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EPA

Environmental Technology Verification Program

Verification Test Plan

Evaluation of Field Polychlorinated Biphenyl (PCB) Detection Technologies

ornl

Oak Ridge National Laboratory

ETV ETV ETV

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Evaluation of Field Polychlorinated Biphenyl (PCB) Detection Technologies

By

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-6120

and

U.S. Environmental Protection Agency
Environmental Sciences Division
National Exposure Research Laboratory
Las Vegas, Nevada 89193-3478

APPROVAL SIGNATURES

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

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

U. S. ENVIRONMENTAL PROTECTION AGENCY

Project Manager: ______________________________ ______________
Eric Koglin **Date**

ESD Quality Manager: ______________________________ ______________
George Brilis **Date**

OAK RIDGE NATIONAL LABORATORY

Program Manager: ______________________________ ______________
Roger Jenkins **Date**

Technical Lead: ______________________________ ______________
Amy Dindal **Date**

QA Specialist: ______________________________ ______________
Janet Wagner **Date**

Statistician: ______________________________ ______________
Charles Bayne **Date**

U. S. DEPARTMENT OF ENERGY, Oak Ridge Operations Office

Program Manager: ______________________________ ______________
Regina Chung **Date**

SITE APPROVAL

ES&H Coordinator: ______________________________ ________________

Fred Smith **Date**

TECHNOLOGY VENDORS

Dexsil Corporation: ______________________________ ________________

Ted Lynn **Date**

Hybrizyme: ______________________________ ________________

Randy Allen **Date**

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EXECUTIVE SUMMARY

EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. The verification study described in this test plan will be conducted by the Site Characterization and Monitoring Technologies Pilot (SCMT), one of 12 pilots of the ETV program. The SCMT pilot is administered by the EPA's National Exposure Research Laboratory in Las Vegas, Nevada. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the test.

This test plan has been developed to describe the verification of two PCB field analytical technologies: Dexsil Corporation's L2000DX and Hybrizyme's DELFIA PCB assay. The purpose of this verification is to obtain performance information regarding the PCB field analytical technology, to compare the results to conventional fixed-laboratory results, and to provide supplemental information (e.g., cost, sample throughput, and training requirements) regarding the operation of the technology. The vendor will have a choice of analyzing PCB-contaminated soils (208 samples), methanol extracts (24 samples), and/or transformer oils (152 samples). Each matrix will include blanks, spikes, and environmentally-contaminated samples. The verification of soil and extracts will be conducted under two climatic conditions. One set of activities will be conducted outdoors, with naturally fluctuating temperatures and relative humidity conditions. A second set will be conducted in a controlled environmental facility, with lower, relatively stable temperatures and relative humidities. The oil analyses will be conducted under the outdoor field conditions only. The soil samples, collected from sites in Ohio, Kentucky, and Tennessee, will have PCB concentrations ranging from approximately 0.1 to 700 parts per million (ppm). Methanol solutions of known PCB concentration will simulate extracted surface wipe samples, and range in PCB concentration from 0 to 100 µg/mL. The oil samples, collected from active and in-active transformers at Oak Ridge National Laboratory, will range in PCB concentration from 0 to 200 ppm.

ABBREVIATIONS AND ACRONYMS

CASD	Chemical and Analytical Sciences Division
DOE	U. S. Department of Energy
EPA	U. S. Environmental Protection Agency
ERA	Environmental Resource Associates
ESD-LV	Environmental Science Division-Las Vegas
ESH&Q	Environmental Safety, Health, and Quality
ETTP	East Tennessee Technology Park
ETV	Environmental Technology Verification Program
ETVR	Environmental Technology Verification Report
fn	false negative result
fp	false positive result
GC	gas chromatography
HASP	Health and Safety Plan
LCS	Laboratory Control Sample
LMES	Lockheed Martin Energy Systems
MS/MSD	matrix spike/matrix spike duplicate
ORNL	Oak Ridge National Laboratory
ORNL-GJ	Oak Ridge National Laboratory, Grand Junction, Colorado
PCBs	polychlorinated biphenyls
PE	performance evaluation
PPE	personal protective equipment
ppm	parts per million, mg/kg for soils or µg/mL for extracts and oils
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RFD	request for disposal
RSD	relative standard deviation

