hours

Sample Set 3

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	<57	<65	a
1,2,4-Trichlorobenzene	<57	<65	
1,2,4-Trimethylbenzene	<57	<65	
1,3,5-Trimethylbenzene	<57	<65	
Naphthalene	<57	<65	
p-Isopropyltoluene	<57	<65	
Tetrachloroethene	<57	<65	

^a % Recovery can not be calculated.

Table 9. Accu Core Performance for Location 7: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	400	940	42
1,2,4-Trichlorobenzene	160	190	84
1,2,4-Trimethylbenzene	150	200	75
1,3,5-Trimethylbenzene	69	87	79
Naphthalene	360	420	86
p-Isopropyltoluene	87	110	79
Tetrachloroethene	330	370	89

Sample Set 2

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	360	530	68
1,2,4-Trichlorobenzene	130	120	108
1,2,4-Trimethylbenzene	130	140	93
1,3,5-Trimethylbenzene	70	<64	>109
Naphthalene	280	270	104
p-Isopropyltoluene	70	71	99
Tetrachloroethene	240	220	109

Table 9 cont'd. Accu Core Performance for Location 7: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,2,4-Trichlorobenzene	79	95	83
1,2,4-Trimethylbenzene	85	<94	>90
Naphthalene	190	190	100
Tetrachloroethene	100	120	83

Table 10. Accu Core Performance for Location 8: Sample Storage at 4 ± 2 °C for48 Hours

Sample Set 1

Screening Results: PID-YES; X-Wand-Yes

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH <u>Field Sample, μg/Kg</u>	<u>% Recovery</u>
1,1,1-Trichloroethane	760	1500	51
1,2,4-Trichlorobenzene	140	190	74
1,2,4-Trimethylbenzene	280	400	70
1,3,5-Trimethylbenzene	130	170	76
Naphthalene	390	510	76
p-Isopropyltoluene	130	190	68
Tetrachloroethene	450	480	94

Sample Set 2

Screening Results: PID-YES; X-Wand-Yes

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	530	930	57
1,2,4-Trichlorobenzene	100	140	71
1,2,4-Trimethylbenzene	220	280	79
1,3,5-Trimethylbenzene	110	130	85
Naphthalene	320	390	82
p-Isopropyltoluene	100	140	71
Tetrachloroethene	300	310	97

Table 10 cont'd. Accu Core Performance for Location 8: Sample Storage at 4 ± 2 °C for48 Hours

Sample Set 3

Screening Results: PID-YES; X-Wand-Yes

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH <u>Field Sample, µg/Kg</u>	<u>% Recovery</u>
1,1,1-Trichloroethane	480	860	56
1,2,4-Trichlorobenzene	95	140	68
1,2,4-Trimethylbenzene	230	310	74
1,3,5-Trimethylbenzene	100	140	71
Naphthalene	280	370	76
p-Isopropyltoluene	110	150	73
Tetrachloroethene	270	300	90

Table 11. Accu Core Performance for Location 9: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	880	1800	49 1,2,4-
Trichlorobenzene	180	220	82
1,2,4-Trimethylbenzene	450	570	79
1,3,5-Trimethylbenzene	190	240	79
Naphthalene	550	650	85
p-Isopropyltoluene	200	250	80
Tetrachloroethene	530	580	91

Sample Set 2

<u>Analyte</u>	Conc. in Accu	Conc. in MeOH	<u>% Recovery</u>
	<u>Core Sample, µg/Kg</u>	<u>Field Sample, µg/Kg</u>	
1,1,1-Trichloroethane	620	1400	44
1,2,4-Trichlorobenzene	140	190	74
1,2,4-Trimethylbenzene	400	520	77
1,3,5-Trimethylbenzene	190	210	90
Naphthalene	480	550	87
p-Isopropyltoluene	190	230	83
Tetrachloroethene	400	460	87

Table 11 cont'd. Accu Core Performance for Location 9: Sample Storage at 4 ± 2 °C for48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	410	610	67
1,2,4-Trichlorobenzene	110	100	110
1,2,4-Trimethylbenzene	340	300	113
1,3,5-Trimethylbenzene	150	130	115
Naphthalene	350	310	113
p-Isopropyltoluene	140	130	108
Tetrachloroethene	260	230	113

Table 12. Accu Core Performance for Location 10: Sample Storage at 4 ± 2 °C for48 Hours

Sample Set 1

Screening Results: PID-YES

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	810	1,300	62
1,2,4-Trichlorobenzene	200	240	83
1,2,4-Trimethylbenzene	690	730	94
1,3,5-Trimethylbenzene	300	290	103
Naphthalene	650	680	96
p-Isopropyltoluene	260	270	96
Tetrachloroethene	580	540	107

