SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION PROGRAM

FINAL DEMONSTRATION PLAN FOR THE EVALUATION OF SOIL SAMPLING AND SOIL GAS SAMPLING TECHNOLOGIES

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APPROVAL SIGNATURES

The primary purpose of this demonstration is to evaluate soil and soil gas sampling technologies on how well they preserve sample integrity and provide representative (relative to conventional methods) samples of soils and soil gas at hazardous waste sites. The demonstration will take place under the sponsorship of the U.S. Environmental Protection Agency's Superfund Innovative Technology Evaluation Program.

This document is intended to ensure that all aspects of the demonstration are documented, scientifically sound, and that operational procedures are conducted within quality assurance and quality control specifications and health and safety regulations.

The signatures of the individuals below indicate concurrence with, and agreement to operate in compliance with, procedures specified in this document.

DEMONSTRATION PLAN

Barry Lesnik Date EPA Technical Project Manager	Noah Heller Date Simulprobe®
Brian Schumacher Date EPA Technical Project Manager	Wes McCall Date Geoprobe® Systems, Inc.
Brian Anderson Date Art's Manufacturing and Supply	Bruce Tucker Date Quadrel Services, Inc.
Wes Bratton Date Applied Research Associates	Mark Wrigley Date W. L. Gore & Associates, Inc.
Jim Clements Date Clements and Associates, Inc.	Eric Hess Date PRC Environmental Management, Inc.

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LIST OF ACRONYMS AND ABBREVIATIONS

AMS	Arts Manufacturing and Supply
ANOVA	analysis of variance
AOC	Administrative Order on Consent
ARA	Applied Research Associates
ASTM	American Society of Testing and Materials
bgs	below ground surface
CaMP	Characterization and Monitoring Program
CCAL	continuing calibration
CFR	Code of Federal Regulations
CLP	contract laboratory program
cm^3	cubic centimeters
CRD-LV	Characterization Research Division—Las Vegas
CRQL	contract-required quantitation limit
CRZ	contamination reduction zone
CSC	Chemical Sales Company
DCA	1,1-dichloroethane
DCE	1,2-dichloroethylene
DoD	Department of Defense
DOE	Department of Energy
DQO	data quality objective
DTD	Dynamic Thermal Desorber
EDD	electronic diskette deliverable
ELCD	electrolytic conductivity detector
EPA	Environmental Protection Agency
ESP	Environmentalist's Subsoil Probe
EV	electron voltage
FIT	Field Investigation Team
GC	gas chromatograph
H_o	hypothesis
HASP	health and safety plan
HSD	health and safety director
HWY	Highway
ICAL	initial calibration
IDW	investigation-derived waste
ITER	innovative technology evaluation report
LCS	laboratory control sample
MDL	method detection limits
mg	milligrams
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
mm	millimeter
MMTP	Monitoring and Measurement Technologies Program
MS	matrix spike

LIST OF ACRONYMS AND ABBREVIATIONS
(Continued)

MSD	matrix spike duplicates
NERL	National Exposure Research Laboratory
NETAC	National Environmental Technology Applications Center
NIOSH	National Institute of Occupational Safety and Health
NPL	National Priorities List
ORD	Office of Research and Development
OSHA	Occupational Safety and Health Act
OSW	Office of Solid Waste
OU	operable unit
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyls
PE	performance evaluation
PETG	polyethylene terephthalate glycol modified copolyester
PID	photo ionization detector
POL	petroleum, oil, and lubricants
PQL	practical quantitation limit
PPE	personal protective equipment
ppmv	parts per million by volume
ppt	parts per trillion
PVC	polyvinyl chloride
QAPP	quality assurance project plan
ppb	parts per billion
ppm	parts per million
QA/QC	quality assurance/quality control
PCE	tetrachloroethylene
PTFE	polytetrafluoroethylene
RCRA	Resource Conservation and Recovery Act of 1976
RPD	relative percent difference
RI/FS	remedial investigation/feasibility study
RSD	relative standard deviation
SBA	Small Business Administration
SD	standard deviation
SHSO	site health and safety officer
SITE	Superfund Innovative Technology Evaluation
SMC	Superior Manufacturing Company
SOG	Standard Operating Guideline
SOP	standard operating procedure
SQL	sample quantitation limit
SRM	standard reference materials
SWMU	solid waste management unit
TCA	1,1,1-trichloroethane
TCE	tetrachloroethylene
TER	technology evaluation record
TPM	technical project manager

LIST OF ACRONYMS AND ABBREVIATIONS
(Continued)

USCS	Unified Soil Classification System
UST	underground storage tank
VOC	volatile organic compound
μg/kg	micrograms per kilogram
μg/L	microgram per liter
%RSD	Percent relative standard deviation

EXECUTIVE SUMMARY

This demonstration was developed under the auspices of the U.S. Environmental Protection Agency's (EPA) Superfund Innovative Technology Evaluation (SITE) Program. The monitoring and measurement technologies evaluations are implemented and managed by the National Exposure Research Laboratory's (NERL) Characterization Research Division (CRD). This document provides the information needed to fairly and thoroughly evaluate how well selected innovative technologies for soil sampling and soil gas sampling perform at hazardous waste sites.

The following soil sampling technologies will be evaluated: (1) Geoprobe® Systems Large-Bore Soil Sampler, (2) JMC Environmentalist's Subsoil Probe, (3) Art's Manufacturing & Supply, Inc.'s, Dual Tube Liner Sampler, and (4) the Simulprobe® Sampler. The following soil gas sampling technologies will be evaluated: Quadrel Service's Emflux® Soil Gas Investigation System and the W.L. Gore & Associates GORE-SORBER®. These two soil gas sampling systems use a passive-type soil gas collection method. In addition, Applied Research Associates Dynamic Thermal Desorber probe will be evaluated. This probe samples and provides quantitative data on soil or soil gas contamination. Although this sampler shares qualities of both a soil and soil gas sampling system, its evaluation will be discussed with the soil gas technology evaluation.

These technologies were developed to provide representative, rapid, and low cost sampling (relative to conventional approaches) of subsurface soil and soil gas at hazardous waste sites while producing little investigation-derived waste. These performance attributes make these technologies appropriate for most site characterization and remediation sampling, and in many cases they complement the rapid data production by on-site analytical technologies.

The performances of the soil and soil gas sampling technologies will be compared to the performances of conventional soil and soil gas sampling technologies; they will be evaluated to see if they provide samples exhibiting the same physical and chemical properties as conventional methods. To focus the evaluation of the soil sampling technologies on the technologies themselves, and not the method of sample packaging for analysis, nonconventional methods for sample packaging will be used. This nonconventional method involves collecting subsamples of a soil core using open-ended syringes. The syringes will remove 5 grams of soil and directly extrude it into a headspace sample vial or into a

methanol-containing vial for analysis. This method of sampling will be used for all of the soil sampling technologies, including the conventional sampling method.

This demonstration will evaluate each innovative technology relative to a conventional method and any developer claims for the following basic subsurface sampling functions: (1) ability to retrieve a sample from a precise depth more than once (soil sample only), (2) ability to provide chemically and physically identical samples (relative to conventional methods) more than once (assuming the media sampled is homogenous) for a range of soil textures and for a range of contaminant concentrations, (3) ability to return the sample to the surface while preserving the sample integrity, including chemical concentration and physical properties (for soil samples only), for a range of external environments, (4) ability to perform these operations in a consistent and measurable time period, and (5) ability to perform these operations for a consistent and measurable cost.

The following sites were selected for this demonstration: the Albert City Small Business Administration (SBA) site, Albert City, Iowa; and the Chemical Sales Company (CSC) site, Denver, Colorado.

The SBA site is the location of a former grease gun manufacturer. The site exhibits soil contamination from chlorinated organic solvents at concentrations in excess of several 1,000 parts per million (ppm). The CSC site is the location of a former chemical supply company. The soils at this site are contaminated in the 1's to 10's of ppm range with chlorinated organic solvents. These sites exhibit volatile organics contamination in a wide range of concentrations, and they are situated on soils of different textural classifications. Based on the contaminants present at these sites, the following target analytes have been identified for this demonstration: vinyl chloride, trichloroethylene (TCE), tetrachloroethylene (PCE), 1,2 dichloroethylene (DCE), and 1,1,1-trichloroethane (TCA).

All of the analytical data needed to evaluate the soil sampling technologies will be provided by an on-site laboratory using two Hewlett Packard 5890 gas chromatographs fitted with electrolytic conductivity detectors. The innovative soil gas technologies will all analyze their own samples using their conventional methods. The conventional soil gas samples will be analyzed by the on-site laboratory.

The demonstration design uses a stratified random sampling design. Each technology, including the conventional methods, will collect multiple samples, within sampling grids, at select target sampling depths. This sampling will allow a direct comparison of the innovative technologies, individually and as a whole group, and the conventional methods. The conventional method for soil sampling will involve hollow-stem auger drilling and split-spoon sampling. The conventional method for soil gas sampling will involve drawing a soil gas sample through a polyethylene sampling tube into a 40-milliliter volatile organics vial for analysis.

The resultant data will be evaluated using analysis of variance or non-parametric statistics. In addition to this quantitative evaluation of the analytical data, data on sample recovery, costs, throughput, and other performance factors will be recorded and reported.

The soil gas samplers will be installed beginning on May 21, 1997. The soil sampler portion of the demonstration is scheduled to take place from June 2 through June 13, 1997. Following the demonstration, PRC will summarize the results of the demonstration in individual technology-specific innovative technology evaluation reports (ITER). These reports will help potential users assess the performance of each technology and their potential application at future site characterization or remediation projects at hazardous waste sites. A complete technology evaluation record containing all supporting data will be maintained by PRC.

CHAPTER 1
INTRODUCTION

This chapter provides an overview of the SITE Program and introduces elements of the soil and soil gas sampling demonstration, such as the purpose, the technologies to be demonstrated, the developers of the technologies, and where and when the demonstration will take place.

1.1 SITE PROGRAM OVERVIEW

The SITE Program evolved in response to the Superfund Amendments and Reauthorization Act of 1986, which recognized the need for a program to explore alternative or innovative technologies for treating hazardous waste and hazardous constituents at Superfund sites. The two primary goals of the SITE Program are to develop and implement (1) treatment technologies for hazardous waste and hazardous constituents remediation, and (2) monitoring and measurement technologies for evaluating the nature and extent of hazardous waste and hazardous constituent contamination.

The SITE Program consists of four related programs: Demonstration, Emerging Technologies, Monitoring and Measurement Technologies, and Technology Transfer. This demonstration will be conducted under the supervision and guidance of the Monitoring and Measurement Technologies Program (MMTP). The goal of MMTP is to encourage the development, demonstration, and use of innovative monitoring, measurement, and characterization technologies at Superfund sites. The MMTP focuses on new technologies that can provide more cost effective, faster, and safer ways to assess the nature and extent of contamination than current technologies. The EPA NERL in Las Vegas, Nevada, implements the MMTP portion of the SITE Program. Under the NERL management, the MMTP portion of the SITE Program is known as the Characterization and Monitoring Program (CaMP).

1.1.1 Selecting Technologies

Technologies are selected based on their potential use at hazardous waste sites, and EPA regional interest in a technology. Each technology is evaluated for its ability to meet one or more of the following criteria:

- Capable of being used in the field or in a mobile laboratory
- Applicable to a variety of Superfund sites
- High potential for resolving problems for which current methods are not satisfactory
- Costs are significantly less than current methods
- Performance is significantly better than current methods in areas such as data quality, sample preparation, or analysis time
- Uses techniques that are easier and safer to perform than current methods

1.1.2 Demonstrating Technologies

After a technology has been accepted into the SITE Program, a cooperative agreement between EPA and the developer is made. The agreement specifies responsibilities for conducting the demonstration and evaluating the technology. The following issues are settled at this time:

- Assessing the maturity of the technology
- Estimating the potential benefits and limitations of the technology
- Identifying demonstration sites that will provide the appropriate analytes in the desired environmental sample media or matrices (contaminants must be present in concentrations amenable to the technology being evaluated)
- Identifying and defining the roles of appropriate demonstration participants, observers, and reviewers
- Arranging analytical support for comparative testing (for example, confirmatory analysis)
- Supplying standard operating procedures (SOP), analysis methodologies, and other relevant protocols
- Preparing a demonstration plan that addresses the experimental design, sampling design, quality assurance/quality control (QA/QC), health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and data output format

- Determining logistical requirements and support (for example, field equipment, power and water sources, mobile laboratory, communications network)
- Anticipating possible corrective actions that may be required during the actual demonstration and providing this information to the demonstration participants

1.1.3 Evaluating Technologies

Innovative technologies are evaluated independently and, when possible, against conventional technologies. Data resulting from a demonstration is used to evaluate the capabilities, limitations, and field applications of each technology. Following the demonstration, all raw and reduced data used to evaluate each technology is contained in a technology evaluation record (TER) and the reduced data and evaluation of each technology is presented in an ITER. These reports are reviewed by EPA and technology developers for technical quality.

1.2 DEMONSTRATION PURPOSE

The purpose of this demonstration is to provide the information needed to fairly and thoroughly demonstrate and evaluate how well selected subsurface soil sampling and soil gas sampling technologies perform relative to conventional methods.

The soil and soil gas sampling technologies will be compared to the performance of a conventional soil and soil gas method, and evaluated to see if they provide samples exhibiting the same chemical and physical properties as conventional methods.

The basic characteristics of a conventional soil sampling method in environmental work are to collect a sample from a specified depth, and return it to the surface with minimal changes to the chemical concentration or physical properties of the sample. A soil sample includes three matrices: (1) soil matrix, (2) sorbed water, and (3) soil gas. The basic characteristics of a conventional soil gas sampling method in environmental work are to collect a gas sample from the immediate area around the end of the sampler, over a time period of minutes; to provide information on volatile contaminant sources, or on relative extent of volatile contamination. Specifically, this demonstration will evaluate each technology relative to a conventional sampling method and developer claims for the following sampling functions:

(1) ability to retrieve a sample for analysis from a precise depth (soils only), (2) ability to provide chemically and physically similar samples relative to conventional methods (assuming the media sampled is homogenous), for a range of soil textures and for a range of contaminant concentrations, (3) ability to return the sample to the surface while preserving the sample integrity, including chemical concentration and physical properties (for soil samples only), for a range of external environments, (4) ability to perform these operations in a consistent and measurable time period, and (5) ability to perform these operations for a consistent and measurable cost.

CHAPTER 2
DEMONSTRATION ORGANIZATION

This chapter identifies the participants involved in this demonstration and describes the primary roles of each participant. It also describes the methods and frequency of communication that will be used in coordinating the demonstration.

2.1 DEMONSTRATION PARTICIPANTS

This demonstration is being conducted under the direction of the EPA Office of Research and Development (ORD), NERL, CRD.

A total of seven technologies will be demonstrated. Four of these are subsurface soil sampling technologies, two are soil gas sampling technologies, and one shares characteristics of both soil and soil gas sampling technologies. Table 2-1 lists the participating technologies, some of their capabilities, and who developed them. Participants in this demonstration are listed in Table 2-2. The specific responsibilities of each participant are outlined in greater detail in Section 2.3.

2.2 COMMUNICATION

PRC will communicate regularly with all participants involved in the demonstration to coordinate activities and to resolve any logistical, technical, or quality assurance (QA) issues that arise as the demonstration progresses. Communication will take place through the points of contact for each organization listed in Table 2-2. The organizational structure for the demonstration, showing lines of communication, is provided on Figure 2-1.

2.3 RESPONSIBILITIES

PRC, in consultation with CRD, will be responsible for the following elements of this demonstration:

- Designing, preparing, overseeing, and implementing all elements of this demonstration plan

- Providing needed logistical support, establishing a communication network, and scheduling and coordinating the activities of all demonstration participants
- Ensuring that an appropriate site and appropriate analytes and matrices are selected for use in the demonstration
- Performing on-site sampling activities including collecting and homogenizing samples, dividing them into replicates, and bottling, labeling, and shipping them as necessary
- Providing monitoring, oversight, or operation of the technologies during the demonstration and documenting the experimental methodology and operation of each technology
- Developing a quality assurance project plan (QAPP) and a health and safety plan (HASP) for the demonstration activities
- Acquiring the necessary analytical data to support the demonstration design
- Managing, evaluating, interpreting, and reporting on data generated by the demonstration
- Evaluating and reporting on the performance of the technologies

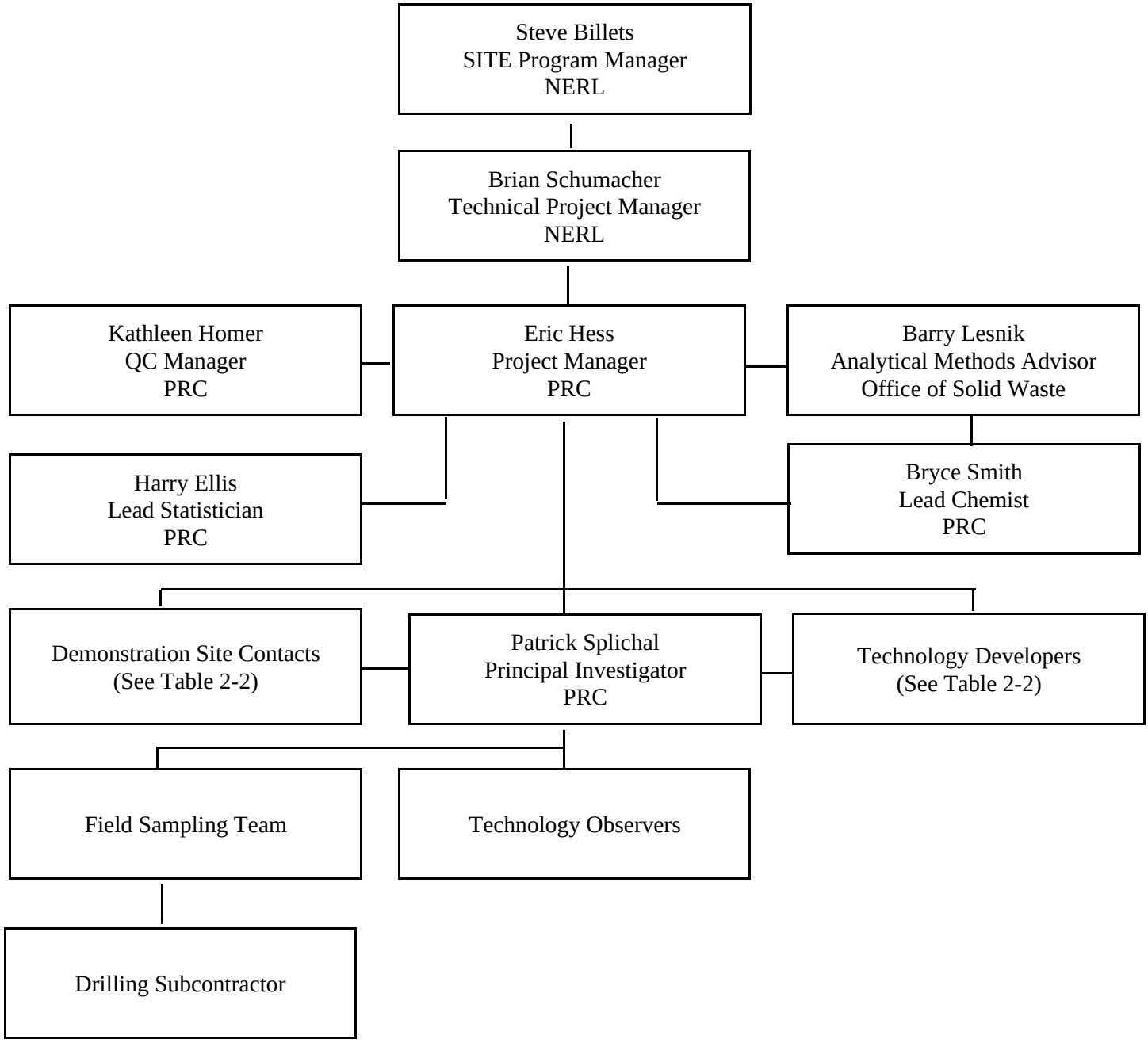
Each technology developer will be responsible for the following:

- Detailed protocols for using their respective technologies
- Review and comment on the demonstration plan and ITERs
- Complete, field-ready systems for demonstration
- Operation of the technologies during the demonstration or training on the operation of the technologies.
- Data reduction and interpretation support, as required
- Logistical, troubleshooting, and other support, as required

The site owners and the EPA contacts for each site will provide the following support:

- Site access
- Site characterization information
- Health and safety information
- Other logistical information and support needed to coordinate access to the site for the field portion of the demonstration

FIGURE 2-1 ORGANIZATIONAL CHART



Note: The field sampling team and technology observers will be assigned by technology. The drilling subcontractor will not change between sites

TABLE 2-1
TECHNOLOGY LIST
SAMPLING TECHNOLOGY DEMONSTRATION

Technology/Comments	Developer	Contact/Title	Address/Phone
SOIL			
JMC Environmentalist's Subsoil Probe. Collects undisturbed soil samples. - Manual system - Equipment has been manufactured since 1978.	JMC	Jim Clements, President	1992 Hunter Avenue Newton, IA 50208 (515) 792-8285 (515) 792-1361 (fax)
Large-Bore Soil Sampler and Macrocore® Soil Sampler. Soil sampling tools that are part of a complete sampling system.	Geoprobe®Systems, Inc.	Wes McCall, Technical Representative	601 N. Broadway Salina, KS 67401 (913) 825-1842 (913) 825-2097 (fax)
Dual Tube Liner Sampler. Continuous or discrete soil sampling tool, part of a sampling system designed for deep probing in site assessment of soils, soil gas, and groundwater.	Art's Manufacturing and Supply	Brian Anderson	105 Harrison St. American Falls, ID 83211 1(800) 635-7330 (208) 226-7280 (fax)
Simulprobe®. Soil, groundwater, soil gas sampler with fiber optic probe for hydrocarbon screening. Driven by hydraulic push rig, drill rig, or cone penetrometer truck. Can collect two media, such as soil and soil gas, simultaneously, uses fiber optic sensor to identify sample points.	Simulprobe® Equipment, Inc.	Noah Heller, President	150 Shoreline Highway Building E Mill Valley, CA 94941 (415) 331-2979 (415) 331-2665
Applied Research Associates Dynamil Thermal Desorber probe. Thermally desorbs volatile and semi-volatile compounds form the soil as the probe is advanced with a cone penetrometer. The desorbed gasses are drawn directly into a gas chromatograph for analysis. This system works continuously as the probe is advanced.	Applied Research Associates	Wes Bratton	Box 120A Waterman Road South Royalton, VT 05068 (802) 763-8348 (802) 763-8283 (fax)
Emflux® Soil Gas Investigation System. Passive soil gas sampling which can monitor volatile and some semi-volatile contaminants.	Quadrel Services, Inc.	Bruce Tucker, Chief Operating Officer	1896 Urbana Pike, Suite 20 Clarksburg, MD 20871 1(800)878-5510 (301) 874-5567 (fax)

TABLE 2-1 (Continued)

TECHNOLOGY LIST
SAMPLING TECHNOLOGY DEMONSTRATION

Technology/Comments	Developer	Contact/Title	Address/Phone
SOIL			
GORE-SORBER®. Soil gas sampler which can monitor volatile and some semi-volatile compounds.	W.L. Gore & Associates, Inc., Environmental Products Group	Mark Wrigley, Product Scientist	101 Lewisville Rd. Elkton, MD 21922-1100 (410) 996-3406 (410) 996-3325 (fax)

TABLE 2-2

DEMONSTRATION PARTICIPANTS
Soil and Soil Gas Sampling Technologies Demonstration

Agency/Company	Point of Contact
EPA Region 7 Superfund Branch 726 Minnesota Ave. Kansas City, KS 66101	Janice Kroone, On-scene Coordinator for the SBA site (913) 551-5190 (phone) (913) 551-5035 (fax)
EPA NERL 944 East Harmon Las Vegas, NV 89119	Steve Billets, SITE Program Manager (702) 798-2232 (phone) (702) 798-2261 (fax) Brian Schumacher, Technical Project Manager (702) 798-2242 (phone) (702) 798-2107 (fax)
EPA Office of Solid Waste (OSW) 401 M St. SW Washington, DC 20460	Barry Lesnik, Analytical Methods Advisor (202) 260-7459 (phone) (202) 260-1381 (fax)
EPA Region 8 Superfund Section USEPA 8EPR-SR 999 18th Street, Suite 500 Denver, CO 80202	Armando Saenz, Regional Project Manager Chemical Sales Site (303) 312-6559 (phone)
Simulprobe® 150 Shoreline Highway, Bldg. E Mill Valley, CA 94941	Noah Heller, President (415) 331-2979 (phone) (415) 331-2665 (fax)
Geoprobe® Systems, Inc. 601 N. Broadway Salina, KS 67401	Wes McCall, Technical Representative (913) 825-1842 (phone) (913) 825-2097 (fax)
Art's Manufacturing and Supply 105 Harrison St. American Falls, ID 83211	Brian Anderson (800) 635-7330 (phone) (208) 226-7280 (fax)
Colorado Department of Public Health and the Environment 4300 Cherry Creek Drive South Denver, CO 80222	Joe Vranka Chemical Sales Site (303) 692-3402 (phone) (303) 759-5355 (fax)
PRC 233 N. Michigan Avenue, Suite 1621 Chicago, IL 60601	Harry Ellis, Lead Statistician (312) 856-8700 (phone) (312) 938-0118 (fax)

TABLE 2-2 (Continued)

DEMONSTRATION PARTICIPANTS
Soil and Soil Gas Sampling Technologies Demonstration

Agency/Company	Point of Contact
PRC 7932 Nieman Road Lenexa, KS 66214	Eric Hess, Project Manager (913) 495-3910 (phone) (913) 894-6295 (fax) Patrick Splichal, Principal Investigator (913) 495-3920 (phone) (913) 894-6295 (fax) Bryce Smith, Lead Chemist (913) 495-3907 (phone) (913) 894-6295 (fax) Kathleen Homer, QC Manager (913) 495-3914 (phone) (913) 894-6295 (fax)
Applied Research Associates Box 120A Waterman Road South Royalton, VT 05068	Wes Bratton (802) 763-8348 (phone) (802) 763-8283 (fax)
W.L Gore & Associates, Inc. Environmental Products Group 101 Lewisville Rd. Elkton, MD 21922	Mark Wrigley, Product Scientist (410) 996-3406 (phone) (410) 996-3325 (fax)
Quadrel Services, Inc. 1896 Urbana Pike Suite 20 Clarksburg, MD 20871	Bruce Tucker, Chief Operating Officer (800) 878-5510 (phone) (301) 874-5567 (fax)
Clements and Associates, Inc. 1992 Hunter Ave. Newton, IA	Jim Clements, President (515) 792-8285 (phone) (515) 792-1361 (fax)

CHAPTER 3
TECHNOLOGY DESCRIPTIONS

This chapter describes the soil and soil gas sampling technologies manufactured by each developer. The descriptions appear verbatim from information provided by the developers. The descriptions include background information and a description of the equipment, the scientific principles behind the technologies, and their application histories. General operating procedures, training and maintenance requirements, and the cost of each technology also are discussed. Wherever provided, technology performance goals are presented, both those specific to the demonstration and those relevant to general applications.

3.1 GEOPROBE® SYSTEMS LARGE-BORE SOIL SAMPLER

The following subsections discuss the Large-Bore Soil Sampler developed by Geoprobe® Systems in detail.

3.1.1 Introduction

The Large-Bore Soil Sampler is a single tube-type, solid barrel, closed-piston sampler advanced by direct-push techniques that can be used cost effectively to collect high integrity, discrete interval, representative samples of unconsolidated materials at depth (Figure 3-1). The sampler is 24-inches long and has a 1.5-inch outside diameter. It is capable of recovering a discrete sample that measures up to 320 milliliters (mL) in volume in the form of a 22-inch x 1-1/16-inch (559 millimeter [mm] x 27 mm) core contained inside a removable liner. The liner is a 24-inch-long by 1-1/8-inch-diameter (610 mm x 29 mm) removable/replaceable, thin-walled tube that fits inside the Large-Bore sample tube. Liners facilitate retrieval of the sample and may be used for storage when applicable.

3.1.2 Components

The components of the Large-Bore Soil Sampler are shown on Figure 3-2. The Large-Bore Soil Sampler is a solid-barrel, closed-piston type sampler with only seven basic components. These are: piston tip, piston rod, drive head, stop-pin, sample tube, cutting shoe, and sample liner. The sampler is 24-inches

long and has an outside diameter of 1.5 inches. The liners are available in clear plastic (cellulose acetate butyrate), brass, stainless-steel, and Teflon® to meet the various sample collection requirements and data quality objectives (DQO) of the specific project. The fully assembled sampler weighs approximately 7.2 pounds.

The Large-Bore Soil Sampler can be driven to depth with the Geoprobe® percussion-probing machine or by manual methods. A sufficient number of drive rods (3- or 4-foot lengths) are required to advance the sampler to the target depth. Extension rods with connectors are needed to insert through the hollow drive rods and release the piston stop-pin. Drive caps and pull caps are needed to advance and then retract the sampler. Hand tools may be needed for disassembly after use. A manual extruder is available to extrude samples from the brass or stainless-steel liners. Geoprobe® Systems has developed grouting equipment that will allow for bottom-up re-entry grouting of the probe holes that meet American Society of Testing and Materials (ASTM) and state requirements.

3.1.3 Parameters or Analytes

When appropriate sample liner materials are used and the Large-Bore Soil Sampler is operated properly, soil samples can be collected for all environmental analytes of interest. This includes volatile organic compounds (VOC), metals (including the Resource Conservation and Recovery Act of 1976 [RCRA] metals), and cationic and anionic compounds, such as dichromate, hydrogen cyanide, nitrates.

3.1.4 Matrices

The Large-Bore Soil Sampler is designed to collect core samples of unconsolidated materials. These unconsolidated materials may include soils, sediments, and waste materials, or mixtures of these. This sampling device works best in medium- to fine-grained cohesive materials such as silty clay soils or sediments. The Large-Bore Soil Sampler also is used successfully in sampling medium- to coarse-grained sandy materials with some fine gravels. This sampling device is not designed for sampling consolidated bedrock, strongly cemented soils or sediments, or materials rich in coarse gravels, cobbles, or boulders.

3.1.5 Costs

The Large-Bore Soil Sampler is available in a kit which includes an extra cutting shoe. The cost of the kit is $305, plus shipping (1997 price list). Replacement parts are available individually if needed. Lease or rental of the Large-Bore Soil Sampler is not available through Geoprobe® Systems. However, it is possible to rent the soil sampler for $15 per week or $38.75 per month from a Geoprobe® rental company. A box of 100 acetate liners costs $130. The Geoprobe® percussion probing machine used to advance the soil sampler can be rented or leased. Information on the costs of all required supporting equipment for the use and operation of the Large-Bore Soil Sampler can be obtained from Geoprobe® Systems.

3.1.6 Training

No specialized training or education is required for operation of the Large-Bore Soil Sampler. Assembly and operation of the soil sampler is simple and can be learned in a matter of minutes. Advancing the sampler into the subsurface using manual methods can be labor intensive. Use of the Geoprobe® percussion-probing machine to advance the sampler minimizes the labor required, but does require some training to learn proper operation of the Geoprobe®. Usually, 2 to 4 hours of hands-on training is all that is required to learn the proper procedures for operating the Geoprobe® to successfully collect soil samples with the Large-Bore Soil Sampler.

The field manager of the project should have basic knowledge and understanding of the subsurface geology and geohydrology in the study area. This is recommended especially when the Large-Bore Soil Sampler is to be used at potentially hazardous waste sites. It is important that any confining layers (aquitards), such as a thick continuous clay layer, are not inadvertently penetrated and then not properly sealed by grouting. Penetration of an aquitard without emplacing a proper abandonment seal when the sampler is removed can allow migration of contaminants into formerly clean aquifers and lithologic units.

General health and safety training (Occupational Safety and Health Act [OSHA] 1910.21) is required for all workers on potentially hazardous waste sites. Following these health and safety guidelines, the

equipment operating procedures, the site specific sampling plan, and equipment decontamination procedures will enable the sampler to meet the project DQO's by obtaining representative samples.

3.1.7 General Operating Procedures

The following is a summary of the recommended operating procedure.

Before use and between each sampling event, the Large-Bore Soil Sampler and any supporting equipment that may come into contact with the sample should be cleaned and decontaminated to meet the project-specific DQOs. Next, the sampler is properly assembled and connected to the leading end of a drive rod and driven into the subsurface using a percussion-probing machine. Additional probe rods are connected in succession to advance the sampler to depth. The sampler remains sealed (closed) by a piston tip as it is being driven. The piston is held in place by a reverse-threaded stop-pin at the trailing end of the sampler. When the sampler tip has reached the top of the desired sampling interval, a series of extension rods, sufficient to reach depth, are coupled together and lowered down the inside diameter of the probe rods. The extension rods are then rotated clockwise using a handle. The male threads on the leading end of the extension rods engage the female threads on the top end of the stop-pin, and the pin is removed. After the extension rods and stop-pin have been removed, the tool string is advanced an additional 24 inches. The piston is displaced inside the sampler body by the soil as the sample is cut. To recover the sample, the sampler is retrieved from the hole and the liner containing the soil sample is removed. The operation is summarized on Figure 3-1. If samples are to be subsampled for chemical analysis, this should be done as soon as possible using appropriate procedures, containers, and preservation methods.

The Large-Bore Soil Sampler should be cleaned and decontaminated between each use to meet project DQO. Excess soil should be thoroughly cleaned from the threads of each part to assure a smooth assembly and to reduce damage to the threads. If burrs develop on the threaded connections after use, a hand file or rotary grinding tool may be used to touch up the fittings. The cutting shoe should be maintained to prevent blockage of the sampler opening. If the cutting shoe becomes broken or damaged by improper use or application of excessive force, it can easily be replaced.

When state or local regulations require, the probe holes can be grouted. Geoprobe® Systems has developed special grouting equipment for direct-push applications. This grouting equipment will allow for re-entry bottom-up grouting of the probe holes that meet ASTM and state requirements. Grouting also can be done by gravity installation of bentonite chips through the open probe hole from the surface when acceptable.

3.1.8 Performance Range

The Large-Bore Soil Sampler has been used to collect samples of unconsolidated materials at depths ranging from ground surface to over 100 feet below grade. The depth that can be reached with the soil sampler is a function of the equipment and methods used to advance the sampler (manual, static vehicle weight, or percussion probing), the formation being penetrated, and operator experience.

3.1.9 Licensing Requirements

Currently, most state and local agencies do not require licensing or permits to collect soil samples with the Large-Bore Soil Sampler. This may be a function of how the samples are collected, either manually, with a direct-push machine, or with a drill rig. Some states do place depth limits on operation without a license or permit, and some states limit sampling to <u>not</u> penetrate the water table. The soil sampler or equipment operator must contact the appropriate state or local agencies to determine if there are any license or permit requirements.

3.1.10 History

Implementation of environmental laws and regulations created a demand for a simple, reliable, and cost effective means of collecting representative soil samples for chemical analyses. In the late 1980s Geoprobe® Systems began development of percussion-probing (direct push) techniques. One of the first soil samplers designed for percussion probing was a simple closed-barrel, piston-type sampler called the Kansas Sampler. This sampler allowed the operator to collect a soil core approximately 1-foot long and 1-inch in diameter that had to be extruded from the unlined sample barrel. The need in the environmental industry for larger sample volumes, collection of samples in liners to minimize sample handling, and

replaceable cutting shoes to reduce equipment costs led to the development of the Large-Bore Soil Sampler.

3.1.11 Applications

The Large-Bore Soil Sampler can be used for many applications. When properly operated, high integrity, representative samples may be obtained. The soil sampler is used during site investigations, Brownfields assessments, remedial investigations/feasibility studies, RCRA facility assessments, RCRA facility investigations, corrective actions, underground storage tank (UST) investigations and removals, and other environmental, and hydrogeological studies. The following is a listing of some of the uses of the Large-Bore Soil Sampler:

- Sampling soils and sediments for chemical analyses for all environmental analytes and other parameters to provide accurate qualitative and quantitative data for determination of presence, absence, and accurate concentrations.
- Sampling to determine vertical and horizontal extent of environmental contamination or other chemical or physical parameters of interest. Resultant analytical data can be plotted to construct isopleth maps when samples are accurately located.
- Closure sampling for RCRA solid waste management units (SWMU) and USTs to meet state and federal regulatory requirements.
- Sampling unconsolidated materials, including soils and sediments, for visual or manual determination of soil types for ASTM and Unified Soil Classification System (USCS) soil classification.
- Sampling soils and sediments to determine subsurface lithology for preparation of boring logs, geologic cross sections, and soils or geologic maps.
- Sampling unconsolidated materials, including soils and sediments, for grain-size analysis and determination of other physical parameters.

3.1.12 Advantages

The Large-Bore Soil Sampler improves on conventional drilling and sampling techniques for unconsolidated materials in many ways. Some of these are summarized below.

- Representative Samples and Sample Integrity/Disturbance: Proper use of the Large-Bore Soil Sampler enables the operator to collect soil samples with minimal physical and chemical disturbance, providing representative samples. Minimal disturbance assures a high degree of sample integrity and data quality. In some standard sampling methods, formation heave or blow-in can result in significantly disturbed samples being collected from an undefinable depth interval.

- Accurate Sampling Interval: Proper use of the Large-Bore Soil Sampler allows the operator to collect representative samples with a high degree of accuracy from the targeted depth. The ability to precisely control the depth to be sampled allows the operator to accurately sample from a predetermined interval. In some standard sampling methods, formation heave or blow-in can result in disturbed samples being collected from an undefinable depth interval.

- Waste Minimization: No drill cuttings are generated during the sample collection process. This eliminates the need to handle, contain, store, sample, analyze, and dispose of potentially hazardous and contaminated drill cuttings during environmental investigations. This will significantly reduce costs. Eliminating drill cuttings also will reduce the potential exposure of site workers, facility employees, local residents, and sensitive environments or species to hazardous contaminants.

- Recovery: Sample recoveries of 100 percent can often be achieved when the Large-Bore Soil Sampler is properly operated. This is a function of the formation being sampled and operator experience.

- Efficiency: The Large-Bore Soil Sampler operated with the Geoprobe® percussion probing machine is one of the most efficient means of collecting a discrete soil sample at depth. Since this is a single-tube system, the use and advancement of an outer casing, or hollow-stem augers, to successfully collect a sample is not required. This minimizes the time and effort required to collect a discrete interval sample and eliminates generation of drill cuttings.

- Location Accessibility: The Large-Bore Soil Sampler can be operated with relatively small mobile, hydraulically-powered direct push equipment, or with manual equipment. This allows the operator to reach many locations not accessible to larger and heavier conventional drilling equipment.

- Drilling Fluids: No drilling fluids are required for the proper operation of the Large-Bore Soil Sampler. The use of drilling fluids, even distilled water to maintain hydraulic equilibrium to prevent formation heave or blow-in, can cause chemical changes in the sample resulting in nonrepresentative samples.

- Surface Disturbance: Minimal disturbance to the ground surface is generated during the sampling process. This is because a hole only 1.5 inches in diameter is created, no drill cuttings are generated, and smaller, lighter equipment can be used to successfully collect samples with the Large-Bore Soil Sampler. This can reduce project costs by eliminating

or minimizing the need to return the sampling location to original condition (e.g., replacing damaged turf or re-leveling the ground surface).

3.1.13 Limitations

The Large-Bore Soil Sampler is not designed to penetrate or collect samples of consolidated bedrock, such as limestone, sandstone, or granites. Samples have been collected of poorly consolidated bedrock formations such as soft or weathered shales, siltstones, or sandstones. If soils are strongly cemented, such as a hard caliche, it may be difficult to collect samples. Some glacial tills or alluvial sediments containing large boulders, cobbles, or abundant coarse gravels may limit penetration, sampling success, and recovery.

3.2 GEOPROBE® SYSTEMS MACRO-CORE® SOIL SAMPLER

Geoprobe® Systems has developed another soil sampler tool called the Macro-Core®. The Macro-Core® is similar in design and function to the Large-Bore Soil Sampler, except that it gives a larger sample. This sampler will not be directly evaluated during this demonstration.

Introduction

The Macro-Core® soil sampler is a single, tube-type, solid-barrel soil sampler advanced by direct-push techniques that can be used cost effectively to collect representative continuous core samples of unconsolidated materials in 4-foot increments from ground surface to depth. The Macro-Core® sampler can be operated as an open-tube sampler or as a closed piston sampler (Figure 3-3). The Macro-Core® sampler is a 48-inch-long x 2.0-inch (1,219 mm x 51 mm)-outside diameter soil sampler capable of recovering a sample that measures up to 1,300 mL in volume in the form of a 45-inch x 1.5-inch (1,143 mm x 38 mm) core. The samples are recovered in a liner inserted inside the Macro-Core® sample tube. Liners are 46 inches (1,168 mm) long and 1.5 inches (38 mm) in diameter. The liners are available in stainless-steel, Teflon®, polyvinyl chloride (PVC), and polyethyle terephthalate glycol modified copolyester (PETG).

Components

The components of the Macro-Core® soil sampler are shown on Figure 3-4. The Macro-Core® soil sampler is a solid, barrel-type sampler that may be operated as an open-tube or closed-piston sampler. The open-tube system has only five basic parts. These include the sample tube, drive head, cutting shoe, sample liner, and either a spacer ring or a core catcher. The closed-piston system operates with these same basic parts, but also includes the closed-piston assembly. This assembly consists of four parts which are not disassembled during normal operation of the sampler.

The sample liners are available in stainless-steel, Teflon®, PVC, and PETG. The spacer ring and core catchers are manufactured of PVC. Vinyl end caps are available to close the ends of the sample liners for sample storage. If samples are to be subsampled for chemical analysis this should be done as soon as possible using appropriate procedures, containers, and preservation methods. The fully assembled Macro-Core® open-tube soil sampler weighs approximately 14.1 pounds. The closed-piston assembly weighs approximately 20 pounds.

The Macro-Core® soil sampler can be driven to depth with the Geoprobe® percussion-probing machine or by manual methods. A sufficient number of drive rods (3- or 4-foot lengths) are required to advance the sampler to the target depth. When operating the closed piston system, the Macro-Core® release rod and extension rods with connectors are needed to insert through the hollow drive rods to release the locked closed-piston assembly. Drive caps and pull caps are needed to advance and then retract the sampler. Hand tools, including the Macro-Core® combination wrench, may be needed for assembly and disassembly after use. Geoprobe® Systems has developed grouting equipment that will allow for re-entry bottom-up grouting of the probe holes that meet ASTM and state requirements.

Parameters or Analytes

When appropriate sample liner materials are used and the Macro-Core® soil sampler is operated properly, soil samples can be collected for all environmental analytes of interest. This includes VOC (including petroleum products), metals (including the RCRA metals), and cationic and anionic compounds, such as dichromate, hydrogen cyanide, nitrates. The Macro-Core® soil sampler can also be used to collect samples to be submitted for biological assays.

Sample Matrices

The Macro-Core® soil sampler is designed to collect core samples of unconsolidated materials. These unconsolidated materials may include soils, sediments, and waste materials, or mixtures of these. This sampling device works best in medium- to fine-grained cohesive materials such as silty clay soils or sediments. The Macro-Core® sampler is also used successfully in sampling medium- to coarse-grained sandy materials with some fine to medium gravels when core catchers are used. This sampling device is not designed for sampling consolidated bedrock, strongly cemented soils or sediments, or materials rich in coarse gravels, cobbles, or boulders.

Costs

The Macro-Core® soil sampler is available in several different kits to meet the specific needs of the sampler. Some of the different kits available and costs are listed below (based on January 1997 prices).

- MC Starter Kit, nickel (Ni)-Plated (fits 1.25-inch probe rods): Cost = $734 + shipping.

  This closed-piston sampling kit includes the following parts:

  1 - MC closed piston kit
  1 - MC drive head
  1 - MC sample tube, Ni-plated
  1 - MC cutting shoe
  1 - MC release rod
  2 - MC combination wrenches
  2 - Nylon brushes for MC tubes

- MC Standard Sampler Kit, Unplated (fits 1-inch rods): Cost = $365 + shipping.
  This open-tube sampling kit includes the following parts:

  1 - MC drive head
  1 - MC sample tube, unplated
  1 - MC cutting shoe

The MC closed-piston kit is available separately ($240 + shipping). Liners are available in boxes of 66 including 66 spacer rings. Core catchers are available in boxes of 25. Replacement parts for the Macro-Core® soil sampler are available individually if needed.

Lease or rental of the Macro-Core® soil sampler is not available through Geoprobe®. However, the open-tube sampling kit can be rented for $20.50 per week or $52.75 per month from a Geoprobe® rental company. The closed-tube sampling kit rents for an additional $14.25 per week or $37.00 per month. A box of 66 PETG liners costs $112. The Geoprobe® percussion-probing machine used to advance the sampler can be rented or leased. Information on the costs of all required supporting equipment for the use and operation of the Macro-Core® soil sampler can be obtained from Geoprobe® Systems.

Training

No specialized training or education is required for operation of the Macro-Core® soil sampler. Assembly and operation of the Macro-Core® sampler is simple and can be learned in a matter of minutes. Operation of the closed-piston system requires proper maintenance to assure reliable operation. Advancing the Macro-Core® sampler into the subsurface using manual methods can be labor intensive. Use of the Geoprobe® percussion-probing machine to advance the Maro-Core® sampler minimizes the labor required, but does require some training to learn proper operation of the Geoprobe®. Usually, 2 to 4 hours of hands-on training is all that is required to learn the proper procedures for operating the Geoprobe® to successfully collect soil samples with the Macro-Core® sampler.

The field manager of the project should have basic knowledge and understanding of the subsurface geology and geohydrology in the study area. This is recommended especially when the Macro-Core® soil sampler is to be used at potentially hazardous waste sites. It is important that confining layers (aquitards), such as a thick continuous clay layer, are not inadvertently penetrated and then not properly sealed by grouting. Penetration of an aquitard without emplacing a proper abandonment seal when the Macro-Core® sampler is removed can allow migration of contaminants into formerly clean aquifers and lithologic units.

General health and safety training (OSHA 1910.21) is required for all workers on potentially hazardous waste sites. Following these health and safety guidelines, the equipment operating procedures, the site-specific sampling plan, and equipment decontamination procedures will enable the Macro-Core® Soil Sampler to meet the project DQOs by obtaining representative samples.

General Operating Procedures

The following is a summary of the recommended operating procedure.

Before use and between each sampling event, the Macro-Core® Soil Sampler and any supporting equipment that may come into contact with the sample, should be cleaned and decontaminated to meet the project-specific DQOs. After decontamination, the properly assembled Macro-Core® sampler is driven into the subsurface using a Geoprobe® percussion-probing machine. Probe rods are connected in succession and driven to advance the Macro-Core® sampler to depth. The Macro-Core® soil sampler may be used as an open-tube or closed-piston sampler.

The simplest use of the Macro-Core® soil sampler is as an open-tube sampler (Figure 3-5). In this method, coring starts at the ground surface with an open-ended sampler. From the ground surface, the Macro-Core® sampler is advanced 48 inches and retrieved from the hole with the first soil core. In stable soils the open-tube sampler is inserted back down the hole to obtain the next core. Geoprobe® operators have reported coring to depths exceeding 30 feet (9 meters [m]) with this method.

In unstable soils which tend to collapse into the core hole, the Macro-Core® soil sampler can be equipped with a closed-piston assembly. This assembly locks into the cutting shoe and prevents soil from entering the sampler as it is advanced down the existing hole.

The Macro-Core® closed-piston sampler is not designed to be driven through undisturbed soil (Figure 3-6). Soil is first removed to sampling depth with an open-tube sampler, or a pilot hole may be made with a Macro-Core® pre-probe. A closed-piston tip is then installed and the Macro-Core® sampler is inserted or driven back down the same hole. When the leading end of the Macro-Core® sampler reaches the top of the next sampling interval, the piston is unlocked using extension rods to insert the Macro-Core® release rod down the inside of the probe rods.

Once the piston is released, the Macro-Core® Soil Sampler is driven another 48 inches. Soil entering the Macro-Core® sampler pushes the piston assembly to the top of the Macro-Core® sampler where it is retrieved upon removal of the soil liner and core. The soil sample is removed from the Macro-Core® sampler by unscrewing the cutting shoe and pulling out the liner. If desired, the sample may be secured

inside the liner by placing a vinyl end cap on each end of the liner. If samples are to be subsampled for chemical analysis this should be done as soon as possible using appropriate procedures, containers, and preservation methods.

Loose soils will sometimes fall out of the Macro-Core® soil sampler as it is retrieved from depth. The Macro-Core® catcher (Figure 3-4) was designed to alleviate this problem. Excellent results are obtained when the core catcher (sometimes called a basket retainer) is used with saturated sands and other noncohesive soils. A core catcher is not necessary when sampling cohesive soils and may actually inhibit sample recovery in some soft soils or sediments. The core catcher is made of PVC and it may be used with either the PVC, PETG, Teflon®, or the stainless-steel liners.

The Macro-Core® Soil Sampler should be cleaned and decontaminated between each use to meet project DQOs. Excess soil should be thoroughly cleaned from the threads of each part to assure a smooth assembly and to reduce damage to the threads. If burrs develop on the threaded connections after use, a hand file or rotary-grinding tool may be used to touch up the fittings. The cutting shoe should be maintained to prevent blockage of the sampler opening. If the cutting shoe becomes broken or damaged by improper use or application of excessive force, it can easily be replaced.

When state or local regulations require, the probe holes can be grouted. This can be done by gravity installation of bentonite chips from the surface when acceptable. Geoprobe® Systems also has developed special grouting equipment for direct push applications. This grouting equipment will allow for re-entry, bottom-up grouting of the probe holes that meet ASTM and state requirements.

Performance Range

The Macro-Core® Soil Sampler has been used to collect samples of unconsolidated materials at depths ranging from ground surface to over 50 feet below grade. The depth that can be reached with the Macro-Core® Soil Sampler is a function of the equipment and methods being used to advance the sampler (manual, static vehicle weight, or percussion probing), the formation being penetrated, and operator experience. The Macro-Core® closed-piston sampler is not designed to be driven through virgin soil or sediments. Soil is first removed to the sampling depth with an open-tube sampler, or a pilot hole may be made to sampling depth with a Macro-Core® pre-probe.

Licensing Requirements

Currently, most state and local agencies do not require licensing or permits to collect soil samples with the Macro-Core® Soil Sampler. This may be a function of how the samples are collected, either manually, with a direct-push machine, or with a drill rig. Some states do place depth limits on operation without a license or permit, and some states limit sampling to not penetrate the water table unless a licensed operator or registered geologist is present. The sampler or equipment operator must contact the appropriate state or local agencies to determine if there are any license or permit requirements.

History

Implementation of environmental laws and regulations created a demand for a simple, reliable, and cost effective means of collecting representative soil samples for chemical analyses and physical identification. The increasing need in the industry to collect larger sample volumes and conduct continuous soil coring with direct-push equipment lead to the development of the Macro-Core® Soil Sampler by Geoprobe® Systems. The Macro-Core® Soil Sampler was initially designed as a simple open-tube sampler with a replaceable cutting shoe and sample liner. This system was designed for sampling in cohesive soils to depths of 15 to 30 feet (5 to 9 meters).

Because the Macro-Core® Soil Sampler was being operated at greater depths in noncohesive materials than allowed for by the initial open-tube system design, borehole slough and caving became a concern. If sloughing or caving occurred, then material from a higher interval in the borehole could be collected at the top of the next sample interval. This led to the development of the Macro-Core® closed-piston system. The closed-piston assembly locks into the cutting shoe and prevents soil from entering the sampler as it is advanced in the existing hole to the top of the next sampling interval. This system allows the operator to collect continuous cores of undisturbed, representative soils and sediments to depths exceeding 50 feet (17 meters).