SCMT	Site Characterization and Monitoring Technology Pilot, ETV
SMO	Sample Management Office
SPE	solid phase extraction
SOP	standard operating procedure
SOW	statement of work
SSM	synthetic soil matrix
SVOCs	semivolatile organic compounds
UPSI	United Power Services Inc.
VOCs	volatile organic compounds

1 INTRODUCTION

This chapter discusses the purpose of the verification and the verification test plan, describes the elements of the verification test plan, and provides an overview of the Environmental Technology Verification (ETV) Program and the technology verification process.

1.1 Verification Objectives

The purpose of this verification test is to evaluate the performance of commercially available field analytical technologies for performing polychlorinated biphenyl (PCB) analyses in soil, methanol extract, and/or transformer oil samples. Specifically, this plan defines the following elements of the verification test:

- Roles and responsibilities of verification test participants;
- Procedures governing verification test activities such as sample collection, preparation, analysis, data collection, and interpretation;
- Experimental design of the verification test;
- Quality assurance (QA) and quality control (QC) procedures for conducting the verification and for assessing the quality of the data generated from the verification; and,
- Health and safety requirements for performing work at hazardous waste sites.

1.2 What is the Environmental Technology Verification Program?

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations and stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology vendors. The program evaluates the performance of innovative technologies by developing verification test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

ETV is a voluntary program that seeks to provide objective performance information to all of the participants in the environmental marketplace and to assist them in making informed technology decisions. ETV does not rank technologies or compare their performance, label or list technologies as acceptable or unacceptable, seek to determine “best available technology,” or approve or disapprove technologies. The program does not evaluate technologies at the bench or pilot scale and does not conduct or support research. Rather, it conducts and reports on testing designed to describe the performance of technologies under a range of environmental conditions and matrices.

The program now operates 12 pilots covering a broad range of environmental areas. ETV has begun with a 5-year pilot phase (1995–2000) to test a wide range of partner and procedural alternatives in various pilot areas, as well as the true market demand for and response to such a program. In these pilots, EPA utilizes the expertise of partner “verification organizations” to design efficient processes for conducting performance tests of innovative technologies. These expert partners are both public and private organizations, including federal laboratories, states, industry consortia, and private sector entities. Verification organizations oversee and report verification activities based on testing and QA protocols developed with input from all major stakeholder/customer groups associated with the technology area. The verification test described in this plan will be administered by the Site Characterization and Monitoring Technologies (SCMT) Pilot, with Oak Ridge National Laboratory (ORNL) serving as the verification organization. (To learn more about ETV, visit ETV's Web site at www.epa.gov/etv and ORNL's web site at www.ornl.gov/etv). The SCMT pilot is administered by EPA's National Exposure Research Laboratory (NERL), Environmental Sciences Division, in Las Vegas, Nevada. Note that seven PCB technologies have already been verified for soils and solvent extracts; the reports can be viewed at either of the above-mentioned web sites.

1.3 Technology Verification Process

The technology verification process is intended to serve as a template for conducting technology verifications that will generate high quality data which can be used to verify technology performance. Four key steps are inherent in the process:

- Needs identification and technology selection
- Verification test planning and implementation
- Report preparation
- Information distribution

1.3.1 Needs Identification and Technology Selection

The first step in the technology verification process is to determine technology needs of the user-community (typically state and Federal regulators and the regulated community). Each Pilot utilizes stakeholder groups. Members of the stakeholder groups come from EPA, the Departments of Energy and Defense, industry, and state regulatory agencies. The stakeholders are invited to identify technology needs and to assist in finding technology vendors with commercially available technologies that meet the needs. Once a technology need is established, a search is conducted to identify suitable technologies. The technology search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searches of industry and trade publications, attendance at related conferences, and leads from technology vendors. The following criteria are used to determine whether a technology is a good candidate for the verification:

- Meets user needs
- May be used in the field or in a mobile laboratory
- Applicable to a variety of environmentally impacted sites
- High potential for resolving problems for which current methods are unsatisfactory
- Costs are competitive with current methods
- Performance is better than current methods in areas such as data quality, sample preparation, or analytical turnaround
- Uses techniques that are easier and safer than current methods
- Is commercially available and field-ready.