Sample Set 2

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	1,300	1,300	100
1,2,4-Trichlorobenzene	250	170	147
1,2,4-Trimethylbenzene	940	600	157
1,3,5-Trimethylbenzene	410	240	171
Naphthalene	820	530	155
p-Isopropyltoluene	360	240	150
Tetrachloroethene	770	410	188

Table 12 cont'd. Accu Core Performance for Location 10: Sample Storage at 4 ± 2 °C for 48 Hours

Sample Set 3

<u>Analyte</u>	Conc. in Accu Core Sample, µg/Kg	Conc. in MeOH Field Sample, µg/Kg	<u>% Recovery</u>
1,1,1-Trichloroethane	600	1,100	54
1,2,4-Trichlorobenzene	140	160	87
1,2,4-Trimethylbenzene	560	650	86
1,3,5-Trimethylbenzene	220	250	88
Naphthalene	520	540	96
p-Isopropyltoluene	200	250	80
Tetrachloroethene	390	390	100

Sample Location/Sample Set	% Recovery Values Group of Six Compounds 1,1,1-Trichloroethar		
		1,1,1 ⁻ 111Cm010cmanc	
Location 2/Sample Set 3	86-106	81	
Location 3/Sample Set 2	86-100	70	
Location 3/Sample Set 3	104-126	65	
Location 4/Sample Set 2	83-95	70	
Location 4/Sample Set 3	78-90	68	
Location 5/Sample Set 2	79-89 ^a	41	
Location 5/Sample Set 3	82-96	59	
Location 7/Sample Set 1	75-89	42	
Location 7/Sample Set 2	93->109	68	
Location7/Sample Set 3	83, >90, 100, 83 ^a	b	
Location 9/Sample Set 1	79-91	49	
Location 9/Sample Set 3	108-115	67	
Location 10/Sample Set 1	83-107	62	
Location 10/Sample Set 3	80-100	54	

Table 13. Performance Study Sample Sets Showing Percent Recovery Values in the Range of
75 to 126% for the Group of Six Compounds (1,2,4-Trichlorobenzene, 1,2,4-
Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene, p-Isopropyltoluene, and
Tetrachloroethene)

^a Percent recovery values for 1,2,4-Trichlorobenzene, 1,2,4-Trimethylbenzene, Naphthalene, and Tetrachloroethene; 1,3,5-Trimethylbenzene and p-Isopropyltoluene were not detected in the Accu Core or methanol-extruded samples from this set.

^b 1,1,1-Trichloroethane was not detected in the Accu Core or methanol-extruded samples from this set.

Compound	Boiling Point, °C at 760 mm Hg ^a
1,1,1-Trichloroethane	74
Tetrachloroethene	121
1,3,5-Trimethylbenzene	165
1,2,4-Trimethylbenzene	169
p-Isopropyltoluene	177
1,2,4-Trichlorobenzene	213
Naphthalene	218

Table 14. Boiling Point Temperatures of Compounds Detected in the Accu Core Performance Study Samples

^a Boiling point values are from the 59th Edition of the CRC Handbook of Chemistry and Physics (CRC Press 1978).

Compound	% Recovery Values		
1,1,1-Trichloroethane	41, 42, 49, 54, 59, 62, 65, 67, 68, 68, 70, 70, 81		
	$\overline{x} = 61\%$ s = 12 % rel. s = 20%		
Tetrachloroethene	83, 83, 86, 89, 90, 91, 95, 95, 96, 100, 105, 107, 109, 113		
	$\bar{x} = 96\%$ s = 10 % rel. s = 10%		
1,3,5-Trimethylbenzene	79, 79, 86, 87, 88, 92, 100, 102, 103, 104, >109, 115		
	Approximate values due to greater than values:		
	$\overline{x} = 95\%$ s = 12 % rel. s = 13%		
1,2,4-Trimethylbenzene	75, 79, 79, 81, 82, 85, 86, 86, >90, 93, 94, 105, 106, 113		
	Approximate values due to greater than value:		
	$\overline{x} = 90\%$ s = 11 % rel. s = 12%		
p-Isopropyltoluene	78, 79, 80, 80, 83, 85, 87, 96, 99, 100, 108, 109		
	$\overline{x} = 90\%$ s = 11 % rel. s = 12%		

Table 15. Percent Recovery Values for the Compounds in the Performance Study
Samples Stored in the Accu Core Samplers for the Locations/Sample
Sets Listed in Table 13