Applications

The Macro-Core® Soil Sampler can be used for many applications. When properly operated, high integrity, representative samples may be obtained. The Macro-Core® sampler is used during site investigations, Brownfields assessments, remedial investigations/feasibility studies, RCRA facility assessments, RCRA facility investigations, corrective actions, UST investigations and removals, and other environmental, soils, and geological studies. The following is a summary of some of the useful applications for the Macro-Core® Sampler:

- Sampling soils and sediments for chemical analyses for all environmental analytes, and other parameters of interest. The samples can provide accurate qualitative and quantitative data for determination of presence, absence, and accurate concentrations of environmental contaminants or other parameters of interest.
- Sampling to determine vertical and horizontal extent of environmental contamination or other chemical or physical parameters of interest. Resultant analytical data can be plotted to construct isopleth maps when samples are accurately located.
- Closure sampling for RCRA SWMUs and USTs to meet state and federal regulatory requirements.
- Sampling unconsolidated materials, including soils and sediments, for visual/manual determination of soil types for ASTM and USCS soil classifications.
- Sampling soils and sediments to determine subsurface lithology for preparation of boring logs, geologic cross sections, and soils or geologic maps.
- Sampling unconsolidated materials, including soils and sediments, for grain-size analysis and determination of other physical parameters.

Advantages

The Macro-Core® Soil Sampler improves on conventional drilling and sampling techniques for unconsolidated materials in many ways. Some of these are summarized below.

- Representative Samples and Sample Integrity/Disturbance: Proper use of the Macro-Core® Soil Sampler enables the operator to collect soil samples with minimal physical and chemical disturbance, providing representative samples. Minimal disturbance assures a high degree of sample integrity and data quality. In some

traditional sampling methods formation heave or blow-in can result in significantly disturbed samples being collected from an indefinable depth interval.

- Accurate Samples: Proper use of the Macro-Core® Soil Sampler allows the operator to collect representative samples with a high degree of accuracy from the targeted depth. In some standard sampling methods formation heave or blow-in can result in samples being collected from an indefinable depth interval.

- Waste Minimization: No drill cuttings are generated during the sample collection process. This eliminates the need to handle, contain, store, sample, analyze, and dispose of potentially hazardous and contaminated drill cuttings during environmental investigations. This will significantly reduce costs. Eliminating drill cuttings also will reduce the potential exposure of site workers, facility employees, local residents, and sensitive environments or species to hazardous contaminants.

- Recovery: Sample recoveries of 100 percent can often be achieved when the Macro-Core® Soil Sampler is properly operated. This is a function of the formation being sampled and operator experience.

- Efficiency: The Macro-Core® Soil Sampler operated with the Geoprobe® percussion-probing machine is one of the most efficient means of collecting continuous soil cores in unconsolidated materials. Since this is a single-tube system, the use and advancement of an outer casing, or hollow-stem augers, to successfully collect a sample is not required. This eliminates generation of drill cuttings.

- Drilling Fluids: No drilling fluids are required for the proper operation of the Macro-Core® Soil Sampler. The use of drilling fluids, even distilled water to maintain hydraulic equilibrium to prevent formation heave or blow-in, can cause chemical changes in the sample resulting in nonrepresentative samples.

- Location Accessibility: The Macro-Core® soil sampler can be operated with relatively small, mobile, hydraulically-powered, direct-push equipment, or with manual equipment. This allows the operator to reach many locations not accessible to larger and heavier conventional drilling equipment.

- Surface Disturbance: Minimal disturbance to the ground surface is generated during the sampling process. This is because a hole only 2 inches in diameter is created, no drill cuttings are generated, and smaller, lighter equipment can be used to successfully collect samples with the Macro-Core® Soil Sampler. This can reduce project costs by eliminating or minimizing the need to return the sampling location to original condition (e.g., replacing damaged turf or re-leveling the ground surface).

Limitations

The Macro-Core® Soil Sampler is not designed to penetrate or collect samples of consolidated bedrock such as limestone, slate, or granites. Samples have been collected of poorly consolidated formations such as soft or weathered shales, siltstones, or sandstones. If soils are strongly cemented, such as a hard caliche, it may be difficult to collect samples. Some glacial tills or alluvial sediments containing large boulders, cobbles, or abundant coarse gravels may limit penetration, sampling success, and recovery.

3.3 ENVIRONMENTALIST'S SUBSOIL PROBE

The following subsections discuss the Environmentalist's Subsoil Probe (ESP) developed by JMC in detail.

3.3.1 Introduction

Many soils with few or no stone fragments greater than 0.5 inch in diameter can be sampled for various contaminants with a small-diameter (0.8 inch), hand-operated percussive soil sampling device to depths of 20 feet.

3.3.2 Components

Basic equipment required to reach a 20 foot depth:

- 1- PN150 ESP (see Figure 3-7: includes the jack, slide hammer and sampling tube)
- 1 - PN157 Master extension
- 5 - PN158 Regular extensions
- 1 - PN215 Electric hammer with adapter (optional)

3.3.3 Parameters or Analytes

When the ESP is operated properly, soil samples can be collected for all environmental analytes of interest. This includes VOCs, SVOCs, and metals (including the RCRA metals).

3.3.4 Matrices

The ESP is designed to collect core samples of unconsolidated materials. These unconsolidated materials may include soils, sediments, and waste materials, or mixtures of these. This sampling device works best in medium- to fine-grained cohesive materials such as silty clay soils or sediments. The ESP also is used successfully in sampling medium- to coarse-grained sandy materials with some fine gravels. This sampling device is not designed for sampling consolidated bedrock, strongly cemented soils or sediments, or materials rich in coarse gravels, cobbles, or boulders.

3.3.5 Costs

Basic costs required to reach a 20 foot depth:

- 1- PN150 Environmentalist's Subsoil Probe (includes the jack, slide hammer and sampling tube): $1826.50
- 1 - PN157 Master extension: $186.70
- 5 - PN158 Regular extensions: $107.20 each
- 1 - PN215 Electric hammer with adapter (optional): $1,345.00

3.3.6 Training

Because of the simplicity of the equipment, reading the owner's manual is usually all the training required.

3.3.7 General Operating Procedures

The following provides a step-by-step instruction for using the ESP (Figures 3-8a through 3-8c):

1. Lay the ESP on the ground and insert liner.
2. Insert hammer assembly into sampling tube.
3. Put pedal depressor into drive-mode position.
4. Tip ESP with hammer assembly to vertical position.
5. Drive sampling tube into the ground.
6. Move pedal depressor to jacking mode position.
7. Release jack lever.
8. Jack sampling tube up 2 feet.
9. Remove hammer assembly and continue jacking.
10. Lay ESP on ground. Unload liner and soil core.
11. Insert new liner.
12. Attach master extension assembly. Check ball plungers, and insert and tape pins.
13. Place ESP vertically over the hole and push sampling tube down hole.
14. Depress pedal slightly with foot pressure and lift ESP 6 to 8 inches.
15. Step down on pedal forcing tube downward.
16. Repeat up and down movement until tube is at the bottom of hole.
17. Insert hammer assembly into the top of master extension.
18. Drive sampling tube into the ground.
19. Move pedal depressor to jacking mode position.
20. Release jack lever.
21. Jack sampling tube up 2 feet.
22. Remove hammer assembly. Finish jacking operation.
23. Return jack lever to upright position and secure with plastic knob.
24. Lay ESP on the ground.
25. Detach master extension adapter from sampling tube.
26. Unload liner and soil core.
27. Load new liner.

28. Reattach master extension adapter to sampling tube. Check pin and ball plunger. Then tape connection.
29. Disconnect master extension adapter from master extension tube.
30. Attach regular extension to master extension adapter. Check pin and ball plunger. Then tape connection.
31. Attach master extension to regular extension. Electrical tape optional.
32. Repeat steps 13 through 28.

3.3.8 Performance Range

None specified.

3.3.9 Licensing Requirements

None specified.

3.3.10 History

Clements Associates, Inc., was asked by American Cyanamid in the late 1980s to develop a hand-operated sampler to take soil samples from their "hot" studies. The contaminants were agricultural chemicals with radioisotope traces which were applied to agricultural soils and were part of what are referred to as "soil dissipation studies" or "environmental fate studies." This equipment is used by several agricultural chemical companies for these and nonradioisotope studies to a maximum depth of 4 feet. Using extensions to the apparatus, depths of 15 to 20 feet are possible in many soils. Much of the physical labor is eliminated by using an electric hammer. The use of extensions and the electric hammer were both suggestions made by several of the environmental consultants using JMC's basic apparatus. In fact, the environmental consultant's interest in the equipment caused JMC to name it the "Environmentalist's Subsoil Probe" or ESP.

3.3.11 Applications

As a sampling device for agricultural chemical soil dissipation studies, the ESP has the following advantages:

- Easily transported. Sites may be several hundred miles apart.
- Field work can be done regardless of field conditions. These are studies where the sampling intervals are predetermined and very rigid.
- Higher speed and lower manpower requirements than other methods.
- Low investment compared to vehicle mounted machines.

Other applications include as a primary sampling device for small jobs requiring less than 50 borings to depths of less than 20 feet or inside buildings, and as a reconnaissance tool for environmental consultants.

3.3.12 Advantages

The ESP offers the following advantages:

- Light weight
- Portable
- Can be a manual operation
- Labor can be minimized by using an electric hammer
- Low cost
- Can be used inside buildings or places with low ceilings
- Can be used on steep terrain, in dense vegetation, or in areas accessible only on foot

3.3.13 Limitations

- Can not core through rocks
- 20-foot depth capacity

3.4 EMFLUX® SOIL GAS INVESTIGATION SYSTEM

The following subsections discuss the Emflux® Soil Gas Investigation System (Emflux®) developed by Quadrel Services (Quadrel).

3.4.1 Introduction

Emflux® is distinguishable by its capacity, through Quadrel's proprietary computer model (which is based on the gravitational phenomenon known as "earth tides"), to predict periods of maximum soil gas emission for any geographic location. The underlying scientific theory was developed specifically to relate empirical data involving gravitational effects to orders-of-magnitude changes in the vertical velocities of gases moving through the earth's crust and to make possible the aforementioned predictions of "favorable" (*i.e.*, relatively high-vertical-velocity) periods. Knowing when these favorable periods occur and how to take advantage of them increases soil gas accuracy in locating and mapping subsurface chemical contamination to formerly unattainable levels.

3.4.2 Components

Standard Emflux® field kits for shallow subsurface sampling (collectors are placed about 4 inches below ground surface [bgs]) are 3 inches high by 9 inches long by 9 inches wide and weigh some 5 pounds. Field kits for surface-based sampling are 9 inches high by 9 inches wide by 19 inches long, and weigh about 25 pounds. Figure 3-9 provides line drawings of key components. For reasons of accuracy and sensitivity Quadrel prefers to use standard laboratory facilities whenever possible but, if required, can provide field gas chromatograph (GC) equipment and mobile laboratories.

The Emflux® sample cartridges themselves contain 100 milligrams (mg) each of selected adsorbent(s) and collect nanogram masses of contaminant. Neither sample volume nor weight is significant.

3.4.3 Parameters or Analytes

The Emflux® System has proved capable of accurately identifying and proportionally patterning a broad spectrum of VOCs and SVOCs, including halogenated compounds, petroleum hydrocarbons (aromatics,

aliphatics, etc.), polynuclear aromatic hydrocarbons (PAH), and methane to concentrations at and below parts per billion (ppb) levels.

3.4.4 Sample Matrices

Emflux® has proved capable of accurately identifying soil gas on open land, beneath artificial surfacing, and under water; in extremes of terrain (*e.g.*, desert, mountain, and arctic regions), weather (*e.g.*, rain, snow, heat, high and low pressures), and soils (*e.g.*, rocky, muddy, clays, caliche), to depths exceeding 200 feet.

3.4.5 Costs

All-inclusive, per-sample costs for Emflux® surveys – from initial planning, mobilization, and field work through demobilization, laboratory analysis, and reporting – range from approximately $85 to about $275, depending on number and location of sites, type of analysis, assignment of field work, number of sample points, number of target compounds, quantity and type of maps, extent of QA/QC, etc.

3.4.6 Training

Complete written instructions for sample-collector deployment and retrieval accompany all Emflux® field kits. When deemed desirable or necessary by a client, Quadrel also furnishes a 16-minute training video. But neither the video nor other forms of training are usually required or needed, on-site or off.

3.4.7 General Operating Procedures

The following field procedures are routinely used during Emflux® soil-gas surveys. Modifications can be and are incorporated from time to time in response to individual project requirements. In all instances, Quadrel adheres to EPA-approved QA/QC practices.

1. Field personnel carry Emflux® components and support equipment to the site and deploy the Emflux® collectors in a prearranged survey pattern. Although Emflux® collectors

require only one person for emplacement and retrieval, the specific number of field personnel required depends on the scope and schedule of the project. Each collector emplacement generally takes less than 2 minutes.

2. For those sample locations covered with debris or vegetation, a field technician clears vegetation and debris exposing the ground surface. Using a hammer and a 3/4-inch-diameter pointed metal stake, the technician creates a hole approximately 3 inches deep. For those locations covered with asphalt or concrete cap, the field technician drills a 1-inch-diameter hole through the cap to the soils beneath. (If necessary, the collector can be sleeved with a 3/4-inch inside diameter (i.d.) copper pipe for either capped or uncapped locations).

3. The technician then removes the solid plastic cap from an Emflux® collector (a glass vial containing an adsorbent cartridge with a length of wire attached to the vial for retrieval) and replaces it with a sampling cap (a plastic cap with a hole covered by screen meshing). The technician inserts the collector, with the sampling cap end facing down, into the hole (Figure 3-10). (The collector is then covered with either local soils for uncapped locations, aluminum foil and a concrete patch.) The collector's location, time and date of emplacement, and other relevant information are recorded on the Field Deployment Form.

4. As a QC check during emplacement and retrieval, the technician takes periodic ambient-air control samples and records the date, time, and location of each. (One or more trip blanks are also included as part of the QC procedures.)

5. Once all Emflux® collectors have been deployed, field personnel schedule collector recovery (approximately 72 hours after emplacement) and depart, taking all no-longer-needed equipment and materials with them.

6. Field personnel retrieve the collectors at the end of the 72-hour exposure period. At each location, a field technician withdraws the collector from its hole and wipes the outside of the vial clean using gauze cloth; following removal of the sampling cap, the threads of the vial are also cleaned. A solid plastic cap is screwed onto the vial and the sample location number is written on the label. The technician then records sample point location, date, time, etc., on the Field Deployment Form.

7. Sampling holes are refilled with soil, sand, or other suitable material. If Collectors have been installed through asphalt or concrete, the hole is filled to grade with a plug of cold patch or cement.

8. Following retrieval, field personnel ship or carry the Emflux® collectors to analytical laboratories under contract to Quadrel. The remaining equipment is returned to Quadrel's preparation facility.

3.4.8 Performance Range

Detection limits associated with Emflux® obviously depend on the equipment and analytical methods used in any given project. However, Emflux® detects nanogram levels of contamination and in general, taking all laboratory methods into account, has a reportable quantitation limit range of 25 nanograms to 1×10^6 nanograms. With regard to source concentrations in the field, the most sensitive Emflux® surveys to date have involved groundwater contamination in the low parts per trillion (ppt).

3.4.9 Licensing Requirements

There are no special licensing requirements needed for using Emflux®.

3.4.10 History

The present form of Emflux® System is the product of some 30 to 35 years of evolution that started with initial work in the uranium industry (where the method was developed in connection with attempts to use radon gas as an exploration tool). On a formal level, initial performance data on Emflux® was collected under the auspices of the National Environmental Technology Applications Center (NETAC) in 1989; subsequent performance data stems from field work done for government and commercial clients on sites in 39 states and in Canada, including projects under the purvue of EPA, Department of Energy (DOE), Department of Defense (DoD) (primarily through the U.S. Army Corps of Engineers), state environmental agencies, and other regulatory bodies. Among the private companies which have used Emflux® are Bechtel Corp., IT Corp., Law Environmental, RUST Environment & Infrastructure, Radian Corp., ABB, SAIC, EG&G, Black & Veach, Brown & Caldwell, Brown & Root, Dames & Moore, and CH2M-Hill. For legitimate and appropriate uses, field data is undoubtedly available from many of the foregoing sources.

During its commercial history, Emflux® has been used on more than 290 major projects, including federal and state superfund sites; army, air force, navy, and marine corps bases (involving dry land and underwater locations, as well as petroleum, oil, and lubricants [POL] and missile sites); sites at five

national laboratories; at national cemeteries; and on industrial and commercial sites; in addition, the technology has been used in commercial and residential real estate transactions.

3.4.11 Applications

In formal evaluations and in 6 years of commercial application on federal, state, and private sites, Emflux® has proved capable of accurately identifying and proportionally patterning a broad spectrum of VOCs and SVOCs—including halogenated compounds, petroleum hydrocarbons (aromatics, aliphatics, etc.), polyaromatic hydrocarbons (PAH), and methane—on open land, beneath artificial surfacing, and under water; in extremes of terrain (*e.g.*, desert, mountain, and arctic regions), weather (*e.g.*, rain, snow, heat, high and low pressures), and soils (*e.g.*, rocky, muddy, clays, caliche), to concentrations at and below ppb, and to depths exceeding 200 feet.

3.4.12 Advantages

Among the greatest advantages of Emflux® is the fact that, based on the initial NETAC evaluation and on all subsequent client follow-on verification work known to us, it has correctly identified all targeted compounds present, has avoided false negatives and false positives, and has accurately tracked changes in subsurface contaminant concentrations through changes in surface or near-surface measurements of soil gas more than 90 percent of the time.

Highly accurate and reproducible, Emflux® also provides an unusually cost effective basis from which to develop intrusive characterization programs and, ultimately, remediation plans. In terms of time, an Emflux® survey requires a sample exposure period of only 72 hours (even less for methane surveys, where field times ordinarily range from 12 to 24 hours). This exposure time is considerably less than that for most passive soil gas methods, which usually require 2- to 4-weeks in the field. In addition, depending on size of survey and type of report desired, Quadrel provides finished reports in 1- to 3-weeks; in no case has the company required more than 3 ½ weeks to produce a report.

Because, among other things, the passive Emflux® system involves simultaneous sample collection by multiple field collectors and, therefore, eliminates the need to move equipment from point to point, decontamination is generally not an issue.

3.4.13 Limitations

As for limitations, the Emflux® system—in company with all other soil gas technologies—is incapable of detecting subsurface contaminants which do not have a vapor phase under conditions at the time of survey or which are located beneath impermeable or clean barriers (such as shale or uncontaminated perched water). Moreover, the effectiveness of the technology is reduced for compounds (such as polychlorinated biphenyls [PCB] and pesticides), which preferentially adhere to soil or other materials.

3.5 GORE-SORBER®

The following subsections discuss the GORE-SORBER® developed by W.L. Gore & Associates.

3.5.1 Introduction

Organic chemicals, if released to the subsurface geologic environment through spills or leaks, will be present in several different phases, including an adsorbed-to-soil phase, non-aqueous liquid phase, and dissolved phase. Each of these phases has the potential, depending on the properties of the chemical, to contribute to the fourth phase, soil vapors. Because soil gases migrate, generally through diffusive transport, toward the surface, this organic vapor phase can be used as an indicator for the possible presence and location of the other three phases in the subsurface.

In essence, soil gas detection relies upon the properties (e.g., vapor pressure, solubility) of organic chemicals in the subsurface, by which they may partition into the pore space of the soil, and diffuse toward the surface. Within the limitations posed by soil and target compound properties, samples of the soil atmosphere can be collected from the shallow subsurface and analyzed to detect organic vapors evolving from deeper adsorbed-, dissolved -, and liquid-phase organic contaminants.

3.5.2 Components

A typical GORE-SORBER® (Figure 3-10) consists of several separate passive sorbent collection devices (sorbers). A typical sorber is 15 to 25 mm long, with a 3 mm I.D., and contains 40 mgs of a suitable granular adsorbent material depending on the specific compounds to be detected. Typically, polymeric

and carbonaceous resins are used for their affinity for a broad range of VOCs and SVOCs. The sorbers are sheathed in the bottom of a 4-foot length of vapor-permeable insertion and retrieval cord. This construction is termed a GORE-SORBER® module. Both the retrieval cord and sorbent container are constructed solely of inert, hydrophobic, microporous GORE-TEX® expanded polytetrafluoroethylene (ePTFE, similar to Teflon® brand PTFE). Figure 3-10 shows a typical GORE-SORBER® module.

A unique feature of ePTFE membranes are that they are hydrophobic and exclude liquid water, yet they do not retard vapor transfer, thus allowing VOC and vapors to freely penetrate the module and collect on the adsorbent material. This ability to protect the sorbent media from contact with ground and soil pore water without retarding soil vapor diffusion facilitates the application of W.L. Gore & Associates soil vapor screening methods in very low permeability and poorly drained soils, and ensures that organic detection is consistently through vapor-phase transfer (across the membrane) and not by direct contact adsorption with contaminated soil or water matrices. Additionally, the module construction facilitates easy installation and retrieval at a desirable installation depth of 2 to 3 feet using simple hand tools.

3.5.3 Parameters or Analytes

The broad range of target chemistry possible with the GORE-SORBER® is provided below:

NOTE:

This is not a comprehensive list of detection or analytical capabilities

VOCs

Vinyl Chloride, methyl t-butyl ether, 1,1-dichloroethane, chloroform, benzene, 1,2-dichloroethane, toluene, tetrachloroethene, ethylbenzene, o-xylene, trans-1,2-dichloroethene, cis-1,2-dichloroethene, 1,1,1-trichloroethane, carbon tetrachloride, trichloroethene, octane, chlorobenzene, m,p-xylene.

SVOCs

1,3,5-trimethylbenzene, 1,2,4-trimethylbenzene, 1,4-dichlorobenzene, undecane, tridecane, pentadecane, naphthalene, 2-methyl naphthalene, acenaphthene, acenaphthylene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene.

Explosives

Nitrobenzene, 2-nitrotoluene, 3-nitrotoluene, 4-nitrotoluene, 1,3-dinitrobenzene, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 1,3,5-trinitrobenzene, 2,4,6-trinitrotoluene, 2-amino-4,6-dinitrotoluene, 4-amino-2,6-dinitrotoluene.

Chemical Agents/Breakdown Products

Mustard (as a TIC), 1,4-dithiane, 1,4-oxathiane, benzothiozole, p-chlorophenylmethylsulfide, p-chlorophenylmethylsulfoxide, p-chlorophenylmethylsulfone, dimethyldisulfide, DIMP (diisopropyl methylphosphonate), DMMP (Dimethyl methylphosphonate), 4-chloroacetophenone, 2-chloroacetophenone.

Pesticides/Herbicides & PCB Congeners

Some capability demonstrated.

3.5.4 Sample Matrices

GORE-SORBER® modules (soil gas collectors) use granular adsorbents housed in a chemically inert, hydrophobic, microporous GORE-TEX® ePTFE membrane. The microporous structure of ePTFE allows vapors to move freely across the membrane and onto the sorbent material. These unique properties of the ePTFE collector housing protect the granular adsorbents from physical contact with soil particulates and water thereby protecting the integrity of the sample, ensuring uniform sample size (no mass gain or loss in the analyzed matrix) and a consistent mechanism of mass transfer onto the adsorbent matrix.

Due to the hydrophobic construction GORE-SORBER® modules can be used to screen water quality in monitoring wells. Dissolved-phase VOCs and SVOCs partition across the membrane and are adsorbed. This application has been of use in monitoring relative changes in water quality within a given well, and has potential applications in sentry well monitoring as an early detection system for advancing groundwater plumes. Typical exposure periods in the screened interval of the well are 2 to 3 days. No well purging is required, therefore no wastes are generated.

3.5.5 Costs

Unit pricing per field-installed collector (including collector, laboratory analysis, mapping, and reporting of results) ranges from $125 to $225, depending on the target chemistry required for the project.

3.5.6 Training

Installation of the modules is performed by the customer and no specialized training of personnel is required.

3.5.7 General Operating Procedures

Installation of the modules is performed by the customer. Although GORE-SORBER® modules can be installed to any depth, a slam bar or electric rotary hammer-drill is typically used to auger a ½ inch-to ¾-inch-diameter pilot hole for the deployment of the modules to an average depth of 2 to 3 feet below grade.

After the pilot hole is completed, modules are inserted into the completed boreholes using the stainless-steel insertion rod supplied by W.L. Gore & Associates. The top of each cord is typically fastened to a cork, which is tamped flush with the ground surface to assist in retrieval of the module, and to seal the annulus of the boring.

Module retrieval requires that field personnel locate the module, remove the cork, grasp the retrieval cord and manually pull the module from each location. Corks are separated from the module and discarded. The exposed modules are resealed in their respective designated shipping vials and placed immediately on ice in the supplied coolers. In addition, trip blanks and water temperature control blanks (provided by W.L. Gore & Associates) are also returned. Coolers are returned along with the chain-of-custody form to W.L. Gore & Associates laboratory in Elkton, Maryland via overnight carrier.

3.5.8 Performance Range

W.L. Gore & Associates suggested module exposure time is approximately 1 to 2 weeks. Results of collector analysis are reported in micrograms per sample. Method detection limits (MDL) for target VOCs and SVOCs typically range from 0.01 micrograms to 0.10 micrograms.

3.5.9 Licensing Requirements

There are no licensing requirements for using the GORE-SORBER®.

3.5.10 History

Soil gas screening technology was used as early as 1929 as a surface geochemical technique in oil and gas exploration. In the early 1980s soil gas screening became widely used as an environmental investigative tool for aiding in the delineation of subsurface organic contamination at industrial facilities and hazardous waste sites. As with any environmental screening tool, the intent of soil gas screening is to reduce the role of soil borings and monitoring wells to one of confirmation rather than exploration.

Soil gas sampling techniques can be broadly divided into two categories, active and passive. Briefly, active, pumped soil gas samples are snapshots of the soil gas environment, require detectable vapor-phase compound concentrations, skilled on-site personnel, and expensive, portable on-site equipment. The burden of maintaining project data QA/QC is on field equipment operators. Active soil gas sampling is generally most appropriate for rapid screening of VOCs emitted from high concentration subsurface source matrices in moderately permeable soils. Passive sampling techniques rely on diffusion and adsorption. Passive samplers can be unobtrusive to install, allow a dynamic equilibrium to develop between the soil gases and the sorbent, and integrate the dynamic flux of vapors produced by fluctuations in barometric pressure, rainfall, and temperature over the duration of sample collection. Sample collection occurs over a period of several days to weeks, thereby, enhancing detection sensitivity to both VOCs and SVOCs present in the soil gases.

Typical sites where active sampling techniques have had limited utility include chemical, asphalt and town-gas plants and refineries where detection of SVOCs such as substituted aromatics, naphthalenes

and heavier polycyclic aromatic compounds are of importance, or at sites where low permeability or poorly drained soil predominate. Additionally, some of the early passive soil gas sampling systems designs were susceptible to interference by the presence of water or high soil moisture, and did not permit easy installation to optimum sampling depths.

The GORE-SORBER® passive soil gas collector (module) was developed to overcome some of the limitations of conventional active soil gas sampling techniques (i.e., sensitivity to detection of SVOCs and performance under a broader range of geologic conditions), and improve upon the design limitations of existing passive collection systems (which impacted quantity and type of adsorbents used, sorbent hydrophobicity, and collector installation depth). This was accomplished by combining GORE-TEX® membrane technology with commercially available high technology adsorbents.

3.5.11 Applications

Because of the broad compound detection capabilities of the GORE-SORBER® passive soil gas system, the technology has applications for virtually any site where organic contamination is present or suspected, and where the nature of the contaminants is unknown. Its intended use is to give preliminary information on the nature and lateral distribution of organic contaminants in the subsurface so that intrusive sampling activity can be more cost effectively performed. Typical applications include soil and groundwater quality characterization at:

- Refineries and fuel storage terminals
- Fire training areas
- Manufactured gas plants
- Solvent manufacturing/distribution facilities
- Dry cleaners
- Airports
- Landfills
- Military sites
- Retail petroleum facilities
- Real estate transfer assessments
- Brownfields sites

In addition to traditional use as an assessment tool, GORE-SORBER® collectors have been used in the remedial action stages of projects, including:

- Siting of remediation system components such as recovery wells and soil vapor extraction or groundwater sparging points.
- Monitoring progress of remedial action efforts.
- The hydrophobic collector design allows application as a water quality screening tool in groundwater monitoring wells.

In general, users should be familiar with the application and interpretation of soil gas screening technology in the environmental characterization and remediation process. Technology users include environmental and engineering consultants, environmental regulators, industrial end-users in the petro chemical industries, purchasers of industrial or commercial real-estate (including Brownfields) and the military.

3.5.12 Advantages

The GORE-SORBER® offers several advantages to current active and passive sampling systems:

- No specialized field personnel required
- Ease of installation to effective sampling depths
- Sensitivity to VOCs, SVOCs and PAHs
- Application in low permeability or wet soils
- Only single analysis needed for VOCs and SVOCs
- Applications in saturated zone monitoring
- All analyses in controlled laboratory environment
- Reproducible sample results
- Positive identification of target compounds and tentative identification of unknowns
- Fast installation and retrieval of collectors (allows for increased sampling density)

3.5.13 Limitations

The GORE-SORBER® has few limitations to current active and passive sampling systems:

- Time for sample collection
- No on-site, real-time analysis

3.6 POWERPROBE 9600 DUAL-TUBE LINER SAMPLER

The following subsections discuss the PowerProbe 9600 Dual Tube Liner Sampler (PowerProbe 9600) developed by Art's Manufacturing and Supply (AMS).

3.6.1 Introduction

Direct-push technology covers the insertion of small diameter, usually less than about 3 inches, probes, samplers, or sensors into the ground. The technique does not require the use of a drilled hole and virtually eliminates the creation of cuttings that may require disposal as hazardous waste. Direct-push technology, as it applies to the hydraulic operated PowerProbe 9600, is significantly faster when compared to conventional drilling techniques. For insertion, it relies on the application of the static weight of the probing system and carrier vehicle with a hydraulic-driven percussion hammer. Tool removal is accomplished with a hydraulic ram.

3.6.2 Components

The PowerProbe 9600 is a dual-tube probing system that uses a temporary cased hole through which samplers may be passed to ensure that the sampling is not influenced by any contaminated soils or materials above the sampling point. Dual-tube tools are available in two sizes, 2 1/8-inch-outside-diameter extension with a 1 1/8-inch-outside-diameter inner extension and a 1 3/4-inch outside-diameter. Outer extension also with a 1 1/8-inch-inner extension. AMS direct push tooling features a double entry thread for faster make-up. All tubular extensions have a 1/4 inch wall thickness to provide long term reliability.

For the site demonstration, AMS proposes to feature the liner sampler for soils (Figure 3-11). This innovative product consists of a metal plastic grabber that is attached to a clear plastic liner on the lower end and a 1 1/8-inch inner extension on the other. It is driven into the soil inside a 1 3/4-inch or 2 1/8-inch dual-tube outer extension to collect the sample; either 1 1/4-inch or 1 ½-inch in diameter by 2 or 4 feet long. The sample may then be removed, leaving the outer extension in the ground. The process may then be repeated, thereby allowing for continuous coring. Consumable items are the plastic liners.

The sample preparation station is designed to provide the means of opening the clear plastic liners currently used to collect direct push soil profile samples. The free standing or truck bed mounted sample preparation station provides a "V" tray to hold the liner in position as a captive hook blade slits one side of the liner from end to end. Four additional "V" trays are provided to hold opened samples for examination. The cutting blade is the only consumable item.

3.6.3 Parameters or Analytes

When appropriate sample liner materials are used and this sampler is operated properly, soil samples can be collected for all environmental analytes of interest. This includes VOCs and SVOCs, metals (including the RCRA metals), and cationic and anionic compounds, such as dichromate, hydrogen cyanide, nitrates, etc.

3.6.4 Sample Matrices

The PowerProbe 9600 will allow collection of 1 ½-inch-diameter by 4-foot-long soil samples in liners, using either a single-tube or dual-tube sampler. The liner sampler is a new (patent pending) AMS sampler that allows collection of the sample directly into the liner. It is fast, effective, and felt to be a significant innovation in sampling for direct-push techniques. However, like other direct-push sampling systems, it will not sample fully consolidated materials or rock. Likewise, filling of the sampler when working in large-grained soils may not be complete.

The models equipped with auger rotation are used to penetrate hard compacted surface soils or soils with a significant cobble content. After auguring 3 to 6 feet in most instances, the AMS dual-tube direct-push

tools are inserted through the auger for sample collection. Overseas, this dual probe/auger feature has been used extensively for soil sampling at 18 to 25 feet followed by installation of 2-inch "monitoring wells" in the open hole.

3.6.5 Costs

The PowerProbe 9600 costs between $29,480 and $33,485, depending on the model selected. The carrier vehicle is not included, but training at the AMS manufacturing facility in American Falls, Idaho, is provided at no cost.

AMS dual-tube tools are available as follows:

- 2-1/8-inch dual-tube tool set with 40 foot depth capability is priced at $4,275.
- 2-1/8-inch dual tube add-on kit is priced at $2,450. This includes additional drive adapters, "Help tools," and additional extensions. Other kits and individual items are available.

Liner Sampler

The liner sampler is supplied complete including the liner grabber, liner sampler drive head adapter, and liner sampler thread protector cap for $575. Heavy-wall (0.065-inch), clear polybutyrate liners cost $3.50 for 2 feet and $5.50 for 4 feet in single purchase quantities.

Stations

The sample preparation station costs $895.

3.6.6 Training

Training in the operation of the PowerProbe 9600 is offered by AMS to all purchasers of their equipment. The training is primarily practical. A comprehensive operating manual is also provided.

3.6.7 General Operating Procedures

The AMS liner sampler is a dual-tube sampler that may be used to collect continuous or discrete 1 ½-inch diameter soil profile samples. It is assembled by first threading a liner onto the lower end of the liner sampler plastic grabber, then attaching a 1-1/8-inch extension to the upper end. The assembly is lowered into the in place 2-1/8-inch outer extension, using additional 1-1/8-inch extensions as needed. The liner sampler is completed with a thread protector cap on the inner extension and a liner sampler drive head adapter on the outer extension. The tool string is then driven 2 or 4 feet further to fill the sampler. It is recovered by removing the inner tools string. Samples 2 or 4 feet long may be collected with a 1-½-inch diameter sampler using the 2-1/8-inch outer extension or 1-1/8-inch diameter sampler using the 1-3/4-inch outer extension.

3.6.8 Performance Range

The PowerProbe 9600 has been used primarily as a site investigation tool for the collection of soil samples and to a lesser extent for groundwater and soil gas using just the direct-push capability. Dependent on soil conditions it has been used to depths over 100 feet, but most operators use it for sampling to about 50 feet.

3.6.9 Licensing Requirements

Licensing for probing varies from state to state.

3.6.10 History

AMS has a long history of innovation in soil sample collection since the inauguration of the company in 1942. In the 1960s, they developed hydraulic-operated core samplers for attachment to either pickup trucks or agricultural tractors. In 1994, they designed and built self-powered, hydraulic-operated, auger drilling machines. These were for pickup truck or trailer mounting and offered an optional hydraulic percussion hammer for direct-push use. The AMS dual-tube tooling evolved at that time as a more efficient means of collecting soil samples without the risk of contaminants in the soils above the sampling point affecting the sample. The design emphasis changed in 1995 to a direct-push machine

with an optional auger system capability. The PowerProbe 9600, as it is configured today, has been on the market for 15 months with units shipped to all parts of the United States and the world, many with the combination direct-push and auguring capability.

3.6.11 Applications

The PowerProbe 9600 is designed as a multifunctional site assessment machine. It may be used to probe or auger into soils and other materials. The additional auger capability will allow the probe to be used in situations where surface and subsurface conditions are not suitable for direct-push access.

AMS has perfected the use of a dual-tube soil sampling system, which will allow either discrete or continuous core dual-tube sampling with the innovative AMS liner grabber soil sampler. This eliminates the need for cleaning a sampler because the sample is collected directly in a liner. It saves significant time, alternatively, allows collection of more samples.

Potential users of this equipment are environmental contractors, environmental engineers, environmental consultants, and others involved in environmental site assessment, hazardous waste cleanup, and long-term monitoring and remediation.

3.6.12 Advantages

FEATURE	ADVANTAGE
Direct-push soil probing	No cuttings for disposal
Direct-push soil sampling	Fast and efficient
Soil sample size 1 ½ inch x 4 foot max.	Use for soil profiling
Standard 4 foot tooling	Speed through fewer makeups
Dual-tube tooling	Seals hole, prevents drawdown, and improves sample integrity, faster sample recovery
Dual-tube liner sampler	Faster sample collection
Dual-tube liner sampler	Liner is the sampler, no decon
Sample preparation station	Allows efficient and safe opening of heavier wall liners

3.6.13 Limitations

FEATURE	LIMITATION
Direct-push soil probing	Some soil conditions limit depth
Soil sample size 1 ½ inch x 4 foot max.	May not be suitable for geotechnical assessment
Standard 4 foot tooling	Increased tool weight per piece
Dual-tube tooling	Penetration in tight soils may be slower
Dual-tube liner sampler	None
Sample preparation station	None

3.7 DYNAMIC THERMAL DESORBER PROBE

The technology information for the Dynamic Thermal Desorber (DTD) probe sampling system developed by Applied Research Associates (ARA) had not been submitted at the time this document was completed. Figure 3-12 shows the basic configuration of the DTD probe.

3.7.1 Introduction

3.7.2 Components

3.7.3 Parameters or Analytes

3.7.4 Sample Matrices

3.7.5 Costs

3.7.6 Training

3.7.7 General Operating Procedures

3.7.8 Performance Range

3.7.9 Licensing Requirements

3.7.10 History

3.7.11 Applications

3.7.12 Advantages

3.7.13 Limitations

3.8 SIMULPROBE®

The technology information for this sampler has not been submitted by Simulprobe® at the time this document was completed.

3.8.1 Introduction

3.8.2 Components

3.8.3 Parameters or Analytes

3.8.4 Sample Matrices

3.8.5 Costs

3.8.6 Training

3.8.7 General Operating Procedures

3.8.8 Performance Range

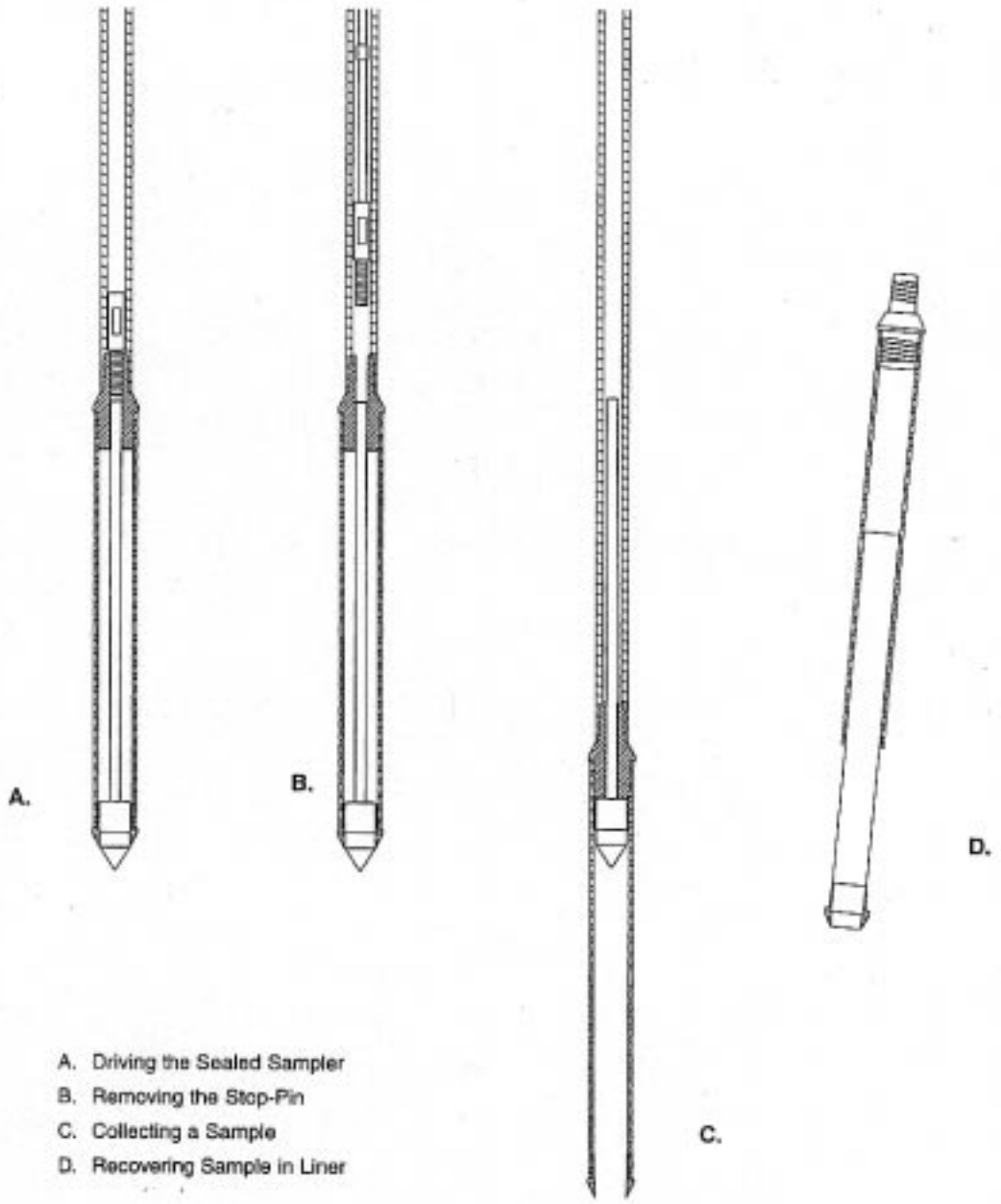
3.8.9 Licensing Requirements

3.8.10 History

3.8.11 Applications

3.8.12 Advantages

3.8.13 Limitations



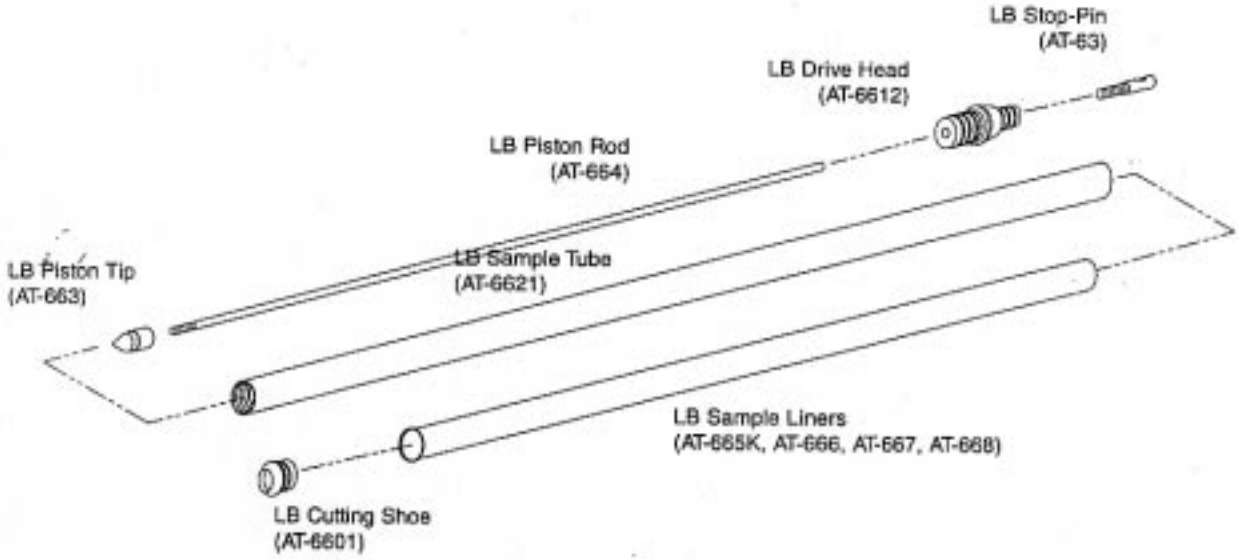
A. Driving the Sealed Sampler
B. Removing the Stop-Pin
C. Collecting a Sample
D. Recovering Sample in Liner

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-1
DRIVING AND SAMPLING
WITH THE LARGE-BORE SOIL SAMPLER
GEOPROBE® SYSTEMS

Source: Modified from Geoprobe® Systems 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

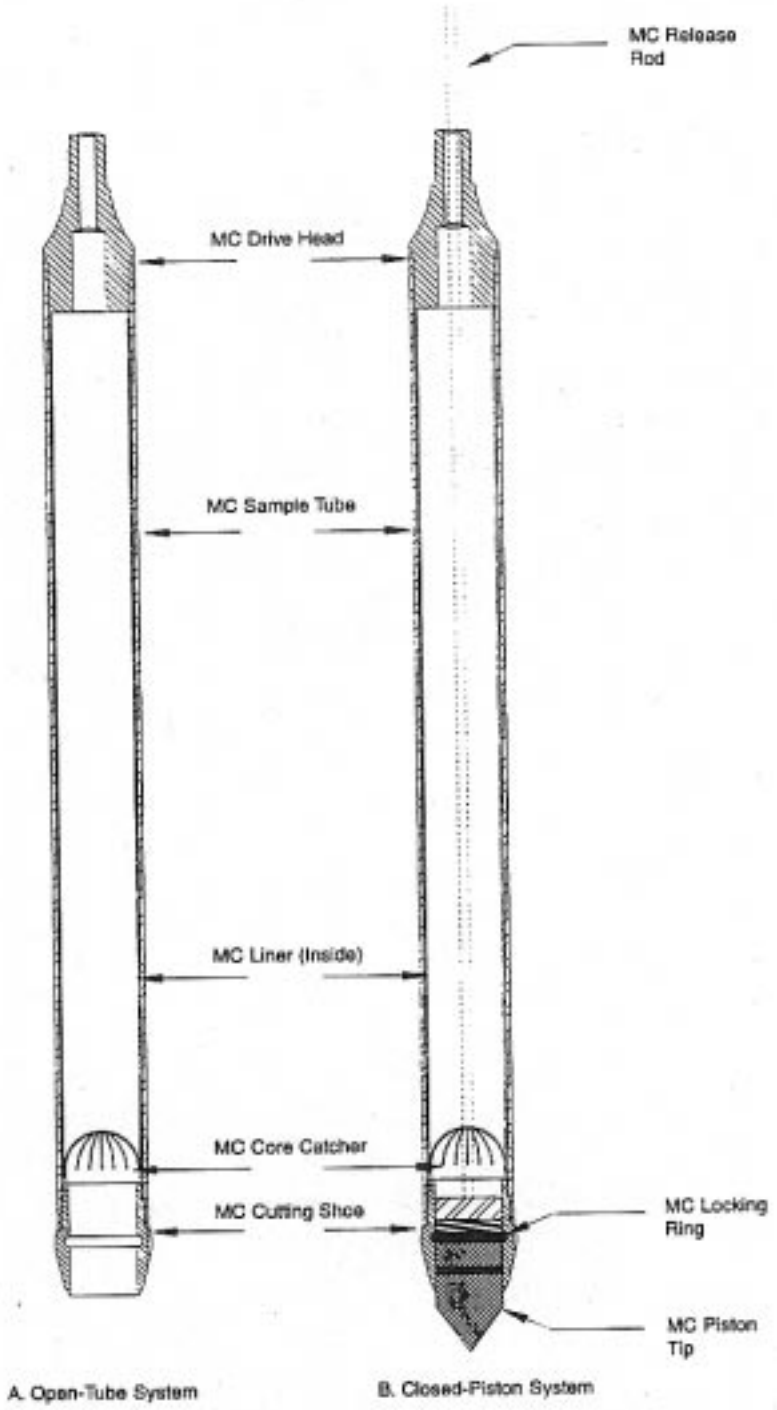


SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-2
LARGE-BORE SOIL SAMPLER PARTS
GEOPROBE® SYSTEMS

Source: Modified from Geoprobe® Systems 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

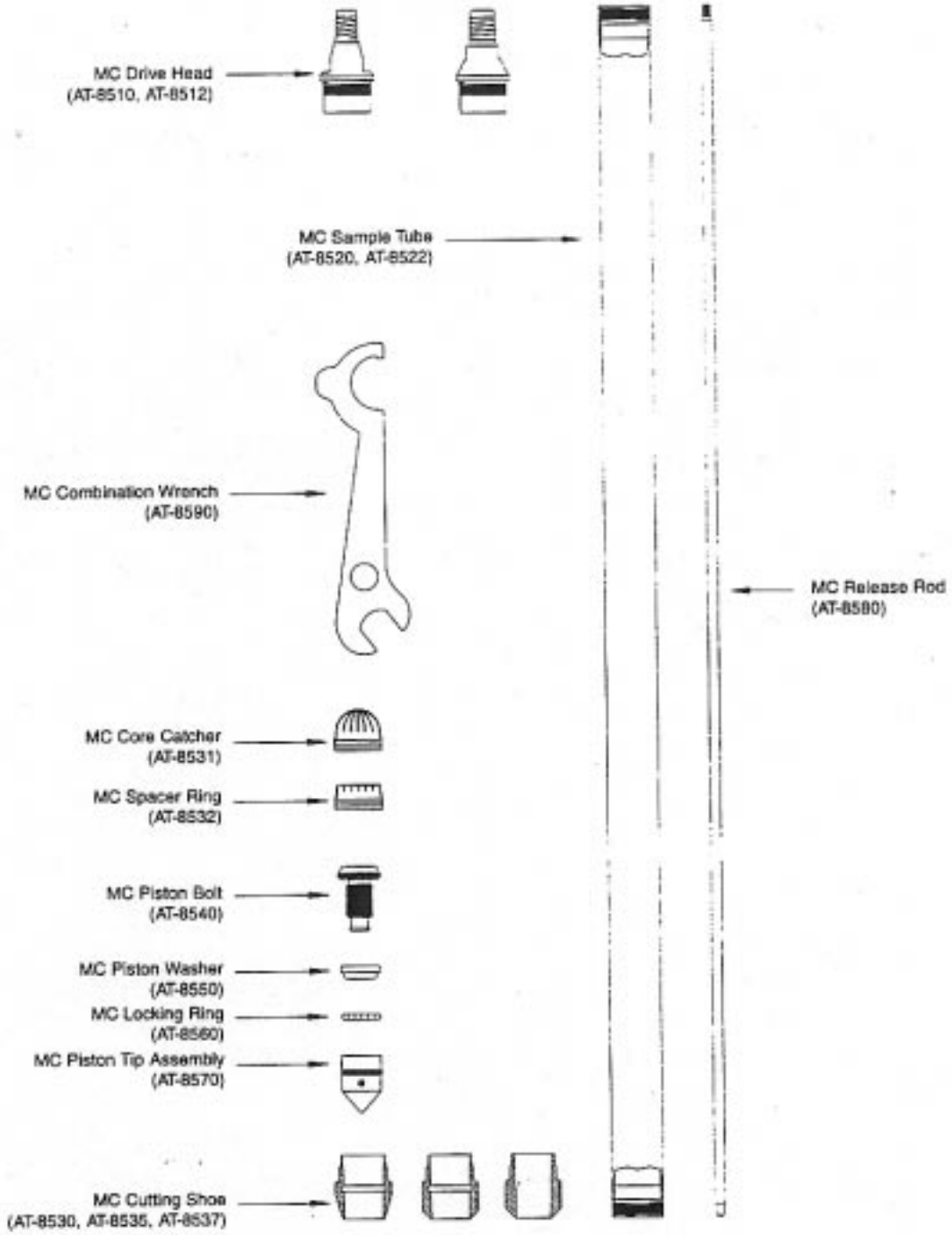


SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-3
MACRO-CORE® SOIL SAMPLER ASSEMBLIES
GEOPROBE® SYSTEMS

Source: Modified from Geoprobe® Systems 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

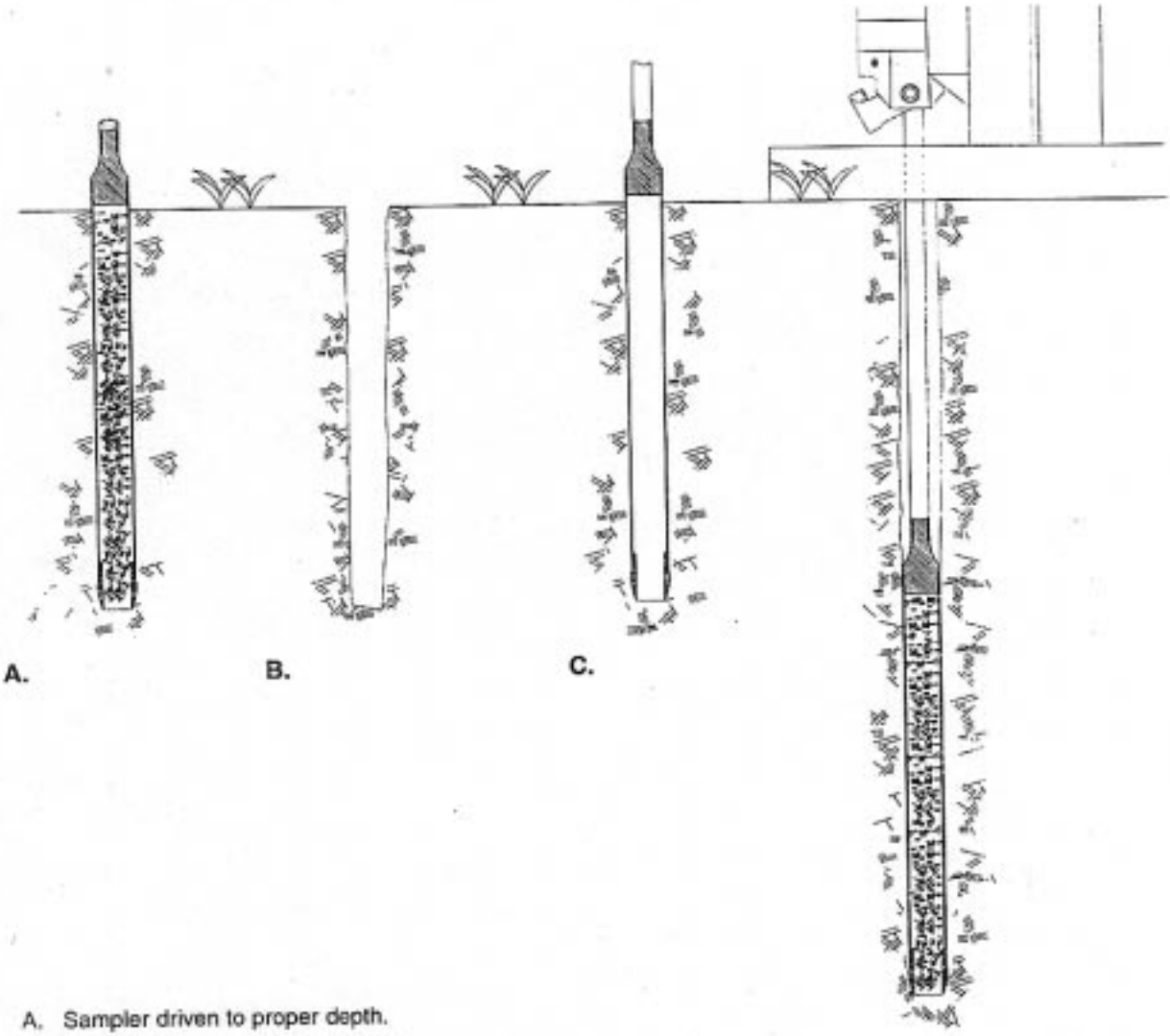


Source: Modified from Geoprobe® Systems 1997.