1.3.2 Verification Planning and Implementation

After a vendor agrees to participate, EPA, the Verification Organization, and the vendor meet to discuss each participants responsibilities in the verification process. In addition, the following issues are addressed:

- Site selection. Identifying sites that will provide the appropriate physical or chemical environment, including contaminated media
- Determining logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network)
- Arranging analytical and sampling support
- Preparing and implementing a verification test plan that addresses the experimental design, sampling design, QA/QC, health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements

1.3.3 Report Preparation

Innovative technologies are evaluated independently and, when possible, against conventional technologies. The technologies being verified are operated by the vendors in the presence of independent observers. The observers are EPA staff, state staff or from a independent third-party organization. The data generated during the verification test are used to evaluate the capabilities, limitations, and field applications of each technology. A data summary and detailed evaluation of each technology are published in an Environmental Technology Verification Report (ETVR). The original complete data set is available upon request.

An important component of the ETVR is the Verification Statement, which consists of three to five pages, using the performance data contained in the report, are issued by EPA and appear on the ETV Internet Web page. The Verification Statement is signed by representatives of EPA and ORNL.

1.3.4 Information Distribution

Producing the ETVR and the Verification Statement represents a first step in the ETV outreach efforts. ETV gets involved in many activities to showcase the technologies that have gone through the verification process. The Program is represented at many environmentally-related technical conferences and exhibitions. ETV representatives also participate in panel sessions at major technical conferences. ETV maintains a traveling exhibit that describes the program, displays the names of the companies that have had technologies verified, and provides literature and reports.

We have been taking advantage of the Web by making the ETVRs available for downloading to anyone interested. The ETVRs and the Verification Statements are available in Portable Document Format (.pdf) on the ETV Web site (http://www.epa.gov/etv).

1.4 Purpose of this Verification Test Plan

The purpose of the verification test plan is to describe the procedures that will be used to verify the performance goals of the technologies participating in this verification. This document incorporates the QA/QC elements needed to provide data of appropriate quality sufficient to reach a credible position regarding performance. This is not a method validation study, nor does it represent every environmental situation which may be appropriate for these technologies. But it will provide data of sufficient quality to make a judgement about the application of the technology under conditions similar to those encountered in the field under normal conditions.

2 VERIFICATION RESPONSIBILITIES AND COMMUNICATION

This section identifies the organizations involved in this verification test and describes the primary responsibilities of each organization. It also describes the methods and frequency of communication that will be used in coordinating the verification activities.

2.1 Verification Organization and Participants

Participants in this verification are listed in Table 2-1. The specific responsibilities of each verification participant are discussed in Section 2.3 This verification test is being coordinated by the Oak Ridge National Laboratory (ORNL) under the direction of the U.S. Environmental Protection Agency's (EPA) Office of Research and Development, National Exposure Research Laboratory, Environmental Sciences Division - Las Vegas, Nevada (ESD-LV). ESD-LV's role is to administer the verification program. ORNL's role is to provide technical and administrative leadership and support in conducting the verification.

Table 2-1. Verification Participants in PCB Field Analytical Technology Verification Test