Compound	% Recovery Values			
1,2,4-Trichlorobenzene	82, 83, 83, 84, 86, 87, 87, 89, 90, 94, 100, 108, 110, 126			
	$\overline{x} = 93\%$ s = 13 % rel. s = 14%			
Naphthalene	83, 85, 85, 86, 86, 88, 89, 96, 96, 100, 100, 104, 104, 113 $\overline{x} = 94\%$ s = 9 % rel. $s = 10%$			

Table 15 cont'd.Percent Recovery Values for the Compounds in the Performance
Study Samples Stored in the Accu Core Samplers for the Locations/
Sample Sets Listed in Table 13

Sample Location/Sample Set	% Recovery Values			
	Group of Six Compounds	1,1,1-Trichloroethane		
Location 2/Sample Set 1	67-96	47		
Location 4/Sample Set 1	73-89	59		
Location 8/Sample Set 1	68-94	51		
Location 8/Sample Set 2	71-97	57		
Location 8/Sample Set 3	68-90	56		
Location 9/Sample Set 2	74-90	44		

Table 16. Range of Percent Recovery Values for the Group of Six Compounds (1,2,4-
Trichlorobenzene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Naphthalene,
p-Isopropyltoluene, and Tetrachloroethene) and 1,1,1-Trichloroethane in Soil Stored
in Accu Core Samplers for Location 2/Sample Set 1, Location 4/Samples Set 1, Location
8/Sample Sets 1-3, and Location 9/Sample Set 2

Compound	% Recovery Values		
1,1,1-Trichloroethane	44, 47, 51, 56, 57, 59		
	$\overline{x} = 52\%$ s = 6 % rel. s = 11%		
Tetrachloroethene	67, 87, 89, 90, 94, 97		
	$\overline{x} = 87\%$ s = 11 % rel. s = 13%		
1,3,5-Trimethylbenzene	71, 76, 80, 85, 90, 96		
	$\overline{x} = 83\%$ s = 9 % rel. s = 11%		
1,2,4-Trimethylbenzene	70, 74, 74, 77, 79, 86		
	x = 77% s = 5 % rel. s = 6%		
p-Isopropyltoluene	68, 71, 73, 73, 83, 85		
	x = 75% s = 7 % rel. s = 9%		

Table 17. Percent Recovery Values for the Compounds in the Performance Study SamplesStored in the Accu Core Samplers for the Locations/Sample Sets Listed in Table16

Compound	% Recovery Values		
1,2,4-Trichlorobenzene	68, 71, 74, 77, 79, 88		
	$\overline{x} = 76\%$ s = 7 % rel. s = 9%		
Naphthalene	76, 76, 82, 84, 87, 89		
	$\overline{x} = 82\%$ s = 5 % rel. s = 6%		

Table 17 cont'd.Percent Recovery Values for the Compounds in the Performance Study
Samples Stored in the Accu Core Samplers for the Locations/
Sample Sets Listed in Table 16

<u>Sample Set 1</u>					
Analyte	EVV ^a <u>Conc, μg/Kg</u>	En Core ^b <u>Conc, µg/Kg</u>	MeOH ^c <u>Conc, µg/Kg</u>	Accu Core ^d <u>Conc, μg/Kg</u>	
Trichloroethene	58	<51	80	120	
Sample Set 2					
Analyte	EVV ^a <u>Conc, μg/Kg</u>	En Core ^b Conc, µg/Kg	MeOH ^c <u>Conc, μg/Kg</u>	Accu Core ^d <u>Conc, μg/Kg</u>	
All analyte concentrations reported below analytical detection limit.					

Table 18. Accu Core Comparison Study Data for Location 2

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab

<u>Sample Set 1</u>					
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d	
	<u>Conc, μg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, μg/Kg</u>	
1,1,1-Trichloroethane	53	<50	87	78	
cis-1,2-Dichloroethene	<50	<50	56	<50	
Sample Set 2					
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d	
	<u>Conc, μg/Kg</u>	<u>Conc, μg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, μg/Kg</u>	
All analyte concentrations reported below analytical detection limit.					

Table 19. Accu Core Comparison Study Data for Location 3

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab

<u>Sample Set 1</u>					
Analyte	EVV ^a <u>Conc, μg/Kg</u>	En Core ^b <u>Conc, µg/Kg</u>	MeOH ^c <u>Conc, µg/Kg</u>	Accu Core ^d <u>Conc, μg/Kg</u>	
1,1, 1-Trichlroethane	<50	<56	<50	63	
<u>Sample Set 2</u>					
Analyte	EVV ^a <u>Conc, μg/Kg</u>	En Core ^b Conc, µg/Kg	MeOH ^c Conc, µg/Kg	Accu Core ^d Conc, μg/Kg	
All analyte concentrations reported below analytical detection limit.					