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-4
MACRO-CORE® SOIL SAMPLER PARTS
GEOPROBE® SYSTEMS

PRC ENVIRONMENTAL MANAGEMENT, INC.



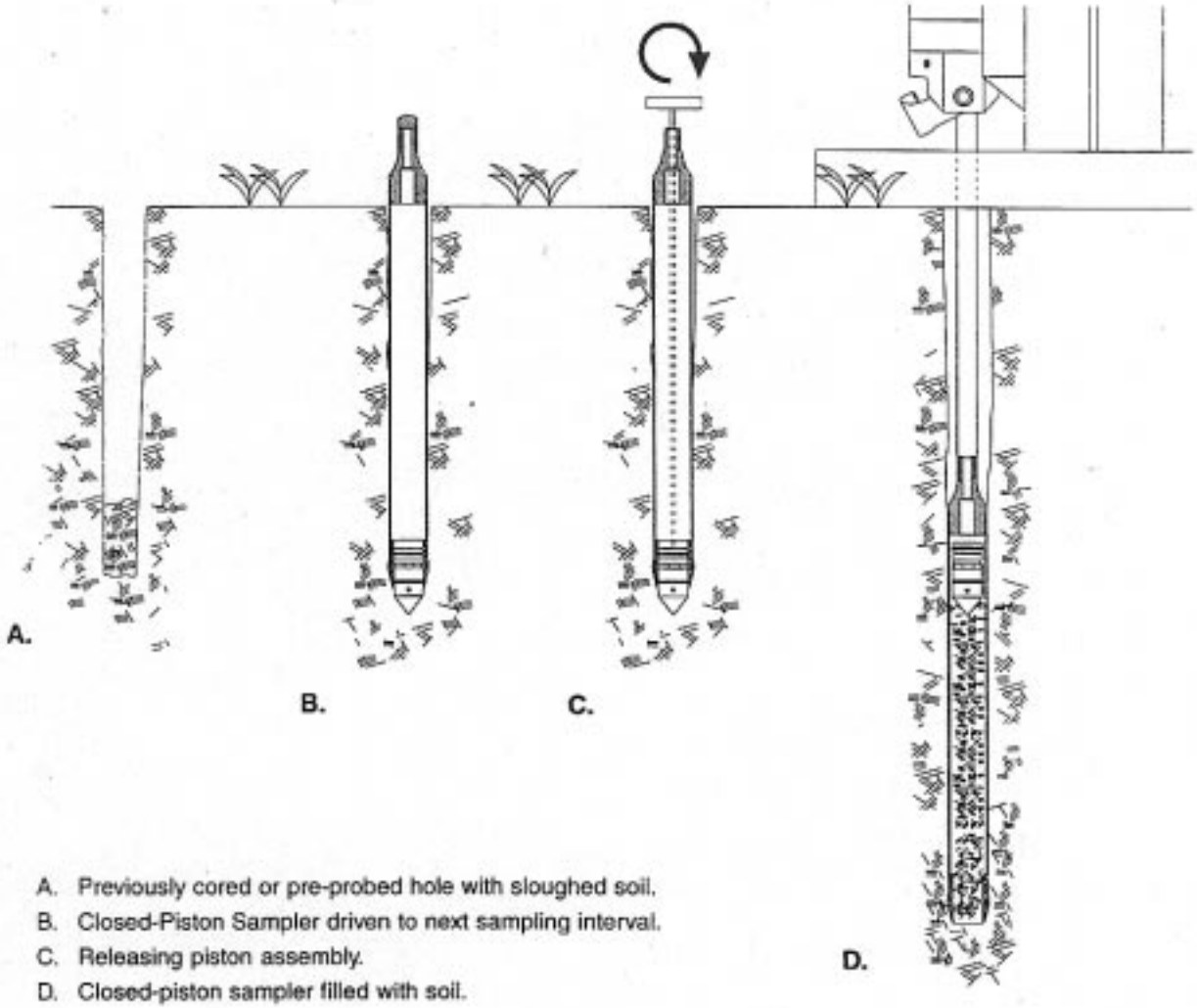
A. Sampler driven to proper depth.
B. Open hole from previous sample.
C. Open-tube sampler driven back down previous hole.
D. Sampler driven to proper depth for sampling second interval.

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-5
PHASES OF MACRO-CORE®
OPEN-TUBE SOIL SAMPLING
GEOPROBE® SYSTEMS

Source: Modified from Geoprobe® Systems 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.



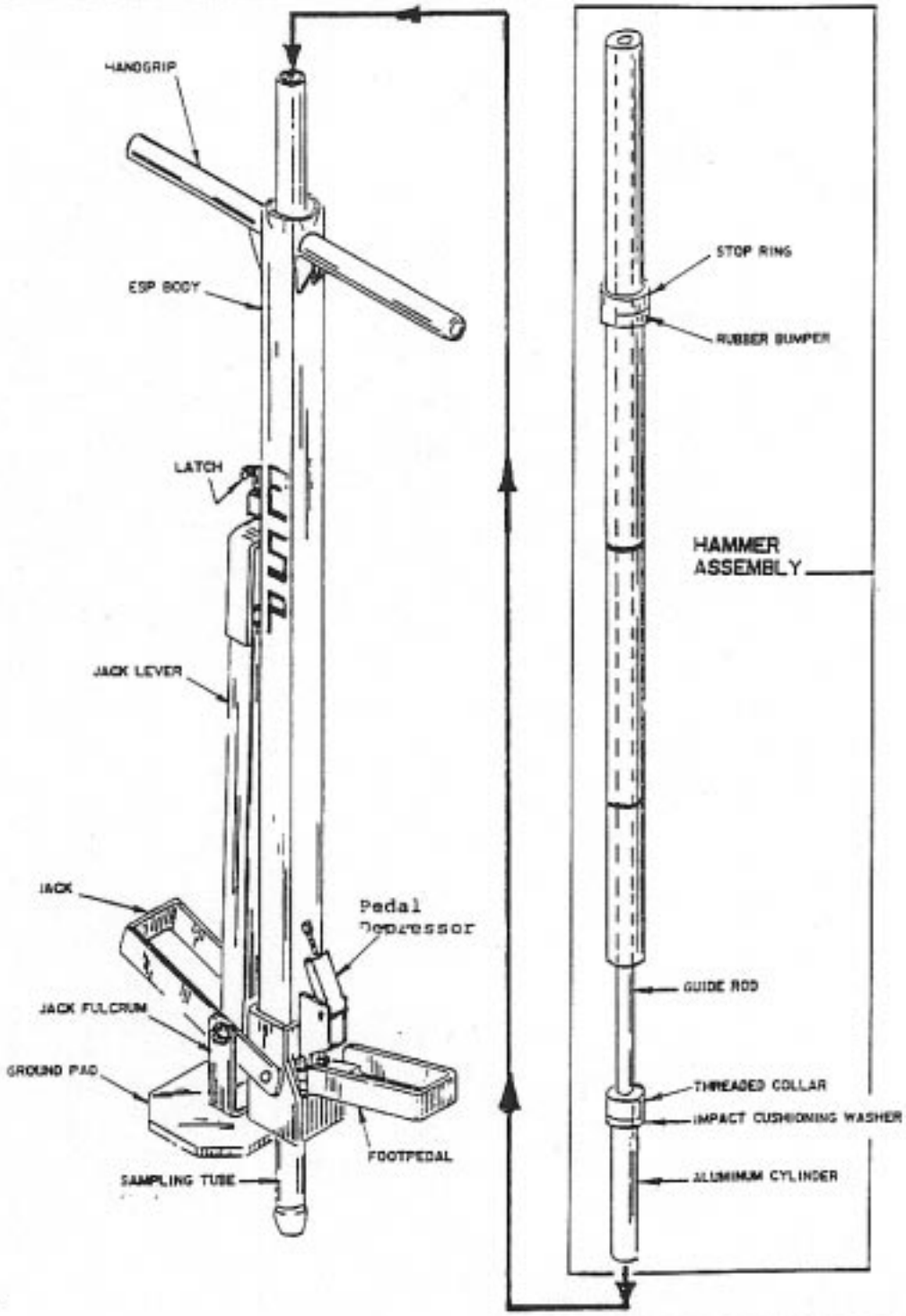
A. Previously cored or pre-probed hole with sloughed soil.
B. Closed-Piston Sampler driven to next sampling interval.
C. Releasing piston assembly.
D. Closed-piston sampler filled with soil.

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-6
PHASES OF MACRO-CORE®
CLOSED-PISTON SOIL SAMPLING
GEOPROBE® SYSTEMS

Source: Modified from Geoprobe® Systems 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

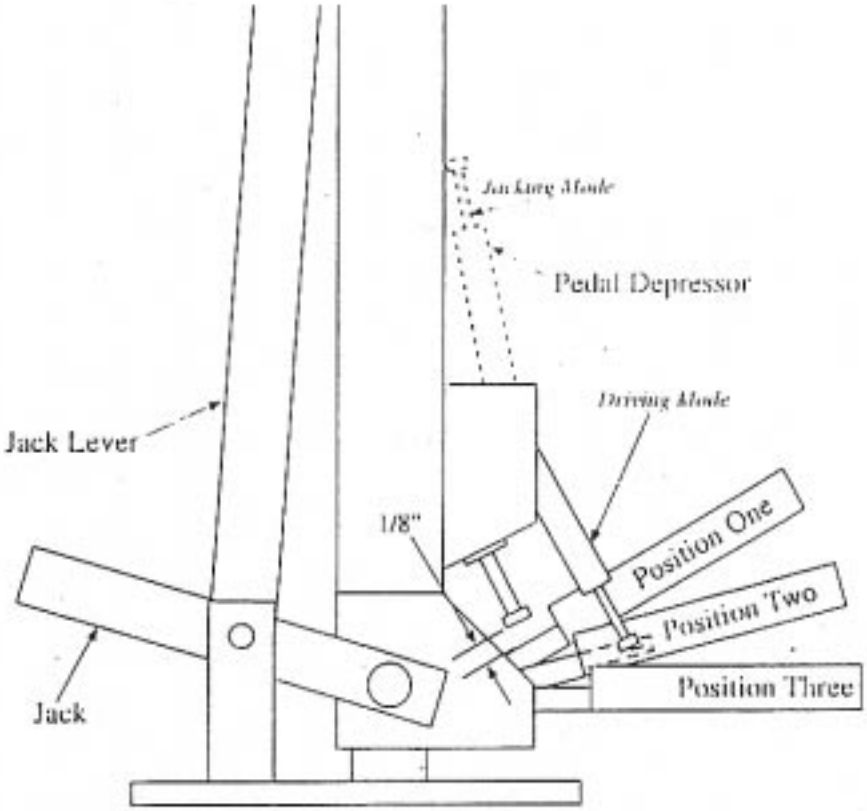


SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-7
JMC ENVIRONMENTALIST'S
SUBSOIL PROBE PARTS
CLEMENTS ASSOCIATES INC.

Source: Modified from JMC 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.



Notes: *The drawing shows the area surrounding the footpedal of the JMC Environmentalist's Sub-Soil Probe. The footpedal has three positions. In position one(jacking mode), the footpedal allows the sampling tube to be retracted, but prevents the tube from sliding back out. In position two (driving mode), the tube moves freely in either direction. And in position three, the footpedal immobilizes the tube.*

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-8A
OPERATION OF JMC
ENVIRONMENTALIST'S SUBSOIL PROBE
CLEMENTS ASSOCIATES INC.

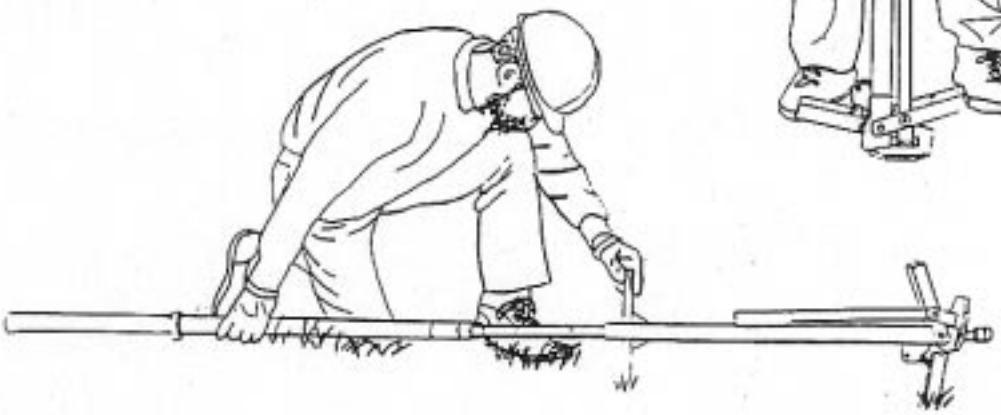
Source: Modified from JMC 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

(above): *Inserting the Liner.*

(below): *Inserting the Hammer Assembly.*

(at right): *Driving the Sampling Tube.*



SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

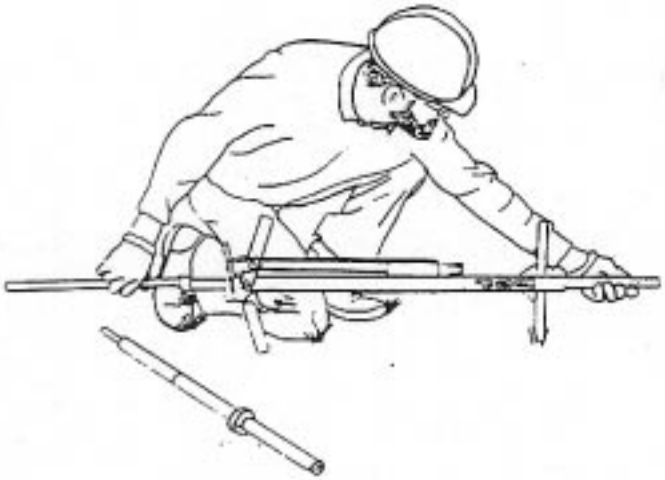
FIGURE 3-8B
OPERATION OF JMC
ENVIRONMENTALIST'S SUBSOIL PROBE
CLEMENTS ASSOCIATES INC.

Source: Modified from JMC 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

(above): *Releasing the Jack Lever.*

(above): *Retrieving the Sampling Tube*



(at left): *Extracting the Liner and Soil Sample.*

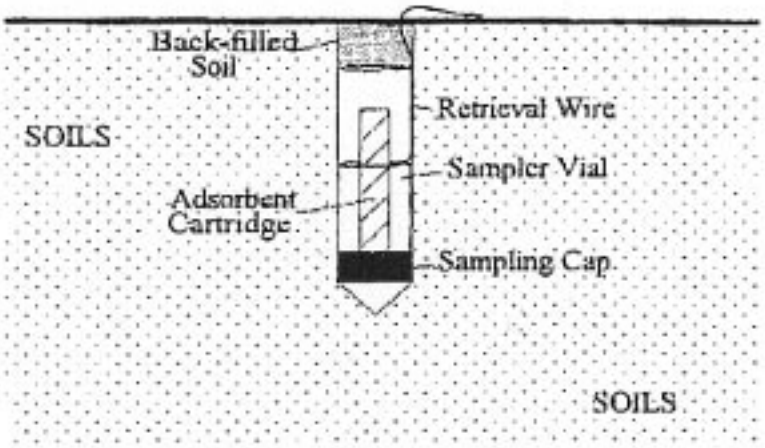
SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-8C
OPERATION OF JMC
ENVIRONMENTALIST'S SUBSOIL PROBE
CLEMENTS ASSOCIATES INC.

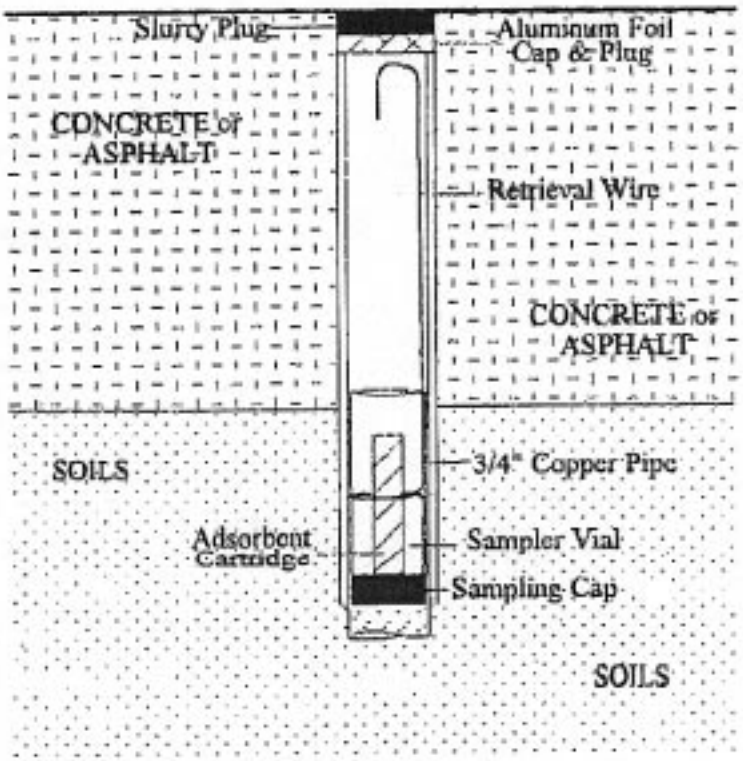
Source: Modified from JMC 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

DEPLOYMENT THROUGH SOILS



DEPLOYMENT THROUGH AN ASPHALT/CONCRETE CAP

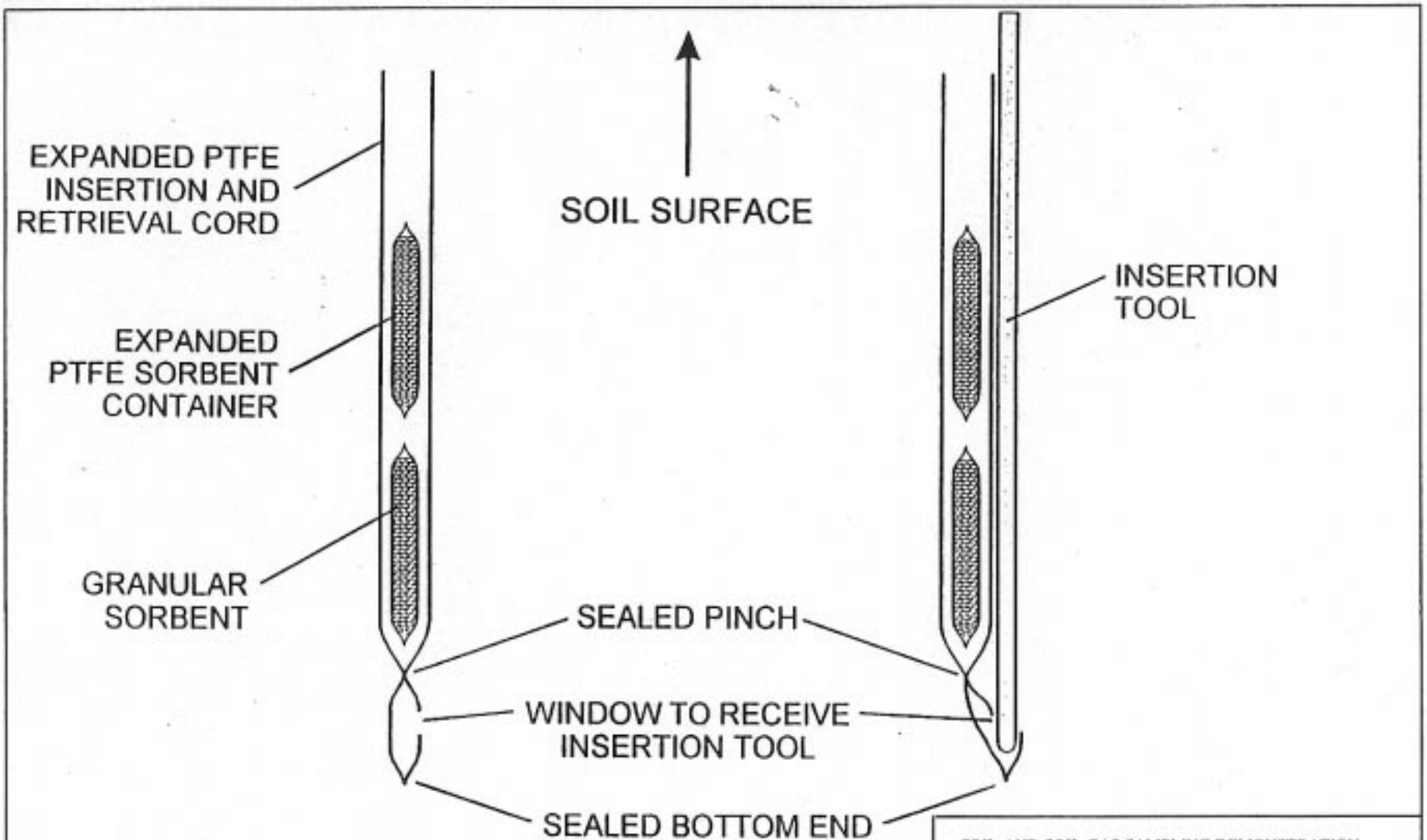


SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-9
EMFLUX® COLLECTOR PARTS
AND DEPLOYMENT OPTIONS
QUADREL SERVICES INC.

Source: Modified from Quadrel 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

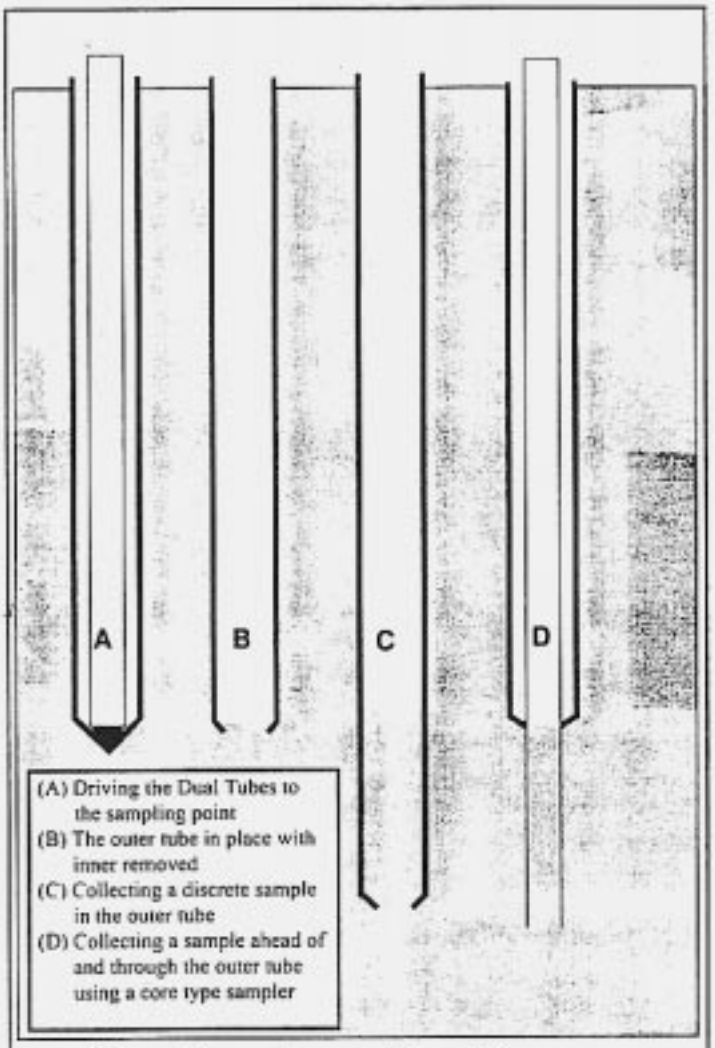


Source: Modified from W.L. Gore & Associates, Inc. 1997.

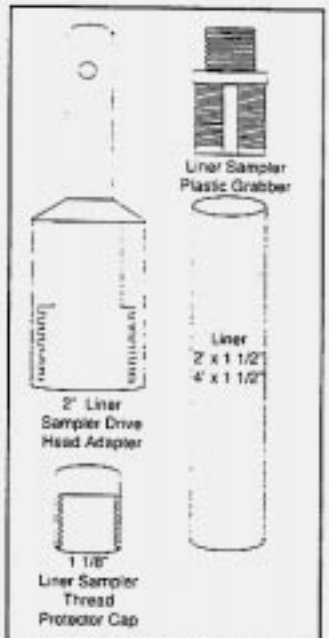
SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-10
GORE-SORBER® SCREENING MODULE
W.L. GORE & ASSOCIATES INC.

PRC ENVIRONMENTAL MANAGEMENT, INC.



DUAL TUBE DIRECT PUSH SAMPLING



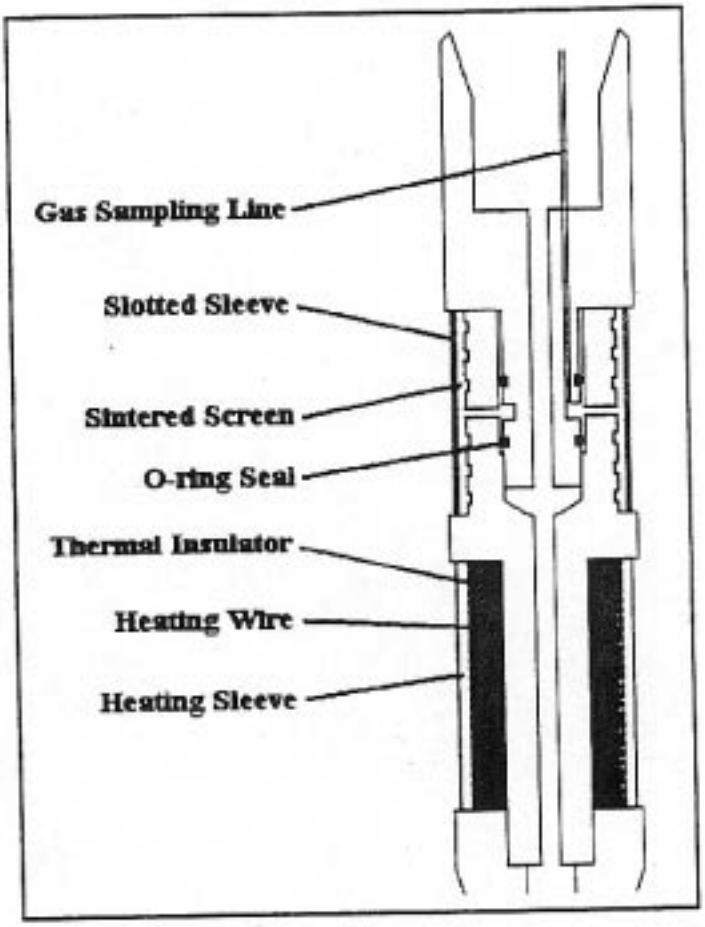
AMS LINER SAMPLER

Source: Modified from AMS 1997.

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-11
POWER PROBE 9600
DUAL TUBE SAMPLER PARTS
ART'S MANUFACTURING & SUPPLY INC.

PRC ENVIRONMENTAL MANAGEMENT, INC.



SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 3-12
THE DYNAMIC THERMAL
DESORPTION PROBE
APPLIED RESEARCH ASSOCIATES INC.

Source: Modified from Precision Sampling Inc. 1997.

PRC ENVIRONMENTAL MANAGEMENT, INC.

CHAPTER 4
DEMONSTRATION SITE DESCRIPTIONS

This demonstration will be conducted at two sites: the SBA site, Albert City, Iowa; and the CSC site, Denver, Colorado.

These sites were chosen because they exhibit different climates, modes of waste deposition, soil textures, and because they are contaminated with a relatively uniform suite of chlorinated VOCs. Chlorinated VOCs will be the target analytes for this demonstration because they are frequently detected at hazardous waste sites, can easily be lost during sample handling, and exhibit relatively high human toxicity characteristics. This chapter discusses the history and characteristics of the demonstration.

4.1 CHEMICAL SALES COMPANY SITE

The CSC site is located in Commerce City, Colorado, and in the northern portion of Denver, Colorado, approximately 5 miles northeast of downtown Denver. The site is divided into three operable units. The predemonstration activities will occur at operable unit (OU) 1. OU1 includes the CSC property, and is also referred to as the Leyden Street Site. The address for OU1 is 4661 Monaco Parkway, Denver, Colorado.

4.1.1 Site History

In 1962, a warehouse was constructed at 4661 Monaco Parkway. Between 1962 and 1976, the warehouse was occupied by Samsonite and then by Rubber Gates Company. These companies reportedly used the facility solely as a product warehouse. CSC purchased and occupied the facility in 1976. All existing surface tanks, and USTs and pipelines were installed between October 1976 and February 1977. This facility was used to receive, blend, store, and distribute various chemicals and acids. Chemicals were transported in bulk to the CSC facility by train and were unloaded along railroad spurs located to the north and south of the CSC warehouse. Operations at the CSC warehouse ceased around 1992.

In 1981, the EPA conducted a random national survey of drinking water systems. The EPA found several organic chemicals in alluvial municipal water supply wells in the area west of Rocky Mountain Arsenal (also in the vicinity of the CSC property). These results were confirmed in 1982 and 1985. In April 1986, the EPA Field Investigation Team (FIT) conducted a soil gas survey near the CSC property and found elevated TCE levels on the north side of the rail spur in the northern portion of the CSC property. The results of several subsequent studies of the FIT team indicated a release of hazardous substances into the soil and groundwater from the CSC property. As a result of these findings, the CSC site was proposed for listing on the National Priorities List (NPL) in 1988 and was placed on the NPL in 1990. In September 1989, EPA and CSC entered into an Administrative Order on Consent (AOC) requiring CSC to conduct a remedial investigation/feasibility study (RI/FS) for CSC OU1. The RI/FS was completed on OU1 in 1991.

4.1.2 Site Characteristics

The current site features at the CSC site consist of the warehouse, a concrete containment pad with a few remaining tanks from the aboveground tank farm, another smaller containment pad on the north side of the railroad spur with aboveground tanks, and multiple areas of stacked drums on the west side of the warehouse and in the northwest corner of the property (Figure 4-1). The drums remain from past CSC activities. The warehouse is currently occupied by Laidlaw, AET Environmental, and Highland. The area immediately adjacent to the west and north of the warehouse is paved. A fence surrounds the property.

The CSC site sits on a large hill. The topography slopes northward toward Sand Creek. Elevations range from 5,200 feet near Sand Creek to 5,265 feet above mean sea level in the southeastern corner of the CSC site. Natural topographic features have been extensively modified by construction and earthwork. A relatively abrupt change in the natural topography occurs immediately north of the fenced-in property, where there is as much as a 50-foot change in the elevation to the north.

The northern portion of the CSC site lies within the Sand Creek flood plain, which is part of the South Platte River System. The topography, distribution of surficial deposits and the materials encountered during drilling suggest that the southern portion of the CSC site (near the CSC warehouse) is a terrace comprised of Slocum Alluvium beneath aeolian sand, silt, and clay. The terrace was most likely formed

be renewed downcutting of the Sand Creek tributary. Borings on the CSC property indicate that the soils in the vadose zone and saturated zone are primarily fine to coarse, poorly sorted sands with some zones of finer-grained silts and clays. The alluvial aquifer also contains some poorly sorted gravel zones. The aquifer appears to be composed of coarser grained sand and gravel near Sand Creek versus the area around the CSC warehouse. The depth to water is about 30 to 40 feet bgs near the CSC warehouse and becomes as shallow as 10 feet bgs near Sand Creek.

The contaminants of concern in the soil and groundwater at OU1 are chlorinated VOCs including: PCE, TCE, 1,2-DCE, 1,1,1-trichloroethane (TCA), 1,1-dichloroethane (DCA), methylene chloride, carbon tetrachloride, and vinyl chloride. Potential sources of the observed contamination include: (1) organic solvents stored at the CSC tank farm; (2) surface drainage from the CSC property; (3) leaks and spills from rail cars during unloading operations; and (4) other documented releases during CSC operations such as a 200-gallon methylene chloride release in 1985 and a 3,700-gallon methanol spill in 1990.

Soil investigations on the CSC property have found chlorinated VOC contamination extending from near the soil surface (less than 5 feet bgs) to the water table (40 feet bgs). The predominant chlorinated VOCs detected in the soil have been PCE, TCE, and 1,1-DCA. Elevated VOC concentrations have been found in areas between the CSC warehouse and the Trammell Crow Building and in areas north of the CSC warehouse. The area of highest VOC contamination is north of the CSC tank farm near the northern railroad spur with PCE concentrations in this area as high as 80 micrograms per kilogram (mg/kg) and TCE and 1,1,1-TCA concentrations as high as 1 mg/kg. Field gas chromatography results have indicated TCE, PCE, 1,1,1-TCA, and 1,1-DCA concentrations as high as 5,000 parts per million by volume (ppmv) from soil headspace samples. The predominant chlorinated VOCs in groundwater also have been PCE, TCE, and 1,1-DCA. The highest concentrations detected have been PCE at 30 micrograms per liter (mg/L) and TCE and 9.5 mg/L.

4.2 ALBERT CITY SMALL BUSINESS ADMINISTRATION SITE

The SBA site is located on Orchard Street in east-central Albert City, Buena Vista County, Iowa. The site is the location of the former Superior Manufacturing Company (SMC) facility. The site property

where the SMC plant was formerly located occupies an area 142 feet north to south and approximately 250 feet east to west and is located on Orchard Street between 1st and 2nd Avenue.

4.2.1 Site History

The site consists of two potential source areas: the former SMC plant property and the former SMC waste storage area. The former SMC plant property is presently owned by the U.S. SBA and B&B Chlorination, Inc. The SMC began its operation in 1924. The company initially made such items as garden tools, vending machines, comb sterilizers, and engine governors. In 1935, the SMC began manufacturing grease guns. Grease gun production reached its peak in 1966 and 1967 at a rate of 6,000 guns per day. The SMC was sold and production ceased in 1967. An estimated 17 million grease guns were manufactured at the SMC plant. Various entities owned the property between 1967 and 1984. From 1984 to present, the SBA has owned the west portion of the former plant property. The east portion of the former plant property is now owned by B&B Chlorination, Inc.

The SMC facility performed metal working, assembling, polishing, degreasing, painting, and other operations. A former SMC employee stated that various solvents were used in the manufacturing of grease guns. He stated that metal shavings wastes coated with oil and solvents were placed in the former waste storage area location approximately 50 feet north of the plant. The oil and solvents were allowed to drain onto the ground. The waste was hauled off site by truck. He also said that metal shavings were stacked in tall piles that extended north from Orchard Street to the abandoned alley in the center of the block. Based on records it is known that degreasers were used at the former SMC plant. Sampling results indicate that PCE, TCE, 1,2-DCE, and vinyl chloride are present in the soil and groundwater at the site.

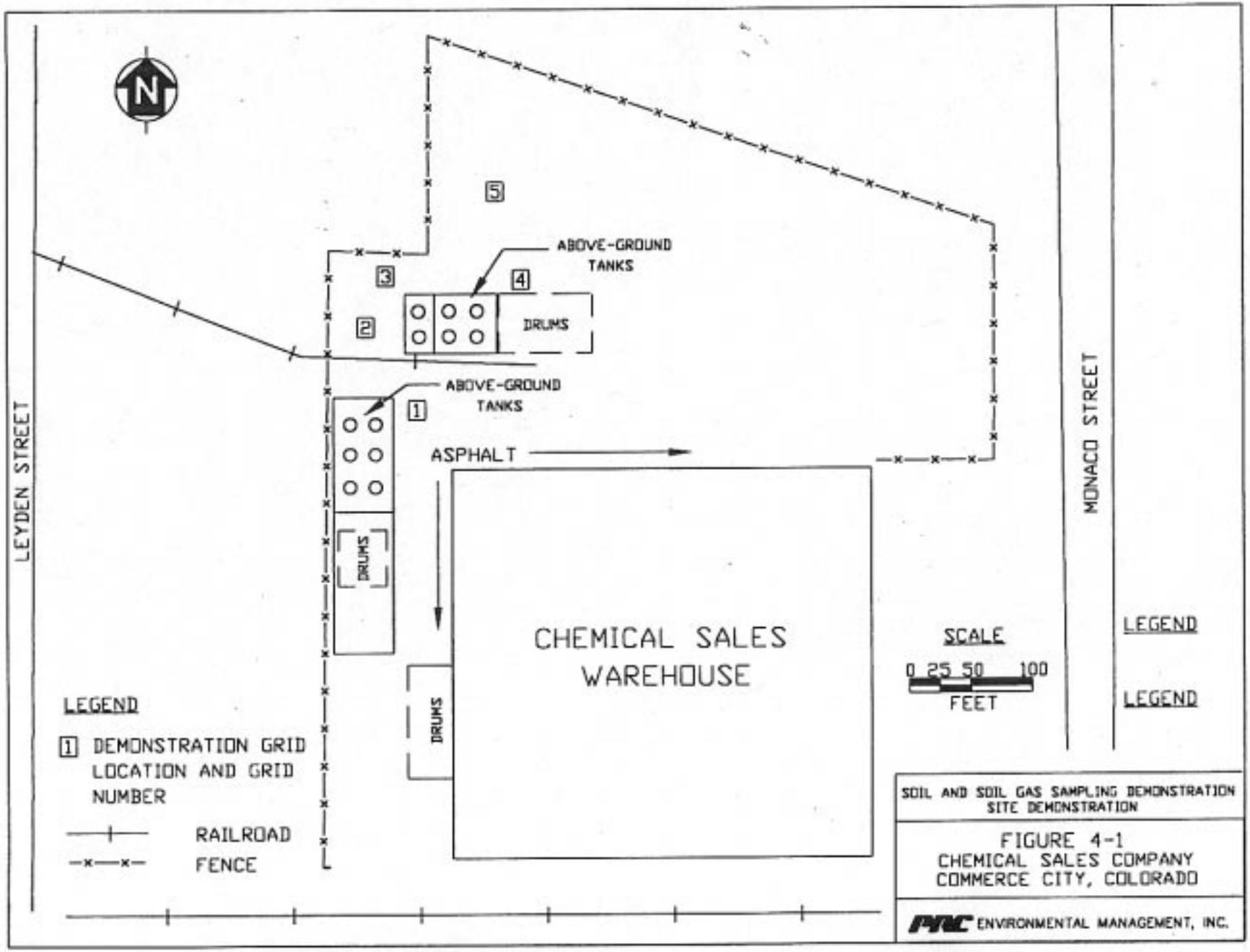
4.2.2 Site Characteristics

The site is located in a commercial area of Albert City approximately 150 feet north of Main Street. The site is bordered by commercial and residential areas. The site consists of the former SMC plant property and a former waste storage area (Figure 4-2). Previous documents indicate the former waste storage area was located approximately 50 feet north of the former SMC plant property, across and on the north side of Orchard Street.

The former SMC plant property is a grass covered, relatively flat unfenced open lot. The former plant buildings have been razed. One pole barn from a former lumber company is the only building currently on the property. Surface water runoff from the site flows into two storm drains; one located on the west side and the other on the southeast side of the property. Stormwater is transported via underground piping to a drainage channel located approximately 3 miles to the southeast of Albert City. Three buildings are present on the former SMC waste storage area property. They are historic buildings that include a garage, a museum, and a school house. The former SMC waste storage area topography slopes to the south.

Poorly drained, loamy soils of the Nicollet series are found throughout the site area. The upper layer of these soils is a black loam, grading to a dark gray loam. Below this the soils become a friable light clay loam, extending to a depth of 60 inches. Underlying these soils is a thick sequence (400 feet or more) of glacial drift materials. The lithology of this glacial drift is generally a light yellowish-gray, sandy clay, with some gravel, pebbles, or boulders. It is likely that the sand-to-clay ratio is highly variable throughout this drift. Groundwater is encountered at about 6 to 7 feet bgs at this site. Recharge of groundwater to monitoring wells near the site is very slow from the glacial drift materials.

PCE, TCE, 1,2-DCE, and vinyl chloride are the primary contaminants found in the soil and the groundwater at the site. These chlorinated VOCs have been found in both surface (0 to 2 feet) and subsurface (3 to 5 feet) soil samples. TCE and 1,2-DCE tend to be found at the highest concentrations in both soil and groundwater. TCE and 1,2-DCE have been found in the soil at 17 and 40 mg/kg, respectively, with vinyl chloride present at 1.4 mg/kg. The maximum concentrations of PCE, TCE, 1,2-DCE, and vinyl chloride found in the groundwater at the site are 0.12, 570, 190, and 3.5 mg/L, respectively. The areas of highest contamination have been found near the center of the former SMC plant property and near the south end of the former SMC waste storage area.

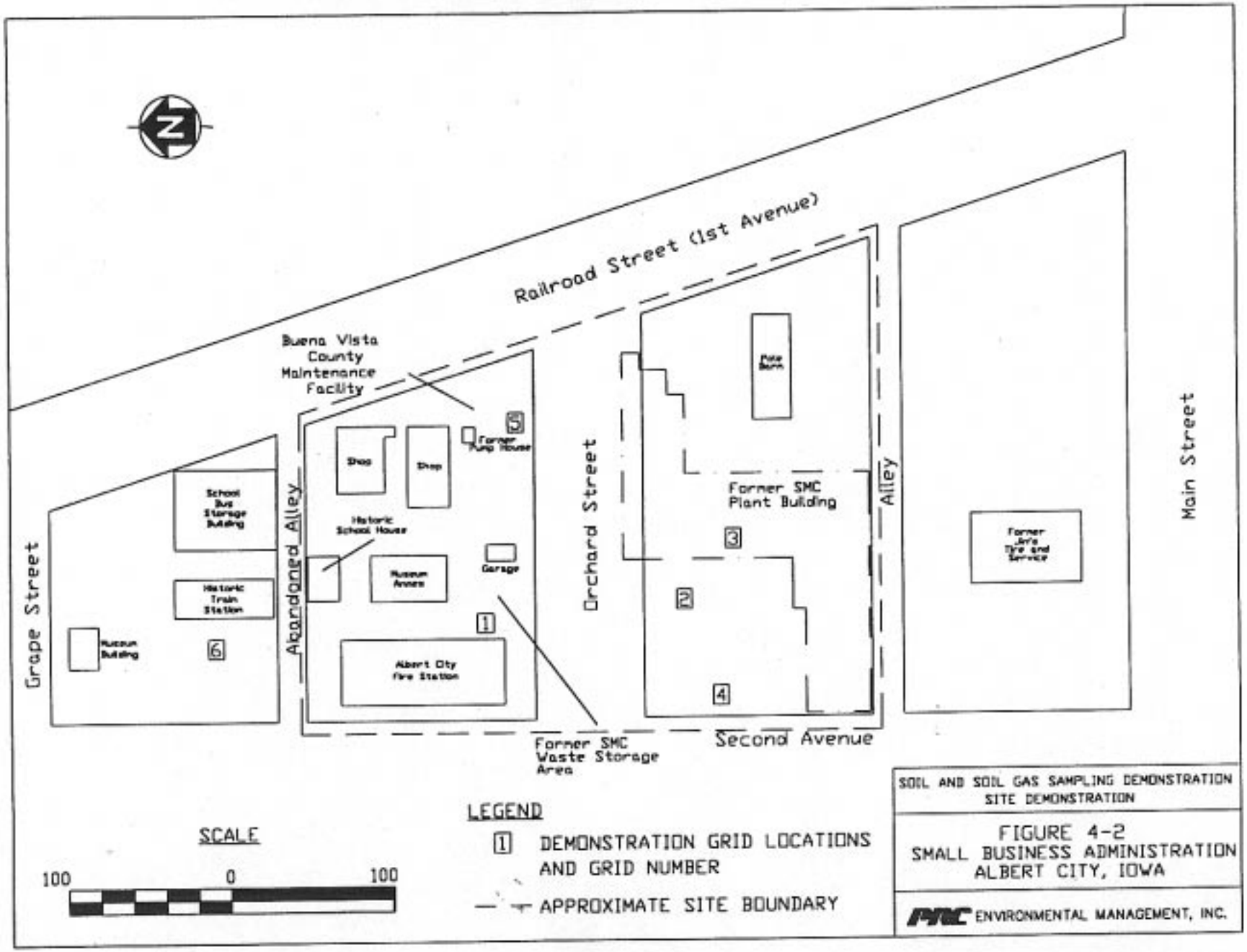


SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 4-1
CHEMICAL SALES COMPANY
COMMERCE CITY, COLORADO

PRC ENVIRONMENTAL MANAGEMENT, INC.

SOURCE: PRC MODIFIED 1997, S:\SAMPLING DEMO\DEMOPLAN\V64-1


Railroad Street (1st Avenue)
Buena Vista County Maintenance Facility
Former Pump House
Shop
Shop
School Bus Storage Building
Historic School House
Museum Annex
Garage
Historic Train Station
Museum Building
Albert City Fire Station
Grape Street
Abandoned Alley
Orchard Street
Former SMC Plant Building
Alley
Former Jim's Tire and Service
Main Street
Second Avenue
Former SMC Waste Storage Area
1
2
3
4
5
6
LEGEND
1 DEMONSTRATION GRID LOCATIONS AND GRID NUMBER
APPROXIMATE SITE BOUNDARY
SCALE
100
0
100
SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION
FIGURE 4-2
SMALL BUSINESS ADMINISTRATION
ALBERT CITY, IOWA
ENVIRONMENTAL MANAGEMENT, INC.

CHAPTER 5
CONFIRMATORY PROCESS

The SITE demonstration process is based on the presence of a statistically valid data set against which quantitative performance goals of a technology may be directly assessed or compared. This data may be generated through direct measurement and monitoring of a technology during the demonstration, or through the use of analytical methods applied to samples collected by the technology or a conventional method. For the soil and soil gas demonstration, many quantitative technology performance indicators will be measured and recorded during technology operation. Examples of these indicators include percent sample recovery, sample cost, sample throughput, maximum and minimum sample volume, and maximum sampling depth. The other quantitative performance indicators include a technology's ability to preserve sample integrity and provide a representative sample relative to conventional methods. These indicators will be assessed through a statistical comparison between sampling data produced by the technologies and a conventional sampling method. This comparison will require physical sample collection by both the technologies and the conventional sampling methods, and chemical analysis of all samples. The first part of this chapter focuses on the standard analytical methods selected; the second portion addresses the methods of data reduction and analysis.

The choice of appropriate standard analytical methods are critical to the success of the demonstration. EPA, PRC, and developers must work closely to select the best combination of analytical methods and appropriate laboratory QA/QC. Since all analytical work for this demonstration will be done in an on-site laboratory using advanced analytical systems, the standard analytical methods and especially the QA/QC requirements must be thoroughly defined. The standard analytical methods and QA/QC presented in this chapter will assure the production of analytical data of equal or greater quality than generally produced by formal fixed-laboratories.

5.1 METHOD SELECTION

The analytical data generated for this demonstration will be produced entirely in an on-site laboratory. The methods used by the on-site laboratory personnel will be PRC's SOG 001-- "Volatile Organic Compounds in Soil Gas, Water and Soil." SOG 003 -- "Weight Balance Operation" will be followed by

the on-site laboratory for weighing soil samples for VOC analysis. PRC SOG 001 is a GC method for the definitive identification and quantification of VOCs in soil. Both SOGs are included in Appendix A.

5.2 STANDARD LABORATORY SELECTION

No outside laboratory is planned for confirmation analysis of the on-site laboratory. The internal QA/QC proposed in Chapter 8 and in the attached SOGs describe methods which will produce analytical data of equal or greater quality than off-site laboratory analysis.