Organization	Point(s) of Contact	Role
Oak Ridge National Laboratory P.O. Box 2008 Bethel Valley Road Bldg. 4500S, MS-6120 Oak Ridge, TN 37831-6120	**Program Manager:** Roger Jenkins phone: (865) 576-8594 fax: (865) 576-7956 email: jenkinsra@ornl.gov **Technical Lead:** Amy Dindal phone: (865) 574-4863 fax: (865) 576-7956 email: dindalab@ornl.gov	verification organization
U. S. EPA National Exposure Research Laboratory Environmental Science Division P.O. Box 93478 Las Vegas, NV 89193-3478	**Project Officer:** Eric Koglin phone: (702) 798-2432 fax: (702) 798-2261 email: koglin.eric@epa.gov	EPA project management
U. S. DOE ORNL Site Office P.O. Box 2008 Bldg. 4500N, MS-6269 Oak Ridge, TN 37831-6269	**Program Coordinator:** Regina Chung phone: (865) 576-9902 fax: (865) 574-9275 email: chungr@ornl.gov	DOE/ORO project management
Dexsil Corporation One Hamden Park Drive Hamden, CT 06517	**Contact:** Ted Lynn 1-800-433-9745 fax: (203) 248-6523 tblynn@dexsil.com	technology vendor
Hybrizyme 2801 Blue Ridge Rd, Suite G-70 Raleigh, NC 27607	**Contact:** Randy Allen phone: (919) 783-9595 fax: (919) 782-9585 rallen@hybrizyme.com	technology vendor
LAS Laboratories 975 Kelly Johnson Drive Las Vegas, NV 89119	no longer in business	reference laboratory for soil analyses
United Power Services Inc. 817 Fesslers Parkway Nashville, TN 37210	**Contact:** Janet Lloyd phone: (615) 255-3700 fax: (615) 256-0915	reference laboratory for oil analyses

2.2 Organization

In Figure 2-1 is presented an organizational chart depicting the lines of communication for the verification.

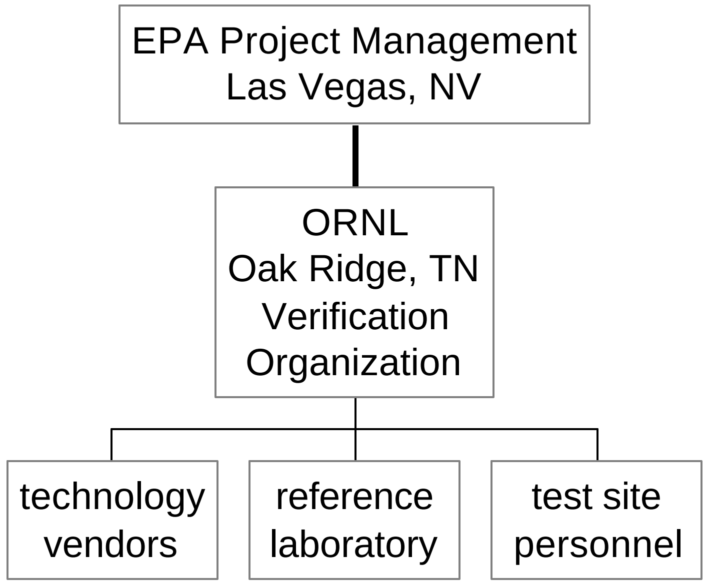

Figure 2-1. Organization chart for the verification test.

2.3 Responsibilities

The following is a delineation of each participant's responsibilities for the verification test. Henceforward, the term "vendor" applies to Dexsil and Hybrizyme.

The Vendor, in consultation with ORNL and EPA, is responsible for the following elements of this verification test:

- Contribute to the design and preparation of the verification test plan;
- Provide detailed procedures for using the technology;
- Prepare field-ready technology for verification;
- Operating and monitoring the technology during the verification;
- Documenting the methodology and operation of the technology during the verification;
- Furnish data in a format that can be compared to reference values;
- Logistical, and other support, as required.

ORNL has responsibilities for:

- Preparing the verification test plan;
- Developing a quality assurance project plan (QAPP) (Section 8 of the verification test plan);
- Preparing a health and safety plan (HASP) (Section 10 of the verification test plan) for the verification activities;
- Developing a test plan for the verification;
- Acquiring the necessary reference analysis data;
- Performing sampling activities (including collecting, homogenizing, dividing into replicates, bottling , labeling, and distributing).