Table 20. Accu Core Comparison Study Data for Location 7

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab

		Sample Set 1		
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d
-	Conc, µg/Kg	Conc, µg/Kg	Conc, µg/Kg	Conc, µg/Kg
1,1,1-Trichloroethane	52	85	93	500
1,2,4-Trimethylbenzene	e < 50	< 50	< 50	310
1,3,5-Trimethylbenzene	e < 50	< 50	< 50	150
cis-1,2-Dichloroethene	< 50	52	68	81
Naphthalene	< 50	< 50	< 50	66
p-Isopropyltoluene	< 50	< 50	< 50	120
s-Butlylbenzene	< 50	< 50	< 50	69
Tetrachloroethene	< 50	< 50	< 50	140
Analyte	EVV ^a <u>Conc, μg/Kg</u>	<u>Sample Set 2</u> En Core ^b <u>Conc, µg/Kg</u>	MeOH ^c Conc, µg/Kg	Accu Core ^d Conc, μg/Kg
	$\underline{\text{conc}}, \underline{\mu g} / \underline{Kg}$	<u>cone, µg/ Kg</u>	$\underline{\text{conc}}, \underline{\mu g}/\underline{Ng}$	
1,1,1-Trichloroethane	240	880	53	<50
1,1-Dichloroethane	<50	90	<50	<50
1,1-Dichloroethene	<50	81	<50	<50
1,2,4-Trichlorobenzene	<50	94	<50	<50
1,2,4-Trimethylbenzene	e 200	460	<50	<50
1,3,5-Trimethylbenzene	e 110	230	<50	<50
cis-1,2-Dichloroethene	70	190	72	<50
Naphthalene	92	210	<50	<50
p-Isopropyltoluene	89	220	<50	<50
Tetrachloroethene	110	300	<50	<50

Table 21. Accu Core Comparison Study Data for Location 8

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab

<u>Sample Set 1</u>							
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d			
	Conc, µg/Kg	Conc, µg/Kg	<u>Conc, µg/Kg</u>	<u>Conc, µg/Kg</u>			
1,1,1-Trichloroethane	52	580	69	260			
1,2,4-Trimethylbenzen	e 64	1,000	< 60	210			
1,3,5-Trimethylbenzen	e < 50	570	< 60	110			
cis-1,2-Dichloroethene	67	150	85	73			
Naphthalene	< 50	180	< 60	< 50			
n-Propylbenzene	< 50	78	< 60	< 50			
p-Isopropyltoluene	< 50	310	< 60	80			
Tetrachloroethene	< 50	600	< 60	120			
<u>Sample Set 2</u>							
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d			
	Conc, µg/Kg	<u>Conc, µg/Kg</u>	Conc, µg/Kg	<u>Conc, µg/Kg</u>			
cis-1,2-Dichloroethene	< 50	56	60	64			

Table 22. Accu Core Comparison Study Data for Location 9

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab

Sample Set 1							
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d			
	<u>Conc, μg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, μg/Kg</u>			
1,1,1-Trichloroethane	< 50	< 50	< 51	89			
cis-1,2-Dichloroethene	51	< 50	66	56			
		Sample Set 2					
Analyte	EVV ^a	En Core ^b	MeOH ^c	Accu Core ^d			
	<u>Conc, μg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, µg/Kg</u>	<u>Conc, μg/Kg</u>			
cis-1,2-Dichloroethene	< 50	< 50	< 50	64			

Table 23. Accu Core Comparison Study Data for Location 10

^a Represents a sub-sampled 5-gram soil sample extruded into a 40-mL empty VOA vial for shipment to the laboratory

^b Represents a sub-sampled 5-gram soil sample collected and shipped in an En Core sampler

^c Represents a sub-sampled 5-gram soil sample extruded into methanol for shipment to the lab



Figure 1. Example of an Accu Core Configuration: Stainless Steel Sampler Sections Arranged in Alternating Order with Clear Acrylic Sections



Figure 2. Accu Core Sampler



Figure 3. Accu Core Sampler Sealed for Sample Shipment to the Laboratory

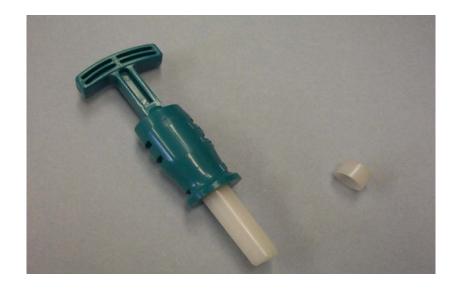


Figure 4a. An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core



Figure 4b. An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core



Figure 4c. An Example of a Coring Tool Used to Collect and Transfer a Sample of Soil from the Subsurface Soil Core



Figure 5. Demonstration of a Soil Sample Being Extruded into an Empty VOA Vial (For actual field work, personnel would be wearing protective gloves.)