5.3 REFEREE LABORATORY SELECTION

No referee laboratory will be selected since most of the technologies do not produce analytical data themselves. The ARA DTD, Emflux®, and GORE-SORBER® will indirectly produce their own data following the developers analytical specifications. This developer analysis is part of their technologies standard application. The experimental design used for this demonstration minimizes the need for a referee laboratory, even for technologies that produce analytical data. The experimental design relies on replicate sampling to define soil chemical and physical heterogeneity, and to assess technology performance (Chapter 6). The replicate sampling allows a comparability assessment between the on-site analytical data and the ARA DTD, Emflux®, and GORE-SORBER® data, without the use of a referee laboratory.

5.4 SPECIAL QC REQUIREMENTS

As described later in Chapter 8 and in the attached analytical SOGs, the on-site laboratory will run numerous blind performance evaluation (PE) samples, laboratory control samples, and, if available, standard reference materials (SRM), to monitor the accuracy of its data. If acceptance limits are not published for the SRMs, acceptable analysis by the on-site laboratory will fall within a window of 80 to 120 percent recovery for the target analytes.

5.5 ON-SITE LABORATORY AUDIT

Mr. Barry Lesnik, Office of Solid Waste, will review and audit the on-site laboratory methods before the demonstration. This audit will identify potential areas of concern, prior to the collection and analysis of critical samples for the demonstration. The audit will address the QC procedures and document any changes to the analysis process. If possible, a second audit will be conducted during the demonstration at one of the sites.

Chapter 8 of the demonstration plan highlights key elements of the audit process and any special procedures which will be addressed.

5.6 STATISTICAL ANALYSIS OF RESULTS

The analytical and other quantitative data collected during the demonstration will be analyzed to allow statistical tests of the hypotheses discussed in Sections 6.1 and 6.2 and summarized in Table 5-1. The nature of the statistical analysis performed is partially dependent on the nature of the distribution of the data. Information of the distribution of the data will not be known until after the data is collected. However, based on analysis of data from previous studies (EPA 1995a,b SCAPS and ROST ITERs) and the nature of the experimental design, the methods of statistical analysis can be decided. Since the predemonstration sampling has identified sampling depths with minimal heterogeneity it is possible that the analytical data will show a normal distribution. Assuming the data exhibit expected distribution patterns, three different analyses will be performed depending on the hypothesis being tested: three-way analysis of variance (ANOVA) for untransformed data, three-way ANOVA for transformed data, and nonparametric analysis. If the data is not normally distributed, only nonparametric methods will be applied to the nontransformed data. In addition to these analyses, several statistical analyses will be performed that test the nature of the data distribution, the adherence to underlying assumptions, and provide multiple comparisons or comparison between an innovative technology and the conventional method. The following text discusses these analyses. For this demonstration, the level of significance for all inferential statistics will be set at a 5 percent probability of making a Type I error. A Type I error involves accepting a false null hypothesis (α=0.05).

5.6.1 ANOVA and Untransformed Data

The factorial experimental design of the sampling plan allows for control of the three factors being evaluated: technology, concentration, and soil type. Typically, data from this type of factorial design can be analyzed using a three-way ANOVA. This approach will be used to analyze some of the data and address some of the test hypotheses using what may be considered untransformed data. The hypotheses and data that can be tested using ANOVA and untransformed data include all of the hypotheses summarized on Table 5-1. The data used for tests of these hypotheses are expressed in units of percent or concentration, and it is probable that the data distribution will be normal or near normal and that the variance will be equal between factors. ANOVA will be performed for each data set collected to test each of these hypotheses (that is, three separate ANOVA procedures will be performed to address each of the hypotheses).

An assumption underlying ANOVA is that the variances within each population being compared are equal. Prior to performing ANOVA, either or both a Cochran's C test or Bartlett-Box test will be performed to test for homogeneity of variance. If the data do not exhibit homogeneity of variance then nonparametric analysis will performed instead of ANOVA. Possible nonparametric analysis is discussed in Section 5.6.3.

ANOVA determines whether there is a significant difference between one or more populations, but does not provide information about individual or multiple comparisons. That is, it would not provide a test of the difference between a given technology and the conventional method or between the conventional method and all other technologies. This requires *post hoc* or multiple comparison tests. To obtain this information, the Tukey or Sheffé multiple comparison tests will be performed. These tests will provide comparison between individual technologies and between an individual technology and the remaining field of technologies. As stated in Section 6.1, although multiple comparison tests may provide comparison between individual innovative technologies, the only three comparisons that will be reported for any given technology will be between that technology and the conventional method, between the technology and the undifferentiated field of all other technologies, and between the conventional method and the undifferentiated field of all other technologies.

Application of the test described above should provide conclusions concerning acceptance or rejection of the stated statistical hypotheses that are clear and can be shown to have been supported by the observed data within the stated statistical limits.

5.6.2 ANOVA and Transformed Data

Direct use of ANOVA for analysis of concentration data may not be valid because the concentration data will not meet the underlying assumption of homogeneity of variance. The two levels of concentration, high and low, will exhibit average concentrations that differ by several orders of magnitude. While the percent variance (expressed as percent of a given concentration) for each concentration range will probably be similar, the absolute variance in concentration units will probably differ by several orders of magnitude. This will violate the assumption of homogeneity of variance. Several approaches can be taken to resolve this problem each offering advantages and disadvantages.

If it is desired to keep the data in concentration units, then use of ANOVA and related tests will not be possible. Tests of the statistical hypotheses can be conducted using nonparametric statistical tests. These are described in Section 5.6.3. An advantage of these nonparametric tests is that the comparison is performed on untransformed data so that few, if any, assumptions are required concerning how the transformation may have changed the nature of data distribution. These tests also do not require any assumptions about the data distribution. A significant disadvantage is that these tests provide answers only to the statistical hypotheses stated, but provide no average or mean values for comparison. It is believed that an important aspect of this demonstration is to not only provide comparison of performance measures as expressed by the statistical hypotheses, but also to provide some quantitative measure of the amount of difference if the hypothesis is rejected. For this demonstration, both parametric tests on transformed data and nonparametric tests on untransformed data will be conducted and reported. This will provide definitive answers to the statistical hypotheses and will provide measures of comparison that involve some assumptions.

A second alternative is to split the data into two sets representing the high and low concentration ranges. This alternative will work only if the concentrations for each group fall within a sufficiently narrow range that homogeneity of variance is still maintained. While it is possible that in each of the high and low concentration groups this condition can be met, this would have to be tested using a Cochran’s C test

or a Bartlett-Box test prior to application of the ANOVA procedure. If each data set did not satisfy the homogeneity of variance assumption, splitting the data set would not be useful. A disadvantage of this alternative is that the data set for comparison is halved. This may require a change in the desired level of significance or confidence interval. Whether this alternative can be used depends heavily on the nature of the data obtained. If the data obtained exhibit the necessary distribution properties for all technologies then this alternative will be used to allow use of ANOVA procedures on raw concentration data.

A third alternative is to transform the concentration data into a form where the variance for the different concentration ranges is equal. Results from prior studies of subsurface sampling indicate that the standard deviation (SD) is, in fact, approximately constant at a level of approximately 100 percent for concentrations ranging from hundreds to millions of parts per billion. Thus, if concentration values were expressed as percent the data would satisfy the homogeneity of variance assumption. For the analysis of data to test hypotheses S3 and SG2 (Table 5-1), the variance data would be expressed as percent of the mean concentration. Specifically, at each grid site the concentrations of each the seven samples collected by a technology would be divided by the mean concentration of all seven samples for that technology. The variance would, thus, be expressed as a percentage of the mean for a relatively narrow range of concentration. Testing of hypothesis S4 (Table 5-1) would require that the final concentration be expressed as a percent of the initial concentration. For the purpose of testing these hypotheses, the transformation to percent units does not present any significant limitations on the inference reached or on the application of the inference.

The transformation of data to test hypotheses S2 (Table 5-1) is slightly more problematic. Two transformations are possible and both represent sufficient change to the meaning of the raw data that it is possible for the application of the inference to be interpreted differently. One transformation involves taking the logarithmic transform of the concentrations. This would result in constant variance over the range of log concentrations and would, thus, satisfy the requirement of homogeneity of variance. It would also, however, mean that the ANOVA result would apply to logarithmic values and it could be interpreted that the conclusions reached cannot be applied to actual concentrations. While this transform may provide a satisfactory test of the statistical hypothesis, interpretation of the means would also require taking the antilog which may be interpreted by some as too complicated. For these reasons, this transform was not selected.

A second transformation that can achieve homogeneity of variance is to transform the data to percent units just as proposed for data addressing hypotheses S3 and SG2 (Table 5-1). The question arises as to which standard to use as a reference. That is, what will the concentrations be expressed as a percentage of? For hypotheses S3 and SG2 (Table 5-1), the reference concentration was the mean concentration for a given grid site for the specific technology. For comparison of concentration and testing of hypotheses S2 (Table 5-1), referencing each technology to itself is not suitable. Since the primary purpose of the demonstration is to compare the technologies with a conventional methodology, the mean of the conventional method concentration at a site grid provides a useful but limited reference. This reference allows meaningful comparison, however, if the conventional method is biased compared to all other technologies, then the inference reached can only be stated in terms relative to the conventional method. A reference that possibly provides the greatest application is the mean of all concentrations for all technologies at a given grid and target depth. Use of this as a reference assumes that all the technologies are adequately and randomly sampling a normally distributed population of concentration existing at the specified depth within the 10.5' x 10.5-foot square area being sampled. This assumption seems appropriate. The large number of samples for each target sampling depth site should also provide the narrowest SD and the greatest probability that the calculated mean represents the true population mean. Hypothesis SG1 (Table 5-1) will be evaluated by transforming both the passive and conventional soil gas data into a percent. This is necessary since the two methods do not produce data in matching units. The conventional soil gas data will be transformed by dividing all the measurements at a given site by the highest reading obtained at the site. This will produce percentages referenced to the high measurement at a site. The ANOVA analysis will be run on this transformed data.

5.6.3 Nonparametric Analysis

The ANOVA procedures described above provide powerful tests of comparison, but require that the data be normally distributed and that the variance be homogeneous. To satisfy these requirements requires transformations of the data that can be interpreted to influence the inference reached concerning the statistical hypotheses. The statistics offered by ANOVA are highly useful, but are not always required to address only acceptance or rejection of the statistical hypotheses. These can be addressed using nonparametric analysis tests that make no assumptions concerning data distribution. For this demonstration, both nonparametric and parametric analysis will be performed.

Numerous nonparametric tests can be used. For this demonstration and testing of the hypotheses, the Mann-Whitney or Wilcoxon Signed Rank test will be used. These tests compare two data sets composed of matched pairs of ranked data and determine if the two methods are similar.

5.6.4 Treatment of Outliers

Before any formal statistical analysis of the data, it is important to determine if any aberrant data was collected that might influence the statistical analysis. Outlier analysis will be performed for each technology. Rejection of an outlier will be considered using the tests outlined in Natrella, 1963.

This method calculates the upper and lower boundaries for outliers to a data set. Data falling outside these boundaries can be considered outliers. This method begins with the determination of the following variables:

α = probability of making a Type I error (for this demonstration $\propto = 0.05$)
μ = mean of the measurements in a data set
δ = standard deviation of the data set
n = number of samples in the data set

Once these variables are determined, Equation 5-1 is used to determine α.

$$\alpha' = 1 - (1-\alpha)^{1/n} \tag{5-1}$$

α' is used to calculate z, the normal deviate, according to Equation 5-2.

$$z = 1-\alpha' \tag{5-2}$$

Finally the outlier boundaries are calculated according to Equation 5-3.

$$\mu \pm (\delta * z) \tag{5-3}$$

If more than one outlier is identified for a given technology at a given target sampling depth, the data for that depth will not be used in the evaluation of the technology.

TABLE 5-1

Test Hypotheses[a]
Soil and Soil Gas Sampling Demonstration

Media	Hypothesis Number	Hypothesis Summary
Soil	S1	Is the percent sample recovery for the innovative technology significantly different from the conventional method of sample collection?
Soil	S2	Is the concentration of contaminants significantly different from the concentration of contaminants in the conventional method's soil sample?
Soil	S3	Is the variance in contaminant concentration for a technology's samples significantly different from the variance in contaminant concentration for the conventional method's samples?
Soil	S4	Is the initial contaminant concentration of a blank soil significantly different than its concentration after being pushed through a contaminated zone by a technology?
Soil Gas	SG1	Is the soil gas concentration reported by the soil gas technology significantly different from the soil gas concentrations reported by the conventional method?
Soil Gas	SG2	Is the variance in contaminant concentration reported by the soil gas technology significantly different from the variance in contaminant concentration for the conventional method?

Note:

[a] These hypotheses will be evaluated for each target depth interval and for data sets combined by factor and as a whole. See Section 6.1.2.2 for a detailed examination of the soil test hypotheses and Section 6.2.2.2 for a detailed examination of the soil gas test hypotheses.

CHAPTER 6
DEMONSTRATION DESIGN

This chapter is divided into five main sections. Section 6.1 presents the demonstration design for the field testing and evaluation of the four soil sampling technologies. Section 6.2 presents the demonstration design for the field testing and evaluation of the two soil gas sampling technologies and the ARA DTD probe. ARA's DTD Probe thermally desorbs volatile and some semi-volatile compounds, and draws them either into a sample container or directly into a GC for analysis. This tool can either provide a relatively continuous measure of desorbable contaminants with depth or it can provide more qualitative compound identification and quantitation at specific depths. Since this technology has characteristics of both a soil and soil gas sampling technology, it will be included in the demonstration associated with soil gas samplers (Section 6.2). This technology will be evaluated for all soil gas test hypotheses and soil sample test hypothesis (S2). Section 6.3 presents the general sampling design and results of predemonstration sampling. (Specific sampling and analysis procedures for the conventional methods are presented in Chapter 7 "Field Operations.") Section 6.4 discusses field data collection and auditing. Section 6.5 presents the field demonstration schedule and the overall demonstration schedule.

The discussion of the demonstration design and data collection will also apply to the conventional sampling methods. This is necessary to allow both quantitative and qualitative comparisons between the technologies and the conventional sampling methods.

6.1 SOIL SAMPLING TECHNOLOGY DEMONSTRATION DESIGN

This section discusses of the demonstration objectives and design, including these basic elements: (1) the precise statement of the problems or hypotheses to be tested, (2) the performance measures (dependent variables) that will be evaluated which will provide the basis for comparison, (3) the factors or independent variables that will be controlled or varied to assess their influence on the performance measures, and (4) the nature of the experimental design and sampling.

6.1.1 Objectives

Fundamental to delineating the nature and extent of contamination at a potential hazardous waste site is the necessity for collecting subsurface soil samples. A subsurface soil sample consists of the soil matrix, the soil gas, and soil moisture. Contamination in a subsurface soil sample eventually achieves a dynamic equilibrium between these three phases. At the present time, one of the most prevalent subsurface soil sampling methods involves the use of the split-spoon sampler technology. While split-spoon samplers have been used to meet DQOs for most environmental sampling, new innovative methods may be faster, easier, less expensive, and equal to or more accurate and precise. Being new and, by definition, nontraditional, it is necessary to demonstrate the performance of these technologies to provide evidence that they work as intended and to eliminate as many nontechnical barriers to their use as possible. Nontechnical barriers may include regulations that preclude the use of the new technologies simply because they did not exist at the time the regulation was enacted, or users who are unwilling to apply new and "unproven" technologies. Through this demonstration, the performance of these new technologies will be compared to a similar technology commonly used in the industry. For this demonstration, split-spoon samplers applied through a hollow-stem auger will be considered the conventional sampling method, and used for comparison. This type of comparison will provide information which will help potential users understand the nature of the results that may be obtained using these technologies and, based on this comparison, evaluate their possible use in the field as a replacement or supplement to the conventional method. The new and innovative technologies will not be directly compared to each other.

Specifically, this demonstration will compare the performance of new and innovative soil sampling technologies to the performance of a conventional soil sampling technology. This comparison will evaluate if they provide samples exhibiting the same relevant properties needed for site investigation as the conventional method. In order to identify specific demonstration objectives, it is necessary to define the basic characteristics of a subsurface soil sampling technology.

The basic characteristics of a subsurface soil sampling technology in environmental work is to collect a representative soil sample, of sufficient volume for analysis, from a specified depth, and return it to the surface with minimal change to the chemical or physical properties of the sample. Specifically, this demonstration will evaluate each new and innovative technology relative to the conventional method or developer claims for the following basic subsurface soil sampling functions: (1) ability to retrieve a

predetermined sample volume from a precise depth for a series of attempts, (2) ability to provide chemically and physically similar samples (relative to corresponding conventional samples) for a series of attempts (assuming the media sampled is homogenous) for a range of soil textures and for a range of sample concentrations, (3) ability to return the sample to the surface while preserving the sample integrity, including chemical concentration and physical properties, for a range of external environments, (4) ability to perform these operations in a consistent and measurable time period, and (5) ability to perform these operations for a consistent and measurable cost.

For the purpose of this demonstration, once the soil sample is removed from the sampler the samples are no longer a part of the sampling technology, but are part of a chemical analytical method. As described in Section 6.4, all samples will be handled in the same fashion after they are removed from the samplers; this is critical to the design of this evaluation.

6.1.2 Statement of the General Problem and Functional Elements

The sampling functions described above provide a basis for identifying meaningful criteria or performance measures for which a comparison between an innovative technology and the conventional method can be performed. The criteria for evaluation of each performance measure must be clearly stated in the form of a statistical hypothesis that can be tested so that conclusions reached are clear and can be shown to have been supported by the observed data within the stated statistical limits. Since it is desirable for the conclusions reached to be applicable to as many environmental investigation sites as possible, the test of each hypothesis must be conducted under a wide range of conditions. These conditions will be discussed below.

Before presenting statistical hypotheses to be tested it is important to address a question about the nature of the comparison (intermethod comparison) and to define the specific performance measures or criteria (dependent variables) that will provide accurate and quantifiable measures of comparison and that meaningfully address the basic question and the specific statistical hypotheses.

6.1.2.1 Intermethod Comparison

The primary goal of the demonstration is to compare the performance of a given innovative subsurface soil sampling technology to the performance of the conventional subsurface soil sampling technology, and to evaluate if it provides samples exhibiting the same relevant properties needed for site investigation, as the conventional method. Hypotheses can be stated that address this question, however, they may be stated in such a fashion that they may not address the larger questions: does the innovative technology provide results that are different from the average of all other innovative technologies or does the conventional method provide different results from the average of all other innovative technologies? If the hypotheses are stated to only address comparison with the conventional method we are not able to test whether the conventional method represents a biased sample, nor whether the innovative technology being evaluated does not exhibit a significant advantage (or disadvantage) over the complete field of available technologies.

To illustrate this point, let us suppose that when the split-spoon sampler is opened for transfer of soil to a vial, some VOCs are lost through volatilization. Further, suppose that a range of technologies do not expose the sample to air during transfer and, therefore, do not allow such a loss of VOCs. An isolated comparison of the technology with the conventional method would indicate that the technology exhibited higher concentrations than the conventional method, however, comparison of the conventional method with all technologies would show that the conventional method was biased low. Similarly, comparison of the innovative technology with all other technologies would show that the technology exhibited concentrations that were the same as the total population of innovative technologies.

To allow the collected data to provide answers to the intermethod comparisons, the statistical hypotheses presented below have been stated so that the following three comparisons can be tested: (1) the measures of performance of a given innovative technology can be compared to similar measures of performance exhibited by the standard subsurface soil sampling method, (2) the measures of performance of a given innovative technology can be compared to similar measures of performance for the range of innovative technologies tested, and (3) the measures of performance of the conventional method can be compared to similar measures of performance for the whole range of innovative technologies tested. Comparison will not be made between any two individual innovative technologies. In addition, for the comparison of an

individual innovative technology with the range of innovative technologies and for the comparison of the conventional method with the range of innovative technologies, the data representing the range of innovative technologies will be a composite of all similar data produced by the innovative technologies. Results from a specific technology will neither be identified, nor be able to be isolated from the data sets representing the range of innovative technologies evaluated.

6.1.2.2 Quantitative Performance Criteria (Dependent Variables)

Meaningful intermethod comparison of performance requires comparison of a wide variety of performance criteria that are each representative of the different functions that are part of sampling presented in the general demonstration objectives outlined in Section 6.1.1. Criteria or measures of performance that allow meaningful comparison can be both quantitative and qualitative.

Examination of the major functional elements of subsurface soil sampling indicate that the quantitative measures of performance or criteria that are important for comparison include: (1) the reliability with which the innovative technologies physically collect soil samples at specified depths in various soil types (percent sample recovery), (2) the degree to which average concentrations of target volatile constituents in the soil samples collected using a given innovative technology agree with average concentrations measured from samples collected using the conventional soil sampling method and with the average concentration exhibited by samples collected using the range of innovative sampling technologies, (3) the degree to which variability of concentrations measured on samples collected using a given innovative technology agree with variability of concentrations measured from samples collected using the conventional soil sampling method and with the variability of the range of innovative sampling technologies, (4) the degree to which the integrity of sample concentrations are preserved when captured samples must pass through intervals of different concentration and properties, (5) how do sample retrieval times compare with those of the conventional sampling method and with the range of innovative sampling technologies, and (6) how do sample costs compare with costs of the conventional sampling method and with the range of costs for the range of innovative sampling technologies.

Summarizing the above quantitative criteria, the following dependent variables will be measured for both the conventional method and the innovative technologies in order to assess the general demonstration objectives:

(1) Proportion of attempts that the technology successfully reached a specified sampling depth and the proportion of sampler volume that was filled with sample

(2) Average concentration of target VOCs: vinyl chloride, TCE, PCE, 1,1,1-TCA, and 1,2-DCE

(3) SD of concentration of target VOCs

(4) Average change in concentration from a reference concentration (soil blank) when the sample is moved through an external environment of different concentration or properties

(5) Average sample retrieval time

(6) Average sample retrieval cost

To provide clear conclusions about the nature of the points of comparison listed above, it is necessary to clearly state the criteria being compared and the exact nature of the comparison in the form of a statistical null hypothesis (H_o). Dependent variables S2 and S3 will not be evaluated through hypothesis testing, rather this data will be recorded and presented in tabular form with similar data from the conventional method. The significance of these variables will need to be assessed by potential users based on project needs. A null hypothesis is then tested and is either accepted or rejected at some level of significance. The general language used to describe the intermethod comparisons is too imprecise to reach clear conclusions about the comparisons. Therefore, precise null hypotheses are stated below. These hypotheses represent the major conclusions that will be tested using the data collected during the demonstration.

For each null hypothesis, there is an alternate hypothesis that for this demonstration is defined as the negative of the null hypothesis. For example, if the null hypothesis states that some property of Method A differs from that of Method B, then the alternate hypothesis is that the property of Method A does <u>not</u> differ from that of Method B.

As noted above, it is desirable to test each null hypothesis at three levels: (1) between an individual technology and the conventional method, (2) between an individual technology and the range of technologies, and (3) between the conventional method and the entire range of technologies. To avoid restating the null hypothesis for each of these three levels of comparison, the null hypotheses presented below are phrased in a generic fashion using representative variables for the methods. Thus, the term Method A may be replaced with either (1) an individual subsurface soil sampling technology, or (2) the standard subsurface soil sampling technology. The term Method B may be replaced with either (1) the standard subsurface soil sampling technology, or (2) the combined and undifferentiated results of the entire range of technologies demonstrated. The four permutations for combinations of Method A and Method B are only unique for three since the comparison of the conventional method with itself is unnecessary.

Using the above conventions the following four null hypotheses for soil, stated both grammatically and mathematically, will be tested in this demonstration:

S1) Comparing Reliability in Physically Recovering a Sample:

The average recovery within the sample chamber for Method A differs from Method B. This factor is dependent on both the technology itself and the operator. Since the operators of the technologies are provided by the developers, the operator effect is considered to have little impact on this evaluation.

$$(H_o: |X_A - X_B| > 0) \qquad (6\text{-}1)$$

where

X_A= average of all sample recoveries, expressed as a percentage of the sample volume chamber that is filled with soil, for Method A

X_B= average of all sample recoveries, expressed as a percentage of the sample volume chamber that is filled with soil, for Method B

S2) Comparing the Chemical Concentration of the Recovered Soil Samples:

The average concentration of target VOCs measured from soil samples collected using Method A differs from Method B.

$$(H_o: |X_A - X_B| > 0) \quad (6\text{-}2)$$

where

X_A= average concentration micrograms per kilogram (μg/kg) of a selected volatile compound measured from soil samples collected using Method A. As discussed below this average is for samples collected at a similar concentration and in a similar soil texture. (This would require that the samples be collected at the same local site and from the same depth.)

S3) Comparing the Variance in Chemical Concentration of the Recovered Soil Samples:

The variance of the population of concentrations of target VOCs measured from soil samples collected using Method A differs from Method B.

$$(H_o: |X_A - X_B| > 0) \quad (6\text{-}3)$$

where

X_A= the variance of the population of concentrations measured for target VOCs from soil samples collected using Method A expressed as a percentage of the mean concentration. As discussed below, this variance is expressed as a percent of the mean concentration for samples collected at a similar concentration and in a similar soil texture. (This would require that the samples be collected at the same local site and from the same depth.)

S4) Comparing the Chemical Concentration of a Blank Soil after it is Advanced through a Contaminated Zone in a Soil Sample:

The average final concentration of target VOCs measured from soil samples collected using Method A differs from the average final concentration of Method B for the same compounds, when the sample within the sample collection chamber has passed through intervals of different concentration and properties. (An assessment of sample integrity.)

$$(H_o: |X_{Afinal} - X_{Bfinal}| > 0) \quad (6\text{-}4)$$

where

X_{afinal}= the average final concentration (expressed as percent of initial concentration) of the population of concentrations measured for target VOCs from soil samples collected

using Method A. Final concentrations would be measured after the sampler, including a sample at an initial concentration, had passed through contaminated soil intervals containing different chemical properties relative to the properties of the sample itself. Initial concentrations are all 100 percent.

Sample throughput will be monitored by the technology observers. Throughput will be measured as the time interval required to set up on a sample point, collect the specified sample, grout the hole, decontaminate samplers, and move to a new sampling point. This information will be recorded for the standard sampling method.

Sample costs will be estimated for each technology. The costs will include purchasing or renting equipment, mobilizing and demobilizing, expendable supplies, and labor to operate the technology. This information will be presented for the reference sampling method.

6.1.2.3 Nonquantitative Performance Criteria (Dependent Variables)

The above performance measures are important to performance comparison and are suitable for statistical analysis. There are also performance criteria that are important to evaluate, but which are difficult to measure or are not suitable for statistical analysis. These following criteria will be observed and recorded, and the results reported in summary tables without statistical comparison between methods:

(1) reliability under the test conditions (failure rate during evaluation)

(2) susceptibility to environmental conditions (ruggedness)

(3) ease of operation

(4) ease of learning to use technology

(5) logistical requirements

(6) degree of physical sample disruption

(7) type and amount of sample handling after collection that is necessary for laboratory-ready samples

(8) degree to which measures of performance meet developer performance claims

6.1.2.4 Operational Specifications

The above quantitative and nonqualitative criteria provide measures of comparison for equivalent or similar performance functions. However, due to design differences there are also differences between technologies that are important to compare, but that simply represent operational specifications. These specifications will be summarized in a table:

(1) minimum and maximum depth of sample retrieval

(2) sample volume and geometry

(3) operating temperature range

(4) operating humidity or moisture range

(5) power and water requirements

(6) weight and size of technology

(7) amount and type of investigation derived waste

(8) amount(s) and type(s) of material necessary to prepare and restore sampling site

(9) the basic procedural steps required for sample retrieval

(10) health and safety aspects of operating the technology

6.1.2.5 Selection of Factors (Independent Variables)

The performance criteria outlined in Section 6.1.1, as well as the statistical hypotheses being tested can be influenced by many factors. These factors can be considered independent variables. These factors can be quantitative (that is, vary continuously), such as concentration or temperature, or qualitative, such as the sampling technology or the soil type. These factors can be either fixed (such as selection of a particular series of values) or random. The influence of these factors must be controlled in the demonstration either by holding the factors constant for all tests (and thereby limiting the demonstration conclusions to those constant conditions), obtaining data for a series of combinations of factors at various

fixed or random values, or randomizing test conditions resulting in a random influence of less important factors.

The act of sampling in the field is subject to many influences, some of which are major and significantly affect the nature of the sample obtained, and some of which do not exert a significant affect and are considered minor.

Major factors are consider to be those factors or variables that in their typical range of values encountered under field conditions are capable of significantly affecting several of the demonstration's performance criteria. Significant affects have the potential to cause false acceptance or rejection of one or more of the statistical hypotheses. Controls for major factors in the experimental design are discussed in Section 6.1.3. The major factors that are believed to most significantly affect the demonstrations performance criteria described above include:

- Sampling Technology
- Soil Type
- Contaminant Concentration
- Contaminant Concentration Heterogeneity
- Operator Skill
- Sample Handling Procedures (after removal from the sampling technology)
- Temperature

The demonstration is designed to allow statistical analysis of the relative influence of the first three factors. The last four factors will be controlled by holding them constant or randomizing the testing to minimize the influence of their fluctuations.

Minor factors are consider to be those factors or variables that in their typical range exert limited influence on most of the performance criteria, but that outside their typical range are capable of affecting several of the performance criteria and, thus, of causing false acceptance or rejection of a null hypothesis.

Controls for minor factors in the experimental design are discussed in Section 6.1.3. The minor factors that are believed to most significantly affect the performance criteria described above include:

- Site Surface Topography
- Site Surface Conditions
- Site Logistics
- Weather (excluding temperature)
- Sample Depth
- Sample Bulk Density
- Analytical Laboratory Methodology
- Order in Sampling Schedule
- Decontamination Procedures and Efficiency

The demonstration has been designed to control for the influence of these minor factors by holding them constant or randomizing the testing to minimize the influence of fluctuations.

6.1.3 EXPERIMENTAL DESIGN

The basic experimental design of the demonstration is to evaluate the effects of the major factors described above in Section 6.1.2 and to provide quantitative data that can be analyzed statistically to test the hypotheses presented in Section 6.1.1. Conducting an experiment that controls and tests for all the major levels for all the factors listed in Section 6.1.2 would require prohibitive time and expense. Even assuming that each major factor had only two levels or occurrence, the number of combinations that would need to be tested would be 2^7 or 128 combinations. To limit the demonstration cost and still provide meaningful answers to the statistical hypotheses posed, the number of experimental conditions was limited to testing only for several levels for each of the first three major factors. The last four major factors will be controlled by various methods.

To design the demonstration, the number of levels for the first three factors (technology, soil type, and contaminant concentration) must be selected. The number of levels for the sampling technology is equal to the number of sampling technologies to be tested. In the demonstration, there will be four soil sampling technologies and the conventional method resulting in five levels.

Soil type as a factor includes numerous subfactors that represent combinations of soil type properties. Several of the most important of these would be the mean grain size, grain size distribution, grain density, mineralogy, grain surface area, porosity, and organic carbon content. Clearly, it is not feasible to control or test for the influence of all of these variables. However, it is also probably not necessary to control for all these variables to address the influence of soil texture on sampling performance. In considering the performance criteria being evaluated, the soil texture variables most likely to influence the performance criteria are mean grain size and grain size distribution. Mineralogy, grain density, porosity, and organic carbon content are likely to exert minor influence on the performance criteria within the range of values they typically exhibit at environmental sites. The performance criteria most likely to be affected by grain size and grain size distribution are the average recovery, the variability of concentration, sample integrity, and the sampling time and cost.

While, clearly, sample grain sizes can be varied over a wide range, selection of two levels, representing a wide range in grain size, could adequately represent the outer limits of influence of grain size and sorting on sampling. Two soil types have been selected: fine-grained soils composed primarily of silts and clays, and coarse-grained soils composed of primarily medium- to coarse-grained sand with little silt or clay fraction.

It is possible that the performance of the various technologies could vary with the concentration of the contaminants contained within the soil sample. To control for this variable, two levels have been selected, high and low. High concentrations would be represented by contaminant concentrations in the range of one to tens of ppm. These concentrations have been selected to approximate soil action levels commonly associated with determining the risk from direct contact. Low concentrations will be represented by contamination in the 10s to 100s of ppb range. These concentrations are commonly associated with action levels designed to be protective of groundwater. Concentrations less than 2.5 times greater than the analytical MDL are being avoided, even though they represent an important concentration range for environmental work, because of the potentially confounding influences of

analytical method error near detection limits, and the heterogeneity of concentration within the site test area. Based on predemonstration analysis, the on-site laboratory has an MDL of 2 μg/kg for the low concentration analysis.

Based on this distribution of factors, the four soil technologies and the conventional method would have the following four combinations of test conditions:

Soil Type	Concentration
Sand	Low
	High
Clay	Low
	High

Analytical data will be obtained for each of these four factor combinations for each of the technologies and the conventional method, which will result in a total of 20 combinations of tests.

The number of samples to be obtained for each test condition is dictated by the desired statistical probability of performing a Type I error (that is, the level of statistical significance), the desired probability of avoiding a Type II error (that is, the power of the test, β), and the acceptable error (δ^2). Previous analysis of the variance in concentration that is exhibited by sampling in the field indicates that concentration heterogeneity or sampling heterogeneity exhibits an SD that is about 100 percent of the mean sample concentration for a wide range of concentrations. To conduct tests of the statistical hypotheses stated in Section 6.1.1 comparing an innovative technology with the conventional method, at a level of significance of α=0.05, and a power of β=0.95 when the error (δ^2) is within 50 percent of the estimated SD, a minimum of seven samples (points of comparison) are required for each of the five technologies (including the conventional method). This represents the number of observations (samples) that must be obtained for each technology, for each combination of factors. Because of the factorial design, fewer samples are required in each test condition to satisfy the number of samples.

Major factors that are not tested at various levels include: contaminant concentration heterogeneity, operator skill, sample handling procedures after the soil core is brought to the surface, and temperature. Contaminant concentration heterogeneity is being handled by repeat sampling within the site location

where sample locations will be assigned randomly. Each cell within the grid is to be 18 inches square. Predemonstration sampling has roughly defined the heterogeneity of the subsurface interval to be sampled. To be used in the demonstration, a target sampling interval in a grid exhibits the following characteristics: the soil texture may not change in dominant grain size; and the concentrations of at least one target analyte may not vary by more than a factor of five, over all the samples collected at a given grid and given depth interval. Since these criteria are relatively conservative, instead of sampling a single location for each combination of factors (four locations), a minimum of two locations will be sampled for each combination of factors (eight locations). This will provide backup data in the event a given grid produces data outside the acceptable criteria. These target sampling locations will be identified as specific depths under 10.5- by 10.5-foot sampling grids. These grids will be further divided into 7 rows and 7 columns producing 49 18- by 18-inch sampling cells. The intersection of a row and column will produce a cell 18 inches on a side (Figure 6-1). The cells in each row will be assigned numbers from 1 to 7. The cell columns will be labeled A to G. Of the 49 possible cells in a sample grid, each technology (including the conventional method) will sample in seven cells (Figure 6-1). The assignment of which cells are to be sampled will be random. In this manner, each technology will be randomly sampling the area within each grid.

Operator skill can exert an influence on several performance criteria. Rather than test for this variable, it has been decided that the technologies will be operated by experienced operators selected by the technology developer. This choice limits application of results concerning performance only to experienced operators, but this is considered appropriate.

Sample handling procedures, once the soil core is brought to the surface, may vary between technologies. This factor will be held constant by using consistent sample handling procedures between all sampling technologies as discussed in Section 7.2.

Temperature can have an influence on volatilization. While it would be desirable to perform tests under various temperature conditions, the influence of temperature is likely to be greatest only under high temperature conditions. The demonstration will be performed under moderate temperature conditions near 70 ^{o}F. Limiting the demonstration to this condition is not believed to significantly limit the applicability of the conclusions reached.

The null hypothesis dealing with the preservation of sample integrity will be assessed differently for technologies that collect a physical soil sample and technologies that analyze a sample in situ (ARA DTD Probe). The degree to which a soil sampler protects chemical integrity of a sample will be evaluated by advancing a sampler filled with an uncontaminated loam soil (blank soil) through a zone of grossly contaminated soil. This zone will be identified in one sampling grid at each site. It will be a minimum of 2 feet thick and exhibit volatile organics contamination in the 10's to 1,000's of ppm, at a minimum. A sampler will be decontaminated and filled with a pre-analyzed loam soil which did not exhibit any detectable volatile organics contamination. The sampler filled with the uncontaminated loam soil will be advanced at least 4 feet into the contaminated zone. After the sampler has penetrated the contaminated zone, it will be withdrawn and the loam soil sampled for analysis. This integrity testing will be conducted in each cell at Grid No. 1 at both sites. At the SBA site, this test will be performed after the collection of the deepest demonstration soil samples in each cell. The filled sampler will be advanced 4 feet past the deepest demonstration sampling point. At the CSC site, samplers filled with blank soil will be advanced from 10 to 14 feet bgs at each cell in Grid No. 1.

During the sampling activities producing the data which will be used to evaluate the quantitative variables, all of the qualitative variables will be monitored, measured, assessed, and recorded by the PRC technology observer.

6.2 SOIL GAS SAMPLING TECHNOLOGY DEMONSTRATION DESIGN

This section focuses on the demonstration and evaluation of the two passive soil gas sampling technologies: the Emflux® and Gore-Sorber® samplers; and the ARA DTD probe. This section discusses the demonstration objectives and design including these basic elements: (1) the precise statement of the problems or hypotheses to be tested that the demonstration will answer, (2) the performance measures (dependent variables) that will be evaluated which will provide the basis for comparison, (3) the factors or independent variables that will be controlled or varied to assess their influence on the performance measures, and (4) the nature of the experimental design and sampling.

6.2.1 Objectives

Identifying the source of subsurface contamination and delineating the relative nature and extent of contamination at a potential hazardous waste site is a common application of soil gas sampling. At the present time, the most prevalent method of collecting soil gas samples involves actively withdrawing a volume of soil gas into a sample container for direct analysis by on-site gas chromatography. This type of sampling collects data in the immediate vicinity of the end of the soil gas pipe, and provides data that may be sensitive to temporal or other variations. The reproducibility of this data is often low. This type of sample cannot distinguish between soil and groundwater soil gas sources. While active soil gas sampling has been used to meet DQOs for most environmental applications, new innovative methods can be easier to deploy, relatively less expensive, more effective in fine-grained soils, relatively unaffected by temporal variation, more sensitive to a wider range of contaminants at low concentrations, more reproducible, and equal to or more accurate and precise. The passive soil gas technologies allow their samplers to reach equilibrium with the soil-soil gas-moisture matrix. This is accomplished by leaving the samplers exposed to the soil over a period of a few days. This integrates contamination over a broader area and volume of the soil matrix and may increase sensitivity.

Being new and by definition nontraditional, it is necessary to demonstrate the performance of these technologies to provide evidence that they work as intended and to eliminate as many nontechnical barriers to their use as possible. Nontechnical barriers could include regulations that preclude the use of the new technologies simply because they did not exist at the time the regulation was enacted, or users who are unwilling to apply new and “unproven” technologies. Through this demonstration, the performance of these new soil gas sampling technologies will be compared to a soil gas collection and analysis method commonly used in the industry. For this demonstration, active soil gas collection by Geoprobe® Systems and AMS and on-site analysis will be considered the conventional method, and used for comparison. The new and innovative technologies will not be compared to each other. This type of comparison will provide information which will help potential users understand the nature of the results that may be obtained using these technologies and, based on this comparison, evaluate their possible use in the field as a replacement or supplement to the conventional method.

Specifically, this demonstration will quantitatively compare the performance of the two innovative passive soil gas sampling technologies and the ARA DTD probe to the performance of a standard active

soil gas sampling technology. The demonstration will evaluate if the innovative technologies provide samples exhibiting the same relevant properties, needed for site investigation, as the conventional methods. Since the unit of measure for the passive soil gas and ARA technologies is mass and the conventional methods report results in either mass per volume or mass per mass, this comparison will be made on transformed data. The passive soil gas and ARA data will be transformed to a percentage relative to the highest reading for that technology at a given site. The conventional method (soil gas) results will also be transformed to a percentage relative to their respective high measurements at a given site. The specific objectives and questions asked during the demonstration of soil gas technologies are described below. The ARA DTD probe will also be evaluated relative to soil sampler hypotheses S2 and S3 (Table 5-1). This evaluation will be at the target depth intervals specified for the soil sampling demonstration. In order to identify specific demonstration objectives, it is necessary to define the basic characteristics of a soil gas sampling technology.

The basic characteristics of a soil gas sampling technology in environmental work is to collect a soil gas sample from a specified depth, provide data identifying contamination source location, and provide data delineating the relative extent of soil gas contamination. Since the soil gas is only a fraction of the soil matrix and its chemical and physical properties are different from water, soil gas data is generally only used to identify the general location of contaminant sources or the relative extent of contamination. Except for the ARA DTD Probe, soil gas measurements cannot identify the exact location of contamination, either in the soil or groundwater. Soil gas sampling is generally applied as a true screening technology, used to optimize the location of soil or groundwater samples. This demonstration is not intended to validate the design or principals of operation of the soil gas samplers, rather, this demonstration is designed to determine how innovative technologies perform relative to the conventional approach and to assess if the conventional approach has a bias. Specifically, this demonstration will evaluate each new and innovative technology relative to the conventional method or developer claims for the following basic soil gas sampling functions: (1) ability to provide chemically similar samples (relative to the conventional method) for a series of attempts (assuming the media sampled is homogenous) for a range of soil textures and for a range of sample concentrations, (2) ability to perform these operations in a consistent and measurable time period, and (3) its ability to perform these operations for a consistent and measurable cost.

For the purpose of this demonstration, these innovative soil gas sampling technologies incorporate both the collection of a sample and the process of analyzing the sample. The analysis is included with the sample collection since both of the soil gas samplers require developer analysis of the samplers and the ARA DTD probe can transfer the soil gas sample directly into a GC for analysis. As described in Chapters 3 and 5, the soil gas samplers will be implanted and analyzed according to the developer specifications. The ARA DTD probe will also be operated according to its developers specifications.

6.2.2 Statement of the General Problem and Functional Elements

The sampling functions described above provide a basis for identifying meaningful criteria or performance measures for which a comparison between a technology and the conventional method can be performed. The criteria for evaluation of each performance measure must be clearly stated in the form of a statistical hypothesis that can be tested so that conclusions reached are clear and can be shown to have been supported by the observed data within the stated statistical limits. Since it is desirable for the conclusions reached to be applicable to as many environmental investigation sites as possible, the test of each hypothesis must be conducted under a wide range of conditions. These conditions will be discussed below.

Before presenting statistical hypotheses to be tested, it is important to address a question about the nature of the comparison (intermethod comparison) and to define the specific performance measures or criteria (dependent variables) that will provide accurate and quantifiable measures of comparison and that meaningfully address the basic question and the specific statistical hypotheses.

6.2.2.1 Intermethod Comparison

The primary goal of the demonstration is to compare the performance of a given soil gas sampling technology to the performance of the conventional method, and to evaluate if it provides samples exhibiting the same relevant properties needed for site investigation as the conventional method. Hypotheses can be stated that address this question, however, they may be stated in such a fashion that they may not address a larger question: does the conventional method provide different results from the average of the innovative technologies? If the hypotheses are stated to only address comparison with the

conventional method, PRC will not be able to test whether the conventional method represents a biased sample.

To allow the collected data to provide answers to the intermethod comparisons mentioned above, the statistical hypotheses presented below have been stated so that the following two comparisons can be tested: (1) the measures of performance of a given technology can be compared to similar measures of performance exhibited by the conventional method, and (2) the measures of performance of the conventional method can be compared to similar measures of performance for the whole range of technologies tested. Comparison will not be made between the individual technologies.

6.2.2.2 Quantitative Performance Criteria (Dependent Variables)

Meaningful intermethod comparison of performance requires comparison of a wide variety of performance criteria that are each representative of the different functions that are part of sampling presented in the general demonstration objectives outlined in Section 6.2.1. Criteria or measures of performance that allow meaningful comparison can be both quantitative and qualitative.

Examination of the major functional elements of soil gas sampling indicate that the quantitative measures of performance or criteria that are important for comparison include: (1) the degree to which average relative concentrations of target VOCs in the soil gas collected using a given technology agree with average relative concentrations measured on samples collected using the conventional method, (2) the degree to which the variability of concentrations of target VOCs in the soil gas collected using a given technology agree with variability concentrations measured on samples collected using the conventional method, (3) how do sample retrieval times compare with those of the standard sampling method and with the range of technologies, and (4) how do sample costs compare with costs of the conventional method and with the range of costs for the range of technologies.

Summarizing the above quantitative criteria, the following dependent variables will be measured for both the conventional method and the technologies in order to assess the general demonstration objectives:

(1) Average concentration of target VOCs in the soil gas samples reported as a percentage of the high average measurement, for a given technology, at a given site. The target analytes for the soil gas sampler demonstration are: vinyl chloride, 1,1,1-TCA, TCE, PCE, DCA, and DCE

(2) SD of concentration of target volatile compounds for soil gas based on nontransformed data

(3) Average sample retrieval time

(4) Average sample retrieval cost

The last two dependent variables associated with sample retrieval time and cost will not be evaluated through statistical testing. Rather, this data will be recorded and presented in tabular form with the same data for the conventional method, in the ITER.

To provide clear conclusions about the nature of the first two points of comparison listed above it is necessary to clearly state the criteria being compared and the exact nature of the comparison in the form of a statistical null hypothesis (H_o). This null hypothesis is then tested and is either accepted or rejected at some level of significance. The general language used to describe the intermethod comparisons is too imprecise to reach clear conclusions about the comparisons. Therefore, precise null hypotheses are stated below. These hypotheses represent the major conclusions that will be tested using the data collected during the demonstration.

For each null hypothesis, there is an alternate hypothesis that for this demonstration is defined to be the negative of the null hypothesis. For example, if the null hypothesis states that some property of Method A differs from that of Method B, then the alternate hypothesis is that the property of Method A does <u>not</u> differ from that of Method B.

As noted above, it is desirable to test each null hypothesis at two levels: (1) between an individual technology and the conventional method, and (2) between the conventional method and the entire range

of technologies. To avoid restating the null hypothesis for each of these two levels of comparison, the null hypotheses presented below are phrased in a generic fashion using representative variables for the methods. Thus, the term Method A may be replaced with either (1) an individual technology, or (2) the conventional method. The term Method B refers to the conventional method. The three permutations for combinations of Method A and Method B are only unique for three since the comparison of the conventional method with itself is unnecessary.

Using the above conventions, the following two null hypotheses for soil gas, stated both grammatically and mathematically, will be tested in this demonstration:

SG1) Comparing the Chemical Concentration of the Recovered Soil Gas Samples:

The relative concentration of target volatile compounds measured as a percentage of the highest measurement for a given technology (Method A), at a given site differs from the relative concentration of target volatile and semivolatile petroleum hydrocarbon compounds in soil gas measured as a percentage of the highest measurement for Method B (standard soil gas sampling method).

$$(H_o: |X_A - X_B| > 0) \qquad (6\text{-}5)$$

where

X_A= relative concentration (as a percentage relative to the high concentration) of target volatile compounds measured in soil gas samples collected using Method A. As discussed below, this relative concentration represents samples collected at a similar concentration, and in a similar soil texture.

SG2) The Variance of the Population of Concentrations of Target Volatile Compounds Measured on Soil Gas Samples Collected using Method A differs from Method B.

$$(H_o: |X_A - X_B| > 0) \qquad (6\text{-}6)$$

where : X_A= the variance of the population of concentrations (μg) measured for target volatile compounds in soil gas samples collected using Method A expressed as a percentage of the mean concentration. As discussed below, this variance is expressed as a percent of the mean concentration, for samples collected at a similar concentration and in a similar soil texture.

Sample throughput will be monitored by technology observers. Throughput will be measured as the time interval required to set up on a sample point, collect the specified sample, grout holes, decontaminate samplers, and move to a new sampling point. This information will also be recorded for the conventional method.

Sample costs will be estimated for each technology. They will include the cost of purchasing or renting equipment, mobilizing and demobilizing, expendable supplies, and labor to operate the technology. This information will also be presented for the conventional method.

6.2.2.3 Nonquantitative Performance Criteria (Dependent Variables)

The above performance measures are important to performance comparison and are suitable for statistical analysis. There are also performance criteria that it are important to evaluate, but which are difficult to measure or are not suitable for statistical analysis. These following criteria will be observed and recorded, and the results reported in summary tables without statistical comparison between methods:

(1) reliability under the test conditions (failure rate during evaluation)

(2) susceptibility to environmental conditions (ruggedness)

(3) ease of operation

(4) ease of learning to use technology

(5) logistical requirements

(6) type and amount of sample handling after collection that is necessary for laboratory-ready samples

(7) degree to which measures of performance meet developer performance claims

6.2.2.4 Operational Specifications

The above quantitative and nonqualitative criteria provide measures of comparison for equivalent or similar performance functions. However, due to design differences there are also differences between technologies that are important to compare but that simply represent operational specifications. These specifications will be summarized in a table:

(1) minimum and maximum depth of sample retrieval

(2) sample volume and geometry

(3) operating temperature range

(4) operating humidity or moisture range

(5) weight and size of technology

(6) amount and type of investigation derived waste

(7) amount(s) and type(s) of material necessary to prepare and restore sampling site

(8) the basic procedural steps required for sample retrieval

(9) health and safety aspects of operating the technology

6.2.2.5 Selection of Factors (Independent Variables)

The performance criteria outlined in Section 6.2.1, as well as the statistical hypotheses being tested can be influenced by many factors. These factors can be considered independent variables. These factors can be quantitative (that is vary continuously) such as concentration or temperature, or qualitative such as the sampling technology or the soil type. These factors can be either fixed (such as selection of a particular series of values) or random. The influence of these factors must be controlled in the demonstration either by holding the factors constant for all tests (and thereby limiting the demonstration conclusions to those constant conditions), obtaining data for a series of combination of factors at various fixed or random values, or randomizing test conditions resulting in a random influence of less important factors.

The act of sampling in the field is subject to many influences some of which are major and significantly affect the nature of the sample obtained and some of which do not exert a significant affect and are considered minor.

Major factors are consider to be those factors or variables that in their typical range of values encountered under field conditions are capable of significantly affecting several of the demonstration's performance criteria. Significant affects have the potential to cause false acceptance or rejection of one or more of the statistical hypotheses. Controls for major factors in the experimental design are discussed in Section 6.2.3. The major factors that are believed to most significantly affect the demonstrations performance criteria described above include:

- Sampling Technology
- Soil Type
- Contaminant Concentration
- Soil Moisture
- Contaminant Concentration Heterogeneity
- Operator Skill
- Temperature

The demonstration is designed to allow statistical analysis of the relative influence of the first two factors for the soil gas samplers. Concentration is not considered a primary factor since these technologies produce a aggregate measure of all soil gas contamination in the soil and gases being released from groundwater, and due to the screening nature of soil gas data; there are no soil gas based action levels. Contaminant concentration will be considered a primary factor for the ARA DTD probe during its evaluation for soil sampler hypotheses S2 and S3. This technology produces soil gas data which can be tied to discrete depth intervals. The last five factors will be controlled by holding them constant or randomizing the testing to minimize the influence of their fluctuations.

Minor factors are consider to be those factors or variables that in their typical range exert limited influence on most of the performance criteria but that outside their typical range are capable of affecting

several of the performance criteria and thus of causing false acceptance or rejection of a null hypotheses. Controls for minor factors in the experimental design are discussed in Section 6.2.3. The minor factors that are believed to most significantly affect the performance criteria described above include:

- Site Surface Topography
- Site Surface Conditions
- Site Logistics
- Weather (excluding temperature)
- Sample Depth
- Analytical Laboratory Methodology
- Order in Sampling Schedule
- Decontamination Procedures and Efficiency

The demonstration has been designed to control for the influence of these minor factors by holding them constant or randomizing the testing to minimize the influence of fluctuations.