ORNL and EPA have coordination and oversight responsibilities for:

- Providing needed logistical support, establishing a communication network, and scheduling and coordinating the activities of all verification participants;
- Auditing the on-site sampling activities;
- Managing, evaluating, interpreting, and reporting on data generated by the verification;
- Evaluating and reporting on the performance of the technologies.
- Site access;
- Characterization information for the site;
- Other logistical information and support needed to coordinate access to the site for the field portion of the verification, such as waste disposal.

3 TECHNOLOGY DESCRIPTIONS

This section provides descriptions of the technologies participating in the verification test. These descriptions were provided by the technology vendors, with minimal editing by ORNL. This section also describes that performance factors that will be assessed based on the data generated during the verification.

3.1 Dexsil Corporation's L2000DX Analyzer

This technology will be verified for the analysis of transformer oils only. The performance of a previous version of this instrument (L2000 PCB/Chloride Analyzer) was verified by ETV for soil and solvent extracts in 1998 [1].

3.1.1 General Technology Description

The L2000DX Analyzer (dimensions: 9" x 9.5" x 4.25") is a field portable instrument, weighing approximately 5 lbs. 12oz., designed to quantify PCB, chlorinated solvents and pesticides in soils, water, transformer oils, and surface wipes. The L2000 can be operated in the field powered by a rechargeable 8 V gel cell, or in the laboratory using 120V A.C. power. PCBs in transformer oil can be quantified over a range of 2 ppm to 2000 ppm. Total time for analysis of transformer oil is 5 minutes.

3.1.2 Transformer Oil Sample Preparation

Five milliliters of the oil is collected in a polyethylene reaction tube. Two glass ampules contained in the reaction tube are broken, introducing metallic sodium to the oil. The mixture is then shaken for ten seconds and allowed to react for a total of one minute. The sodium strips the covalently bonded chlorine atoms off the PCB molecule. An aqueous extraction solution is added to the reaction tube to adjust the pH, destroy the excess sodium, and to extract and isolate the newly formed chloride ions in a buffered aqueous solution. The aqueous layer is decanted, filtered, and collected in an analysis vial. The ion specific electrode is put into this aqueous solution to measure the millivolt potential. The potential is then converted to the equivalent PCB concentration.

3.1.3 Instrument Calibration

A one point calibration is performed prior to sample analysis. The analyst simply follows the menu driven instructions prompted in the LCD. When prompted, the instrument will ask if the calibration solution is ready. The analyst inserts the ion specific electrode into the 50 ppm chloride solution, then pushes the yes button. The instrument will then prompt the user when calibrated. Additional calibration is required when the instrument prompts the user, approximately every fifteen minutes.

3.1.4 Sample Analysis

The analyst chooses the appropriate Aroclor from the programmed menu. If the Aroclor is not known or if there is a mixture of Aroclors, Aroclor 1242 should be employed for the most conservative results. To analyze the sample, the electrode is placed into the aqueous extract solution and the enter button is pushed. After approximately 30 seconds, the PCB concentration of the samples (in ppm) is then displayed on the L2000DX LCD.

3.2 Hybrizyme's DELFIA PCB Assay

This technology will be verified for the analysis of soils and solvent extracts.

3.2.1 General Technology Description

The Hybrizyme bioassay contains an antibody that binds polychlorinated biphenyls (PCB) and has been developed for the quantitative detection of PCB in sample extracts. The signals from sample extracts are compared with a control signal to determine the relative amount of PCB present.

Figure 3-1. Hybriyzme's DELFIA PCB Assay.

The Hybrizyme DELFIA™ PCB assay is a solid phase fluoroimmunoassay. During an incubation with sample and PCB Antibody, any PCB that is present is bound to the antibody. A second antibody, which binds the PCB Antibody, is attached to the microtiter plate wells, and traps the Ab-PCB complex. The first wash step removes matrix interferences that may be in the sample. A Europium-labeled PCB compound (PCB Tracer) is then allowed to bind to any PCB Antibody binding sites that are empty. A wash step separates antibody-bound and free tracer. Following the wash step, the addition of Enhancement Solution forms highly fluorescent chelates with the bound europium ions. The amount of fluorescence measured is inversely proportional to the concentration of PCB in the sample. Hybrizyme PCB DELFIA Reagent Kit provides 40 duplicate sample analysis. Retail price is $25 per samples result (includes duplicates and controls)

3.2.2 Sensitivity

The sensitivity of the test is determined by the dilution factor used during sample processing. The detection limit is defined as the minimum concentration of PCB that can be distinguished from a blank standard with 95% confidence. Hybrizyme reports that a detection limit of 0.1 ppm Aroclor 1248 in methanol (0.2 ppm in soil) has been demonstrated with this product.