Figure 6. A Soil Sample That Has Been Extruded from a Coring Tool into a 40-mL VOA Vial Containing Methanol



Figure 7. Shown Left to Right, an En Core Sampler T-handle, a 5-Gram En Core Sampler, a 25-Gram En Core Sampler, and an En Core Sampler Extrusion Tool

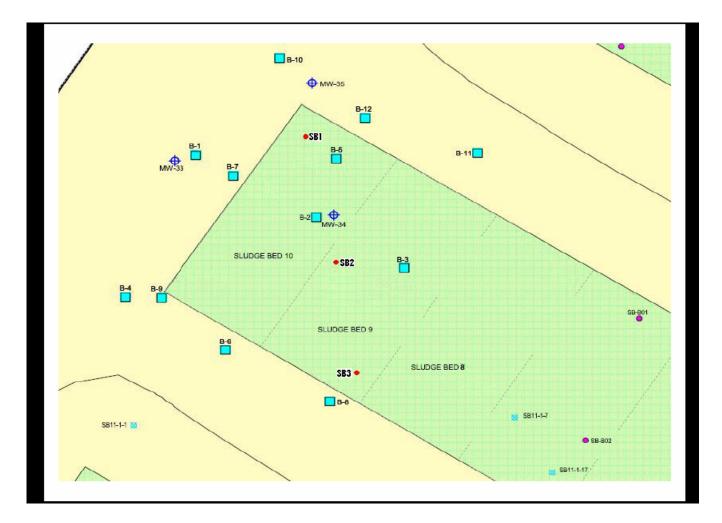


Figure 8. Locations SB1, SB2, and SB3 in Sludge Beds 9 and 10 Sampled in the Preliminary Study



Figure 9. Geoprobe Equipment at the Accu Core Validation Study Sampling Site

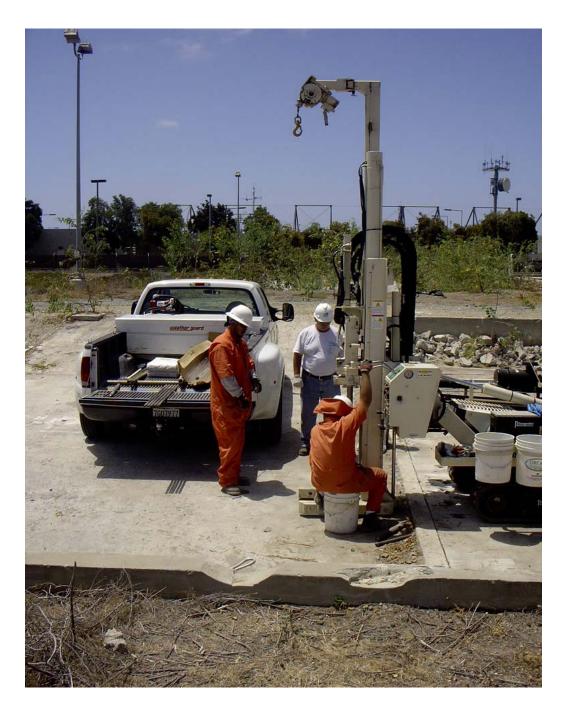


Figure 10. Geoprobe Equipment Being Used to Collect a Soil Boring for the Accu Core Validation Study

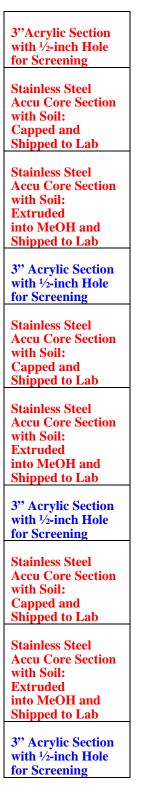


Figure 11. Accu Core Liner Configuration for the Soil Borings Collected for the Performance Study



Figure 12. A Soil Boring Collected for the Accu Core Performance Study



Figure 13. Accu Core Liner Configuration for the Soil Borings Collected for the Comparison Study



Figure 14. A Soil Boring Collected for the Accu Core Comparison Study