6.2.3 Experimental Design

The basic experimental design of the demonstration is to evaluate the effects of the major factors described above in Section 6.2.2 and to provide quantitative data that can be analyzed statistically to test the hypotheses presented in Section 6.2.1. Conducting an experiment that controls and tests for all the major levels for all the factors listed in Section 6.2.2 would require prohibitive time and expense. Even assuming that each major factor had only two levels or occurrence, the number of combinations that would need to be tested would be 2^7 or 128 combinations. To limit the demonstration cost and still provide meaningful answers to the statistical hypotheses posed, the number of experimental conditions was limited to testing only for several levels for each of the first two major factors. The last five major factors will be controlled by various methods.

To design the demonstration, the number of levels for the first two factors (technology and soil type) must be selected. Levels of contaminant concentration must also be determined for the evaluation of the ARA DTD probe (This has been discussed in Section 6.1.3). The number of levels for the soil gas sampling technologies is equal to the number of technologies to be tested. In the demonstration, there will be three technologies and the conventional method resulting in four levels. Soil type as a factor includes numerous subfactors that represent combinations of soil type properties. Several of the most important of these would be the mean grain size, grain size distribution, grain density, mineralogy, grain surface area, porosity, and organic carbon content. Clearly, it is not feasible to control or test for the influence of all of these variables. However, it is also probably not necessary to control for all these variables to address the influence of soil texture on sampling performance. In considering the performance criteria being evaluated, the soil texture variables most likely to influence the performance criteria are mean grain size and grain size distribution. Mineralogy, grain density, porosity, and organic carbon content are likely to exert minor influence on the performance criteria within the range of values they typically exhibit at environmental sites. The performance criteria most likely to be affected by grain size and grain size distribution are the average sample recovery, the variability of concentration, and the sampling time and cost.

While, clearly, sample grains sizes can be varied over a wide range, selection of two levels, representing a wide range in grain size, could adequately represent the outer limits of influence of grain size and sorting on sampling. Two soil types have been selected: fine-grained soils composed of primarily silts and clays, and coarse-grained soils composed of primarily medium- to coarse-grained sand with little silt or clay fraction.

It is possible that the performance of the ARA DTD probe could vary with the concentration of the contaminants contained within the soil sample. To control for this variable two levels have been selected, high and low. This follows the experimental design used for the soil sampling technologies. High concentrations would be represented by soil contaminant concentrations of 10s to 100s of ppm. These concentrations have been selected to approximate soil gas concentrations indicative of source locations. Low concentrations will be represented by soil contamination in the range of 50 to 100 ppb. These concentrations can be associated with boundary concentration for delineating the extent of groundwater or soil contamination.

Based on this distribution of factors, each of the technologies (and the conventional method) would have the following combinations of test conditions:

Soil Gas Technologies	**ARA DTD Probe** (Hypotheses S2 and S3)	
Soil Type	Soil Type	Concentration
Sand	Sand	Low High
Clay	Clay	Low High

Observations will be obtained of each of these factor combinations for each of the technologies and the conventional method, which will result in a total of two combinations of tests for each soil gas technologies for the two null hypotheses and four combinations of tests for the ARA DTD probe relative to soil sampler hypotheses S2 and S3.

The number of samples to be obtained for each test condition is dictated by the desired statistical probability of performing a Type I error (that is, the level of statistical significance, α), the desired probability of avoiding a Type II error (that is, the power of the test, β), and the acceptable error (δ^2). Previous analysis of the variance in concentration that is exhibited by sampling in the field indicates that concentration heterogeneity or sampling heterogeneity exhibits an SD that is about 100 percent of the mean sample concentration for a wide range of concentrations. To conduct tests of the statistical hypotheses stated in Section 6.2.1 comparing an innovative method with the conventional method, at a level of significance of α=0.05, and a power of β=0.95 when the error (δ^2) is within 50 percent of the estimated standard deviation, a minimum of seven samples (points of comparison) are required for each of the technologies and the conventional method.

This represents the number of observations that must be obtained for each technology for each combination of factors. Because of the factorial design, fewer samples are required in each test condition to satisfy the number of samples.

Major factors that are not tested at various levels include: contaminant concentration heterogeneity, operator skill, surface sample handling procedures, and temperature. Contaminant concentration

heterogeneity is being handled by repeat sampling within the site location where sample locations will be assigned randomly. Each cell within the grid is to be 18 inches square. For evaluation of ARA's DTD probe for soil null hypotheses S2 and S3, predemonstration sampling has roughly defined the heterogeneity of soil contamination at a given location and depth (Section 6.1.3). For the soil gas sampler evaluation, a minimum of two locations will be sampled for each combination of factors. This will provide backup data in the event a given grid produces data with excessive variability, a factor of 10 for soil gas measurements. The soil gas technologies will be evaluated in the same grids that are used to evaluated the soil sampling technologies. The soil gas samples will be collected within 1 week of the start of soil sampling at a given demonstration site. The ARA DTD Probe will conduct sampling during the soil sampling phase of the demonstration. The assignment of which cells are to be sampled will be random. In this manner, each technology will be randomly sampling the normally distributed population within the sampling interval. The two soil gas technologies will be sampling in cells which will be used by soil sampling technologies. These two technologies will sample at the outer edges of their assigned sample cells so that when they are later used by the soil sampling technologies or the conventional soil gas or ARA DTD Probe their samples will not disturb the soil samplers. The soil gas sampling technologies will be collecting a single sample no deeper than 3 feet below grade in each cell. The maximum diameter of the soil gas technologies is 1-inch. With this in mind, their use on the edge of a potential soil sampling cell will not measurably impact the performance of a soil sampling technology, the conventional soil gas, or the ARA DTD Probe collecting a sample in the same cell.

Operator skill can exert an influence on several performance criteria. Rather than test for this variable, it has been decided that the technologies will be implanted and analyzed by experienced personnel selected by the technology developer. This choice limits application of results concerning performance only to experienced operators but this is considered appropriate.

Sample handling and analysis procedures vary between technologies. These are considered part of the overall technology and are therefore tested by the technology factor. Since experienced operators will be performing sample deployment and sample analysis it is also assumed that the influence of this factor is controlled.

During the sampling activities producing the data which will be used to evaluate the quantitative variable, all of the qualitative variables will be assessed and recorded.

6.3 SAMPLING PLAN

This section will overview the sample collection activities and discuss the results of the predemonstration sampling.

6.3.1 Sampling Operations

The implementation of the demonstration is divided into two sections. The first section deals with the soil sampling technologies; the second section deals with the soil gas sampling technologies.

6.3.1.1 Soil Sampling

As noted in Section 6.1 there are a minimum of eight target depths at which soil sampling will be conducted in the demonstration. Eight target depths provide two target depths for each primary factor combination. Predemonstration sampling has identified an additional 2 grids at each site that will be sampled if time allows. The experimental design requires only one target depth for each combination. It is possible that the subsurface may exhibit heterogeneities that were not revealed by the predemonstration sampling. These heterogeneities could result in different performance measures that are unrelated to the technologies being compared. Increasing the number of target sampling depths will give PRC the option of eliminating grids that exhibit excessive heterogeneity, without compromising the demonstration. Another way to help insure against the possible influence of subsurface heterogeneity is to divide the grid into 7 rows and 7 columns. The intersection of a row and column will produce a cell 18 inches on a side (Figure 6-1). The cells in each row will be assigned numbers from 1 to 7. The cell columns will be labeled A to G. Random numbers will be assigned to each technology. The technology with the highest random number will use cell 1, the second highest cell 2, and so forth. This procedure will be repeated anew for each column so that each technology will be assigned one cell in each column. This stratified random sampling should provide a statistically valid sample of the subsurface for each technology.

These target depths have been sampled and identified during the predemonstration. More than one target depth will be located in some of the grids. The predemonstration sampling has shown that each target depth has acceptable heterogeneity (based on the demonstration criteria) in the desired combination of

factors being tested. All of the target sampling depths will be contained within 10.5- x 10.5-foot square grids which have been subdivided into 49 cells. Predemonstration sampling was conducted at the center and outer four corners of each grid area.

A total of seven samples will be collected by each technology at each of the target depths. Since there are a minimum of eight target sampling depths with unique combinations of factors the total number of samples collected and analyzed for each technology will be at least 8 x 7 or 56. If the additional grids are sampled (2 at the CSC site will be sampled), this increases the number of samples to at least 70. For the four soil technologies and the standard method, the total minimum number of samples analyzed during the demonstration will be 70 x 5 or 350. These samples will be analyzed for the concentrations of the target VOCs. This data will be statistically analyzed as discussed Chapter 5. Grid Nos. 1, 2,3, 4, and 5 will be used at the SBA site and Grid Nos. 1, 2, 3, and 4 will be used at the CSC site.

The null hypothesis dealing with the preservation of sample integrity will be assessed by advancing a sampler filled with an uncontaminated loam soil through a zone of grossly contaminated soil. This zone will be identified in one grid at each site. It will be a minimum of 2 feet thick and exhibit volatile organics contamination in the 10's to 100's of ppm, at a minimum. This evaluation will be conducted at Grid No. 1 at each site. Prior to this test, a sampler will be decontaminated and filled with a pre-analyzed loam soil, which did not exhibit any detectable volatile organics contamination. At Grid No. 1 at the SBA site, the sampler filled with the loam soil will be advanced down the sampled boreholes at a given cell. This sampler will be advanced 4 feet past the depth of the last demonstration sample in the cell. At the CSC site, the sampler filled with the loam soil will be advanced from 10 to 14 feet below grade. After the sampler has penetrated the contaminated zone, it will be withdrawn and the loam soil sampled for analysis. This integrity testing will be conducted in each cell at Grid No. 1 at both sites.

Besides collecting samples for chemical analysis and assessment of the preservation of sample integrity, data will be obtained to evaluate: (1) the proportion of attempts that the technology successfully reached a specified sampling depth, (2) the proportion of sampler volume that was filled with sample, (3) the average sample retrieval time, and (4) the average sample retrieval cost. This data will be obtained through direct observation during the demonstration and through data supplied by the developers.

For measurements of the performance criteria of the proportion of successful attempts, an attempt will be considered as having begun with the technology breaking the surface. An attempt will be considered unsuccessful if the technology either fails to reach the specified depth during the advancement or surface measurements indicate that the technology did not correctly reach the specified depth. The designation of the type of surface measurement used to gauge penetration depth will be agreed upon with the developer prior to the demonstration. If a technology experiences a failed attempt, it will be allowed to repeat the attempt within the same cell so that a representative sample for analysis can be obtained. This repeat sampling will only be conducted when all the surrounding cells have been sampled. All attempts, including repeat attempts, will be considered in the statistics of proportion of attempts. The proportion of successful attempts based on all attempts will be reported and compared to the other technologies.

The proportion of a sampler that was filled with sample will be measured by the sample packaging team. The length of sample retrieved will be expressed as a percentage of the total length the sampler was advanced in an open mode. The proportion of the sampler filled will be recorded for all sampling events, including repeat sampling events. Data for all sampling events, including repeat sampling events will be considered in the statistics. The average, minimum, maximum, and SD of the proportion of the sampler filled will be reported and compared to the other technologies.

To augment the evaluation of successful sampling attempts and the percent recovery Grid No. 5 at the CSC site will be used. AMS, Geoprobe®, Simulprobe®, and the conventional technology will collect samples in the saturated sands from 35- to 40-foot bgs. If a developer desires to use a continuous sampling approach for this test, it will be allowed. This test will be repeated for each of the sampling systems.

Sample retrieval time will be considered to begin when the technology breaks the ground surface and to end when the sample is delivered to the sample packaging team, the hole has been grouted, and the sampler has been decontaminated. Times for all successful attempts, including repeat attempts, will be recorded and will be considered in the statistics. All attempts that result in a sample being collected for analysis will be considered successful attempts. The average, minimum, maximum, and SD of the time for sample retrieval will be reported.

Sample costs will be computed based on criteria supplied by the developer and documented to represent widely quoted commercial costs. These cost estimates will be based on mobilization and demobilization estimates, equipment and manpower time, and required disposable supplies. Average, minimum and maximum sample costs will be reported.

The following is a list of the sampling sequence to be followed at each grid:

1. One technology will be assigned a grid for sampling. Only one technology will be allowed to sample at a grid at any given time. Technologies will be assigned to morning or afternoon groups. Technologies in the morning group will have access to grids between 0730 and 1230. Technologies in the afternoon group will have access to grids between 1300 and 1800. Once a technology begins to sample a grid, it cannot move to another grid until all its sampling has been completed at that grid.

2. When a technology retrieves a soil sample, the technology operator will: (1) cap the ends of the sampler, (2) mark the location where the sample should be collected, and the intended length of the sample (3), mark the cell column and row on the sampler, (4) indicate what technology collected the sampler, and (5) deliver the sample to the sample packaging team.

3. When a technology has collected all its samples at a cell, the technology operator will grout the open holes and decontaminate the sampling equipment, as appropriate given their standard operational procedures.

Specific target sampling depths and technology sampling assignments are shown in Appendix B.

6.3.1.2 Soil Gas Demonstration Sampling

As noted in Section 6.2, the experimental design requires a minimum of two unique sampling grids, one grid for each combination of primary demonstration factors. To provide a safety factor similar to the one discussed above for the soil sampling technologies demonstration, Grid Nos. 1, 2, 4, 5, and 6 will be used at the SBA site, and Grid Nos. 1, 2, 4, and 5 will be used at the CSC site.

It is possible that the soil gas sampling may reveal heterogeneities that were not revealed by the predemonstration sampling. These heterogeneities could result in different performance measures that are unrelated to the technologies being compared. Increasing the number of soil gas sampling grids will

help eliminate the impact of such heterogeneities on the determinative statistics. Another way to help insure against the possible influence of subsurface heterogeneity, is to divide each grid into seven rows and seven columns. The intersection of a row and column will produce a cell approximately 18 inches on a side. The grid rows will be assigned numbers from 1 to 7 and the columns will be assigned letters A to G. Random numbers will be assigned to each technology. The technology with the highest random number will use cell 1, the second highest cell 2, and so forth. This procedure will be repeated anew after each technology is assigned a grid square in a column. This stratified random sampling should provide a statistically valid sample of the soil gas at a given location, for each technology.

A total of seven cells will be sampled by each technology at each sampling grids. Since there are a total of nine soil sampling grids the total number of soil gas samples collected and analyzed for each technology will be 7 x 9 or 63. Combining the analyses of the two passive soil gas technologies, the ARA DTD probe, and the conventional method, the total number of soil gas samples analyzed during the demonstration will be 63 x 4 or 252. These samples will be analyzed for the concentration of the target volatile compounds or according to developer's requirements. Each passive soil gas developer uses proprietary methods for analyzing its sampler, and the ARA DTD probe also produces its own data; therefore, the on-site laboratory will only analyze the 63 active soil gas samples collected by the conventional method. This data will be statistically analyzed as discussed Chapter 5.

Besides collecting samples for chemical analysis during the retrieval of the 63 samples by each technology, data will be obtained to assess: (1) the proportion of attempts that the technology successfully collected a usable sample, (2) the average sample retrieval time, and (3) the average sample retrieval and analysis cost.

For measurements of the performance criteria of the proportion of successful attempts, an attempt will be considered as having begun with the technology breaking the surface. An attempt will be considered unsuccessful if the technology either fails to reach the specified depth during the advance or insufficient sample was collected to allow analysis. Any sampler failures for the passive soil gas technologies will not be allowed to resample. This is due to the time associated with their installation and collection and since the soil sampling will have started. If the ARA DTD probe experiences a failed attempt, it will be allowed to repeat the attempt within the same cell so that a representative sample for analysis can be obtained. This will only be allowed after all adjacent cells have been sampled. All attempts, including

repeat attempts, will be considered in the statistics of proportion of attempts. The proportion of successful attempts based on all attempts will be reported and compared to the other technologies.

Sample retrieval time will be considered to begin when the technology breaks the ground surface and to end when the sample is delivered for analysis. Times for all successful attempts, including repeat attempts, will be recorded and will be considered in the statistics. All attempts that result in a sample being placed in the cooler will be considered successful attempts. Once the sample has been collected, the time required to deliver a analytical result will be recorded. These results will be considered final results and must be submitted in a paper and electronic format. The average, minimum, maximum, and SD of the time for sample retrieval and sample analysis will be reported and compared to the other technologies.

Sample costs will be computed based on criteria supplied by the developer and documented to represent widely quoted commercial costs. These cost estimates should be based on a equipment and manpower time and materials basis so that variations in time and materials will result in variations in per sample cost. Average, minimum, maximum, and SD of per sample cost will be reported and compared to the other technologies.

The soil gas sampling technologies will be installed and collected between May 21 and May 31, 1997. The conventional method sampling and ARA DTD probe sampling will be conducted in concert with the soil sampling technologies.

A geologic cross-section to a depth of 28 feet for the CSC site is shown on Figure 6-2. This cross-section was generated from continuous soil cores collected at the center of five grids, which are shown on Figure 4-1. The soils at the CSC site are much more heterogeneous than at the SBA site. In general, the top 0.5 to 1.5 feet is dry, gravelly, coarse sand. Grid 1 has about 4 inches of asphalt at the surface. Except at Grid 4, the soil material underlying the gravelly coarse sand was a moist, fine to medium, sandy loam or loamy fine to medium sand. The sandy loam was a dark brown color, whereas the loamy sand was more of a reddish-brown color. The loamy sand was cohesive. In general, this sandy loam or loamy sand got slightly coarser-grained with depth and extended to a depth of 7.5 to 11 feet bgs. At Grids 3, 4, and 5,

there was a 1- to 3-foot thick, slightly moist, dark brown silt loam layer found at a depth between 1 and 5 feet bgs.

Below the sandy loam or loamy sand, there was an abrupt textural change to finer-grained materials which lie between 7.5 and 19 feet bgs. These finer-grained materials were predominantly slightly moist, dark brown to reddish brown, silts and clays with some zones of sandy clays. The predominant soil textures in this zone were silt loams, silty clay loams, silty clays, and sandy clays. The silty clays and sandy clays were stiff and hard and were only slightly moist. The silt loams and silty clay loams were more moist and less stiff. At grid 5 there is a 2-foot interval of moist, sandy loam at 12 to 14 feet bgs.

From 18 feet to 28 feet, the lithology was dominated by moist, sandy soils. Again, there was an abrupt textural change from the finer-grained materials above these sandy soils. As can be seen on Figure 6-2, these sandy soils vary from sandy loams and fine loamy sands at shallower depths to medium- to coarse-grained sands at a depth of 26 feet bgs. In general, these sands were coarser-grained than those found in the upper 8 feet of the soil profile. These sands were generally light brown to light gray in color, noncohesive and some contain significant amounts of gravel. Below about 25 to 26 feet bgs, more fines were found in the soil cores and the texture graded back to loamy sands or sandy loams. These soils in the bottom 2 feet of the soil profile were darker brown in color and were more cohesive than the sands above them.

The lithologic description of soils at the SBA site is based on continuous cores collected to a depth of 16 or 20 feet at the center of the six grids shown on Figure 4-2. The lithology at the SBA site was fairly consistent across the site. The top 2 to 4 feet of soil was a moist, black to dark gray silty clay loam. This silty clay loam usually had increasing clay content with depth and the color became lighter with depth. This top layer tended to be thicker (4 feet) in the vicinity of grid 6 and got shallower toward the south in the vicinity of grids 2,3, and 4. Under the silty clay loam was a tan, moist, silty clay material that extended down to 6 to 8 feet bgs. In highly contaminated areas, such as at grids 1 and 2, this silty clay material was gray probably due to the anaerobic conditions. This silty clay material also was heavily mottled (orange and brown mottles) due to the fluctuating water table found at a depth of 6 to 7 feet bgs. From 8 to 20 feet, the site was underlain by a moist to very moist, stiff, mottled (orange and gray), tan, sandy clay to clay till that contained approximately 10 percent coarse fragments. The coarse fragments

were generally angular and small (less than ½-inch in diameter). Even though the clay till was below the water table, the soil cores were moist but not obviously saturated.

Ten distinct target depths were sampled at five grids at the SBA site; three depths at Grid 1, two depths at Grid 2, one depth at Grid 3, two depths at Grid 4, and two depths at Grid 5. Five of the target depths were high concentration intervals (tens of ppm) and five of the target depths were low concentration intervals (tens of ppb). As expected the primary VOCs found in the soil samples were vinyl chloride, c-1,2-DCE, TCE and PCE. VOCs c-1,2-DCE and TCE were detected at the highest concentrations. Vinyl chloride was found only at the 3-foot interval at Grid 1. PRC used the c-1,2-DCE and TCE concentration variability within a grid to determine the target soil sample locations for the demonstration. PRC initially looked for all concentrations to be within a factor of 5 and a %RSD to be less than 50 percent to be an acceptable target depth location. The factor and %RSD values for all target depths sampled at the SBA site during the predemonstration are shown in Table 6-1.

The two primary target depths chosen for high concentrations are depths of 9.5 and 13.5 feet at Grid 1. If time permits, a third target depth will be sampled at 9.5 feet at Grid 3. These target depths exhibited c-1,2-DCE and TCE concentrations in the tens to hundreds of ppm. The 9.5-foot depth at Grid 3 exhibited a factor of 8.85 for TCE; however, one corner sample appeared to be an outlier with a TCE concentration of 5 to 8 times greater than the other four samples. Removing this sample produced a factor of 2.95 and an RSD of 49.67 percent for TCE. The target depths chosen for the low concentration samples (tens of ppb) are 3.5 feet at Grid 2, 13.5 feet at Grid 5, and if time permits, 9.5 feet at Grid 4. The factor and %RSD for c-1,2-DCE at 13.5 feet at Grid 5 appear large; however, the removal of one data point that had a concentration ten times higher than the other four resulted in a factor of 1.50 and a RSD of 16.5 percent.

Twelve distinct target depths were sampled at five grids at the CSC site; two depths at Grid 1, three depths at Grid 2, three depth at Grid 3, two depths at Grid 4, and two depths at Grid 5. Two of the target depths were high concentration intervals (hundreds of ppb to low ppm) and ten of the target depths were low concentration intervals (tens of ppb). The primary VOCs detected at the CSC site were 1,1,1-TCA, TCE, and PCE. PCE was the only VOC found at ppm levels in the sandy intervals.

The target depths were chosen based primarily on texture and to a lesser extent on contaminant concentration. None of the silts or clays were sampled during the target depth sampling (four corners). It was difficult to find contaminant concentrations at ppm levels in the sandy soils. PRC primarily targeted a shallow depth (about 3 feet) for the higher concentrations in the sandy loam; a depth of 5.5 to 7.5 feet bgs for lower contaminant concentrations in the fine sand, and then a third depth below 18 feet for lower contaminant concentrations in the medium to coarse sands.

The factors and %RSDs for all VOCs at all target depths at the CSC site are shown in Table 6-2. The two high concentration intervals (3 feet at Grids 2 and 3) showed acceptable factors of less than 5 for all VOCs and RSDs less than 56 percent. Although not sampled at the corners during the predemonstration, PRC proposes to sample at 3 feet at Grid 1 as a backup target depth for the high concentration zone. In choosing the low concentration target depths, it was discovered that the medium to coarse sands found below 18 feet had the greatest contaminant concentration variability and had contaminant concentrations less than 10 ppb and, thus, were not suitable for the demonstration. The three target depths chosen for the low concentration samples are 7.5 feet at Grid 3, 6.5 feet at Grid 4, and 6.5 feet at Grid 1. Again PRC is proposing a third target depth in case contaminant variability is too great at one of the locations. The factor for PCE at 6.5 feet at Grid 4 does not appear acceptable; however, the removal of one data point which had a concentration 7 to 8 times less than all other concentrations resulted in a factor of 1.65 and a RSD of 20.4 percent.

6.3.2 Predemonstration Sampling and Analysis

A predemonstration sampling and analysis event was conducted as part of this demonstration. The objectives of predemonstration sampling were to establish the geographic location of the sampling grids, identify soil sampling target sampling depth(s), and to roughly determine the heterogeneity of concentration that is exhibited at each grid location and target sampling depth. This sampling has also confirmed that the proposed sites provide the appropriate combination of factors that are being tested.

Predemonstration sampling was conducted at the SBA site between April 1 and 11, 1997. Predemonstration sampling was conducted at the CSC site between April 20 and 25, 1997. A total of 11 sampling grids were investigated, 6 at the SBA site and 5 at the CSC site. The analytical data for the sampling at these grids is shown on Tables 6-1 and 6-2. Of these 11 grids, all will be used during the

demonstration. Tables 6-1 and 6-2, and Appendix B identify the demonstration sampling grids and the target sampling depths for the soil samplers.

At each of the grids sampled during the predemonstration, a single continuous core was collected at the center of the 10.5-foot-square sampling area (Table 6-1). Analysis of samples from this core confirmed that the site exhibited the desired combination of factors over a vertical interval of not less than 1 foot. After this location had been sampled, four additional boreholes were advanced and sampled in each of the outer four corners of the 10.5-foot by 10.5-foot grid area. These corner locations were sampled at the depth interval of interest identified by the initial sample location in the center of the grid (Table 6-2). These samples were analyzed for the target analytes and for the physical properties of interest (such as soil type and moisture level). The samples from the surrounding locations exhibited an acceptably limited variance in the properties of interest such as soil moisture, soil texture, and contaminant concentration.

Adjacent to each of the soil sampling grids an conventional method sample was collected. Analysis of samples from these locations confirmed that active soil gas methods could be used at the site and that soil gas contamination was detectable by the conventional method.

6.4 Field Data Collection

One PRC observer will be assigned to a minimum of two technologies. This person will record observations on the technology operation, and collect data to evaluate the technology's performance.

Each technology developer will be responsible for the operation of its technology. Operation of a soil sampling technology involves advancing to the proper sampling depth, retrieving the filled sampler, presenting the sampler to the PRC sample packaging team for decontamination of the technology after each use, and for properly grouting the holes. The soil gas developers will implant, collect, and analyze their samplers. The ARA DTD operator will also analyze the samples collected by the ARA technology. If possible, only one PRC observer will be used for both technologies.

6.5 Demonstration Schedule

The demonstration schedule is detailed in Table 6-3. An overall project schedule is shown on Table 6-4. Once the demonstration starts, it will continue without interruption until all sampling is completed. Based on this schedule, including travel between sites, the demonstration sampling should be completed by June 14, 1997.

TABLE 6-1

SAMPLING GRID VARIABILITY AT TARGET DEPTHS
SBA Site, Albert City, Iowa

	Vinyl Chloride	1,2-DCE	TCE	PCE
Grid 1—Depth of 3 feet (High Concentration Interval)				
%RSD	97.99	108.46	ND	ND
Factor	16.19	518.21	ND	ND
***Grid 1—Depth of 9.5 feet (High Concentration Interval)**				
%RSD	ND	7.80	57.74	106.10
Factor	ND	1.17	4.46	58.51
***Grid 1—Depth of 13.5 feet (High Concentration Interval)**				
%RSD	ND	85.58	54.26	ND
Factor	ND	12.29	2.86	ND
***Grid 2—Depth of 3.5 feet (Low Concentration Interval)**				
%RSD	ND	ND	21.28	ND
Factor	ND	ND	1.71	ND
Grid 2—Depth of 8.5 feet (High Concentration Interval)				
%RSD	ND	ND	53.10	113.27
Factor	ND	ND	4.32	6.99
****Grid 3—Depth of 9.5 feet (High Concentration Interval)**				
%RSD	ND	28.95	103.93	ND
Factor	ND	2.17	8.85	ND
Grid 4—Depth of 3.0 feet (Low Concentration Interval)				
%RSD	ND	ND	85.48	ND
Factor	ND	ND	5.45	ND
****Grid 4—Depth of 9.5 feet (Low Concentration Interval**				
%RSD	ND	74.06	72.08	ND
Factor	ND	4.79	3.70	ND

TABLE 6-1 (Continued)

SAMPLING GRID VARIABILITY AT TARGET DEPTHS
SBA Site, Albert City, Iowa

	Vinyl Chloride	1,2-DCE	TCE	PCE
Grid 5—Depth of 4.0 feet (Low Concentration Interval)				
%RSD	ND	137.72	102.83	ND
Factor	ND	292.59	32.20	ND
***Grid 5—Depth of 13.5 feet (Low Concentration Interval)**				
%RSD	ND	130.96	42.04	ND
Factor	ND	10.28	2.93	ND

Notes:

* Primary target soil sampling depth for demonstration.
** Secondary target soil sampling depth for demonstration, if time permits.

%RSD Percent relative standard deviation
1,2-DCE 1,2-dichloroethene
ND No data
TCE Trichloroethene
PCE Tetrachloroethene

TABLE 6-2

SAMPLING GRID VARIABILITY AT TARGET DEPTHS
CSC Site, Denver, Colorado

	1,2-DCE	1,1,1-TCA	TCE	PCE
***Grid 1—Depth of 6.5 feet (Low Concentration Interval)**				
%RSD	50.95	61.46	45.96	45.93
Factor	11.50	5.73	6.33	3.01
Grid 1—Depth of 19.5 feet (Low Concentration Interval)				
%RSD	ND	ND	ND	196.21
Factor	ND	ND	ND	57.21
***Grid 2—Depth of 3.0 feet (High Concentration Interval)**				
%RSD	ND	11.71	ND	55.87
Factor	ND	1.26	ND	4.31
Grid 2—Depth of 6.5 feet (Low Concentration Interval)				
%RSD	32.78	40.53	39.62	64.41
Factor	1.97	2.78	2.55	4.92
Grid 2—Depth of 20.5 feet (Low Concentration Interval)				
%RSD	65.51	83.95	87.53	73.02
Factor	5.55	16.49	12.58	5.74
***Grid 3—Depth of 3.0 feet (High Concentration Interval)**				
%RSD	ND	29.75	31.14	20.42
Factor	ND	1.97	2.10	1.61
***Grid 3—Depth of 7.5 feet (Low Concentration Interval)**				
%RSD	25.28	69.80	46.12	35.69
Factor	2.15	5.38	3.17	2.83
Grid 3—Depth of 22.5 feet (Low Concentration Interval				
%RSD	106.56	75.18	57.91	63.46
Factor	16.43	5.50	5.80	5.66

TABLE 6-2 (Continued)

SAMPLING GRID VARIABILITY AT TARGET DEPTHS
CSC Site, Denver, Colorado

	1,2-DCE	1,1,1-TCA	TCE	PCE
***Grid 4—Depth of 6.5 feet (Low Concentration Interval)**				
%RSD	14.71	18.50	17.51	50.46
Factor	1.39	1.47	1.50	8.23
Grid 4—Depth of 20.5 feet (Low Concentration Interval)				
%RSD	17.33	18.58	24.99	29.23
Factor	1.42	1.58	1.80	2.04
Grid 5—Depth of 5.5 (Low Concentration Interval)				
%RSD	ND	52.57	16.25	79.95
Factor	ND	2.81	1.45	8.47
Grid 5—Depth of 16.5 feet (Low Concentration Interval)				
%RSD	49.25	48.62	29.86	64.71
Factor	4.84	2.97	1.99	5.76

Notes:

* Primary target soil sampling depth for demonstration.

%RSD Percent relative standard deviation
1,1,1-TCA 1,1,1-trichloroethane
1,2-DCE 1,2-dichloroethene
ND No data
TCE Trichloroethene
PCE Tetrachloroethene

TABLE 6-3

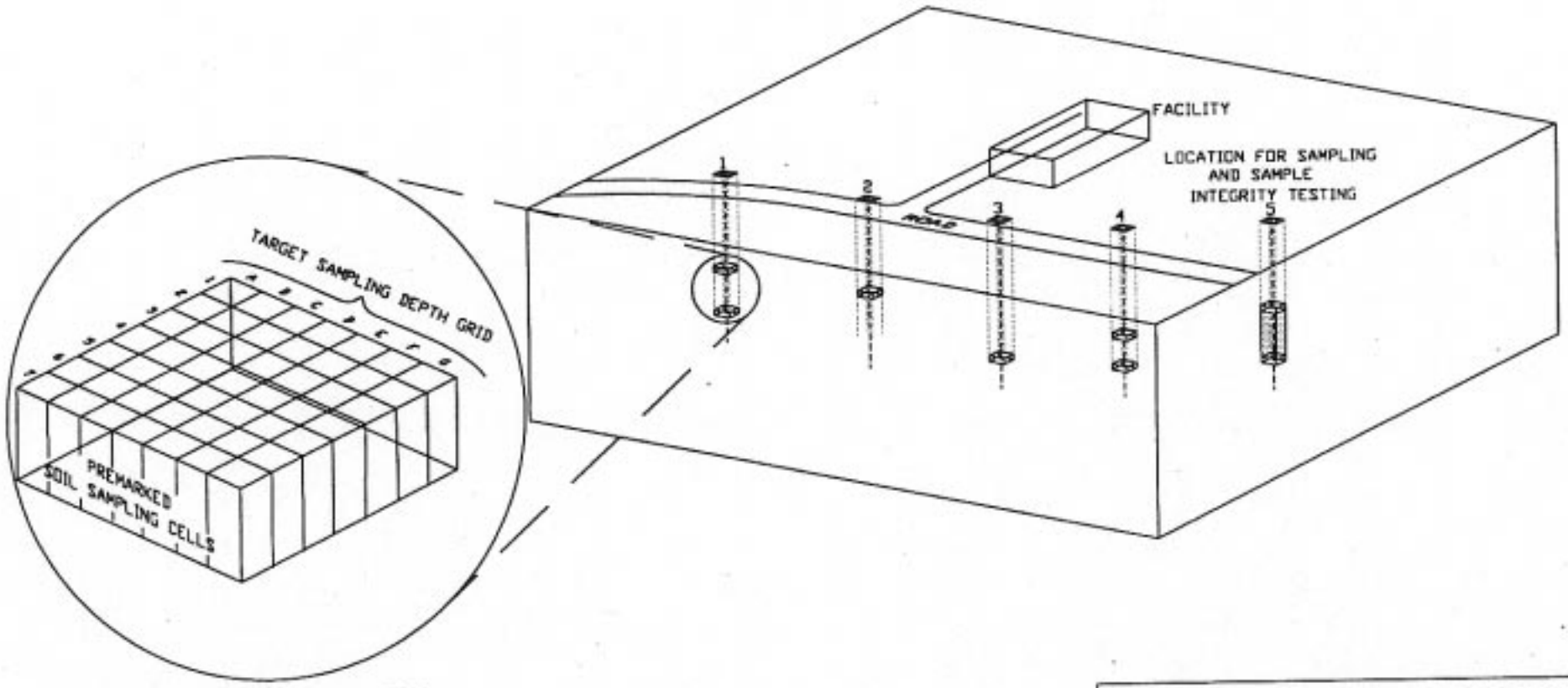
1997 FIELD SCHEDULE
SOIL AND SOIL GAS SAMPLING DEMONSTRATION

Date	Activity
May 21	W.L. Gore implants its sampler at the SBA site.
May 22	Quadrel implants its sampler at the SBA site.
May 23	W.L. Gore and Quadrel implant their samplers at the CSC site.
May 25	Quadrel removes its sampler from the SBA site.
May 26	Quadrel removes its sampler from the CSC site.
May 31	W.L. Gore removes its sampler from the SBA site. PRC mobilizes the on-site laboratory to the SBA site.
June 1	W.L. Gore removes its sampler from the CSC site. PRC mobilizes the remainder of its demonstration team to the SBA site. PRC conducts an all-participant health and safety briefing at the Sail Inn at 2000 hours.
June 2 and 3	Morning and afternoon technologies collect demonstration data at the SBA site.
June 4	Technologies finish any remaining demonstration data collection. Conventional method soil and soil gas sampling begins in the afternoon. Either Geoprobe® or AMS will conduct conventional method soil gas sampling.
June 5 and 6	Conventional sampling.
June 7 and 8	Mobilize to the CSC site.
June 9 and 10	Morning and afternoon technologies collect demonstration data at the CSC site.
June 11	Technologies finish any remaining demonstration data collection. Conventional method soil and soil gas sampling begins in the afternoon. Either Geoprobe® or AMS will conduct conventional method soil gas sampling.
June 12 and 13	Conventional sampling.
June 14	Field portion of the demonstration complete.

TABLE 6-4

DEMONSTRATION SCHEDULE
SOIL AND SOIL GAS SAMPLING

Action	Planned	Completed
Developers' Conference–developer describes technology and learns about the demonstration process	November 13, 1996	November 13, 1996
Review proposals and notify developer of decision	November 20, 1996	November 20, 1996
Receive letters of intent from the developers	December 30, 1996	December 2, 1996
Identify appropriate sites for demonstration	February 17, 1997	February 21, 1997
Select conventional methods	January 31, 1997	January 30, 1997
Distribute a draft demonstration plan based on the "Guidance Manual for the Preparation of Characterization and Monitoring Technology Demonstration Plans"	March 10, 1997	March 10, 1997
Receive comments on the draft demonstration plan from all participants, including external reviewers	April 10, 1997	April 18, 1997
Conduct predemonstration activities–critical element in the final selection of technologies and sites. Final test of conventional methods performance and demonstration plan.	April 11, 1997	April 21, 1997
Receive data from predemonstration activities.	April 25, 1997	April 25, 1997
Conference call with developers	April 15, 1997	April 15, 1997
Revise and approve final demonstration plan–need concurrence from all participants	May 9, 1997	
Conduct field demonstration–three to four days at each site, including one visitors day.	May 31, 1997	
Complete audit reports/receive reference laboratory data/report preliminary findings	June 30, 1997	
Conference call with developers	July 2, 1997	
Complete draft ITERs–Concurrent EPA review	August 8, 1997	
Conference call with developers	August 22, 1997	
Complete developer review	August 29, 1997	
Complete external peer review	September 25, 1997	
Issue final reports with verification statements	October 22, 1997	



STRATIFIED RANDOM SAMPLING GRID

LEGEND

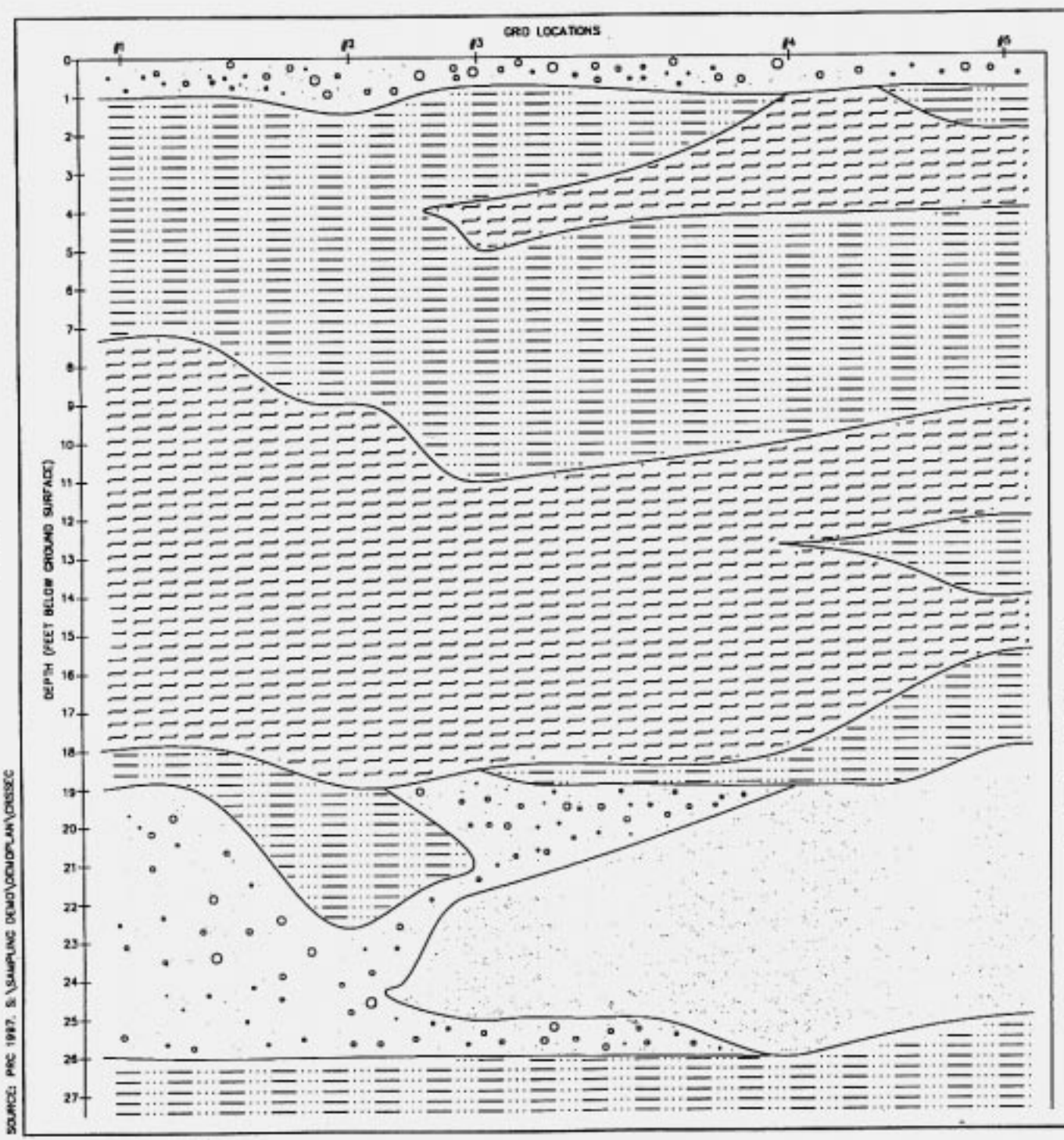
- SAMPLE INTEGRITY TEST ZONE
- 1-7 = ROW IDENTIFIERS
- A-G = COLUMN HEADERS
- 1 UNIQUE SAMPLING GRID
- TARGETED DEPTH INTERVAL

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 6-1
TYPICAL SAMPLING LOCATIONS
AND STRATIFIED RANDOM SAMPLING GRID

PRC ENVIRONMENTAL MANAGEMENT, INC.

SOURCE: PRC MODIFIED 1997. S:\SAMPLING_DEMO\DESIGN\ANY\FIG-1



HORIZONTAL SCALE:

0 50 100

FEET

VERTICAL EXAGGERATION 13X

LEGEND

- SILT AND CLAYS DOMINANT (SILT LOAM, SILTY CLAY, SANDY CLAY)
- SANDY LOAMS AND LOAMY SANDS
- FINE TO MEDIUM SANDS
- MEDIUM TO COARSE SANDS
- #1 GRID LOCATION AND NUMBER

SOIL AND SOIL GAS SAMPLING DEMONSTRATION
SITE DEMONSTRATION

FIGURE 6-2
GEOLOGIC CROSS SECTION
CHEMICAL SALES COMPANY
DENVER, COLORADO

PRC ENVIRONMENTAL MANAGEMENT, INC.

SOURCE: PRC 1997, S:\SAMPLING DEMO\DEMOPLAN\CRSSEC

CHAPTER 7
SAMPLING AND ANALYSIS

The sampling and analysis plan for this demonstration specifies procedures that will be used to ensure the consistency and integrity of samples, and control within sample variation. In addition, this plan outlines the sample collection procedures necessary to meet the demonstration purpose and objectives. Careful adherence to these procedures will ensure that samples analyzed using the technologies to be evaluated are comparable to samples analyzed by the on-site laboratory using standard analytical methods.

7.1 COMMUNICATION AND DOCUMENTATION

PRC will communicate regularly with the demonstration participants to coordinate all field activities associated with this demonstration and to resolve any logistical, technical, or QA issues that may arise as the demonstration progresses. The successful implementation of the demonstration will require detailed coordination and constant communication between all demonstration participants.

All field activities will be thoroughly documented. Field documentation will include field logbooks, photographs, field data sheets, and chain-of-custody forms. Data reporting forms are discussed in Chapter 8. The field team leader will be responsible for maintaining all field documentation. Field notes will be kept in a bound logbook. Each page will be sequentially numbered and labeled with the project name and number. Completed pages will be signed and dated by the individual responsible for the entries. Errors will have one line drawn through them and this line will be initialed and dated.

All photographs will be logged in the field logbook. These entries will include the time, date, direction, subject of the photograph, and the identity of the photographer. Specific notes about each sample collected will be written on sample field sheets, as well as in the field logbook. Any deviations from the approved final demonstration plan will be thoroughly documented in the field logbook and communicated to the EPA technical project manager (TPM) and any other parties that may be affected by the change.

Original field sheets and chain-of-custody forms will accompany all samples that are shipped to the reference laboratory. Copies of field sheets and chain-of-custody forms for all samples will be maintained in the project file.

7.2 SAMPLE COLLECTION PROCEDURES

Sampling personnel will collect and handle samples using the procedures described below. This section describes the procedures for collecting soil and soil gas samples during this demonstration. All field activities will conform with requirements of the HASP and with all requirements in this demonstration plan.

Sampling personnel will maintain communication with the PRC field team leader and the site contacts during field activities. If unanticipated or unusual situations are encountered that may alter the sampling design, sampling location, or data quality, the situation will be discussed with the PRC field team leader, the PRC project manager, the EPA TPM, the developers, and the site contacts before changes to the approved demonstration plan are made. Any deviations from the approved demonstration plan will be thoroughly documented.

Bar coding will be used to track samples. The bar code sample labels will be preprinted for each technology and reflect the grids, cells, and target depths they will be sampling. The bar codes will be designed as follows: AMS-AGR1-A6-B. This example sample number, which will be printed along with a corresponding scanable bar code, indicates that the sample is for the AMS technology, the sample was collected at the SBA site grid No. 1, in cell A6, at the second (deepest) target sampling depth.

7.2.1 Soil Sampling Procedures

Soil samples for this demonstration will be collected either with the technologies being demonstrated or with the conventional method for subsurface soil sampling. The technologies will be operated by the developers and according to their standard operating procedures. The standard soil sampling method will involve the collection of discrete depth interval samples using an auger drill rig, 3.25-inch inside diameter hollow stem augers, and an 18-inch long by 2-inch diameter, acetate lined, split spoon sampler.

The collection of the split spoon samples will follow PRC SOPs. Copies of the SOPs are available upon request.

The purpose of both the technology sampling and the conventional method sampling is to bring a subsurface soil core to the surface for sampling. The method of this sampling or subsampling has been designed to minimize the impact of subsampling and sample packaging. This is necessary to eliminate as much as possible, potential influence on observed performance due to nontechnology factors. These methods of soil core subsampling are described below.

7.2.1.1 Purgeable Volatile Organics Sampling

Soil samples for each soil sampling technology, including the conventional method, will be collected using the same procedure. Once the filled soil sampler is returned to the soil surface the sampler will be delivered to the sample collection team. The sample collection team will then expose the soil core and collect a subsample for on-site analysis in one of two ways: (1) low concentration method, or (2) high concentration method.

The low concentration method will be for target sample depths believed to exhibit contamination in the less than 200 micrograms per kilogram (μg/kg) level. The high concentration sample collection method will be for target sampling intervals exhibiting contamination levels above 200 μg/kg. Predemonstration sampling was used to classify target sampling depths as low or high concentration.

Samples from low concentration target sampling depths will be collected as a 2-gram aliquots. The 5-gram aliquots will be collected using a disposable syringe with the tip cut off and the rubber plunger tip removed. The syringe will be pushed into the sample to the point that 3 to 3.5 cubic centimeters (cm^3) of soil is contained in the syringe. The soil core in the syringe will be directly extruded into a 22-mL headspace vial, that contains 5.0 mL of distilled (organic free) water. Once the soil core has been extruded into the headspace vial, the vial will be sealed with a crimp-top septum cap. A new syringe will be used to collect each aliquot. Each aliquot will be collocated. The headspace vial will be numbered according to the technology it represents, the sample grid and cell from which it was collected, and the depth of sample collection. This sample number will be applied to each sample container through the use of self-adhesive bar-coded stickers. This data along with textural descriptions (based on the United

States Department of Agriculture Classification) of the sample will be recorded on field data sheets. Two collocated samples are being collected to allow reanalysis of a sample for cases where a sample's contamination level was outside calibration ranges or where one sample was accidentally opened or destroyed prior to analysis. After the samples are properly packaged and labeled, they will be stored in a refrigerated (4°C) area until they are analyzed. All samples will be analyzed within 48 hours of collection.

Samples from the high concentration target sampling depths will be collected with disposable syringes as described above. Again two subsamples will be collected from the technology's sample, in the event that one of the subsamples is destroyed prior to analysis. The 5-grams of soil will be directly extruded into 40-mL vials fitted with a Teflon®-lined septum screw cap. The vials will already contain 10 mL of pesticide-grade methanol. The 40-mL vials will be numbered in the same fashion as the low concentration samples. The sample number and textural descriptions (based on the U.S. Department of Agriculture classification) of the sample will be recorded on field data sheets. After the samples are properly packaged and labeled, they will be stored in a refrigerated (4°C) area until they are analyzed. All high concentration samples will be analyzed within 48 hours of collection.

7.2.1.2 Soil Moisture and Soil Texture Analysis Sampling

To correct all analytical data for moisture content and to have soil available for textural analysis if it is deemed necessary, it will be necessary to collect a collocated soil sample for soil moisture content determination. These samples will only be collected from the conventional method's sample core. At each target sampling depth, a collocated sample weighing approximately 100 grams will be collected. This collocated sample will be collected with a disposable plastic teaspoon and placed directly into a 4- or 2-ounce glass jar. These samples will be collected after all samples for chemical analysis have been collected, and they will be collocated within 1-inch of the VOC sample location. The jars will be numbered in the same manner as the high and low concentration samples. After the samples are properly packaged and labeled, they will be stored at ambient temperatures until soil moisture content measurements are made. Soil moisture measurements will be made within 10 days of sample collection. The leftover soil will be archived for possible future textural analysis.

7.2.1.3 Sample Storage, Packaging, and Shipping

After collection and until analysis, all samples for chemical analysis will be stored in coolers. Technical requirements for holding times for soil samples being analyzed for VOCs have been established for the analytical methods being used. For the purpose of this demonstration, the samples will be analyzed for VOCs within 2 days of sample collection. Samples will be analyzed for soil moisture content within a period of 10 days from collection.

7.2.1.4 Decontamination

Disposable clothing or sampling equipment coming into contact with grossly contaminated material will be double-bagged and handled as investigation-derived waste (IDW). IDW will be properly disposed of according to the requirements in the EPA "IDW Management Guidance Manual—Second Draft" (1990). Only material that has come into contact with grossly contaminated material will be treated as IDW. Contamination avoidance practices will be used whenever possible to minimize the volume of IDW generated. Waste not coming into contact with grossly contaminated material will be disposed of in a local sanitary landfill.

Nondisposable sampling equipment will be decontaminated by scrubbing with an Alconox solution and water with a final water rinse. If the wet decontamination method is not adequate to remove soil particles, a high-pressure hot water cleaning unit may be used for decontamination. All decontamination water will be contained in 30- or 50-gallon steel drums pending analysis by the on-site laboratory. Depending on the concentration of contaminants in the decontamination water, the water will either be disposed of at a local publicly owned treatment works or by a licensed hazardous waste disposal contractor. If a technology developer has an alternative method of decontamination, that method will be used instead of the method described above.

No rinsate samples will be collected during this demonstration since the soil matrix is the target and volatile organics are the compounds of interest. All reuseable sampling tools will be washed to remove residual soil and then heated and dried.

7.2.1.5 Sample Hole Abandonment

All soil sampling holes will be backfilled with a neat Type II Portland cement grout, or a hydrated granular bentonite grout.

7.2.2 Conventional Method Soil Gas Sampling Procedures

The conventional method for collecting soil gas samples will involve an active collection process. Both the AMS and Geoprobe® systems will be used for this application. A 1-inch-diameter hollow rod will be driven to a target sample depth. This rod will be fitted with an expendable drive point. Once the rod has reached the target depth it will be withdrawn approximately 6 inches. The expendable drive point will remain in place, producing a 6-inch void space from which a soil gas sample can be collected. Once the rod has been retracted 6 inches, a 0.25-inch-inside diameter, high-density, polyethylene tube will be lowered into the drive rod. The end of the tubing will be fitted with a reverse threaded barbed-fitting. The barb will be inserted into the tubing and when the tubing reaches the end of the drive rod, the reverse threaded end will be threaded into the expendable drive point holder at the end of the drive rod. A butyl rubber o-ring around the threaded end of the barb fitting assures an airtight seal between the tubing and the end of the drive rod.