3.2.3 Cross-Reactivity

The ability of the assay to detect various Aroclors is shown in the Table 3-1.

3.2.4 Quantitative Assay Procedure

The quantitative detection of PCB in sample extracts is performed by comparing the test response of sample extracts to the test response of a control.

Table 3-1. Summary of DELFIA PCB Assay's Cross-Reactivity

Aroclor	% Reactivity
1262	110
1260	130
1254	160
1248	100
1242	40
1016	25
1232	20

1. A.C.S. reagent grade methanol is used as the Control. Perform each determination in duplicate for the Control and samples. All sample extracts must be in methanol for analysis. All reagents and samples must be brought to room temperature prior to use.

2. Prepare the needed volume of PCB Tracer Solution by diluting 50 µL of PCB Tracer stock solution in 1.5 mL of PCB Assay Buffer per strip of wells used. Example: If three strips of wells will be used, dilute 150 µL of tracer stock solution into 4.5 mL of PCB Assay Buffer. Use within one hour of preparation.

3. Prepare the PCB Antibody Solution by diluting 50 µL of PCB Antibody stock solution in 1.5 mL of PCB Assay Buffer per strip of wells used. Use within one hour of preparation.

4. Place the required number of microtitration strips in a strip frame. Wash the strips using the "PREWASH" program of the platewasher. Tap the strips upside-down gently on a paper towel to blot away any excess wash solution that may remain in the wells.

5. Pipet 100 µL of the diluted PCB Antibody Solution into each well.

6. Pipet 4 µL of each Control or sample into a well using the sequence shown in the table below. Use columns 1 and 2 for Controls on each strip of wells used.

Table 3-2. Chart for well use

	1	2	3	4	5	6	7	8	9	10	11	12
A	control	control	1st UK	1st UK	2nd UK	2nd UK	3rd UK	3rd UK	4th UK	4th UK	5th UK	5th UK
B	control	control	6th UK	6th UK	7th UK	7th UK	etc.					

7. Shake the wells for 15 minutes.

8. Wash the strips using the "3 WASHES" program on the platewasher. Tap the strips upside-down gently on a paper towel to blot away any excess wash solution that may remain in the wells.

9. Pipet 100 µL of the diluted PCB Tracer Solution into each well.

10. Shake the wells for 5 minutes.

11. Wash the strips using the "3 WASHES" program on the platewasher. Tap the strips upside-down gently on a paper towel to blot away any excess wash solution that may remain in the wells.

12. Add 150 µL of Enhancement Solution to each well.

13. Select "PCB Quant" from the list of protocols in the Time-Resolved Fluorometer and measure the fluorescence in each well. The protocol will automatically shake the wells for one minute and calculate the concentration of PCB in the extracts. The amount of PCB in the sample must be correlated using the sample processing concentration factor or dilution factor.

4 SAMPLE DESCRIPTIONS

This section discusses the history and characteristics of the verification test samples.

4.1 Soil Sample Descriptions

4.1.1 Environmentally-Contaminated Samples

Oak Ridge, Tennessee, is located in the Tennessee River Valley, 25 miles northwest of Knoxville. Three Department of Energy (DOE) facilities are located in Oak Ridge: ORNL, the Y-12 plant, and the East Tennessee Technology Park (ETTP). Chemical processing and production of components for nuclear devices have occurred at the Y-12 Plant, and ETTP is a former gaseous diffusion uranium enrichment plant. At both facilities, industrial processing associated with nuclear weapons production has resulted in the production of millions of kilograms of PCB-contaminated soils. Two other DOE facilities—the Paducah plant in Paducah, Kentucky, and the Portsmouth plant in Piketon, Ohio—are also gaseous diffusion facilities with a history of PCB contamination. During the remediation of the PCB-contaminated areas at the three DOE sites, soils were excavated from the ground where the PCB contamination occurred, packaged in containers ranging in size from 55-gallon to 110-gallon drums, and stored as PCB waste. Samples from these repositories, referred to as "Oak Ridge", "Portsmouth", and "Paducah" samples, will be used in this verification. The characteristics of these soils are summarized in Table 4-1.