Once the tubing is in place, a 40-mL evacuated VOC vial will be attached to the top end of the tubing. The VOA vial will be evacuated using a 60 cubic centimeter (cc), plastic syringe. The syringe will pull a 60 cc vacuum on the closed VOA vial for 10 seconds. A volume-calibrated vacuum system will be attached to the end of the polyethylene tube attached to the end of the hollow rod. The vacuum system will remove a volume of air equal to the tubing volume. At this point, the vacuum will be shut off and the sampling string will be allowed to equilibrate with ambient air pressure. The system will be closed so that equilibration will occur only by drawing soil gas into the sample tubing. When no vacuum is left in the tubing, a double end hypodermic type needle will be inserted into the tubing. The needle will be inserted into the tyson tubing that connects the polyethylene tubing with the vacuum pump. The exposed end of the needle is sealed with a soft rubber sheath. The evacuated VOA vial will be pushed onto the exposed needle. The needle will penetrate the VOA vial's septum and Expose the soil gas in the tubing to the vacuum in the VOA vial. This will cause the soil gas to fill the VOA vial. The VOA vial will be allowed to collect a sample for 30 seconds at the CSC site and for two minutes at the SBA site. The

predemonstration soil gas sampling showed that those times were sufficient to allow equilibration. The VOA vial containing the soil gas sample will be numbered according to the sample grid and cell from which it was collected. After the samples are properly labeled, they will be analyzed within 24 hours of collection. While waiting to be analyzed, the soil gas samples will be stored at ambient temperatures. Sampling of the soil gas inside the VOA vial will be achieved by withdrawing 1 to 5 mL of sample using a glass syringe inserted through the small rubber septum in the center of the sampling bulb. The withdrawn sample will then be directly injected into a GC for analysis.

All reusable soil gas sampling equipment will be decontaminated by heating. The VOA vials and needles are disposable. The tubing will be discarded after a single use. The drive rod will be heated until it is too hot to touch by a 100,000-British thermal units (BTU) portable propane heater.

7.3 PERSONNEL REQUIREMENTS

All PRC employees working at each site will comply with the requirements of 29 Code of Federal Regulations (CFR) 1910. All developer technology operators will also be required to comply with the requirements of 29 CFR 1910.

7.4 LOGISTICAL NEEDS

PRC will maintain and operate the on-site laboratory used during this demonstration. This laboratory will require an external power source for operation. PRC will provide all the personnel and supplies needed to sample the samples collected by developers, and to contain samples. PRC will subcontract a commercial drilling company to collect the standard subsurface soil samples. Geoprobe® and AMS will conduct the standard active soil gas sampling. PRC will act as an agent of EPA to arrange for, and oversee, the disposal of IDW.

CHAPTER 8
QUALITY ASSURANCE PROJECT PLAN

The QAPP for this demonstration describes those procedures that will be implemented to assure data quality and integrity for the on-site laboratory. Careful adherence to these procedures will ensure that data generated from the on-site laboratory will meet the desired DQOs and will provide sound analytical results that can be used for a comparison of the sampling technologies.

8.1 PURPOSE AND SCOPE

The primary purpose of this QAPP is to outline steps that will be taken by the on-site laboratory to ensure that data resulting from this demonstration is of known quality and that a sufficient number of critical measurements are taken. This QAPP also details the QA/QC criteria that will be used to validate the on-site laboratory results. According to the NERL-CRD statement of work for this demonstration, this demonstration is classified as a Category II project. This chapter of the demonstration plan addresses the key elements that are required for Category II projects prepared according to guidelines in the EPA guidance documents "Preparing Perfect Project Plans" (1989) and the "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (1983).

The analytical method used by the on-site laboratory will be included in specific standard operating guidelines (SOG) for this demonstration. The on-site laboratory will use PRC SOG 001-- "Volatile Organic Compounds in Soil Gas, Water and Soil" for volatile analysis of soils, and soil gas. SOG 003--"Weight Balance Operation" will be followed by the on-site laboratory for weighing soil samples for volatile and semivolatile analysis. PRC SOG 001 is a GC method using a single column to identify and quantify VOCs in soil, water, and soil gas. PRC SOG 001 is attached in Appendix A. The target compounds for this demonstration include TCE, PCE, vinyl chloride, 1,2-DCE, and TCA.

Data generated by the on-site laboratory will be evaluated to determine the level of data quality produced. The on-site laboratory is expected to produce definitive data as classified by EPA in document titled "Data Quality Objectives Process for Superfund" (1993). Definitive data is described as being generated using rigorous analytical methods; and data is analyte specific, with confirmation of analyte identity and concentration. The raw data must be in the form of paper printouts or computer-generated

electronic files. The data can also be generated on- or off-site only if the QA/QC requirements are met. The following are QA/QC elements required for definitive data quality as described in guidance document listed previously (EPA 1993):

- Sample documentation (location, date and time collected, batch, etc.)
- Chain of Custody (when appropriate)
- Sampling design approach (systematic, simple or stratified random, judgmental, etc.)
- Initial and continuing calibration
- Determination and documentation of detection limits
- Analyte(s) identification
- Analyte(s) quantification
- QC blanks (trip, method, rinsate)
- Matrix spike recoveries
- Extraction efficiency (surrogates)
- Performance Evaluation (PE) samples (when specified)
- Measurement of analytical method precision (laboratory duplicates)
- Measurement of overall system precision from sample collection to analysis (i.e., field duplicates)

Adherence to the QA/QC requirements of this QAPP will ensure that definitive level data quality is generated by the on-site laboratory.

8.2 QUALITY ASSURANCE RESPONSIBILITIES

The PRC project manager is responsible for coordinating the preparation of a QAPP for the demonstration plan and its approval by the EPA TPM and the developers. The PRC project manager will ensure that the QAPP is implemented during all demonstration activities. The PRC analytical QA manager for the demonstration will review and approve the QAPP and will provide independent QA oversight of all demonstration activities.

Samples will collected and analyzed on-site by PRC's on-site laboratory using modified EPA-approved methods. Many individuals will be responsible for sampling and analysis QA/QC throughout the

demonstration. Primary responsibility for ensuring that sampling activities comply with the requirements of the sampling plan (Chapter 7) will rest with the PRC field team leader.

PRC's field chemist will be responsible for following written SOGs, for supplying information required for the evaluation of the individual sampling technologies, for sample receipt and log-in, recording daily activities in a laboratory notebook, recording preparation of working analytical standards, recording instrument maintenance in the instrument maintenance log book, keeping control charts for matrix spike and surrogate recoveries, and recording daily analytical results.

8.3 DATA QUALITY PARAMETERS

The data obtained during the demonstration must be of sound quality for conclusions to be drawn on the sampling technologies. For all measurement and monitoring activities conducted for EPA, the agency requires that data quality parameters be established based on the proposed end uses of the data. Data quality parameters include five indicators of data quality referred to as the PARCC parameters: precision, accuracy, representativeness, completeness, and comparability.

Definitive level quality on-site laboratory results are essential for meeting the purpose and objectives of this demonstration. Therefore, the PARCC parameters, which will be used as indicators of data quality, will be closely evaluated to determine the quality of data generated by the on-site laboratory.

The following subsections detail each of the PARCC parameters and include specific QA/QC samples which will be used to evaluate the quality of data by the on-site laboratory.

8.3.1 Precision

Precision is defined as the degree of mutual agreement among replicate measurements and provides an estimate of random error. Precision for the on-site laboratory will be expressed as relative percent difference (RPD) of laboratory duplicates and matrix spike (MS) and matrix spike duplicates (MSD).

Field duplicate samples will not be collected during the demonstration. Instead the replicate samples collected at each target sampling depth will provide a total precision data for sample heterogeneity,

sample collection, field preparation, handling and transportation procedures and analysis. Laboratory duplicate samples for the methanol flood (high concentration) samples will provide precision data for the analytical method sample extraction, preparation and final analysis. A laboratory duplicate sample cannot be prepared for the low concentration (headspace) samples. Analytical precision for these low concentration samples will be monitored through laboratory control sample (LCS). MS and MSD samples will provide precision data for the extraction and analytical methods for all samples. These duplicates will be analyzed at a frequency of one per batch of 20 samples by the on-site laboratory. (LCS data used to monitor analytical precision for the low concentration method must produce RSDs of less than 20 percent.) Initially, MS and MSD RPDs and laboratory duplicate RPDs must be less than or equal to 50 percent for the precision to be acceptable. However, once a minimum of 25 MS and MSD RPD and laboratory duplicate RPD results have been attained, the on-site laboratory will produce a control chart of RPDs with a 95 percent confidence interval around the mean. Subsequently, the upper and lower control limits will serve as control limits for acceptable precision for MS and MSDs and laboratory duplicates. This will provide a better representation of the effect of the matrix.

8.3.2 Accuracy

Accuracy refers to the difference between a true value and a measured value of a sample. Any deviation from the true value is a measure of bias. Bias can be influenced by such factors as standard preparations procedures, calibrations, efficiency of sample preparation for target analytes, completeness of the extraction process, interferences, and systematic or carryover contamination from one sample to the next.

Accuracy of the on-site laboratory will be determined by the analysis of LCSs and PE samples, MS and MSD percent recoveries, and sample surrogate recovery results. An LCS consists of a clean MS with a known concentration of certified standard purchased from a vendor other than the vendor from which the calibration standards were purchased. Accuracy of the extraction and analytical method will be evaluated through the comparison of LCS percent recoveries for each target analyte to known concentration spiked in the sample. LCSs will be analyzed at frequency of one per batch of 20 samples.

Surrogates will be spiked in every sample to monitor extraction efficiency.

A PE sample is a certified sample that is purchased from a reputable vendor, which comes with certified values with acceptable concentration ranges for each target analyte. The PE sample will be submitted to the on-site laboratory "single blind." This means that the laboratory will know that the sample is a PE sample but will not know the concentrations of the analytes in the PE sample. For the accuracy to be acceptable the on-site laboratory reported values for the target analytes must fall within the acceptable ranges supplied by the vendor.

Advisory control limits for LCS, surrogate, and MS/MSD percent recoveries will be between 50 and 150 percent. However, once a minimum of 25 total MS and MSD, LES, and surrogate percent recovery results are obtained by the on-site laboratory a control chart of percent recoveries will be produced. The control chart will represent a 95 percent confidence interval around the mean percent recovery. The upper and lower control limit will then be used to assess acceptable percent recoveries of the target analytes.

8.3.3 Representativeness

Representativeness refers to the degree to which the data accurately and precisely represents the characteristics or conditions of the parameter which the data represents. In this project, representativeness will be ensured by executing consistent sample collection processes, which include sample locations, sampling procedures, sample storage, sample packaging, sample shipping, sampling equipment decontamination and proper laboratory subsampling procedure. Representativeness will further be ensured by optimizing each method to its maximum capability to provide results that represent the most accurate and precise measurement that it is capable of.

8.3.4 Completeness

Completeness refers to the amount of data collected from the measurement process compared to the amount of data that was expected to be obtained. For this project, completeness refers to the proportion of valid acceptable data generated using each of the innovative technologies under investigation. The completeness objective for data generated during this demonstration is 95 percent.

8.3.5 Comparability

Comparability refers to the confidence with which one data set can be compared to another. Comparability will be evaluated with ICAL and CCAL data as well as PE samples. If the ICAL and CCAL data is maintained within control limits and PE sample analysis falls within published acceptance levels, the on-site data will be considered comparable..

8.4 CALIBRATION PROCEDURES, QUALITY CONTROL CHECKS, AND CORRECTIVE ACTION

Calibration procedures, method-specific QC requirements, and corrective action associated with nonconformance QC for the on-site laboratory are listed in Table 8-1.

8.4.1 Method Detection Limit Studies

The MDL is the minimum concentration of a compound that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL takes into account sample matrix and preparation. The on-site laboratory will demonstrate the MDLs for all analyses.

Annual- or demonstration-specific MDL studies will be performed for soil and water matrices or more frequently if any method or instrumentation changes occur. Each MDL study shall consist of seven replicates spiked with all target analytes of interest at a level no greater than the required quantitation limit. The replicates will be extracted and analyzed in the same manner as routine samples. If multiple instruments are used, all instruments will be included in the MDL study, and the reported MDL will be representative of the least sensitive instrument. All MDLs will meet the required quantitation limits or else the situation will be considered out of control and corrective action will be taken.

The MDL for each target compound will be determined in accordance with the procedures specified in Chapter 1, “Quality Control,” in “Test Methods for Evaluating Solid Waste” Physical and Chemical Methods, EPA Office of Solid Waste, EPA Publication SW-846 (EPA 1986).

8.4.2 Sample Quantitation Limits

Sample quantitation limits (SQL), also referred to as practical quantitation limits (PQL), are contract-required quantitation limits (CRQL) for individual sample characteristics. The CRQL is a chemical-specific level that a laboratory should be able to routinely detect and quantitate in a given sample matrix; it is usually defined in the analytical method. The SQL takes into account changes to the preparation and analytical methodology, such as use of a smaller sample aliquot or dilution of the sample extract, and physical characteristics of sample, such as sample matrix and percent moisture, that will alter the ability to detect the analyte in an environmental sample. The on-site laboratory will calculate and report SQLs for all environmental samples analyzed during this demonstration.

8.5 DATA REDUCTION, VALIDATION, AND REPORTING

To maintain good data quality, specific procedures will be followed during data reduction, validation, and reporting. These procedures are detailed below. These procedures will be implemented for the on-site and confirmatory laboratory data.

8.5.1 Data Reduction

Data reduction will be performed by the field chemist in the on-site laboratory. The on-site laboratory will produce data in field logbooks, hard copy chromatograms and reports, hard copy spreadsheet reports, and floppy disks or computer cassettes containing both chromatographic and spreadsheet data. This data will be reduced to produce a report detailing the analytical results. Data reduction will be performed following the formats and requirements of pertinent SOGs or the laboratory QA plan. This will include qualified data that failed one or more of the QC checks and providing definitions for each code used.

The on-site laboratory will produce results for individual target compounds based on internal or external calibrations. The on-site laboratory's calibration procedures outlined in SOGs 001 and 002 will be used during this demonstration. The on-site laboratory will produce direct readouts of analyte concentrations. The concentrations reported by the on-site laboratory will be reported in units of μg/kg for soils and

microgram per liter (μg/L) for waters. The concentrations will be corrected for dry weight.

8.5.2 Data Validation

The field chemist will verify the completion of the appropriate data forms and the completeness and correctness of data acquisition and reduction. The field analytical leader will review calculations and inspect laboratory logbooks and data sheets to verify accuracy, completeness, and adherence to the specific analytical method protocols. Calibration and QC data will also be examined by the field analytical leader. The field analytical team leader will also verify that all instrument systems are in control and that QA objectives for accuracy, precision, completeness, and MDL or SQL have been met.

Analytical outlier data are defined as those QC data lying outside a specific QC objective window for precision and accuracy for a given analytical method. Should QC data be outside of control limits, the on-site laboratory will investigate the cause of the problem. If the problem involves an analytical problem, the sample will be reanalyzed. If the problem can be attributed to the sample matrix, the result will be flagged with a data qualifier. This data qualifier will be included and explained in the final analytical report submitted by the laboratory.

PRC will further perform a data validation on 10 percent of the on-site laboratory data following the EPA's "National Functional Guidelines for Organic Data Review" (EPA 1991). The criteria reviewed during a cursory data validation includes the following QC parameters:

- Holding times
- Calibration
- Blanks
- Surrogate recovery
- MS/MSD recovery
- Blank spike or LCS recovery

- Internal standard performance
- Overall assessment of data for a SDG
- Laboratory and field duplicate sample analysis
- PE sample results

8.5.3 Data Reporting

Data packages will contain all of the information described below and all raw data for samples from on sample delivery group (SDG). A SDG is defined as a group of up to 20 samples of a similar matrix, submitted from one client over a maximum period of 14 days. Data qualifiers, as described in the contract laboratory program (CLP) statement of work for organics analysis will be included if necessary. The on-site laboratory data package will consist of a case narrative, sample results, QA/QC summaries, and all associated raw data.

Data reports generated from the analysis of samples using the on-site laboratory methods will include the following information.

- Analytical results for each parameter including units of measurement
- Data and time of sample analysis
- Summaries of QC data including control limits and identification of QC outliers
- Qualifications for analytical data effected by outlier QC data, along with definition of the outlier
- A data assessment that details the analytical procedures, QC procedures employed, QC outliers, qualifications, corrective actions, and assessment of the data to the PARCC parameters

Data from the on-site laboratory will be provided in both hardcopy and electronic diskette deliverable (EDD) format. This data will be loaded into the SITE analytical database specific to this demonstration. The laboratory will verify all EDDs internally before issue. The EDD will correspond exactly to the hard copy data, and no duplicated data shall be submitted. Electronic and hard copy data will be retained for a minimum of 3 and 10 years, respectively, after final data submittal. The laboratories will use a magnetic

tape storage device or other similar storage device that can record for long-term, off-line storage. All raw data will be retained on magnetic tape in accordance with appropriate instrument.

8.7 PERFORMANCE AND SYSTEM AUDITS

An audit evaluates the capability and performance of a measurement system or its components, and identifies problems warranting correction. Two types of audits may be conducted during the field work for this demonstration: performance and system audits. Audits will be performed at scheduled intervals by the PRC project manager, PRC QA manager, TPM, and by Mr. Barry Lesnik from EPA's OSW. The auditor(s) will complete an audit report. Upon completion of the audit, the auditor(s) will prepare and submit an audit report to the PRC project manager and PRC QA manager. The auditor(s) can issue a corrective action request to identify and schedule specific corrective actions to be undertaken and completed by the project managers. Completion of the corrective action is verified by the auditor(s).

8.7.1 Performance Audit

A performance audit is a review of the existing project and QC data to determine the accuracy of a total measurement system or a component of the system. This will be conducted by ordering PE samples from a certified supplier and submitting these to the on-site laboratory "single-blind." Single blind means that the laboratory will know the sample is a PE sample, but they will not know which target compounds are present or their concentrations. The control limits will be used to evaluate the on-site laboratory method performance.

The on-site laboratory will analyze these PE samples at the beginning of each working day before sample analysis begins to check the accuracy of the calibration.

8.7.2 System Audits

A system audit is used to verify adherence to QA policies and SOGs. This type of audit will consist of an on-site review of measurement systems, including facilities, equipment and personnel. Additionally, procedures for measurement, QC, and documentation will be evaluated. System audits will be scheduled

by the PRC project manager and conducted by the TPM. An audit checklist will be used to audit the on-site and confirmatory laboratories. The auditor(s) will then submit an audit summary report to the PRC project manager.

8.8 PREVENTIVE MAINTENANCE

Regularly scheduled preventive maintenance procedures are performed to keep all field and laboratory equipment in good working condition and are discussed below.

8.8.1 On-site Field Laboratory Equipment

Detailed information regarding maintenance and servicing of field analytical equipment is available in the instruction manual of the specific instrument to be used. Service and maintenance information will be recorded in logbooks by the field chemist. Instrument problems encountered during field work will be recorded and, if possible, remedied in the field. Spare replacement parts will be kept on hand, if needed. Specific preventive maintenance procedures will follow manufacturer recommendations.

Preventive maintenance tasks will be the responsibility of the field chemist. Additional details on equipment maintenance procedures are presented in PRC SOG 001 and the instruction manual for the specific equipment.

8.9 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports provide management with the necessary information to monitor data quality effectively. It is anticipated that the following types of QA reports will be prepared as part of this demonstration project.

8.9.1 Audit Reports

Any QA audits or inspection that take place in the field while the demonstration is being conducted will be formally reported by the auditor(s) to the PRC analytical QC manager and the PRC project manager who will forward them to the EPA TPM.

8.9.2 Corrective Action Reports

Any QA/QC corrective actions that take place in the field while the demonstration is being conducted will be informally reported by the field chemist to the PRC analytical QC manager and the PRC project manager who will forward them to the EPA TPM.

TABLE 8-1

CALIBRATION PROCEDURES, QUALITY CONTROL CHECKS, AND CORRECTIVE ACTION
ON-SITE LABORATORY ANALYSIS
Page 1 of 2

QC Parameter	Frequency	Control Limits	Corrective Action
Initial calibration (6 levels)	Before sample analysis is initiated	20 %RSD for each target analyte, except for vinyl chloride which may have a 25% RSD	1. Use linear regression equation of the line if the correlation coefficient is greater than or equal to 0.995. The low or high level may be eliminated if it is determined that the level is significantly affecting the %RSD and the %RSD falls to less than 20% 2. If the ICAL is still not acceptable check the results of the PE sample. If the PE sample results are within the acceptance ranges then the ICAL can be considered acceptable for quantification
Continuing calibration (medium level)	After every 12 samples or within a 12-hour analytical sequence	15 %D for each target analyte, 20% for vinyl chloride	1. Reanalyze CCAL 2. Evaluate instrument and calibration standards for possible problems and reanalyze the samples following the out-of-control CCAL following a new ICAL
Method blank	With each batch of 20 samples of a similar matrix	No target analytes detected above the SQL	1. Reextract and reanalyze another method blank 2. Qualify the associated samples
Instrument blank	Minimum of once daily and after highly concentrated samples to check for carry-over	No target analytes detected above the SQL	1. Evaluate the instrument for possible contamination and try and correct the problem. Then analyze another instrument blank before sample analysis can be reinitiated
Surrogate standards	All samples and MS/MSDs analyzed	The upper and lower control limits will serve as QC limits	1. Reanalyze 2. Qualify sample

TABLE 8-1

CALIBRATION PROCEDURES, QUALITY CONTROL CHECKS, AND CORRECTIVE ACTION
ON-SITE LABORATORY ANALYSIS
Page 2 of 2

QC Parameter	Frequency	Control Limits	Corrective Action
MS/MSD percent recovery	One per 20 samples of the same matrix	Initial advisory control limits of 50 to 150 percent recovery. After control charts are produced the upper and lower control limits will serve as QC limits 50 percent RPD	1. Check the LCS result 2. Repeat analysis 3. Data cannot be qualified based on MS/MSD recoveries alone
PE samples	One per working day	Within published acceptance limits. If no acceptance limits are specified use 50 to 150 percent recovery	1. Determine the source of the problem and correct 2. Reextract and reanalyze 3. Check the LCS result 4. If unacceptable results still occur remake the calibration standards and reanalyze the PE and all associated samples
Laboratory duplicates	One per 20 samples of the same matrix for methanol-flood only	50 percent RPD	1. Check MS/MSD 2. Repeat analysis 3. Qualify data
LCS	One per 20 samples	Initial advisory control limits of 50 to 150 percent recovery. After control charts are produced the upper and lower control limits will serve as QC limits (for precision assessment, RSDs ≤ 20 percent	1. Determine the source of the problem and correct 2. Reanalyze LCS 3. Check PE results 4. If unacceptable results still occur qualify the data

Notes:

PE	Performance evaluation sample	%RSD	Percent relative standard deviation
SQL	Sample quantitation limit	RPD	Relative percent difference
MS	Matrix spike	ICAL	initial calibration
MSD	Matrix spike duplicate	CCAL	continuing calibration
%D	Percent difference		

The MDL for each target compound will be determined in accordance with the procedures specified in Chapter 1, “Quality Control,” in “Test Methods for Evaluating Solid Waste” Physical and Chemical Methods, EPA Office of Solid Waste, EPA Publication SW-846.

CHAPTER 9
DATA MANAGEMENT AND ANALYSIS

The data management system will use computerized data files and hard copy documentation, such as field and laboratory sheets and hardbound logbooks, to store all technology and conventional method analytical data. This system will be used to form statistical analyses of the data as described in Chapters 5 and 6 and to verify that the data meets the data quality parameters established in Chapter 8.

This chapter describes the procedures that will be used for obtaining and entering data into this system, and for analyzing the data after it has been entered.

9.1 LABORATORY DATA MANAGEMENT ACTIVITIES

All soil samples will be collected and documented as described in Chapter 7. Each sample will be labeled with a unique sample number assigned in the field. Each sample will be submitted for analysis accompanied by a field sheet containing additional information about the sample. Once a sample has been submitted for analysis, data associated with the sample will be managed as described below.

9.1.1 Reference Data Management

Samples submitted for chemical analysis will be analyzed by the on-site laboratory. Chain-of-custody forms will not be used since samples will not be shipped off site. Samples will be tracked by field sheets and logbooks. The tracking of samples from submittal to the on-site laboratory through analysis will be tracked from each analytical step using bar codes and bar code scanners. The sample numbers will be applied to sample containers in an alphanumeric and bar-code format. After samples are analyzed, the data will be reduced, validated, and reported as described in Chapter 8.

Validated sample results will be entered into the data management system. In addition to sample results, QA/QC summary forms for the on-site analyses will be entered. These forms will allow verification of the quality of data generated by these methods. All data transcribed will be double-checked for accuracy.

9.1.2 Technology Data Management

The operator of each technology will record the technology sample number and corresponding reference sample numbers. The operators will be responsible for obtaining, reducing, interpreting, validating, and reporting data associated with their technology's performance. Each operator will be required to provide the field team supervisor with copies of the results obtained from each sampling point, as well as any graphical data used for the delineation of site contamination.

The operators also will be responsible for obtaining information about the assigned technology. This information will include a general description of the technology and how it is used in the field. Each operator will take notes on specific aspects of the technology. These notes will be based on a checklist created for each technology before the demonstration activities begin. The checklist will provide information that will be used in the ITER. In general, the checklists will contain the following items:

- Description of equipment used
- Logistical considerations including size and weight of technologies, power requirements, and other accessories needed, but not provided by the developer
- Historical uses and applications of the technology
- Estimated cost of the equipment or the cost of using the equipment
- Number of people required to operate equipment
- Qualifications of technology operator
- Training required for technology operator
- Description of data each technology can produce and a description of the operational mode required for producing this data
- Reliability under the test conditions (failure rate during evaluation)
- Susceptibility to environmental conditions (ruggedness)
- Ease of operation
- Ease of learning to use technology
- Logistical requirements

- Degree of physical sample disruption
- Type and amount of sample handling after collection that is necessary for laboratory-ready samples
- Degree to which measures of performance met developer performance claims
- Description of the amount of time required for data interpretation
- Description of the reports and graphics that each technology will produce
- Specific problems or breakdowns occurring during the demonstration
- Matrix interferences found during the demonstration

The operators will be responsible for reading the approved demonstration plan, as well as any other information submitted by the developers. A copy of the completed checklists will be included in the TER. Notes taken by each technology operator will be documented in a hardbound logbook and will be used as a reference when preparing the ITER.

CHAPTER 10
HEALTH AND SAFETY PLAN

This chapter describes specific health and safety procedures that will be used during the field work to be performed at the demonstration sites. The demonstration sites include: the SBA site, Albert City, Iowa; and the CSC Site, Denver, Colorado.

The purpose of the HASP is to define the requirements and designate the protocols to be followed during the field work specified under OSHA 29 Code of Federal Regulations (CFR) 1910.120(b) Final Rule. All demonstration personnel, subcontractors, and visitors on site must be informed of site emergency response procedures and any potential fire, explosion, health, or safety hazards related to demonstration activities. A copy of the HASP will be provided to all demonstration personnel, subcontractors, and site visitors who may be exposed to dangerous conditions during the demonstration.

This HASP must be reviewed and approved by the PRC project manager. A HASP compliance agreement form must be signed by all field personnel before they enter each site. Any revisions to this plan must be approved by the PRC project manager.

10.1 HEALTH AND SAFETY PLAN ENFORCEMENT

The project manager, field site supervisor, health and safety director, and site health and safety officer (SHSO) will be responsible for implementing and enforcing the health and safety provisions of this HASP. Their duties are described in the following subsections.

10.1.1 Project Manager and Field Site Supervisor

The project manager will ultimately be responsible for ensuring that all demonstration participants abide by the requirements of this HASP. The field site supervisor will oversee and direct field activities, including subcontractor activities, and also is responsible for ensuring compliance with this HASP.

10.1.2 Health and Safety Director

The Health and Safety Director (HSD) will be responsible for coordinating the technical aspects of the health and safety program. The HSD will act in an advisory capacity to the SHSO and will report to the project manager. Liaison with subcontractor or site visitor representatives on matters relating to health and safety will be handled by the HSD or SHSO. The HSD will be responsible for maintaining up-to-date records of HASP-related documentation and HASP participants. This documentation will include the following:

- Documentation of the physician's examination of each employee (Chapter 10.10, Medical Surveillance)
- The training record for each employee who has completed the training necessary to perform his or her job
- Documentation of a fit test for each employee required to wear respiratory protection equipment meeting the requirements of OSHA 29 CFR 1910.134 and American National Standards Institute Z88.2-1980
- Task-specific air monitoring information (regarding drilling, drumming of waste, and other activities)

Anyone who does not meet HASP requirements will not be allowed to conduct field work.

10.1.3 Site Health and Safety Officer

The SHSO will be responsible for implementing and enforcing the requirements of this HASP in the field. The SHSO will have advanced field work experience and will be familiar with health and safety requirements specific to the demonstration. The SHSO will ensure that a Safety Meeting Sign-off Sheet is signed by all employees who are to perform field work, and that each employee completes a Daily Site Log before leaving the site.

10.2 VISITORS

All visitors to operations at the site will be required to read the HASP and sign a compliance agreement form. Visitors will be expected to comply with relevant OSHA requirements. Visitors will also be expected to provide their own personal protection equipment (PPE) as required by the HASP.

Any visitors who do not adhere to the provisions of the HASP will be ordered to leave the work area. Visitors who have not met OSHA training and medical surveillance requirements will not be permitted to enter areas where exposure to hazardous materials is possible. Exceptions will be strongly discouraged, but they can be made on a case-by-case basis under the following conditions: (1) respirators are not required, (2) visitors' time on site is limited, (3) visitors are given a pre-entry briefing, (4) visitors are accompanied by trained personnel at all times, and (5) SHSO approval is obtained.

10.3 DEMONSTRATION-SPECIFIC HAZARD EVALUATION

The hazards associated with this demonstration include worker exposure to VOCs, exposure to weather extremes, and physical hazards associated with the technologies equipment and work on active or abandoned industrial facilities.

This demonstration will occur in May and June of 1997; therefore, the possibility for hot weather exists. In addition to heat exposure, it is possible that the demonstration team will be working during precipitation events. Heat stress hazards are discussed in Section 10.12.

General hazards associated with active or abandoned industrial facilities include trip and fall hazards, surficial debris, unmaintained structures, and manufacturing processes themselves.

10.4 EXPOSURE PATHWAYS

Exposure to VOC contaminants during field activities may occur through inhalation, dermal contact, or ingestion. Descriptions of these exposure pathways are provided below.

10.4.1 Inhalation

One possible exposure pathway for contaminants may be through inhalation. Personnel will monitor the concentrations of airborne contaminants with an air monitoring instrument, such as a Microtip photoionization detector (PID) with an 10.2 electron voltage (EV) lamp. Level D personal protection will generally be used, but when the monitoring instruments indicate potential problems, personal protection will be upgraded to Level C.

10.4.2 Dermal Contact

Dermal contact with contaminated soil may occur at each site during demonstration activities. Dermal contact will be prevented with the use of PPE, such as inner and outer gloves. Safe personal protection procedures are described in Section 10.9.

10.4.3 Ingestion

Ingestion, although unlikely, may occur if personnel demonstrate a lack of proper hygiene or decontamination. Section 10.14.2 discusses safe work practices that may prevent ingestion of contaminated material.

10.5 HEALTH EFFECTS

This section describes the possible health effects of exposure to VOCs. Health effect information has been determined by the National Institute for Occupational Safety and Health (NIOSH). Low, medium, or high levels of VOCs may be encountered during sampling activities at the demonstration sites.

Acute symptoms of exposure to the VOCs detected at the demonstration sites are listed in Tables 10-1 through 10-3. Chronic symptoms of exposure to the VOCs include: liver lung, and kidney damage, dermatitis, and cancer.

Exposure to VOCs in soil will be controlled through proper use of PPE and real-time air monitoring. The need for respiratory protection (air purifying respirators) will be based on real-time air monitoring

results. If the concentration of VOCs exceeds 5 ppm above background concentrations in the breathing zone, as measured with an air-monitoring instrument, the level of protection will be upgraded from Level D to Level C.

10.6 PHYSICAL HAZARDS

Physical hazards associated with sampling and other field activities present a potential threat to on-site personnel. Dangers are posed by utility and power lines, unseen obstacles, noise, heat, and poor illumination.

Injuries may result from the following:

- Accidents due to slipping, tripping, or falling
- Improper lifting techniques
- Moving or rotating equipment
- Equipment mobilization and operation (for example, electrocution from contact with overhead or underground power lines)
- Improperly maintained equipment

Injuries resulting from physical hazards can be avoided by adopting safe work practices and by using caution when working with machinery. Safe work practices to be used during all field activities are described in Section 10.14.2. To ensure a safe work place, the SHSO will conduct and document regular safety meetings to make sure that all personnel are informed of any potential physical hazards related to the site.

10.7 TRAINING REQUIREMENTS

All personnel, subcontractors, and site visitors who may be exposed to hazardous on-site conditions at the demonstration sites will be required to meet the training requirements outlined in OSHA 29 CFR 1910.120, which covers hazardous waste operations and emergency response. All personnel, subcontractors, and visitors entering the site will be required to read this HASP and sign the compliance

agreement form. All site workers will be required to sign a Safety Meeting Sign-off Sheet as well.

Before field activities begin, a briefing will be presented by the SHSO for all personnel who will participate in field activities. The following topics will be addressed during the briefing:

- Names of the SHSO and the designated alternate
- Site history
- Hazardous chemicals that may be encountered during on-site activities
- Physical hazards that may be encountered on site
- Training requirements
- Levels of protection to be used for specific work tasks
- Work tasks
- Environmental surveillance equipment use and maintenance
- Action levels (Section 10.11.3, Monitoring Parameters) and identification of situations requiring an upgrade or downgrade in levels of protection
- Site control measures, including site control zones, communications, and safe work practices (Section 10.14)
- Emergency communication signals and codes
- Decontamination procedures
- Environmental accident emergency procedures (in case contamination spreads outside the exclusion zone)
- Personnel exposure and accident emergency procedures (in case of exposure to hazardous substances, falls, and other hazardous situations)
- Fire and explosion emergency procedures
- Emergency telephone numbers
- Emergency routes

Any other health- and safety-related topics that may arise before field activities begin also will be discussed at the briefing.

Issues that arise during implementation of field activities will be addressed during "tailgate" safety meetings to be held daily before the shift begins. Any changes in procedures or site-specific health- and safety-related matters will be addressed during these meetings.

10.8 PERSONAL PROTECTION REQUIREMENTS

PPE will be worn to protect personnel from known or suspected physical hazards, and air, soil, and groundwater contamination. The levels of personal protection to be used for work tasks have been selected based on known or anticipated physical hazards and concentrations of contaminants that may be encountered on site, and their chemical properties, toxicity, exposure routes, and contaminant matrices. The following sections describe levels of protection, protective equipment and clothing, limitations of protective clothing, duration of work tasks, and respirator selection, use, and maintenance.

10.8.1 Levels of Protection

Personnel will wear protective equipment when field activities involve known or suspected atmospheric contamination or when direct contact with skin-affecting substances may occur. Full-face respirators will protect lungs, the gastrointestinal tract, and eyes against airborne contaminants. Chemical-resistant clothing will protect the skin from contact with skin-destructive and absorbable chemicals.

For this demonstration, the levels of protection and necessary components for each are classified under two categories according to the degrees of protection afforded:

Level D:	This level provides minimal protection against chemical hazards. Worn only as a work uniform; not to be worn in areas posing respiratory or skin hazards.
Level C:	Worn when the criteria described for Level C protection in Section 10.11.3 for using air-purifying respirators are met, and a lesser level of skin protection is needed.

Field activities for this demonstration will be conducted in Level D. The demonstration team will monitor the ambient air for airborne contaminants. If the concentration of airborne contaminants exceeds 5 ppm in the breathing zone, personnel will upgrade their level of protection to Level C.

10.8.2 Protective Equipment and Clothing

The following general levels of protection and the associated PPE ensembles have been selected for use by personnel during sampling and field screening activities. Because the anticipated hazard level is low, field work will be performed using Level D protection. If site conditions or the results of air monitoring performed during field activities warrant Level C protection, all personnel will upgrade to Level C protection. Descriptions of equipment and clothing required for Levels D and C protection are provided below.

- Level D
  - -- Coveralls or work clothes, if applicable
  - -- Steel-toed boots with shanks
  - -- Hard hat (face shield optional)
  - -- Disposable outer gloves (polyvinyl chloride or nitrile), if applicable
  - -- Safety glasses or goggles
  - -- Chemical-resistant clothing (Tyvek® or Saranex®), if applicable
  - -- Disposable boot covers (when entering wet or muddy areas with known elevated contamination levels, such as previously excavated waste areas)
  - -- Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)
- Level C
  - -- Coveralls or work clothes, if applicable
  - -- Chemical-resistant clothing (Tyvek® or Saranex®) if a direct contact hazard exists
  - -- Outer gloves (neoprene or nitrile)
  - -- Inner gloves (nitrile or polyvinyl chloride)
  - -- Steel-toed boots with shanks
  - -- Disposable boot covers or chemical-resistant outer boots

- -- Full- or half-face, air-purifying respirator with NIOSH- or OSHA-approved cartridges to protect against organic vapors, dust, fumes, and mists (cartridges will be changed at the end of each shift or at breakthrough, whichever occurs first)
- -- Safety glasses or goggles (with half-face respirator only)
- -- Hard hat (face shield optional)
- -- Hearing protection (for areas with a noise level exceeding 85 decibels on the A-weighted scale)

10.8.3 Limitations of Protective Clothing

PPE clothing ensembles designated for use during field activities have been selected to provide protection against contaminants at known or anticipated concentrations in soil. However, no protective garment, glove, or boot is entirely chemical-resistant, nor does any protective clothing provide protection against all types of chemicals. Permeation of a given chemical through PPE depends on contaminant concentrations, environmental conditions, the physical condition of the protective garment, and the resistance of the garment to the specific contaminant. Chemical permeation may continue even after the source of contamination has been removed from the garment.

To obtain optimum use from PPE, the following procedures will be followed by all personnel:

- When using Tyvek® or Saranex® coveralls, don a new, clean garment after each rest break or at the beginning of each shift.
- Inspect all clothing, gloves, and boots both before and during use for the following:
  - -- Imperfect seams
  - -- Nonuniform coatings
  - -- Tears
  - -- Poorly functioning closures
- Inspect reusable garments, boots, and gloves both before and during use for visible signs of chemical permeation such as the following:
  - -- Swelling
  - -- Discoloration
  - -- Stiffness
  - -- Brittleness

-- Cracks

-- Any sign of puncture

-- Any sign of abrasion

Reusable gloves, boots, or coveralls exhibiting any of the characteristics listed above must be discarded. PPE clothing used in areas with known or suspected elevated concentrations of contaminants should not be reused. Reusable PPE will be decontaminated according to the procedures described in Section 10.15 and will be neatly stored in the support zone away from work zones.

10.8.4 Duration of Work Tasks

The duration of field activities involving use of PPE will be established by the SHSO or a designee and will be based on ambient temperature and weather conditions, the capacity of personnel to work in the designated level of PPE, and the limitations of the PPE. All rest breaks will be taken in the support zone after decontamination and removal of PPE.

10.8.5 Respirator Selection, Use, and Maintenance

All personnel and subcontractors taking part in field activities must fulfill worker provisions outlined in OSHA 29 CFR 1910.120. All demonstration personnel will be informed of the proper use, maintenance, and limitations of air-purifying respirators during the daily safety briefing, if applicable. All personnel must complete a qualitative fit test for the respirator to be used on site.

Respirator use is not anticipated at the demonstration sites. However, if respirator use becomes necessary, a full-face air-purifying respirator equipped with NIOSH- or OSHA-approved cartridges will be selected for use to protect against organic vapors. Respirators will be selected by the SHSO based on knowledge of the substances that may be present at the sites and the concentrations of compounds previously encountered at the sites. Air-purifying respirators will be used only when they can provide protection against the substances encountered at the sites.

Respirators will be inspected daily and any necessary repairs will be made during the time of inspection. Damaged respirators will be properly disposed of. Respirators issued to personnel will be cleaned and

disinfected in the support zone at least weekly. When a respirator is used by more than one person, the respirator will be cleaned and disinfected after each use. After being cleaned, respirators will be placed in clean plastic bags and stored in the support zone. The following respirator inspection and cleaning procedures will be followed whenever respirator protection is used:

- Daily inspection and checkout procedures:
  - -- Visually inspect the entire unit for obvious damage and deteriorated rubber.
  - -- Inspect the face-piece harness for damage.
  - -- Inspect the lens for damage and make sure the face piece has the proper seal.
  - -- Pull the plastic cover off the exhalation valve and check the valve for debris and tears in the neoprene that could cause leakage.
  - -- Unscrew the cartridges of both inhalation valves and visually inspect the neoprene valves for tears. Make sure the inhalation valves and cartridge receptacle gaskets are in place.
  - -- Make sure the speaking diaphragm retainer ring is hand-tight.
  - -- Don the respirator, and perform a negative pressure test.
- Weekly cleaning procedures:
  - -- Disassemble the respirator in the support zone by removing the cartridges, damaging them to prevent accidental reuse, and discarding them. To clean the respirator thoroughly, remove the inhalation and exhalation valves, speaking diaphragm, and any hoses.
  - -- To clean the respirator, dissolve cleaning and disinfecting solution (usually provided by the manufacturer) in warm water in an appropriate tub. With gloved hands, swirl the respirator in the tub for at least 1 minute. A soft brush may be used to facilitate cleaning.
  - -- Rinse the cleaned and disinfected respirator thoroughly with potable water to remove all traces of detergent and disinfectant. This step is very important in preventing dermatitis.
  - -- Air dry the respirator on a clean surface. The respirator may also be hung upside down, but care must be taken not to damage or distort the face piece.
  - -- Reassemble the clean, dry, respirator and inspect it in an area separate from the disassembly area to avoid contamination. Inspect the respirator carefully for detergent or soap residue left by inadequate rinsing. Residue appears most often under the seat of the exhalation valve and can cause valve leakage or sticking.
- Procedures to follow after routine use in the exclusion zone:
  - -- Wash and rinse the respirator in the support zone with soap and warm water.

-- At a minimum, wipe the respirator with disinfectant wipes that have been soaked in benzoalkaloid or isopropyl alcohol. Allow the respirator to air dry in the support zone.

The effectiveness of the respiratory protection program will be continuously monitored by the SHSO or designee. Monitoring of worker stress levels during activities that require respiratory protection will also be performed by the SHSO or designee.

10.9 MEDICAL SURVEILLANCE

The PRC Health and Safety Program is based on employee training, medical surveillance, and the use of PPE. Additional relevant aspects of the program include preparation of site-specific HASP, air monitoring, the hazard communication program, the hearing conservation program, the respiratory protection program, protection from radiation, the confined space entry program, protection from heat and cold stress, accident reporting and investigation, safe work practices, and decontamination. This program applies not only to hazardous waste site work but also to PRC employees that may be exposed to unsafe or unhealthful conditions. Guidance has been developed for ergonomic hazards, reproductive hazards, bloodborne pathogens, lead exposure, cadmium exposure, and a group of special hazards.

The primary regulations upon which the Health and Safety Program is based include the following:

- 29 CFR 1910.120, Hazardous Waste Operations and Emergency Response
- 29 CFR 1910.1200 and 1926.59, Hazard Communication
- 29 CFR 1910., Subpart I, Personal Protective Equipment
- 29 CFR 1910.95, Occupational Noise Exposure
- 29 CFR 1910.146, Permit-Required Confined Spaces
- 10 CFR Part 20, Standards for Protection Against Radiation
- 29 CFR 1910.20 and 1926.22, Access to Employee Exposure and Medical Records
- 29 CFR 1910.151 and 1926.50, Medical Services and First Aid

PRC field workers shall receive 40 hours of initial hazardous waste training before conducting any field work as specified in 29 CFR 1910.120. Field workers shall receive 8 hours of refresher training annually after the initial 40 hours of training. On-site managers and supervisors responsible for employees

engaged in hazardous waste operations shall receive at least 8 hours of specialized training in addition to initial and refresher training. PRC field workers should also be trained in cardiopulmonary resuscitation and standard first aid procedures.

The PRC medical surveillance program is designed to monitor the health of PRC personnel who may be exposed to toxic substances and hazardous conditions in the work environment. The medical surveillance program applies only to PRC personnel who have completed (or will complete) the health and safety training required under 29 CFR 1910.120(e), Training. The medical surveillance program will achieve the following:

- Determine the employees' medical fitness for hazardous work
- Determine the employees' ability to use respirators
- Identify individuals who may be at increased risk and limit their field activities to reduce risk
- Detect through annual, biennial, or special examinations the early onset of symptoms that may be related to occupational exposure
- Determine the medical status of personnel leaving the PRC medical surveillance program through an exit exam

Each health and safety program participant shall undergo a baseline examination before conducting any field work. Examined employees must sign an Affidavit of Participation stating that he or she will participate in all stages of the medical surveillance program.

Each participant shall also undergo either an annual or biennial examination based on the number of hours of field work conducted within 12 months of the previous examination. Employees who conduct 240 hours (30 days) or more of field work within 12 months of their previous examination shall have an annual examination. Employees who conduct less than 240 hours of field work are eligible for a biennial examination, but may have an annual examination if desired. The time interval between examinations shall not exceed 24 months.

Each program participant terminating employment with PRC or transferring to a position not requiring field work shall receive an exit examination. If another examination was conducted within 6 months of termination or transfer, this requirement will be waived. Employees who decline to undergo the exit examination must sign a waiver releasing PRC of liabilities arising from employment.

A special examination should be performed if an injury occurs or if an employee develops signs or symptoms indication possible overexposure to hazardous substances. This examination may be specific to the exposure or injury or may follow the annual examination protocol, as determined by the attending physician. In case of injury, the patient should immediately be transferred to a nearby trauma center for emergency treatment.

10.10 ENVIRONMENTAL SURVEILLANCE

Air monitoring will be performed during designated sampling and other field activities to protect personnel against exposure to airborne hazardous substances and to determine appropriate levels of PPE for work tasks. The following sections discuss initial air monitoring, periodic air monitoring, monitoring parameters, use and maintenance of survey equipment, and heat stress monitoring.

10.10.1 Initial Air Monitoring

Initial air monitoring of the work area will be performed before beginning site activities. This monitoring will be performed using real-time field survey instrumentation, such as a Microtip PID, to determine the concentrations of airborne organic contaminants. Airborne contaminants also will be monitored at the beginning of each work day to identify background contaminant concentrations and to detect any potentially hazardous situation that might have developed during off-shift periods.

10.10.2 Periodic Air Monitoring

Periodic air monitoring will be performed during all site activities. This type of monitoring will be performed as a minimum requirement when the following situations arise:

- Work begins on a different portion of the site
- Workers experience physical difficulties

Required survey instrumentation, sampling procedures, and monitoring procedures are specified in Section 10.11.3. Sampling methods will be subject to review by the SHSO.

10.10.3 Monitoring Parameters

Air monitoring for organic vapors will be performed at shoulder height (in the breathing zone) on personnel most likely to be exposed to potentially hazardous concentrations of contaminants. The following instrument and monitoring frequency may be used to monitor for organic vapors during site activities.

- Instrument: Hand-held PID
- Activity: Direct Real-Time Air Monitoring
- Monitoring Frequency: Monitoring will occur continuously during field activities, especially upon sample retrieval.
- General action levels
  - -- Situation: Concentration of organic vapors below 5 ppm in the breathing zone
    - Action: Continue investigation at Level D without respiratory protection and continue monitoring
  - -- Situation: Concentration of organic vapors at or above 5 ppm in the breathing zone
    - Action: Notify SHSO; upgrade to Level C protection and continue work unless otherwise specified

10.11 USE AND MAINTENANCE OF SURVEY EQUIPMENT

All personnel using field survey equipment will be briefed on its operation, limitations, and maintenance by the SHSO. Maintenance and calibration will be performed according to manufacturer guidelines by a designated individual familiar with the devices. Repairs, maintenance, and routine calibration of this equipment will be recorded in an equipment maintenance logbook that will be signed by the trained service technician. The equipment maintenance logbook for each piece of equipment will be kept in the carrying case for that equipment.

Air monitoring equipment will be calibrated before work begins each day. Only routine maintenance (such as charging batteries and cleaning the probe tip) will be performed by field personnel. Any additional maintenance will be performed by a trained service technician.

10.12 HEAT STRESS MONITORING

Heat and cold stress are serious conditions commonly encountered during field work. The likelihood of a temperature-related illness depends on factors such as physical activity, clothing, wind, humidity, working and living conditions, and the age and state of health of the employee. Although OSHA does not have regulations to limit temperature exposures, PRC follows guidelines from the American Red Cross and the American Conference of Governmental Industrial Hygienists. This chapter provides PRC's temperature stress guideline and discussions of heat stress. Cold stress will not be discussed because extreme cold temperatures are not expected to be encountered during this demonstration.

Temperature stress will be reduced by using engineering controls, safe work practices, and management techniques. PRC field workers will be trained to recognize and respond to temperature-related illnesses during refresher training, cardiopulmonary resuscitation, and first aid courses. Field workers should monitor themselves and coworkers for signs of temperature-related illnesses. The SHSO is responsible for initiating rest schedules during field work.

The possibility of a heat-related injury during field work is low to moderate because the demonstration is taking place during the months of May and early June, and because some types of PPE increase the body's work load and decrease the body's means of cooling. Heat stress symptoms include heat cramps, heat exhaustion, and heat stroke. Heat stroke is the most serious condition and can be life-threatening.

Depending on the degree and nature of possible heat stress to be encountered on site, the SHSO will choose from the following heat stress control actions:

- Provide adequate liquids to replace lost body fluids. These liquids can be water, commercial mixes combined with potable water, or commercial liquids (such as Gatorade®).

- Establish a work regimen that will provide adequate rest periods for cooling down. This action may require additional work shifts or earlier or later work schedules to avoid midday heat.
- Provide cooling devices, such as vortex tubes or cooling vests, to be worn beneath protective garments.
- Require removal of impermeable protective garments during rest periods.
- Ensure that all rest periods are taken in a shaded rest area, if possible.
- Regulate rest periods, and ensure that workers will not be assigned other tasks during rest periods.
- Notify all workers of health hazards and the importance of adequate rest, acclimatization, and proper diet.
- Teach workers to recognize heat stress and to conduct first aid to prevent heat stress.

10.13 SITE CONTROL

Work areas on or near the demonstration sites will, depending on results of environmental monitoring, be divided into three zones: an exclusion zone, a contamination reduction zone (CRZ), also known as decontamination zone, and a support zone. Generally, the exclusion zone will be designated by barricade tape or traffic cones. Access to a contaminated exclusion zone will be through the CRZ, and it will be restricted to authorized personnel. The support zone will be the area where supplies are staged and samples are packaged. A daily roster with the date of each person's entrance into the contaminated zone; the person's name, signature, and organization; the time of entry; and the time of exit will be kept for all personnel working in such an area. Any visitors to the area must present proper identification and be authorized to be on site. Visitors must comply with all provisions of this HASP. The SHSO will identify work areas that visitors or personnel are authorized to enter and will enforce site control measures. The following subsections discuss site control zones, safe work practices, HASP enforcement, and complaints.