In Oak Ridge, excavation activities occurred between 1991 and 1995. The Oak Ridge samples were comprised of PCB-contaminated soils from both Y-12 and ETTP. Five different sources of PCB contamination resulted in soil excavations from various dikes, drainage ditches, and catch basins. Some of the soils are EPA-listed hazardous waste due to the presence of other contaminants (e.g. diesel fuels).

A population of over 5,000 drums containing PCB-contaminated soils was generated from 1986 to 1987 during the remediation of the East Drainage Ditch at the Portsmouth Gaseous Diffusion Plant. The ditch was reported to have three primary sources of potential contamination: (1) treated effluent from a radioactive liquid treatment facility, (2) run-off from a biodegradation plot where waste oil and sludge were disposed, and (3) storm sewer discharges. In addition, waste oil was reportedly used for weed control in the ditch. Aside from PCB contamination, no other major hazardous contaminants were detected in these soils. As such, no EPA hazardous waste codes are assigned to this waste.

Twenty-nine drums of PCB-contaminated soils from the Paducah plant were generated as part of a spill cleanup activity at an organic waste storage area (C-746-R). The waste is considered a listed hazardous waste for spent solvents (EPA hazardous waste code F001) because it is known to contain trichloroethylene. Other volatile organic compounds, such as xylene, dichlorobenzene, and cresol, were also detected in the preliminary analyses of some of the Paducah samples.

4.1.2 Performance Evaluation (PE) and Blank Samples

Pre-prepared certified PE samples were obtained from Environmental Resource Associates (ERA) and EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center for use in this study. The soils purchased from ERA (Arvada, CO) were prepared using ERA's semivolatile blank soil matrix. This matrix is a top soil that has been dried, sieved, and homogenized. Particle size is approximately 60 mesh. The soil is approximately 40% clay. Samples acquired from the EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center were prepared using contaminated soils from various sites around the country in the following manner: The original soils were homogenized and diluted with a synthetic soil matrix (SSM). The SSM had a known matrix of 6% gravel, 31% sand, and 43% silt/clay; the remaining 20% was top soil. The dilution of the original soils was performed by mixing known amounts of contaminated soil with the SSM in a blender for no less than 12 hours. The samples were also spiked with target pesticides (BHC, methoxychlor, and endrin ketone) to introduce some compounds that were likely to be present in an actual environmental soil. The hydrocarbon background from the original sample and the spiked pesticides produced a challenging matrix. The soil that will be used as the uncontaminated (blank) soil is a Captina silt loam from Roane County, Tennessee. It is slightly acidic (pH ~5) and low in organic carbons (~1.5%). The soil composition is 7.7% sand, 29.8% clay, and 62.5% silt [2]. The environmental soil samples were characterized in terms of composition (% sand, % gravel, % silt/ clay, etc.), total organic carbon, and pH. This data will be reported in the technology verification report.

4.2 Extract Sample Descriptions

Traditionally, the amount of PCBs on a contaminated surface is determined by wiping the surface with a cotton pad saturated with hexane. The pad is then taken to the laboratory, extracted with additional hexane, and analyzed by gas chromatography. Unlike soil samples that can be more readily homogenized and divided, equivalent wipe samples (i.e., contaminated surfaces or post-wipe pads) are not easily obtainable. Therefore, for this study, interference-free solutions of PCBs prepared in methanol will be analyzed to simulate an extracted surface wipe pad. Extract sample analyses will provide evaluation data that primarily relies on the technology's performance rather than elements critical to the entire method (i.e., sample collection and preparation). For these samples, the vendor results will be compared to the nominal concentration values only, instead of a reference laboratory result.