10.13.1 Site Control Zones

The SHSO will establish site control zones after initial monitoring of site conditions. Should the conditions change during field activities, the SHSO will reevaluate the current site control zones and establish new zones, if necessary.

10.13.2 Safe Work Practices

Safe work practice requirements for field activities will include the following:

- All personnel will enter a designated exclusion zone only through the contamination reduction corridor. All personnel leaving an exclusion zone must exit through the contamination reduction corridor and undergo the CRZ decontamination procedure.
- Only equipment necessary to complete sampling will be permitted within an exclusion zone. All nonessential equipment will remain within the support zone.
- All personnel will avoid contact with potentially contaminated substances. Walking through puddles or mud and kneeling on the ground will be avoided whenever possible.
- Equipment will not be placed on potentially contaminated surfaces.
- Food and beverages will not be permitted in the exclusion zone or CRZ. Possession or use of tobacco products and application of cosmetics also are prohibited in these areas.
- Matches and lighters will not be permitted in the exclusion or CRZ zones.
- During rest periods, all personnel will be required to observe each other for signs of toxic exposure and heat stress. Indications of adverse effects include, but are not limited to, the following:
  - -- Changes in complexion and skin discoloration
  - -- Changes in coordination
  - -- Changes in demeanor
  - -- Excessive salivation and pupillary response
  - -- Changes in speech patterns
- Personnel will inform each other of nonvisual effects of illness, such as the following:
  - -- Headache
  - -- Dizziness
  - -- Nausea
  - -- Blurred vision
  - -- Cramps

-- Irritation of eyes, skin, or the respiratory tract

The following paragraphs describe safe work practices regarding avoidance of trip and fall hazards, performance of activities near utility and power lines, avoidance of excessive noise exposure, illumination, sanitation, working near bodies of water, and site housekeeping.

Avoidance of Trip and Fall Hazards

Personnel will be informed of any potential trip and fall hazards during regular health and safety meetings. Whenever possible, trip and fall hazards will be eliminated or clearly identified with yellow caution tape.

Field Activities Near Utility and Power Lines

Field activities will proceed with caution in any area where historical data or instrument surveys indicate the presence of utility lines (such as gas, telephone, water, and other lines). All field activity locations will be coordinated by the project manager.

The demonstration sites have overhead power lines in certain areas of the sites. The project manager and SHSO will be responsible for ensuring that field activities, especially drilling or probing, will not place equipment or personnel near power lines. However, if site activities near power lines are required, necessary arrangements to turn off the power will be coordinated by the project supervisor.

Illumination

Outdoor work will not be performed after sunset or when a lack of natural illumination makes outdoor work difficult.

Sanitation

Potable water, drinking cups, nonpotable water, toilet facilities, washing facilities, and other sanitation requirements will be provided in compliance with specifications of OSHA 29 CFR 1910.120(n).

Site Housekeeping

Potentially hazardous wastes generated during field activities will be drummed, if necessary, and handled in accordance with RCRA requirements. Nonhazardous waste and debris will be disposed of as standard municipal waste.

10.13.3 Health and Safety Plan Enforcement

The SHSO will be responsible for enforcement of the HASP during field sampling activities. Personnel who fail to follow HASP procedures will face disciplinary action that may, at a maximum, include dismissal from the site.

At least one copy of this HASP will be available to all personnel at all times. Any necessary changes in HASP procedures will be made at the beginning of each work day by the SHSO.

10.13.4 Complaints

Personnel will be encouraged to report to the SHSO any conditions or practices that they consider detrimental to their health or safety or that they believe are in violation of applicable health and safety standards. Such complaints may be made orally or in writing. Personnel who believe that an imminent danger threatens human health or the environment will be encouraged to bring the matter to the immediate attention of the SHSO for resolution.

10.14 DECONTAMINATION

Decontamination is the process of removing or neutralizing contaminants from personnel or equipment. When properly conducted, decontamination procedures protect personnel from contaminants that may have accumulated on PPE, tools, and other equipment. Proper decontamination also prevents transport of potentially harmful materials to unaffected areas. Personnel and equipment decontamination procedures are described in the following subsections.

10.14.1 Personnel Decontamination

Minimal personnel decontamination is anticipated at the demonstration sites because disposable PPE will be used. If necessary, personnel decontamination will be completed according to the guidance given in the "Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities" (U.S. Department of Health and Human Services 1985). Personnel and PPE will be decontaminated with potable water or a mixture of detergent and water. Liquid and solid wastes produced during decontamination will be collected and drummed.

The following decontamination procedures will be conducted if personnel decontamination is required:

- Wash neoprene boots (or disposable booties) with a Liquinox® or Alconox® solution, and rinse them with water. Remove and retain neoprene boots for reuse, if possible. Place disposable booties in plastic bags for disposal.
- Wash outer gloves in a Liquinox® or Alconox® solution and rinse them in water. Remove outer gloves and place them in a plastic bag for disposal.
- Remove the Tyvek® or Saranex® suit and place it in a plastic bag for disposal.
- Remove the air-purifying respirator, if used, and place the spent filter in a plastic bag for disposal. The filter may be changed daily or at longer intervals, depending on the use and application. Clean and disinfect the respirator with towelettes or a non-phosphate cleaning solution. Place it in a plastic bag for storage.
- Remove inner gloves and place them in a plastic bag for disposal.
- Thoroughly wash hands and face with water and soap.

Used, disposable PPE will be collected in plastic bags and placed in fiberboard drums and disposed of as municipal waste, unless otherwise specified. Further personnel decontamination procedures may be established as needed.

10.14.2 Equipment Decontamination

Decontamination of all nondisposable sampling and field monitoring equipment used during field activities will be required. The equipment decontamination procedures described in the following paragraphs are based on guidelines appropriate for low-level contamination. When appropriate,

Liquinox® or Alconox® cleaning solutions and deionized water rinses will be used to decontaminate equipment. Wastewater from equipment decontamination activities will be placed in a 55-gallon drum. A representative sample will be collected from the wastewater and analyzed for contaminants of concern before a decision regarding its disposal is made.

Sampling Equipment

Sampling equipment, such as stainless-steel spades, spoons, and stainless-steel or aluminum pans, will be decontaminated before and after each use. Potable water will be used for the following sampling equipment decontamination procedures:

- Scrub the equipment with a brush in a bucket containing Liquinox®, or Alconox® solution and potable water.
- Triple-rinse the equipment with water, and allow it to air dry.
- Reassemble the equipment and place it in a clean area on plastic.

Field Monitoring Equipment

This equipment will be cleaned daily by wiping it with isopropyl alcohol. Also, the equipment will be kept in a bag to minimize the exposure to contaminants.

10.15 EMERGENCY CONTINGENCY PLANNING

The SHSO will be notified of any on-site emergencies and will be responsible for ensuring that appropriate emergency procedures are followed. Standard emergency procedures to be used by personnel are described in the following subsections. All subcontractors, developers, and visitors will be informed about emergency procedures and the location of the nearest hospital. A copy of this HASP will be available to all personnel before field work begins.

10.15.1 Injury in the Exclusion or Contamination Reduction Zones

In the event of an injury in the exclusion or CRZ zones, all personnel will exit the exclusion zone and assemble at the decontamination line, and the SHSO will be immediately notified if necessary. The SHSO will contact the HSD, and together they will evaluate the nature and extent of the injury. The affected person will be decontaminated to the extent practical before being moved to the support zone. Appropriate first aid procedures will be performed, an immediate request for an ambulance will be made (if necessary), and the designated medical facility will be notified (if necessary). Emergency numbers for each site are provided in Section 10.16.5. No personnel will re-enter the exclusion zone until the cause of injury or illness is determined and re-entry is considered safe. In case of severe injury, the SHSO will implement procedures to minimize the possibility of further injury. If the need to transport the patient to a medical facility supersedes the need to decontaminate the patient, the medical facility will be notified that the patient has not been decontaminated before the patient arrives. Documentation requirements are outlined in Section 10.10.2.

10.15.2 Injury in the Support Zone

If an injury occurs in the support zone, the SHSO will be notified immediately. Appropriate first aid will be administered and, if necessary, the injured individual will be transported to the designated medical facility. If the injury does not affect the safety or performance of site personnel, operations will continue. Documentation requirements are outlined in Section 10.10.2.

10.15.3 Fire or Explosion

In the event of a fire or explosion at the site, the local fire department will be contacted as soon as possible, and an evacuation of the site will begin immediately.

10.15.4 Protective Equipment Failure

If personnel in the exclusion zone experience a failure of protective equipment that affects his or her personal protection, all personnel will immediately leave the exclusion zone. Re-entry to the exclusion

zone will not be permitted until the protective equipment has been repaired or replaced and the cause of equipment failure has been determined and is no longer considered a threat.

10.15.5 Emergency Information Telephone Numbers

The following emergency telephone numbers are applicable to the demonstration sites:

Police	911
Ambulance	911
Fire Department	911

The following national emergency contacts are applicable to the demonstration sites:

Poison Control Center:	1 (800) 822-3232
National Response Center:	1 (800) 424-8802
CHEMTREC Chemical Transportation Emergency Center:	1 (800) 424-9300

10.15.6 Hospital Route Directions

Before performing any field activities, demonstration personnel will conduct a pre-emergency hospital run from each site to the hospital. Directions to the nearest hospital from each site appear below:

<u>Albert City Site</u>

Go south on Highway (HWY) 197 from Albert City to HWY 3. Go west (right) on HWY 3 approximately 10 miles to HWY 71 South. Go south (left) on HWY 71 approximately 9 miles to enter the City of Storm Lake. Once in Storm Lake, continue south on Lake Avenue until you come to 5th Street. Turn west (right) on 5th Street and continue on until you reach 1525 W. 5th Street, Buena Vista County Hospital.

Chemical Sales Company Site

Go south on Interstate 270 to Interstate 70 west. Go west on Interstate 70 to HWY 2 (Colorado Boulevard). Turn south (left) on Colorado Boulevard to Martin Luther King Boulevard. Turn west (right) on Martin Luther King Boulevard to South University Boulevard. Turn south (left) on South University Boulevard. Presbyterian-Denver Hospital will be on the right side of the road.

TABLE 10-1

ACUTE SYMPTOMS FOR CHEMICALS OF CONCERN AT THE DEMONSTRATION SITES

CHEMICAL	ACUTE SYMPTOMS
PCE	eye, nose, and throat irritant; nausea; flush face, neck; dizzy; headache; vertigo; sleepiness
TCE	eye, skin irritant; headache; vertigo; fatigue; giddiness; tremor; nausea, vomit; sleepiness
1,2-DCE	eye irritant; respiratory system; central nervous system; depression
1,1,1-TCA	eye, nose irritant; central nervous system; depression;
Vinyl Chloride	weakness; abdominal pain; frostbite; gastrointestinal bleeding

Notes:

PCE Tetrachloroethylene
TCE Trichloroethylene
DCE Dichloroethylene
TCA Trichloroethane
DCA Dichloroethane

CHAPTER 11
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APPENDIX A

PRC SOGs

Volatile Organic Compounds in Soil Gas, Water, Soil and Weight Balance Operation

STANDARD OPERATING GUIDELINE

PRC ENVIRONMENTAL MANAGEMENT, INC.
ON-SITE LABORATORY
SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION

VOLATILE ORGANIC COMPOUNDS IN SOIL GAS, SOIL, AND WATER

METHOD 001

INITIAL ISSUE

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1.0 SCOPE AND APPLICATION

This document serves as the standard operating procedure (SOP) for the analytical method used to determine the concentrations of volatile organic compounds (VOC) in soil, water, and soil gas for the evaluation of measurement and monitoring technololgies during demonstrations under the Superfund Innovative Technology Evaluation (SITE) program. The method is designed to be used at an on-site laboratory to give definitive, accurate results. Those results then can be used to guide evaluate innovative technologies. Several classes of VOCs can be determined with this method, including halogenated VOCs (XVOC), non-halogenated VOCs, and volatile petroleum hydrocarbons (VPH).

This method is similar to the method SW-846 5021, 8000, 8010, 8015, and 8020 in the U.S. Environmental Protection Agency (EPA), Office of Solid Waste and Emergency Response, “Test Methods for Evaluating Solid Waste.” (EPA 1986). This method incorporates the screening protocols presented in the SW-846 method, and refines that method to include the identification and quantification of VOCs in soil, water, and soil gas using a single column and dual detector system to provide analyte identification and quantitation.

This method is designed to be used in an on-site laboratory (OSL) to provide definitive, accurate results that can be used to evaluate measurement and monitoring technologies during SITE demonstrations. It is a performance-based method, meaning that each time this method is used, performance of the method will be evaluated to determine data quality. Data derived from this method may be used for specific SITE demonstration purposes that will vary depending on the quality of data generated. The minimum level of data quality that can be expected through the use of this method is screening with definitive confirmation as defined in the EPA guidance document “Data Quality Objectives Process for Superfund” (EPA 1993). The level of data quality generated by this method is determined through the use of quality control (QC) samples to document the accuracy and precision of the method and on the comparability of analytical results produced through the use of this method to analytical results produced from formal laboratory analysis of the same samples using EPA-approved analytical methods. Table 1 lists the VOCs which can be detected using this method. It also lists the method reporting limits (MRL) for each VOC in water, soil, and soil gas.

2.0 SUMMARY OF METHOD

This VOC SOP details how to use a gas chromatograph and other related equipment to determine parts per billion (ppb) levels of VOCs in soil, water, and soil gas. The soil and water analyses are completed by combining automated headspace sampling with gas chromatography (GC) analysis. The soil gas analysis is accomplished by a direct injection GC method. Quantitation and identification are based on relative peak areas and relative retention times using the external standard method.

2.1 SOIL AND WATER METHOD

A measured amount of a soil or water sample is placed into a headspace vial. The vials are sealed and allowed to equilibrate in the headspace analyzer at a constant temperature at or near the boiling point of the target analyte. Water samples must not be heated above 95ºC to prevent sample loss and equipment damage. A headspace sample is automatically withdrawn from the headspace and injected onto a temperature-programmed gas chromatograph typically equipped with a megabore capillary column and a packed or capillary injection port. The GC megabore capillary column effluent is directed through an electron capture detector (ECD) then to a flame ionization detector (FID) connected in series. To produce definitive confirmation results a dual column (i.e. DB-634/VRX capillary column made by J&W Scientific) connected to two ECDs for XVOC identification and quantitation or two FIDs for non-halogenated aromatic and VPHs identification and qauntitation. A photoionization detector (PID) may be used in place of an FID. An electrolytic conductivity detector (ELCD) may also be used in conjunction with the PID, FID, or ECD. The ELCD is halogenated compound specific and provides better resolution of vinyl chloride than the ECD. Sample peak patterns and peak responses are then compared to VOC standards to identify and quantitate the concentration of VOCs in the sample. The PID is used to detect aromatic and nonhalogenated VOCs, and the ECD is used to detect XVOCs. The detectors may be connected in series. The ECD and FID can be connected in series with the FID second in series due to its destructive nature. If this is not possible, the column flow can be split at the detection end of the capillary column into each defector using a “Y” fused silica connector in combination with a guard column. However, the splitting of the column at the detector end will not produce definitive confirmation of analytes.

2.2 SOIL GAS METHOD

A soil gas sample is collected in a 1-liter Tedlar bag or a 250-mL gas sampling bulb. A specific volume of gas is withdrawn from the Tedlar bag or sampling bulb with a ground glass syringe and the volume is directly injected onto a temperature-programmed gas chromatograph typically equipped with a megabore capillary column. The GC megabore capillary column effluent is directed through an ECD, FID, ELCD, or PID or combination of them all. Sample peak patterns and peak responses are then compared to VOC standards to identify and quantitate the concentration of VOCs in the sample. The PID and FID are used to detect aromatic and nonhalogenated VOCs, and the ECD and ELCD are used to detect halogenated VOCs. The detectors may be connected in series. The ECD and FID can be connected in series with the FID second in series due to its destructive nature. If this is not possible, then the column flow at the detector end can be split into each detector using a "Y" fused silica connector in combination with a guard column. Dual column confirmation can also be accomplished as discussed in the previous section.

3.0 INTERFERENCES

Interferences may be introduced through such means as carrier gas contamination, carryover from extremely contaminated samples, and sample matrix effects. Potential for carrier gas contamination is reduced by using ultra-high purity (UHP 99.999 percent) carrier gases. The use of carrier gas filters also will help eliminate these problems. The analysis of highly contaminated samples followed by the analysis of cleaner samples frequently results in sample carryover contamination. Sample carryover contamination can be reduced by using good laboratory techniques. This may include baking glassware, such as soil-gas sample syringes and headspace vials, in a laboratory oven before and between usage. If sampling bulbs are used for soil gas collection, they also must be decontaminated between sample collection. This is accomplished by heating the bulbs in an oven at 100℃ while pulling clean air through them with a vacuum pump. Furthermore, laboratory reagent blanks are used to demonstrate the analytical system is free from contamination.

The use of syringes and other sample handling equipment containing Teflon® should be avoided. Some VOCs, especially 1,1,1-trichloroethane, are strongly sorbed to Teflon® surfaces. This may cause sample carryover contamination. All syringes should be properly decontaminated by flushing with a solvent such as hexane or methanol and by drying in an oven at 100ºC.

TABLE 1

TARGET COMPOUND LIST AND MRLs FOR VOCs IN SOIL, WATER, AND SOIL GAS

(Sheet 1 of 2)

Volatile Organic Compound	CAS Registry Number	MRL[a] Soil Gas (ng/L)[b]	Soil (μg/kg)	Water (μg/L)
Benzene	71-43-2	25	5	1.0
Bromodichloromethane	75-25-4	1.0	5	0.5
Bromomethane (Methyl Bromide)	74-83-9	100	5	5
2-Butanone (Methyl Ethyl Ketone)	78-93-3	25	5	5
Chlorobenzene	108-90-7	25	5	5
(2-Chloroethoxy)ethene (2-Chloroethyl Vinyl Ether)	110-75-8	NR	NR	NR
Chloroethane	75-00-3	NR	NR	NR
Chloroethylene (Vinyl Chloride)	75-01-4	100	20	20
Carbon Tetrachloride	56-23-5	1.0	5	0.10
Dibromochloromethane	124-48-1	1.0	5	0.5
1,2-Dichlorobenzene	95-50-1	25	5	5
1,3-Dichlorobenzene	541-73-1	25	5	5
1,4-Dichlorobenzene	106-46-7	25	5	5
1,1-Dichloroethane (Ethylidene Chloride)	75-34-3	25	5	5
1,2-Dichloroethane (Ethylene Dichloride)	107-06-2	25	5	5
1,1-Dichloroethene (Vinylidene Chloride)	75-35-4	25	5	5
trans-1,2-Dichloroethene (Acetylene Dichloride)	540-59-0	25	5	5
Dichloromethane (Methylene Chloride)	75-09-2	25	10.0	10.0
1,2-Dichloropropane (Propylene Dichloride)	78-87-5	NR	NR	NR
1,3-Dichloropropene (Dichloropropylene)	542-75-6	NR	NR	NR
o,m,p-Dimethylbenzene (Xylenes)	1330-20-7	25	10	1.0
1,4-Dioxane	123-91-1	1	50	50
Ethylbenzene	100-41-4	25	5	1.0
2-Hexanone	591-78-6	NR	NR	NR
Methylbenzene (Toluene)	108-88-3	25	5	1.0

Notes:

MRL Method reporting limits
NR Not reported by this method

[a] Specific method reporting limits are highly matrix dependent. These quantitation limits are provided for guidance and may not always be achievable. Also, they are calculated on an as-received basis

[b] Soil gas concentrations are reported as nanograms/liter of air. One liter of air weighs approximately one gram; therefore, on a weight basis this is equivalent to parts per billion

TABLE 1

TARGET COMPOUND LIST AND MRLs FOR VOCs IN SOIL, WATER, AND SOIL GAS

(Sheet 2 of 2)

Volatile Organic Compound	CAS Registry Number	MRL[a] Soil Gas (ng/L)[b]	Soil (μg/kg)	Water (μg/L)
Methyl tert-Butyl Ether	1634-04-4	25	5	5
4-Methyl-2-Pentanone (Methyl Isobutyl Ketone)	108-10-1	NR	20	20
2-Propenal (Acrolein, Acrylaldehyde)	107-02-8	100	20	20
2-Propenenitrile (Acrylonitrile)	107-13-1	100	20	20
Styrene	100-42-5	NR	NR	NR
1,1,1,2-Tetrachlorethane	630-20-6	1.0	5	1.0
1,1,2,2-Tetrachloroethane	79-34-5	1.05	5	1.0
Tetrachloroethene (Tetrachloroethylene)	127-18-4	1.0	5	0.25
Tribromoethane (Bromoform)	75-25-2	100	5	2.5
1,1,1-Trichloroethane (Methyl Chloroform)	71-55-6	1.0	5	0.5
1,1,2-Trichloroethane (Vinyl Trichloride)	79-00-5	1.0	5	1.5
Trichloroethene (Trichloroethylene)	79-01-6	1.0	5	0.5
Trichlorofluoromethane	75-69-4	1.0	5	0.10
Trichloromethane (Chloroform)	67-66-3	1.0	5	0.5
Unleaded Gasoline			1,000	1,000
Diesel Fuel			10,000	1,000
Jet propellant (JP)-4			10,000	1,000
JP-5			10,000	1,000
JP-8			10,000	1,000
Commeercial Jet Fuel A			10,000	1,000

Notes:

NR Not reported by this method

[a] Specific method reporting limits are highly matrix dependent. These quantitation limits are provided for guidance and may not always be achievable. Also, they are calculated on an as-received basis

[b] Soil gas concentrations are reported as nanograms/liter of air. One liter of air weighs approximately one gram; therefore, on a weight basis this is equivalent to parts per billion

Also, the presence of some VPHs (i.e. gasoline or diesel fuel) will produce interference with non-halogenated aromatics such as benzene, toluene, ethyl benzene, and xylenes (BTEX). This problem can be compensated for by the use of dual column confirmation.

4.0 APPARATUS AND MATERIALS

The analytical system used for this VOC method is described in the following sections.

4.1 ANALYTICAL INSTRUMENTATION

The analytical equipment required for the VOC method includes a headspace autosampler, GC, columns (dual or single), detectors, carrier gases and gas purifiers. Details on each piece of equipment are given in the following sections.

4.1.1 Headspace Autosampler

The Tekmar 7000 Headspace Analyzer (HSA) and the Tekmar 7050 sample carosel or equivalent system is used for the VOC analysis of soil and water samples. The HSA is a temperature programmable system. Samples are heated to a predetermined temperature in the sample carousel and automatically injected onto the GC column via a heated valve and transfer line. A 1-mL sample loop typically is used for most analyses, but larger volume loops (2 ml or 5 ml) may be utilized to lower detection limits. However, the use of larger volume loops may decrease the precision of the method.

4.1.2 Gas Chromatograph

The GC typically used for the analysis of VOCs using this method is a Hewlett Packard (HP) - 5890 Series II, or equivalent. This GC is temperature programmable and includes heated injector ports and detector ports and electronic pressure control (EPC). The injectors used are designed for packed columns but are modified to accept megabore capillary columns. A capillary injection may be used in place of a packed injection port. Typically the capillary injection port will provide a narrower retention

time window. The GC includes pressure valves and flow control valves for the precise delivery of carrier gases. GC data is collected using a data acquisition software system loaded on to a personal computer that is compatible with the GC used. The data system stores "real-time"chromatographic data and is able to integrate peak areas and peak heights. Chromatogram comparisons and baseline correction also can be performed.

The GC also includes carrier gas connection fittings, column connections and fittings, carrier gas leak detectors, septums, syringes, and all other required accessories. The GC will also need to be equipped with an adapter kit (from Tekmar) for the transfer line from the HSA to be plumbed correctly into the injection port of the GC.

4.1.3 Columns

The following analytical columns are recommended for the analysis of VOCs in soil, water, and soil gas. The first three are megabore capillary columns; the fourth is a packed column. The DB-624 is the recommended column for analysis of the VOCs listed in Table 1. Columns other than those listed below may be used. The analyst should ensure that these columns provide resolution and performance sufficient to meet the needs of the specific project.

Column 1: DB-624, 30-meter (m) by 0.53-millimeter (mm) or 0.45-mm I.D. (inner diameter) column coated with 1.4 µm of the liquid phase. (J&W Scientific)

Column 2: DB-624, 75-m by 0.53-mm I.D. (inner diameter) column coated with 1.4 µm of the liquid phase. (J&W Scientific)

Column 3: DB-VRX, 75-m by 0.45-mm I.D. column coated with 2.55 μm of the liquid phase. (J&W Scientific)

Column 4: DB-624/VRX (dual column), 75-m by 0.45-mm I.D column coated with 2.55 μm of the liquid phase. (J&W Scientific)

Column 5: DB-502.2, 105-m by 0.53-mm I.D. column coated with 3.0 μm of the liquid phase. (J&W Scientific)

Column 6: DB-502.2, 75-m by 0.45-mm I.D. column coated with 2.55 μm of the liquid phase. (J&W Scientific)

Other suppliers which manufacturer comprable columns include Hewlett-Packard, Restek, and Supelco, Inc.

4.1.4 Detectors

The detectors used for the analysis of VOCs are a FID and an ECD. A FID is used to detect aromatic and nonhalogenated VOCs. An ECD is used to detect halogenated VOCs. The FID and ECD are operated at a temperature of 300ºC. Detector range and attenuation are set according to manufacturers recommendations and must provide an adequate signal to determine concentrations of target compounds equal to the MRL listed in Tables 1. A make-up carrier gas is supplied directly to the detectors to stabilize the detector signal.

A PID may be used in place of the FID or ECD and the ELCD in place of the ECD for select compounds. The ELCD will provide better resolution and separation of solvents with low boiling points (i.e. vinyl chloride). For dual column confirmation either two ECDs or two FIDs must be used depending on the target analytes. The FID and ELCD cannot be used in combination with each other using a “Y” splitter at the detector end. The ELCD causes enough back pressure that all of the column flow will flow toward the path of least resistance, which is to the FID.

4.1.5 Carrier Gases And Gas Purifiers

The use of ultra-high purity (UHP) carrier gases is recommended. UHP nitrogen is the preferred carrier gas. Helium also may be used. The detector make-up gas is the same as the carrier gas.

Gas purifiers are recommended to further improve the quality of the carrier gas. A water and oxygen trap may be used (Supelco's OMI-1 purifier). A hydrocarbon trap also may be used (Supelco's HC purifier). These purifiers should be routinely changed as recommended by the manufacturer.

Flame gases for the FID are air and hydrogen. These gases are regulated through restrictors to proportion the ratio of each gas reaching the detector. Extreme caution should be employed with the use of hydrogen due to its extreme flammability. All connections should be checked with a leak detector to ensure that no leaks exist. This is especially true when the instrument is to be operated while unattended.

4.2 LABORATORY EQUIPMENT

Other associated laboratory equipment used during sample preparation and analysis is described below.

- Hamilton Syringes: 10-µL (5 minimum) for working standard preparation
- Micro Pipettes: 10-, 50-, and 100 μL (Drummond or equivalent)
- Ground Glass Syringes: 2- and 5-mL volumes for direct injection of soil gas samples
- Gas Sampling Bulbs: 250-mL glass bulbs for collection of soil gas samples
- Tedlar Bags: 1-liter Tedlar gas sampling bags for collection of soil gas samples (Option for glass bulbs)
- Vials: 2-mL glass with Teflon®-lined cap for storage of stock standards and calibration standards
- Pasteur Pipets: 5.75- and 9-inch sizes, disposable glass
- Volumetric Pipets: 1-, 10-, and 25-μL
- Headspace Vials: 22-mL vials with Teflon®-lined septa and crimp caps
- Crimper Pliers: used to securely fasten crimp tops to headspace vials
- Stainless-steel spatulas
- Top Loader Balance: 0.01-gram accuracy
- Drying Oven: 200°C limit
- Solvent Squeeze Bottles: 500-mL, for cleaning spatulas and other lab-supplies
- Pipet Bulbs: both large and small sizes to fit Volumetric and Pasteur pipets
- Bubble Flow Meter: used to check GC column flows
- Labels: assorted sizes, for marking sample and standard containers
- Sharpie Markers: black, for labeling sample and standard containers
- Paper Towels: for general cleanup uses
- Gloves: polypropylene or latex, for personal protection
- Safety Glasses: for personal protection
- 10 mL and 100 mL volumetric flasks
- Ice box or cooler with blue ice to store standards and samples separately
- Fused silica “Y” splitters for splitting flow
- Fused silica guard column for splitting flow
- Packed or capillary injection port glass inserts
- Graphite ferrules
- 11mm septa

5.0 REAGENTS

The reagents needed for sample extraction and analysis are described below.

5.1 SOLVENTS

Solvent extraction is not employed for the headspace analysis of soil or water samples for VOCs. Some common laboratory solvents may be used for preparation of calibration standards for VOC analysis. These solvents are described below:

- Hexane: pesticide quality or equivalent
- Acetone: pesticide quality or equivalent
- Methanol: purge-and-trap grade or equivalent
- Methylene Chloride: pesticide quality or equivalent

5.2 SAMPLE PREPARATION REAGENTS

The following reagents may be used in the preparation of soil samples for VOC analysis.

- Sodium Sulfate: granular, pesticide quality or equivalent
- Deionized Water: organic free grade water or equivalent, for preparation of reagent blanks
- Sand: purified, preanalyzed sand for the preparation of reagent blanks

5.3 STOCK STANDARD SOLUTIONS

Stock standards must be purchased from reputable vendors for the VOCs listed in Table 1. Standards for individual analytes may be purchased, or commercial or custom multicomponent standards may be used. These standards come with a certificate of analysis stating the purity of each component.

Typically a custom mix is obtained from Ultra Scientific for the analysis of multi chloride VOCs. Appendix A lists the compounds and their corresponding concentrations. The standards are in a solution of methanol with VOC concentrations of 100 µg/mL to 2,000 µg/mL. Diesel Fuel No. 2 and JP stock standards may be in a solution of methylene chloride or hexane. The standards are received in sealed ampules. When preparing the calibration standards, the ampule is broken and the needed amount is withdrawn. The remainder of the stock standard solution is then transferred to a 2-mL glass vial with a Teflon-lined screwcap.

Stock standards are to be stored at 4°C away from light in glass amber containers. Stock standards should be checked frequently for signs of degradation or evaporation. Stock standards should be replaced after six months. They should be replaced sooner if comparisons with check standards indicate a problem with the stock standards.

Additionally, for soil gas analysis commercially available gas standards may be used as stock standards. Only a limited number of compounds are currently available in these standards. The initial cost of the gas standards is high, but they maintain their integrity, lasting for up to two years.

5.4 CALIBRATION STANDARD SOLUTIONS

Calibration standards are prepared from the stock standard solutions at a minimum of six levels for each VOC of interest. The lowest level calibration standard for each matrix should correspond to the MRLs presented in Tables 1. The other calibration standards should define the linear range of the GC for the analytes of interest. Typically this range encompasses one order of magnitude of concentration. The concentration of the standards will vary depending on the VOC of interest as indicated by the MRLs listed in Table 1. If site-specific data quality objectives require otherwise, other calibration ranges may be used.

Typically separate standard solutions are made for benzene, ethyl benzene, toluene, and xylenes (BTEX), chlorinated VOCs, and the individual aliphatic petroleum hydrocarbons. BTEX and chlorinated VOC standard solutions are typically separated because of the co-elution of 1,2-dichloroethane and benzene on

the FID. Also Diesel Fuel No. 2 and JP working standards must be prepared in a solution of hexane because they are insoluble in methanol.

Calibration standards are stored at 4°C away from light in glass amber vials. Calibration standards should be checked frequently for signs of degradation or evaporation, especially prior to usage. Calibration standards should be replaced every six months. They should be replaced sooner if comparisons with check standards indicate a problem.

If the gas stock standards are used for calibration, the appropriate volumes of the standard are directly injected onto the GC column for analysis. The volumes injected should define the MRLs of the analytes of interest and the linear range of the GC for these compounds.

5.5 SURROGATE STANDARD SOLUTIONS

The OSL VOC method for soil and water samples can be monitored for the performance of the headspace extraction and the analytical system by using a surrogate standard. The surrogate standard of choice for chlorinated VOC analysis is cis-1,3-dichloropropene and alpha, alpha, alpha,-trifluorotoluene (∝,∝,∝-TFT) for BTEX and TPH analysis. If used, the surrogate standard should be added to all standards, method blanks, samples, and MS/MSD samples. Several other surrogate standards may be used for this method including 2-bromo-1-chloropropane (2BCP), bromochloromethane, 1, 4-dichlorobutane, and 4-bromofluorobenzene. The surrogate standard used will depend on the VOC analytes of interest. A stock standard of individual surrogates or a mixture of the surrogates is purchased from a reputable vendor and treated as all stock standards (see Section 5.3). The surrogate standard is added to all soil standards at the 100 µg/kg level and water standards at the 100 µg/L level. A dilution of the surrogate stock solution is made to produce a surrogate spike solution to add to the samples. This surrogate spike solution is prepared so that when it is added to a sample during the extraction process it will yield a concentration of 100 µg/kg or 100 µg/L. The concentration of the surrogate in each sample is compared to the concentration of the surrogate standard in the calibrating standards to determine the performance of the headspace analyzer and the analytical system.

Surrogate spikes may also be used when analyzing soil gas samples. The appropriate amount of the surrogate is injected into the soil gas sample bulb or Tedlar bag and allowed to equilibrate. The spike concentration should be within the calibration range.

Surrogate standard solutions are to be stored at 4°C away from light in glass amber vials. Surrogate standard solutions should be checked frequently for signs of degradation or evaporation. The surrogate standard solutions should be replaced every six months, or sooner if comparisons to standards indicate a problem.

6.0 SAMPLE COLLECTION, PRESERVATION, HANDLING, AND DOCUMENTATION

Sample collection, preservation, handling, and documentation are described in the following sections.

6.1 SAMPLE COLLECTION

Soil and water samples will be collected in 40-mL glass vials with Teflon-lined caps. Two vials will be filled for each sample, and four vials will be filled when a sample and duplicate are required. The maximum amount of sample required for analysis is 10 grams of soil or 10 mL of water. The extra sample is collected for quality control samples and in case the sample leaks or container breaks.

Soil gas samples will be collected in either a 250-mL gas sampling bulb, a 1-liter Tedlar bag, or a 40-mL VOA vial. Only one gas sampling bulb or Tedlar bag is required for each sample since multiple aliquots may be withdrawn for duplicates or quality control samples. The amount of sample required for analysis is approximately 10 mL.

6.2 SAMPLE PRESERVATION

All soil and water samples are stored at 4°C in a refrigerator or in a cooler on ice. No chemical preservatives are required for VOC analyses because the analyses are generally accomplished within 24 hours of collection. The holding time for unpreserved VOCs in soil or water is 7 days. Since this

method is designed to provide results quickly, holding times should not be exceeded. If water samples are to be held longer than 48 hours or to be shipped to the laboratory, hydrochloric acid should be added to each vial until pH is less than 2.

Soil gas samples should be stored at room temperature away from sunlight because some VOCs degrade in sunlight. The holding time for VOCs in soil gas is 12 hours. Since this method is designed to provide results quickly, holding times should not be exceeded.

6.3 SAMPLE HANDLING

Many VOCs are suspected carcinogens or are otherwise hazardous to health. Care should be taken when collecting, handling, extracting, or analyzing samples. The health and safety plan should be closely examined and adhered to by all sample collection personnel. This plan should be followed at all times by anyone involved with any portion of the project. Safety equipment, including safety glasses, gloves, lab coats, and chemical resistant clothing, should be worn at all times when handling samples.

6.4 SAMPLE DOCUMENTATION

Sample locations, numbers, times, and dates will be recorded in a field logbook at the time of collection. Project-specific sample designations should be used whenever possible. Sample numbering schemes should include a unique identifier for each sample collected and submitted to the OSL for analysis. The sample numbering scheme may be as simple as the following: Soil sampling may be numbered beginning with SS-001, water samples will be numbered beginning with W-001. Soil gas samples will be numbered beginning with SG-001. Field duplicate samples will be assigned a "FD" suffix, laboratory duplicate samples with a “LD” suffix, and field blank samples will be assigned a "B" suffix. A “D” suffix will be used when samples are diluted and analyzed. This numbering scheme can be changed on a project specific basis. Regardless of the numbering nomenclature used, each sample collected should have a unique sample number.

Analytical results from the use of this method will be documented in a logbook or on sample result forms and in electronic form in demonstration specific database. This documentation will include all calibration results, QC sample results, corrective actions, and sample results. This summary will include a description of the method used for analysis, QC sample results, sample results, and an evaluation of the accuracy precision and comparability of the analytical data. This data assessment report is usually incorporated into an environmental investigative report.

An analytical logbook will be kept to record the samples turned over to the on-site laboratory. This logbook will be reviewed to ensure that all samples received were analyzed within the required holding times. The logbook will be electronically generated using bar code labels assigned to each sample collected in the field. Once the sample enters the OSL it will be scanned and logged electronically. At the end of each working day the field chemist will check the samples logged in and make sure they are analyzed within an appropriate time.

7.0 SAMPLE PREPARATION AND ANALYSIS PROCEDURE

The following sections describe the preparation procedures for soil, soil gas, and water samples.

7.1 SOIL AND WATER SAMPLE PREPARATION

The soil and water sample preparation for VOCs is as follows:

1. For soil analysis, weigh 5 grams (+/- 0.1 grams) of soil and place it into a tared and labeled 22-mL headspace vial. For water analysis, volumetrically pipet 5 mL of sample to the 22-mL headspace vial. Sample mass or volume may be increased to attain lower detection limits or decreased for high concentration samples. The same mass or volume should be used for standards and samples.

2. Add surrogate spike solution to all samples, and matrix spike solution to all matrix spike and matrix spike duplicate samples.

3. Cap and crimp the vials for insertion into the headspace analyzer.

4. The sample now is ready for headspace autosampling and GC analysis.

7.2 SOIL GAS SAMPLE PREPARATION

There is no sample preparation required for the analysis of soil gas samples. The soil gas samples are collected in a gas sampling bulb, 40-mL VOA vial, or a Tedlar® bag and delivered to the mobile lab for analysis. A ground glass syringe is used to extract 2 mL (or less) of soil gas from the sample container for direct injection onto the GC column for analysis. Larger volumes of soil gas may be injected onto the column to achieve lower detection limits or smaller volumes may be injected for highly contaminated samples. The use of larger soil gas volumes for analysis may lead to more rapid deterioration of the column liquid phase and oxidation of the ECD foil.

Surrogate spikes or matrix spikes may be prepared by injecting the appropriate amount of the analytes of interest into the gas sampling bulb or Tedlar® bag. The spike concentration should fall within the instrument calibration range.

7.3 HIGH CONCENTRATION SOIL ANALYSIS

If high concentration soil samples are to be analyzed, the following sample preparation should be performed:

1. Transfer 2g of sample from the 40 mL VOA vial into a tared 22 mL sample vial and add 10 mL of methanol.

2. Mix by shaking for 10 minutes at room temperature. Decant 2 mL of methanol to a screw-top vial with Teflon®-faced septa and seal.

3. Withdraw appropriate volume of extract (Table 2) and inject into a 22 mL vial.

TABLE 2

QUANTITY OF METHANOL EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATION SOILS AND SEDIMENTS[a]

Approximate Concentration Range (μg/kg)	Volume of Methanol Extract
5,000 to 10,000	100 μL
1,000 to 20,000	50 μL
5,000 to 100,000	10 μL
25,000 to 500,000	100 μL of 1/50 dilution

[a] Table was reproduced from SW-846 Method 5021 "Volatile Organic Compounds in Soils and Other Solid Matrices Using Equilibrium Headspace Analysis," 3rd Edition, Revision 0, January 1995.

7.4 GAS CHROMATOGRAPHIC CONDITIONS

Table 3 lists the gas chromatographic conditions for each of the first four columns listed in Section 4.1.2 of the OSL VOC method.

7.5 HEADSPACE ANALYZER PARAMETERS

Table 4 lists the time and temperature parameters of the HSA for the SITE VOC method.

7.6 CALIBRATION

Calibration of the analytical system is performed by using the external calibration method. This method requires the analysis of six levels of standards for each VOC of interest. To obtain acceptable %RSD and %D values, a standard mix should be used and serially diluted for initial and continuing calibration analysis. The standards are evaluated for retention time criteria and linearity. The evaluation of the standards is performed through the initial calibration and the continuing calibration. Appendix B of this document shows chromatograms of chlorinated and aromatic VOCs.

These chromatograms were obtained from the DB-624 column. This column was attached to a packed column injection port and was operated at the conditions listed in Table 3.

7.6.1 Initial Calibration

Initial calibration (ICAL) must be performed before the analysis of any sample. The ICAL determines the retention time windows for the analytes of interest, and it establishes the linearity of the analytical system.

The ICAL begins with the analysis of six standards which define the linear range of the GC for analytes of interest. The sequence described on Table 5 of this CSL VOC method illustrates the analytical run sequence for the analysis of VOCs. Generally, the low-level standard is analyzed first, followed by the midlevel standard and finally by the high-level standard. The concentrations of these standards were

given in Section 5.4. These standards can then be used to determine the retention time windows and linearity.

This OSL VOC method is written with the intent of conducting analyses for halogenated, nonhalogenated, and aromatic VOC groups either individually or simultaneously. Simultaneous analysis may be accomplished by connecting the PID and FID or the PID and ECD detectors in series.

Each VOC exhibits a specific retention time on the GC column used for the analysis. The detectors also exhibit varying degrees of sensitivity for each of the VOCs. The PID is very sensitive to aromatic compounds, the FID is sensitive to the aliphatic compounds, and the ECD exhibits great sensitivity to the halogenated compounds. These characteristics enable the identification of each VOC. For aliphatic petroleum hydrocarbons, a specific retention time range and pattern recognition is used to identify these compounds (Diesel fuel No. 2, unleaded gasoline, and JPs). Although, specific peaks that are unique to the multi-response VOC can be used for identification.

Retention times for each peak are determined by calculating the mean of the peak's retention time from each of the six levels of standards used in the ICAL (Equation 1). For megabore capillary columns, the retention time window is defined as plus or minus 1.5 percent from the mean retention time (Equation 2). For packed columns, the retention time window is defined as plus or minus three times the standard deviation of the peak in the three levels of standard (Equation 3). Equation 4 is that for standard deviation.

$$\text{mean RT} = (RT1 + RT2 + RT3) / 3 \qquad (1)$$

where

mean RT = mean retention time.

RT1 = retention time of the peak in the low-level standard.

RT2 = retention time of the peak in the midlevel standard.

RT3 = retention time of the peak in the high-level standard.

RT window = mean RT - (1.5 percent * mean RT), and mean RT + (1.5 percent * mean RT) (2)

TABLE 3

GAS CHROMATOGRAPHIC COLUMN CONDITIONS

GC Parameter	Columns 1 and 2	Column 3	Column 4
Injector Temperature	220°C	220°C	220°C
PID Temperature	200°C	200°C	200°C
FID Temperature	300°C	300°C	300°C
ECD Temperature	300°C	300°C	300°C
Column Flow Rate	10 mL/minute	10 mL/minute	10 mL/minute
Make-up Flow Rate	50 mL/minute	50 mL/minute	50 mL/minute
Initial Column Temperature	50°C	50°C	50°C
Initial Hold Time	0.5 minutes	0.5 minutes	0.5 minutes
Ramp Rate (1)	6°C/minute	6°C/minute	6°C/minute
Final Temperature (1)	145°C	145°C	145°C
Final Hold Time (1)	0.0 minutes	0.0 minutes	0.0 minutes
Ramp Rate (2)	60°C/minute	60°C/minute	60°C/minute
Final Temperature (2)	200°C	200°C	200°C
Final Hold Time (2)	0.5 minutes	0.5 minutes	0.5 minutes
Ramp Rate (3)	60°C/minute	60°C/minute	60°C/minute
Final Temperature (3)	220°C	220°C	220°C
Final Hold Time (3)	0.75 minutes	0.75 minutes	0.75 minutes
Sample Injection Volume[a]	2 mL	2 mL	2 mL

Notes:

Column 1 = DB-624, 30m length, J&W Scientific
Column 2 = DB-624, 75m length, J&W Scientific
Column 3 = DB-VRX, J&W Scientific.
Column 4 = DB-624/VRX, J&W Scientific.

Gas chromatographic conditions may need to be optimized for each GC and analytical column used. These conditions are given only as guidelines.

[a] For headspace analysis of soil and water, the headspace sample loop controls the volume of gas injected. For analysis of soil gas, ground glass syringe is used to control the volume of gas directly injected.

TABLE 4

HEADSPACE AUTOSAMPLER PARAMETERS

HSA Parameter	Setting
Platten Temperature	70° C
Platten Equilibration	10 minutes
Sample Equilibration	20 minutes
Valve Temperature	150° C
Transfer Line Temperature	200° C
Mixer time	1.0 minute
Mixer power setting	3
Equilibration time	10 minutes
Vial pressurization	1.0 minute
Pressure equilibration	0.5 minute
Loop fill	0.5 minute
Sample inject	1.0 minute

Note:

HSA parameters may need to be optimized for each HSA - GC system. These parameters are given only as guidelines.

TABLE 5

ANALYTICAL RUN SEQUENCE FOR VOCs

- Method blank
- VOC standard 1, low-level[1]
- VOC standard 2
- VOC standard 3, mid-level
- VOC standard 4
- VOC standard 5, high-level
- System blank (consist of empty vial)
- Sample Analysis (may continue for up to 12 hours, and may include any number of system blanks to reduce the risk of carryover from contaminated samples)
- System blank
- Continuing Calibration: VOC standard 3, midlevel[2]
- System Blank
- Sample Analysis (may continue as long as continuing calibration is acceptable)
- System blank
- Sequence may continue provided that the continuing calibration is acceptable. If continuing calibration is unacceptable a new initial calibration must be performed before sample analysis can continue.

Notes:

[1] The VOC standards will consist of site-specific analytes of interest. Commercially available multicomponent standards, custom multicomponent standards, or individual analyte standards may be used as required.

[2] Continuing calibration is performed at least once every 12 hours or 12 samples.

where

RT window = the area of a chromatogram where an analyte peak may elute and be considered the analyte.

mean RT = the mean retention time of a peak as determined from Equation 1.

$$\text{RT window} = \text{mean RT} - (3 \times \text{SD}), \text{ and mean RT} + (3 \times \text{SD}) \quad (3)$$

where

RT window = the area of a chromatogram where an analyte peak may elute and be considered the analyte.

mean RT = the mean retention time of a peak as determined from Equation 1.

SD = the standard deviation of the retention time as determined by Equation 4.

$$\text{SD} = [\Sigma\, (\text{RT}^n - \text{mean RT})^2) / (n - 1)\,]^{1/2} \quad (4)$$

where

Σ = the sum of.

RT^n = each of the three peak retention times.

mean RT = the mean retention time of a peak as determined from Equation 1.

n = the number of RT^ns used in this calculation (3).

½ = square root.

Alternatively, the analyte retention time window may be defined using the integrator computer software program. The average retention time of each analyte is calculated from the analysis of the six standards. This retention time is entered into the integrator program, and a three percent retention time window is chosen. A larger retention time window may be required for later eluting peaks.

Linearity for the VOC standards is determined in the ICAL sequence. The measure of linearity is found by evaluating the percent relative standard deviation (%RSD) of the six initial calibration factors (CF).

The CF is calculated by using Equation 5. The same peaks used for retention time identification are used to calculate the CF.

$$CF = \text{peak response} / \text{mass injected} \quad (5)$$

where

CF = calibration factor.
peak response = peak area/height readout from integrator (total area for particular retention time range for multi-response VOCs may be used in place of individual peak areas).
mass injected = nanograms (ng) injected.

The standards are determined to be linear if the %RSD is less than or equal to 20 percent for all compounds except vinyl chloride (25 percent). %RSD is determined from Equation 6.

$$\%RSD = (SD / \text{mean } CF) \times 100 \quad (6)$$

where

%RSD = Percent relative standard deviation.
SD = Standard deviation of the three calibration factors.
mean CF = Mean calibration factor.

High %RSD values can be due to degradation, evaporation of the calibration standards degradation or breakdown of the VOC in hot injection port liner, or instrument problems. IF the %RSD is greater than 20 percent the low or high point of the initial calibration may be eliminated if is determined that this is the cause of the unacceptable %RSD. If the %RSD is still greater than 20 percent, the linear regression equation of a line may be used in place of the average calibration factor for sample quantitation if the correlation coefficient is greater than or equal to 0.995 for each target analyte. However, the line must be forced through zero to avoid over estimation of results near the detection limit. Standards must be compared to laboratory control samples (LCS) discussed in section 8.4, or to performance evaluation samples to determine the accuracy of the calibration as discussed in Section 8.6. If the standards are found to be the cause of the high %RSD, an evaluation of the instrument is performed to determine the cause of the high %RSD. When %RSDs above 20 percent are encountered, the instrument must be evaluated to determine the cause of the high %RSDs. However, if a PE sample has been analyzed and the results are within the acceptance ranges for all of the target compounds, the ICAL may be considered acceptable and useable for identification and quantification.

Actions which can be taken include: changing the septum, leak checking the column, cleaning the glass injection port liner, and making new standards. After the situation is corrected, a new ICAL should be performed and analyzed to ensure that the analytical system linearity is within control.

7.6.2 Continuing Calibration

Continuing calibrations (CCAL) should be performed and analyzed at a frequency of every 12 samples or 12 hours to ensure that the analytical system is in control. Continuing calibrations are performed by analyzing the midlevel standard of the VOCs of interest. The retention time windows are evaluated and adjusted as necessary. The calibration factor for each VOC is calculated to determine if instrument response is in control.

The retention time of the peak for each VOC used in the CCAL is to be examined. These individual peaks should fall within the retention time windows developed in the ICAL. If the individual peaks are outside of the retention time windows, new windows should be determined. This is performed by using the CCAL retention time as the retention time mean and using the windows found in Equation 2 or Equation 3.

The calibration factors of the CCAL VOCs are found using Equation 5. These calibration factors are compared to the average calibration factors determined from the ICAL. The percent difference (%D) is determined for each VOC by using Equation 7.

$$\%D = [(\text{avg CF} - \text{CCAL CF}) / \{\text{avg CF}\}] \times 100 \qquad (7)$$

where

%D = Percent difference.
CCAL CF = continuing calibration - calibration factor.
avg CF = average calibration factor for each VOC determined in the ICAL.

The %D for the CCAL must be within ± 15 percent for all comounds except vinyl chloride (20%) to continue sample analysis. If the %D is greater than 15 percent, the cause of the problem must be found and corrected. Once the problem has been corrected a CCAL can be analyzed to verify the validity of the

retention time windows and calibration factors from the ICAL. If the %D is still greater than 15 percent, a new ICAL must be performed and the samples analyzed after the CCAL re-analyzed..

7.7 GAS CHROMATOGRAPHIC ANALYSIS

After completing an acceptable initial calibration, sample analysis may begin. Samples are analyzed by injecting sample headspace extracts onto the GC. Sample analysis should begin with the analysis of the method blank. Samples believed to be clean should be analyzed before those believed to be highly contaminated. The analyst may not always be able to determine clean samples from highly contaminated samples. Dilutions should be performed on highly colored samples. If VOCs are not found in the dilutions, a more concentrated sample extract must be analyzed.

When highly contaminated samples cause carryover to subsequent samples, analysis must be discontinued. The system must be cleaned either through blank matrix injections or by disassembling the analytical system and solvent cleansing each component. The primary sources of contamination include the vial needle in the HSA, the septa, the glass injection port liner, the injection port, the column, the detector, the sample volume loop on the HSA, and the transfer line from the HSA to the GC. Depending on the amount of contamination, any or all of these may require cleaning. Once cleaned, the system should be reassembled and checked to ensure that no carryover contamination is present by analyzing a method blank and an instrument blank. CCALs should be analyzed to evaluate the validity of the ICAL. If CCAL requirements are not met, a new ICAL must be performed.

One method of identifying potential highly contaminated VOC samples is to have the sampling team screen each sample with a portable PID or FID (such as the Photovac Microtip, HNU Systems 101 Portable PID, or Foxboro Organic Vapor Analyzer) as each is collected. The results can be recorded on the sample label or in the logbook for the field chemist. This information may be used to determine if smaller sample mass (soil) or volume (water or soil gas) may be required to obtain results within the calibration range. MRLs for all of the VOCs will be adjusted by any dilution factor used for the sample.

When an individual sample peak elutes within the retention time window of coeluting multi-response VOCs, definitive positive identificaiton can not be performed. The sample may contain any percentage of one or both of the coeluting VOCs. Therefore false positives are reported. In this situation the analyst should follow the following procedure to determine which of the coeluting compounds is actually present:

1. Calculate the following ratios:

$$R_1 = \frac{\text{Area of multi-response peak of interest in the medium level standard}}{\text{Area of multi-response major peak (non-coeluting) in the medium level standard}}$$

$$R_2 = \frac{\text{Area of coeluting multi-response peak in the sample}}{\text{Area of multi-response major peak in the sample}}$$

2. If $R_2 > R_1$, then area of the coeluting peak in the sample is mainly due to the presence of the individual coeluting compound and not the multi-response coeluting compound.

3. Calculate the area due to the multi-response peak:

$$A_M = R_1 \times \text{Area of the major multi-response peak in the sample.}$$

4. Calculate the area of the coeluting peak due to the individual compound.

$$A_I = \text{Area of coeluting peak in the sample} - A_M$$

Each day the GC system is monitored through the use of the CCAL. Retention time windows for the VOCs are adjusted, if needed. The detector response of each of the CCAL VOCs is evaluated through the comparison of peak area or height. The CCAL standards are compared to the ICAL standards by monitoring the percent differences of the ICAL calibration factors to the CCAL calibration factors. If the CCAL data is acceptable, sample analysis may continue. If the CCAL is unacceptable a new ICAL should be performed.

7.8 CLEANUP

No sample cleanup is required; however, sodium sulfate or calcium chloride (2g) may be added to soil samples to minimize moisture collecting on the vial septa during headspace sampling.

7.9 CALCULATIONS

Sample calculations for VOCs are performed by comparing peak responses for samples to the average calibration factor found from the ICAL. The calculation for soil samples is shown in Equation 8.

$$\text{Conc.} = [(\text{peak response}/\text{avg CF}) / \text{sample mass}] \times \text{DF} \quad (8)$$

where

Conc. = concentration of VOC in the soil sample (µg/kg).
peak response = peak area/height readout from integrator.
avg CF = average calibration factor for each VOC as determined in the ICAL.
sample mass = mass of soil sample (grams) in headspace vial.
DF = dilution factor

The same equation is used for water sample calculation with the exceptions that concentration is in µg/L and sample volume (mL) is used in the calculation in place of sample mass.

If the same sample mass (soil) or sample volume (water) is used in the preparation of both the standards and samples, then the headspace volume cancels out in the equation. Also, since the headspace sample loop is a constant volume, this variable cancels out of the concentration calculations.

Soil gas concentrations are calculated by using Equation 9.

$$\text{Conc.} = (\text{peak response}/\text{avg CF}) / \text{sample volume} \quad (9)$$

where

Conc. = concentration of VOC in the soil gas sample (ng/L: where L = one liter of air).
peak response = peak area/height readout from integrator.
avg CF = average calibration factor for each VOC as determined in the ICAL.
sample volume = volume of soil gas sample (liters) directly injected onto the GC column.

Since one liter of air weighs approximately one gram, the units for soil gas concentration (ng/L) are approximately equal to parts per billion on a mass basis.

8.0 QUALITY CONTROL

Quality control for this OSL VOC method includes: equipment blanks, method blanks, surrogate spikes, matrix spike-matrix spike duplicates (MS and MSD), laboratory duplicate samples, laboratory control samples (LCS), and PE samples. Each of these QC samples is presented below.

8.1 METHOD BLANKS

Method blanks are analyzed with each batch of samples analyzed. A batch is defined as up to 20 samples of a similar matrix prepared and analyzed together. Method blanks monitor laboratory-induced contaminants or interferences. A method blank consist of a headspace vial filled with the same volume or mass as the sample matrix being analyzed and spiked with an appropriate amount of surrogate solution. To be acceptable, a method blank must not contain any analyte above the MRLs. Method blanks must not have peaks which interfere with any analyte and must not have large, broad peaks present in the chromatogram. If any of these conditions are present in method blanks, the cause of the problem must be identified, and all samples extracted and analyzed with the method blanks must be analyzed again. When method blank samples provide positive results for target VOCs, the analyst must

determine the appropriate corrective action. Corrective action may involve the qualification of the analytical data, or may involve reanalysis of all of the samples associated with the method blank sample. Generally, if the concentration of the VOC in the method blank sample is less than one-half the project-specific action level, qualification may be the only corrective action required.

In soil gas analysis, a method blank includes a decontaminated soil gas syringe blank and a decontaminated soil gas sampling bulb blank. Generally, the decontaminated glass syringe and glass sample bulb that exhibited the highest sample concentration the previous day will be used for the method blank analysis to ensure decontamination was thorough.

8.2 SURROGATE SPIKE RECOVERY

Surrogate spikes may be used to determine the headspace extraction efficiency of the SITE VOC method. Surrogates are added to all standards, blanks, samples, and matrix spikes performed. The surrogate standard for headspace VOC analysis are given in Section 5.5. The CF and retention time windows for the surrogate standards are determined during the ICAL. The CF and retention time windows are monitored daily through the CCALs. %Ds for the surrogate standards must be less than or equal to 35 percent in the CCALs. The surrogate recovery for each blank and sample is calculated by Equation 10.

$$\% R = (\text{sample amt.} / \text{spiked amount}) \times 100 \qquad (10)$$

where

% R = percent recovery.
sample amt. = amount found in the sample (µg/L, or µg/kg).
spiked amount = amount spiked into sample (µg/L, or µg/kg).

The advisory control limits for the surrogate recovery in both soil and water is 50 to 150 percent. These control limits are for advisory purposes only. If more than 25 samples of soil or water matrix are analyzed then advisory control limits (site-specific) may be calculated using equations 1 through 4. The upper and lower control limit will correspond to the 95 percent confidence interval around the

mean. If surrogate recovery is outside of the control limits, corrective action will be required. Corrective action includes reextraction and reanalysis of the samples. If surrogate recoveries are frequently outside of the control limits, preparing new surrogate spike standards or calibration standards may be necessary. Samples with surrogate recoveries outside of the advisory control limits need to be qualified as estimated (J) and explained in the final report.

8.3 MATRIX SPIKE RECOVERY

Matrix spike and matrix spike duplicate samples (MS-MSD) are used to evaluate the extraction efficiency of the headspace autosampler and to evaluate the precision of the analysis. The MS-MSDs are prepared with each batch of samples. A batch is defined as up to 20 samples of a similar matrix prepared and analyzed together. The matrix spike is added to the MS-MSD samples at approximately 10 times the MRL. The concentration of the matrix spike found in the spiked samples is calculated using Equation 8 or Equation 9. The recovery of the spiked samples is calculated using Equation 11.

$$\%R = [(\text{amt. found in Sp} - \text{amt. found in sample}) / \text{amt. spiked}] \times 100 \qquad (11)$$

where

%R = percent recovery for either the MS or the MSD.
amt. found in Sp = amount of VOC found in the spiked sample.
amt. found in sample = amount of VOC found in the original sample.
amt. spiked = amount of VOC added to the MS or the MSD.

The precision of the analysis may be determined through the use of the MS-MSD. The recoveries of the MS-MSD are compared, and a relative percent difference (RPD) is calculated. The RPD value obtained can be used to determine method precision. The calculation for RPD is given in Equation 12.

$$RPD = [(MS\ \%R - MSD\ \%R) / \{(MS\ \%R + MSD\ \%R) / 2\}] \times 100 \qquad (12)$$

where

RPD = relative percent difference.
MS %R = matrix spike percent recovery.
MSD %R = matrix spike duplicate percent recovery.

RPD is always used as an absolute value, or a positive number. The advisory control limits for spike recovery in both soil and water are 50 to 150 percent. The advisory control limits for RPD in both soil and water are 50 percent. Site-specific control limits may be calculated using equations 1 through 4, if more than 25 MS-MSD samples are analyzed.

The advisory control limits may not always be achievable. This may be due to the sample matrix. This is especially true for soil samples. If the spike recovery or the RPD is outside of the advisory control limits, another MS-MSD should be performed. If recovery or RPD is still outside of the advisory control limits, the values should be qualified with an (M) for matrix interference and explained in the final sample report.

8.4 LABORATORY CONTROL SAMPLES

Laboratory control samples (LCS) are used to evaluate the accuracy of the analysis. The LCS samples are obtained from outside sources other than those obtained for calibration standards and contain a known amount of the VOCs. These samples are analyzed following the same sample preparation and analysis procedures used for true samples. The results obtained from the analysis are then compared to the known true values. When no control limits are provided, the advisory control limts of 50 to 150 percent recovery should be used. When values obtained from the LCS fall outside of the control limits, the LCS and all samples associated with it must be analyzed again. If the LCS %R is still outside the advisory control limits, a new ICAL must be performed and found to be acceptable before sample analysis can continue. Site-specific control limits may be calculated using equations 1 through 4, if more than 25 LCS samples are analyzed.

LCS samples are analyzed when available. These samples are expensive and may not be readily available when VOC analysis is needed. Results from LCS sample analysis will be stored along with the final report.

8.5 LABORATORY DUPLICATE SAMPLES

Duplicate samples are two analyses performed on the same sample. Duplicate samples may be used to monitor the precision of the sampling and analysis procedures used. Duplicate samples are analyzed with each batch of up to 20 samples. Results from the duplicate samples are compared by evaluating the RPD of both results. Equation 13 shows the terms and the calculation for RPD.

$$RPD = [(Result\ 1 - Result\ 2) / \{(Result\ 1 + Result\ 2) / 2\}] \times 100 \qquad (13)$$

where

RPD = Relative percent difference.
Result 1 = Result from first sample analysis.
Result 2 = Result from second sample analysis.

RPD is always used as an absolute value, or a positive number. RPD values of below 50 percent are considered acceptable. This may not always be achievable. Sample matrix, nonhomogeneity, and organic content can affect the duplicate RPD. When RPD values of above 50 percent are obtained, another duplicate should be prepared and analyzed to gather more data. Usually the reported value will come from the highest result obtained. Qualification of the sample data may be performed to indicate that the RPD did not fall within the advisory control limit of 50 percent.

8.6 PERFORMANCE EVALUATION SAMPLE

PE samples are used to evaluate the accuracy of the analysis. The PE samples are obtained from outside sources and contain certified concnetrations of VOCs. These samples are analyzed following the same sample extraction and analysis procedures used for true samples. The results obtained from the analysis are then compared to the certified acceptable range of concentrations for each target compound of the PE. Control limits are usually provided from the supplier of the PE. The results obtained should fall within the published range of acceptance values. When no control limits are provided, the range from 50 to 150 percent will be used. When values obtained from the PE fall outside of the control limits, the source of the problem must be found and corrected. Once the problem has been corrected, the PE sample and all

samples extracted and analyzed with it must be reexracted and reanalyzed.

PE samples are analyzed when available. These samples are expensive and may not be readily available when VOC analysis is needed. Whenever possible, PE samples should be used to demonstrate the accuracy of this method.

9.0 METHOD PERFORMANCE

The OSL VOC method will be used to determine concentrations of VOCs found in soil gas, water, and soil samples. Method performance for this performance-based method will be evaluated for each project for which it is used to determineVOCs. Method performance will begin with an initial assessment of the capabilities to perform analysis. The accuracy and precision of the method is continually evaluated through the use of QC samples throughout the duration of the project. The comparability of the method is determined through a comparison of the results generated from the use of this method to results determined through the analysis of the same sample using EPA-approved methods. This process is normally performed after completion of the project. A data assessment report will be prepared for each project that will document the performance of this method. The following subsections detail procedures that will be used to assess the performance of this method.

9.1 INITIAL ASSESSMENT

Before using this method for any project, an initial assessment should be performed to determine its applicability for the site. The ability of this method to perform satisfactorily can be effected by many variables including, sample matrix, interferences, site-specific action limits, and target analyte lists.

The type of sample matrix at each site should be researched before using this method to determine possible problems that may be encountered. Generally, water samples provide a minimum of sample matrix problems that may effect this method. Water samples with high concentrations of suspended or dissolved solids may impact the ability of this method to perform satisfactorily. The analysis of soil samples with this method can be impacted by the soil matrix. High amounts of organic material in soil

samples can decrease the sample extraction efficiency and may provide chromatographic interferences that can effect analytical results. Clay also can reduce the extraction efficiency of this method. Alternative sample preparation steps, such as separator funnel extractions for water samples and sonication or soxhlet extraction for soil samples, may need to be employed to overcome problems associated with sample matrix

Interferences can effect the identification and quantification of samples analyzed using this method. Section 3.2 of this SOP describes particular sample matrix interferences that can effect this method. Interferences include those caused by sample matrix, high concentrations of target and non-target organic analytes in the sample, and interferences from multiresponse components that may be present in the samples. Other analytical columns can sometimes be used to overcome interference problems. Interference problems are difficult to anticipate and prepare for. In some extreme circumstances, interferences can eliminate the ability of this method to perform satisfactorily. When this occurs the use of this method should be suspended. Samples should then be sent to formal laboratories using EPA-approved methods and that are experienced in performing sample cleanups to eliminate sample matrix interferences.

MRLs should be less than the site-specific action limits applicable to each site. The MRLs for each of the VOCs that can be analyzed with this method are listed in Table 1. These MRLs are based primarily on the concentration of the low level standard that is used for analysis multiplied by the concentration or dilution factor used for sample preparation. In most cases these MRLs are sufficient to meet project needs. For some projects where the site-specific action limits are close to the MRLs, method detection limit studies may be performed to provide site-specific MRLs. The method detection limit study is performed by spiking seven aliquots of a sample with all of the site-specific target analytes. These samples are then extracted and analyzed using the same procedures outlined in this SOG. Standard deviations are calculated for each of the VOC results, and a 99-percent upper confidence interval is calculated and used for the site-specific MRL. The method detection limit study is recommended for all projects with an adequate budget to allow for such a study. This provides an experimental determination of the MRLs and provides sufficient documentation to satisfy most scientific queries concerning the accuracy of the MRLs.

Site-specific target analyte lists should be reviewed prior to the start of a project to determine that this method is capable of determining all VOC target analytes. Table 1 lists the VOCs that can be determined with this method. In some cases, VOC compounds that are not shown in Table 1 will need to be determined. When this occurs, the ability of this method must be documented to show that it is applicable to the VOC compound. This procedure generally begins with a method detection limit study to show that the compound can be analyzed with this method, and that MRLs that are less than the site-specific action limit can be achieved. Throughout the project, quality control samples are prepared, analyzed, and evaluated to document the ability of the method to analyze the VOC compounds.

9.2 ACCURACY AND PRECISION ASSESSMENT

The accuracy and precision of the analysis will be evaluated using the quality control samples stipulated in Section 8 of this SOP. The following paragraphs discuss how accuracy and precision will be assessed.

9.2.1 Accuracy

Accuracy of the analysis is evaluated through the use of PE samples, LCSs, MS and MSD samples, and surrogate standards. Accuracy is assessed through the use of percent recoveries calculated by comparing the result generated from the use of this method to a reference value accepted as a true value or the expected value. Control limits are detailed for each quality control sample in the appropriate subsection of Section 8.0, and are reiterated below.

- PE sample - within published acceptance range, when no acceptance range is provided the range of 50 to 150 percent recovery will be used
- LCS - 50 to 150 percent recovery or site specific control charts
- MS and MSD - 50 to 150 percent recovery or site specific control charts
- Surrogate standards - 50 to 150 percent recovery or site specific control charts

Method accuracy is considered acceptable when all of the quality control sample percent recoveries fall within the above-listed ranges. For large projects that involve the analysis of a large number of samples

(greater than 25 samples), site-specific control limits can be calculated. These control limits are calculated by, first determining the mean percent recovery for each VOC, second calculating the 95 percent confidence interval based upon the calculated standard deviation of percent recovery for each VOC, and lastly comparing each percent recovery to the appropriate confidence interval. This procedure allows for project-specific control limits to be applied that may be more stringent than the general control limits described above.

When method accuracy is determined to be unacceptable (by percent recoveries that fall outside of the prescribed acceptance ranges), corrective actions will be used. These corrective actions are described in Table 6.

9.2.2 Precision

Precision of the analytical method is evaluated through the use of laboratory duplicate samples and MS and MSD samples. The laboratory duplicate result is compared to the original sample result through an RPD calculation. The control limit for laboratory duplicate sample RPD is 50 percent. The percent recovery of the MS sample also is compared to the percent recovery of the MSD sample through the use of the RPD calculation. The control limit for MS and MSD sample RPD also is 50 percent. Again, when precision control limits are exceeded corrective action will be used. These corrective actions also are shown in Table 10.

9.3 COMPARABILITY ASSESSMENT

The comparability assessment will be performed through a comparison of split sample results generated from this method and those of a formal laboratory performing analysis with EPA-approved methods. This will be performed by comparing the detection limits and the analytical results for each method.

The primary comparability rule is to be used for determining whether detected analytical results is considered good, fair, or poor.

TABLE 6

SUMMARY OF QUALITY CONTROL SAMPLE CRITERIA

QC Check	Frequency	Acceptance Criteria	Corrective Action
Method Blank	One per analytical batch	Less than MRLs	1. Assess the source of the contamination 2. If concentration of contaminant in sample is greater than one-tenth of the site-specific action limit, reprepare and reanlyze method blank and all associated samples. 3. If concentration of contaminant in sample is greater than one-tenth of the site-specific action limit, qualify analytical data.
PE Sample	One per project (minimum)	Within published acceptance limits. If no acceptance limits are specified use 50 to 150 percent recovery	1. Determine the source of the problem and correct 2. Reextract and reanalyze all samples 3. If unacceptable results still occur repeat step 1
LCS	One per initial calibration	50 to 150 percent recovery or site specific control charts	1. Determine the source of the problem and correct 2. Reanalyze LCS
MS and MSD	One per analytical batch	50 to 150 percent recovery or site specific control charts 50 percent RPD	1. Check LCS result 2. Repeat analysis 3. Qualify data
QC Check	Frequency	Acceptance Criteria	Corrective Action
Surrogate standards	All samples analyzed	50 to 150 percent recovery or site specific control charts	1. Repeat analysis 2. Qualify data
Laboratory duplicate	One per analytical batch	50 percent RPD	1. Repeat analysis 2. Qualify data

Notes:

MRL Method reporting limit
PE Performance evaluation
LCS Laboratory control sample
MS Matrix spike
MSD Matrix spike duplicate
RPD Relative percent difference

- Good comparability is defined as agreement of 0 to 67 RPD between the OSL value and the formal laboratory value; 0- to 2-fold difference

- Fair comparability is defined as agreement of 68 to 164 RPD between the OSL value and the formal laboratory value; 2- to 10-fold difference

- Poor comparability is defined as agreement greater than 164 RPD; greater than 10-fold difference

Data that is determined to provide good comparability may be considered definitive data. The MRLs and the accuracy and precision assessment must be taken into account when making this determination.

A secondary rule can also be used for sample results less than 5 times the highest detection limit (between the OSL and the formal laboratory). The following rule applies for VOC analytes:

- For water samples, the absolute difference between the results should be less than 2 times the highest detection limit

- For soil samples, the absolute difference between the results should be less than 5 times the highest detection limit

Generally, when one of the results being compared is undetected, the secondary rule shall apply and the undetected results should be considered zero in the calculations. In addition, false negatives reported by the OSL laboratory must be identified and included in the report. The following are scenarios which must be addressed:

- The OSL detection limit is less than the formal laboratory's
  The OSL reports an undetected result
  The formal laboratory reports a positive result greater than the OSL detection limit
  The OSL result is considered a false negative

- The OSL detection limit is greater than the formal laboratory's
  The OSL reports an undetected result
  The formal laboratory reports a positive result that is less than the OSL detection limit
  The OSL result is considered acceptable

- The OSL detection limit is greater than the formal laboratory's
  The OSL reports an undetected result

The formal laboratory reports a positive result that is greater than the OSL detection limit
The OSL result is considered a false negative

- The OSL detection limit is equal to the formal laboratory's
  The OSL reports an undetected result
  The formal laboratory reports a positive result that is greater than the OSL's detection limit
  The OSL result is considered a false negative

- The OSL detection limit is equal the formal laboratory's
  The OSL reports an undetected result
  The formal laboratory reports a positive result that is less than the formal laboratory's detection limit (J coded)
  The OSL result is considered acceptable

- The OSL detection limit is equal or lesser than the formal laboratory's
  The OSL reports a detected result
  The formal laboratory reports an undetected result
  The OSL result is considered a false positive

Results that have been catergorized as providing fair or poor comparability results will be statistically evaluated through the use of a linear regression equation. This statistical evaluation may provide a statistical approach to the application of correction factors that may be required to allow for more comparable data between the two data sets. When appropriate, these factors can be applied to the data obtained through the use of this method to provide data reviewers a more accurate representation of contaminant concentrations delineated by this data. Results that provide poor comparability, and that cannot be mathematically corrected will not be used for environmental investigation decision-making process.

An additional assessment of the OSL's capability of detecting presence and absence of the VOCs of concern will be determined by the following criteria (Llewellyn, 1993):

- False-negative rate should be <5 percent when measured at two times the action level
- False-positive rate should be <10 percent when measured at 0.5 times the action level

9.4 DATA ASSESSMENT REPORT

Most projecst that use this method will require a data assessment report that summarized the analytical results, QC procedures used along with QC results, and summaries of the accuracy and precision assessment and the comparability assessment. This report also should describe the objectives of the investigation, the level of data quality obtained from the use of this method, and specific uses or objectives that the dat a can be used for. The scope of the data assessment report will vary depending on the size and scope of the project, and should be written in a succint, but comprehensive style.

10.0 DISPOSAL OF LABORATORY WASTE

Waste derived from extraction and analysis of soil and water samples from the OSL will be mixed with investigation derived waste (IDW) when appropriate and disposed of following the guidelines described in the RI/FS workplan. If the OSL waste cannot be mixed with the RI/FS IDW, the waste will be containerized and disposed of at a permitted facility.

11.0 LABORATORY NOTEBOOK

A laboratory notebook must be kept by the field chemist on a daily basis. All standard preparation, ICAL, CCAL, any problems encountered, and QA/QC results and information must be documented. Pages should be signed and dated at the bottom. Any errors should be lined out, initialled, and dated.

12.0 REFERENCES

EPA. 1986. "Test Methods for Evaluating Solid Waste, Third Edition." SW-846. Prepared by Office of Solid Waste and Emergency Response. With promulgated revisions through 1995.

EPA. 1993. "Data Quality Objectives Process for Superfund." Prepared for the Contract Laboratory Program. EPA 1540 IR-94-012. February.

Williams, Llewellyn R. 1993. "Methods for the EPA's Regulatory Programs." *Environmental Testing & Analysis*. Page 36 through 39. January/February.

APPENDIX A

CUSTOM VOLATILE MIX

Certificate of Analysis

Custom Volatile Standard

Catalog Number: CUS-1427

Lot Number: J-1150

This ULTRAstandard(TM) solution was gravimetrically prepared at the nominal concentration stated on the box label. The true value for each analyte, determined gravimetrically, is listed below.

Component	Weight/mL*
methylene chloride	1005.0 µg
trans-1,2-dichloroethene	1005.0 µg
1,1-dichloroethane	1004.0 µg
cis-1,2-dichloroethene	1003.0 µg
1,2-dichloroethane	1005.0 µg
chloroform	50.1 µg
1,1,1-trichloroethane	50.1 µg
trichloroethene	50.1 µg
bromodichloromethane	50.1 µg
dibromochloromethane	50.1 µg
1,1-dichloroethene	251.2 µg
1,1,2-trichloroethane	150.7 µg
1,1,2,2-tetrachloroethane	100.3 µg
bromoform	250.5 µg
1,2-dibromoethane	100.5 µg
tetrachloroethene	25.1 µg
1,1,1,2-tetrachloroethane	25.1 µg
trichlorofluoromethane	10.0 µg
carbon tetrachloride	10.0 µg
vinyl chloride	2512.0 µg

Solvent: methanol

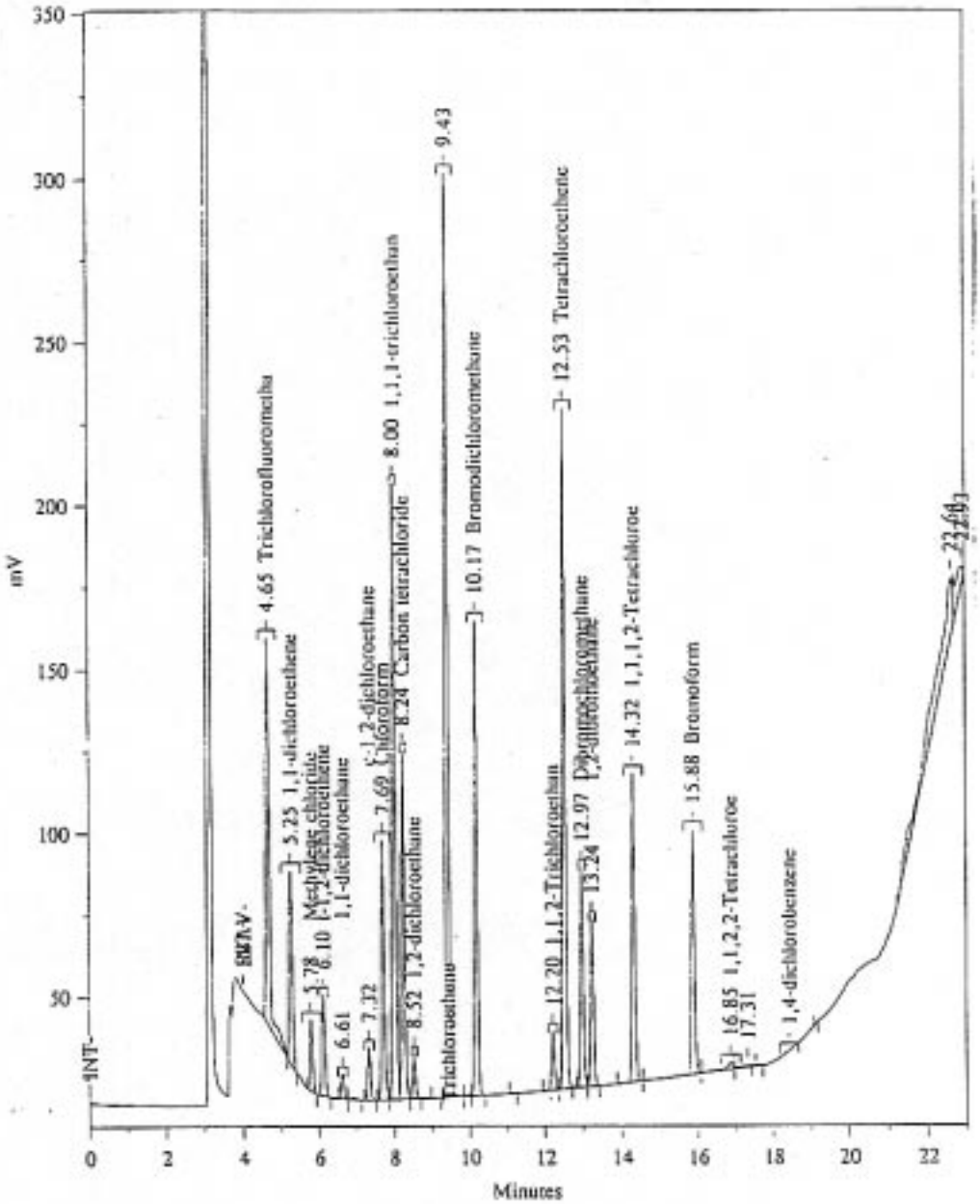
* All weights are traceable through N.I.S.T. Test No. 732/221797

ULTRA SCIENTIFIC
ISO 9001 Registered
250 Smith Street, North Kingstown, RI 02852 • 401 294 9400 • 800 338 1754

APPENDIX B

SAMPLE CHROMATOGRAMS

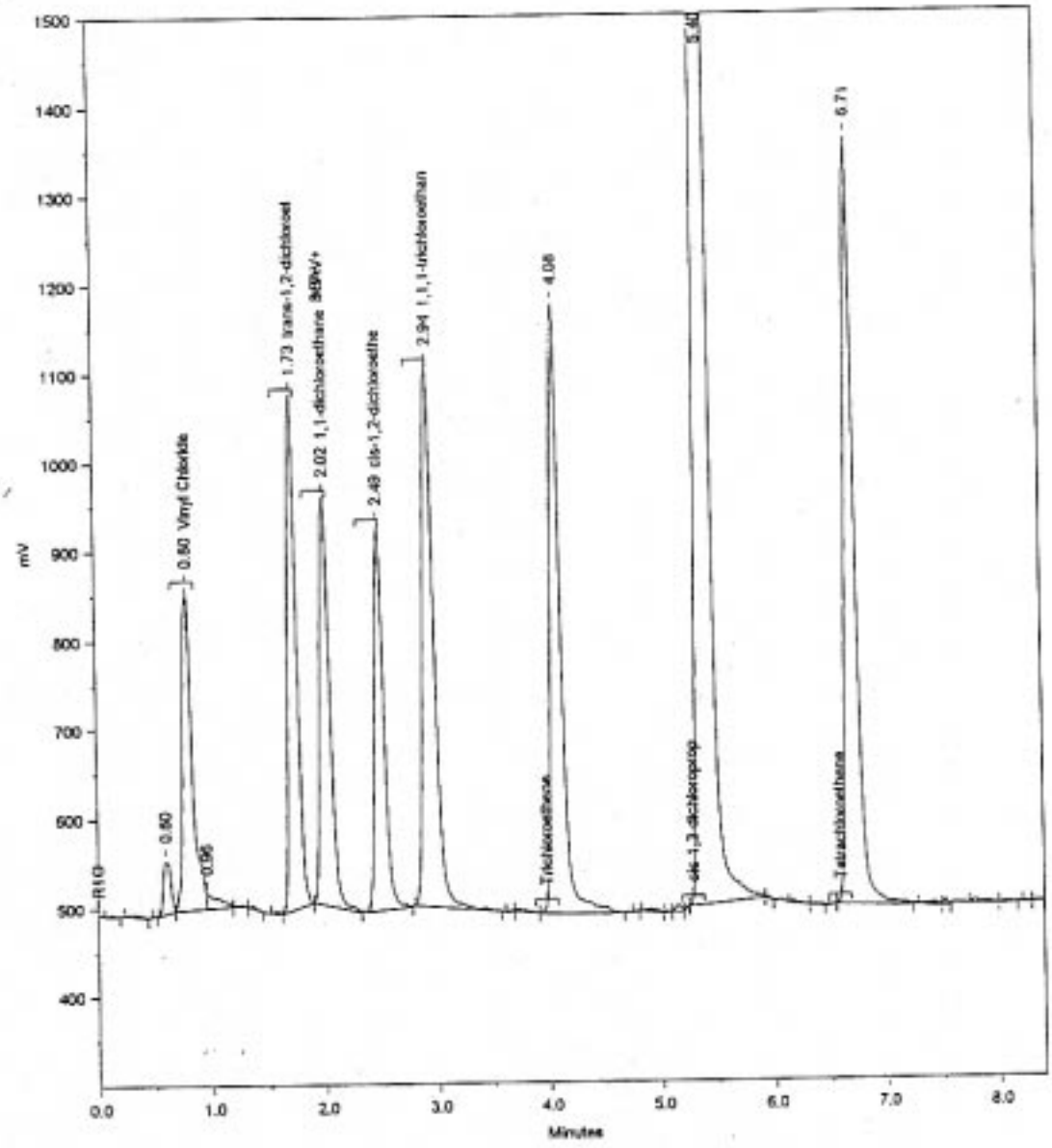
Detector: ECD
Column: DB-624, 0.53mm I.D., 75m
Initial Temp.: 50°C
Initial Time: 0.5 min.
Rate 1: 6°C/min.
Final Temp. 1: 150°C
Rate 2: 20°C/min.
Final Temp. 2: 200°C
Final Time: 1 min.



SITE SOG 001
FIGURE 1 CLORINATED VOLATILE CONTAMINANT CHROMATOGRAPH
PRC Environmental Management, Inc.

Source: PRC 1997.

Detector: ELCD
Column: DB-624, 0.53mm I.D., 30m
Initial Temp.: 35°C
Initial Time: 2 min.
Rate: 6°C/min.
Final Temp.: 70°C
Final Time: 0.5 min.



Source: PRC 1997.

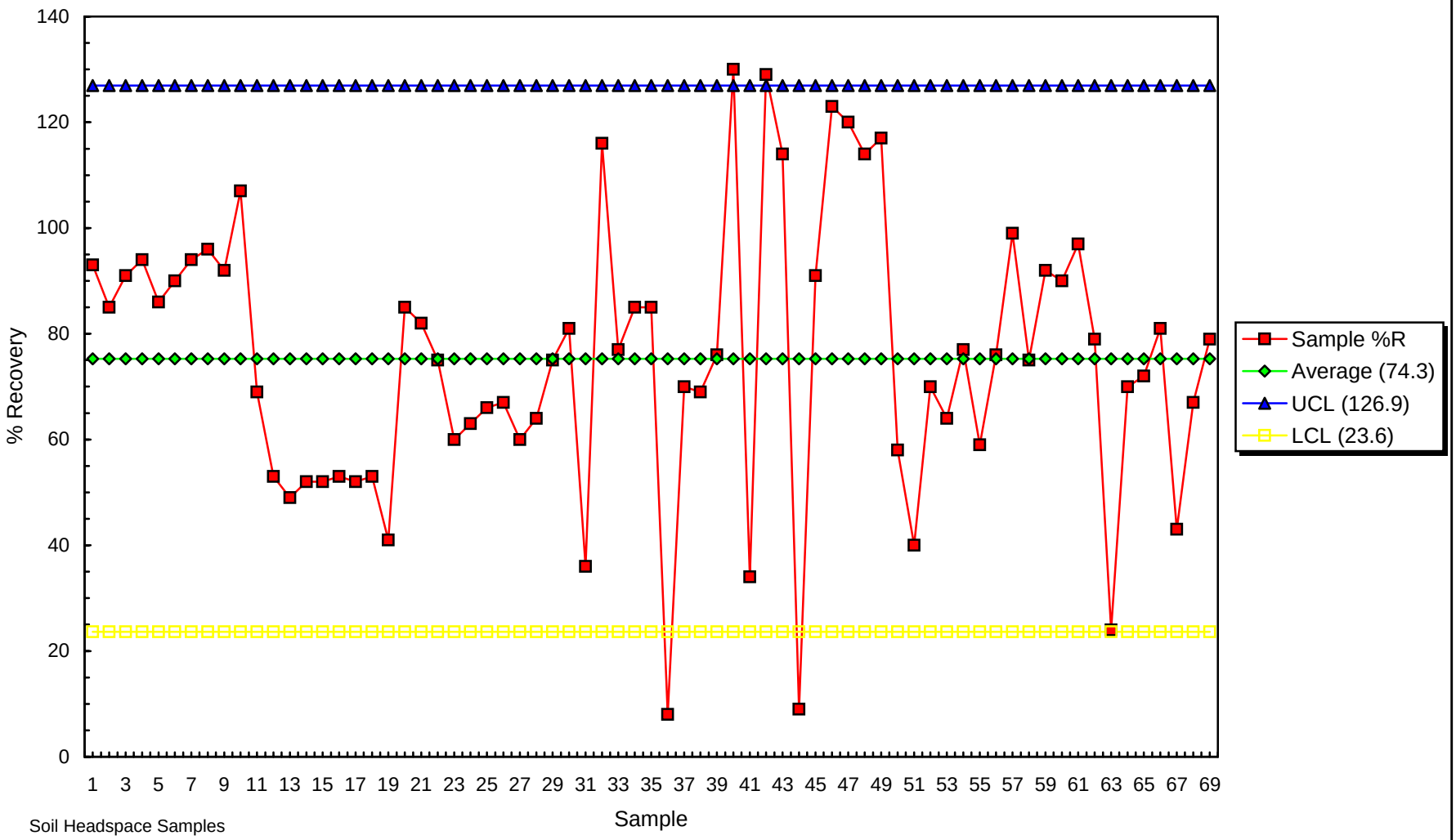
SITE SOG 001
FIGURE 2 CLORINATED VOLATILE CONTAMINANT CHROMATOGRAPH
PRC Environmental Management, Inc.

APPENDIX C

SAMPLE RECOVERY CONTROL CHARTS

SURROGATE CONTROL CHART

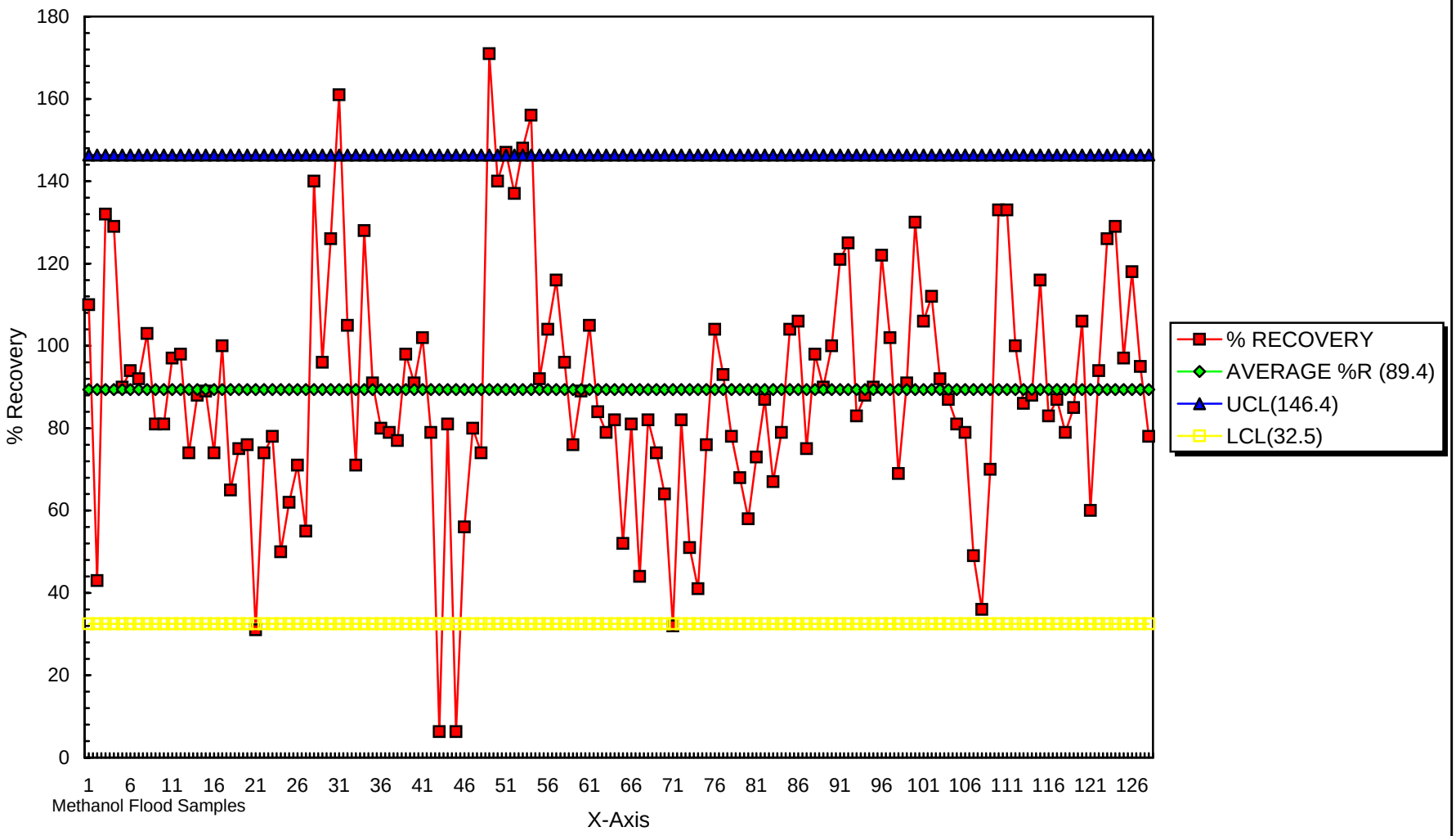
Chemical Sales - cis-1,3-dichloropropene



Soil Headspace Samples

SURROGATE CONTROL CHART

Chemical Sales - cis-1,3-dichloropropene



METHOD APPROVAL FORM

PRC ENVIRONMENTAL MANAGEMENT, INC.
ON-SITE LABORATORY
SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION

TOP LOADER BALANCE CALIBRATION

METHOD 003

INITIAL ISSUE

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1.0 SCOPE AND APPLICATION 1

2.0 SUMMARY OF METHOD 1

2.1 TOP LOADER BALANCE CALIBRATION 1

1.0 SCOPE AND APPLICATION

This document serves as the standard operating procedure (SOP) for the analytical method used to determine the weight of soils for volatile organic compound (VOC)and semivolatile organic compound (SVOC) for the evaluation of measurement and monitoring technololgies during demonstrations under the Superfund Innovative Technology Evaluation (SITE) program. The method is designed to be used at an on-site laboratory to give definitive, accurate results. Those results then can be used to guide evaluate innovative technologies. Several classes of VOCs can be determined with this method, including halogenated VOCs (XVOC), non-halogenated VOCs, and volatile petroleum hydrocarbons (VPH), and SVOCs.

2.0 SUMMARY OF METHOD

This SOP details how to use and calibrate a top loader balance to determine soil weights for the analysis of VOCs and SVOCs in soil. The soil analyses are completed by combining automated headspace sampling with gas chromatography (GC) for VOCs or solvent extraction and GC analysis for SVOCs.

2.1 Top Loader Balance Calibration

The top loader balance will be calibrated according to instrument manufacturers instruction. The balance must be calibrated at the beginning of each work day, and the results recorded in the balance logbook.

APPENDIX B

TARGET SAMPLING DEPTHS AND TECHNOLOGY SAMPLING ASSIGNMENTS

SBA Site, Albert City, Iowa
Grid Number 1 - Target Depth (9.5 and 13.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	ACT	CLE	ACT	AMS (QUA)	CLE (QUA)	SIM (GOR)	ARA
2	ARA (QUA)	REF	REF	GEO	ARA (GOR)	REF	AMS
3	REF	AMS (QUA)	CLE	SIM	GEO	CLE	SIM
4	SIM	ARA	AMS (GOR)	REF (GOR)	REF	ACT	GEO (GOR)
5	CLE (GOR)	GEO	GEO	ARA	AMS	GEO (QUA)	CLE
6	GEO	ACT (GOR)	SIM (QUA)	ACT	SIM	ARA	ACT
7	AMS	SIM	ARA	CLE	ACT	AMS	REF (QUA)

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

SBA Site, Albert City, Iowa
Grid Number 2 - Target Depth (3.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	GEO	CLE	REF (GOR)	SIM (QUA)	ARA	REF	CLE
2	REF	GEO	AMS	ACT	GEO	AMS (GOR)	GEO
3	AMS (QUA)	AMS (GOR)	ACT	GEO	CLE (QUA)	ARA	ARA
4	ACT	REF	GEO	CLE	SIM	SIM	REF (GOR)
5	SIM (GOR)	ARA	SIM	ARA (GOR)	REF	ACT	CLE (QUA)
6	CLE	ACT	CLE (QUA)	REF	ACT (GOR)	CLE	AMS
7	ARA	SIM (QUA)	ARA	AMS	AMS	GEO (QUA)	ACT

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

SBA Site, Albert City, Iowa
Grid Number 3 - Target Depth (Additional if Time Permits 9.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	BLK	SIM	AMS	GEO	REF	CLE	AMS
2	REF	AMS	CLE	AMS	GEO	BLK	SIM
3	GEO	CLE	SIM	SIM	SIM	ARA	CLE
4	SIM	BLK	REF	BLK	CLE	AMS	ARA
5	CLE	ARA	ARA	CLE	AMS	REF	GEO
6	AMS	REF	GEO	REF	ARA	GEO	REF
7	ARA	GEO	BLK	ARA	BLK	SIM	BLK

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
BLK	Blank cell		

SBA Site, Albert City, Iowa
Grid Number 4 - Target Depth (9.5 feet/Additional if Time Permits)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	REF	AMS	ACT	REF	AMS	AMS	ACT (QUA)
2	GEO	SIM	CLE	ACT (GOR)	CLE	REF	REF
3	ACT (GOR)	REF	REF	AMS (QUA)	ARA	ACT	SIM (GOR)
4	AMS	ARA	SIM (QUA)	GEO	ACT	GEO (QUA)	ARA
5	ARA	ACT (GOR)	AMS	CLE	GEO	ARA (GOR)	CLE
6	SIM	CLE (QUA)	ARA (GOR)	ARA	SIM (QUA)	SIM	AMS
7	CLE (QUA)	GEO	GEO	SIM	REF (GOR)	CLE	GEO

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

SBA Site, Albert City, Iowa
Grid Number 5 - Target Depth A (13.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	CLE	REF	ARA	GEO	AMS	SIM	ARA (QUA)
2	ARA	AMS	REF (GOR)	SIM	ACT (GOR)	AMS	SIM
3	REF	SIM	CLE	CLE	REF (QUA)	REF (GOR)	AMS
4	ACT (GOR)	CLE (QUA)	ACT (QUA)	REF	CLE	GEO	REF
5	GEO	ARA (GOR)	GEO	AMS	GEO	ARA (QUA)	CLE (GOR)
6	SIM	ACT	SIM	ARA (GOR)	ARA	CLE	GEO
7	AMS (QUA)	GEO	AMS	ACT (QUA)	SIM	ACT	ACT

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

SBA Site, Albert City, Iowa
Grid Number 6 - Soil Gas Only
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	GOR			GOR	QUA		
2			ACT	QUA		QUA	
3		ACT	GOR			ACT	
4	ACT	QUA		ACT	GOR		QUA
5		GOR	QUA			GOR	ACT
6	QUA						GOR
7					ACT		

Notes:

ACT	Active Soil Gas (only at 5.0 feet bgs)
GOR	W.L Gore and Associates (only at 3.0 feet bgs)
QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)

CSC Site, Denver, Colorado
Grid Number 1 - Target Depth A (3.0 and 6.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	CLE	AMS (QUA)	CLE	SIM (QUA)	REF	REF	CLE (QUA)
2	SIM	SIM	ARA	AMS	ACT (GOR)	SIM (GOR)	SIM
3	REF (GOR)	REF	REF	CLE	AMS	ACT	AMS
4	GEO	CLE	ACT (GOR)	ACT	ARA	GEO (QUA)	ARA
5	ACT	GEO	AMS (QUA)	REF	GEO	ARA	GEO (GOR)
6	AMS	ACT (GOR)	SIM	GEO (GOR)	SIM	CLE	ACT
7	ARA (QUA)	ARA	GEO	ARA	CLE (QUA)	AMS	REF

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

CSC Site, Denver, Colorado
Grid Number 2 - Target Depth A (3.0 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	REF	ARA (QUA)	REF	SIM (GOR)	ACT	SIM	REF (GOR)
2	SIM (QUA)	CLE	GEO	ARA	GEO	ACT	SIM
3	ARA	AMS	SIM	CLE	REF (GOR)	AMS	GEO (QUA)
4	ACT	ACT	AMS (GOR)	ACT (QUA)	AMS	CLE (QUA)	ACT
5	CLE	GEO	ACT	GEO	ARA (QUA)	REF	ARA
6	GEO (GOR)	REF	CLE (QUA)	REF	CLE	GEO	CLE
7	AMS	SIM (GOR)	ARA	AMS	SIM	ARA (GOR)	AMS

Notes:

AMS	Art's Manufacturing and Supply	GOR	W.L Gore and Associates (only at 3.0 feet bgs)
GEO	Geoprobe Systems	QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)
SIM	Simulprobe		
CLE	Clements and Associates		
ARA	Applied Research Associates		
REF	Conventional Sampling Method		
ACT	Active Soil Gas (only at 5.0 feet bgs)		

CSC Site, Denver, Colorado
Grid Number 3 - Target Depth A (3.0 and 7.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	ARA	REF	GEO	AMS	SIM	GEO	BLK
2	REF	AMS	REF	ARA	BLK	SIM	SIM
3	GEO	SIM	BLK	BLK	CLE	ARA	AMS
4	AMS	CLE	AMS	GEO	AMS	CLE	REF
5	CLE	BLK	ARA	CLE	GEO	AMS	CLE
6	BLK	ARA	CLE	REF	REF	REF	GEO
7	SIM	GEO	SIM	SIM	ARA	BLK	ARA

Notes:

AMS	Art's Manufacturing and Supply
GEO	Geoprobe Systems
SIM	Simulprobe
CLE	Clements and Associates
ARA	Applied Research Associates
REF	Conventional Sampling Method
BLK	Blank cell
GOR	W.L Gore and Associates (only at 3.0 feet bgs)
QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)

CSC Site, Denver, Colorado
Grid Number 4 - Target Depth A (6.5 feet)
Soil Gas and Soil Sampling Demonstration

	A	B	C	D	E	F	G
1	REF (GOR)	AMS	ACT	SIM	SIM (QUA)	ACT	AMS
2	SIM	SIM (QUA)	ARA	GEO (QUA)	ARA	ARA	GEO
3	CLE (QUA)	ACT	GEO	REF	CLE (GOR)	REF	ACT
4	ACT	ARA (GOR)	SIM	AMS	ACT	AMS (QUA)	CLE
5	GEO	CLE	AMS (GOR)	ARA	REF	CLE	REF
6	AMS	REF	CLE	ACT	GEO	SIM	ARA (QUA)
7	ARA	GEO	REF (QUA)	CLE (GOR)	AMS	GEO (GOR)	SIM (GOR)

Notes:

AMS	Art's Manufacturing and Supply
GEO	Geoprobe Systems
SIM	Simulprobe
CLE	Clements and Associates
ARA	Applied Research Associates
REF	Conventional Sampling Method
ACT	Active Soil Gas (only at 5.0 feet bgs)
GOR	W.L Gore and Associates (only at 3.0 feet bgs)
QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)

CSC Site, Denver, Colorado
Grid Number 5
Saturated Sand Sampling Test

	A	B	C	D	E	F	G
1	ACT	REF	REF	CLE	REF	ACT	ARA (GOR)
2	REF	SIM (GOR)	ARA	ARA (QUA)	CLE	GEO	REF
3	ARA	GEO	ACT	GEO (GOR)	GEO (QUA)	CLE (QUA)	SIM
4	SIM	ARA	AMS	REF	ACT	ARA	CLE (QUA)
5	AMS (GOR)	ACT (QUA)	GEO	ACT	AMS	SIM	GEO
6	CLE	AMS	SIM (GOR)	AMS	SIM	REF	AMS
7	GEO (QUA)	CLE	CLE (QUA)	SIM	ARA (GOR)	AMS (GOR)	ACT

Notes:

AMS	Art's Manufacturing and Supply
GEO	Geoprobe Systems
SIM	Simulprobe
CLE	Clements and Associates
ARA	Applied Research Associates
REF	Conventional Sampling Method
ACT	Active Soil Gas (only at 5.0 feet bgs)
GOR	W.L Gore and Associates (only at 3.0 feet bgs)
QUA	Quadrel Services, Inc. (only at 4.0 inches bgs)