4.3 Transformer Oil Sample Descriptions

4.3.1 Environmentally-Contaminated Samples

Oils contaminated with various levels of PCBs were collected from active and in-active transformers at ORNL. These transformer oils have been in-service for decades. Because of the lack of computerized records, historical information about these oils (such as when the PCBs were added, what are the oil's chemical characteristics, etc.) is unavailable. It is thought that these are all composed from mineral oil. The concentration range of these samples is < 5 ppm to nearly 200 ppm PCBs, consisting of single and multiple Aroclor mixtures (primarily 1242, 1254, and 1260, although other Aroclors may be present). Because most of the native total PCB concentrations in these samples were less than 50 ppm, ORNL augmented the Aroclor concentration of several of these samples to increase the total PCB concentration. The spiking procedure is described in Section 5.

Table 4-1. Summary of Environmentally-Contaminated Soil Sample Descriptions

Location	Request for Disposal (RFD) #	Drum #	Description
Oak Ridge	40022	02	Soil from spill cleanup at the Y-12 Plant in Oak Ridge, Tennessee. This soil is PCB-contaminated soil excavated in 1992.
Oak Ridge	40267	01 02 03 04	Soil from the Elza Gate area, a DOE Formerly Utilized Sites Remedial Action Program site in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1992.
Oak Ridge	24375	01 02 03	Catch-basin sediment from the K-711 area (old Powerhouse Area) at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated storm drain sediment that was excavated in 1991.
Oak Ridge	43275	01 02	Soil from the K-25 Building area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1993.
Oak Ridge	134555	03	Soil from the K-707 area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil from a dike spillage that was excavated in 1995.
Paducah	97002	01 02 03 04	Soil from the DOE Paducah Gaseous Diffusion Plant in Kentucky. This soil is PCB-contaminated soil from a spill cleanup at the C-746-R (Organic Waste Storage Area) that was excavated in 1989.
Portsmouth	7515	858 1069 1096 1898 2143 2528 3281 538 940 4096	Soil from the DOE Portsmouth Gaseous Diffusion Plant in Ohio. This soil is PCB-contaminated soil from a probable PCB oil spill into the East Drainage Ditch that was excavated in 1986.

4.3.2 Performance Evaluation and Blank Samples

PE samples and certified blanks were obtained from ERA. The oil used as blank and as the spiking material was purchased from Calumet Lubricants (Princeton, LA). It is called transformer oil, with its chemical name being a "severely hydrotreated light naphthenic petroleum oil", CAS # 64742-53-6. PE samples were prepared at concentrations ranging from 5 to 175 ppm, containing single Aroclors (1254 and 1260) and 50:50 mixtures of 1254 and 1260. (See Section 7 for more detailed information.)

5 SAMPLE COLLECTION AND PREPARATION

5.1 Soil Sample Collection

In Appendix A is presented the sample collection plan that was utilized in 1997 to collect the soil samples. This plan specifies the procedures that were used to ensure the consistency and integrity of the samples. In addition, this plan outlines the sample collection procedures necessary to meet the verification test purpose and objectives.

5.1.1 Sample Collection Procedures

Sampling occurred at the K-25 site for several days over the period of April 17 through May 7, 1997. Portsmouth and Oak Ridge Reservation soils were collected from B-25 storage boxes and from 55-gallon drums. Figure 6-1 is a photo of the sampling team acquiring some PCB soil samples from a 55-gallon drum.

Soil was collected from the top of the drum and placed in a plastic bag. The soil was then sifted by hand to remove rocks and other large debris, and placed in a plastic-lined 5-gallon container. Figure 6-2 shows the samplers performing this procedure. The amount of soil collected half-filled the 5-gallon container, amounting to approximately 12 kg of soil. Once the sifting was completed, the plastic liner was then removed from the container. To homogenize the soil sample, the liner was rolled on the ground in a back and forth motion, such the sample was kneaded and thoroughly mixed. Two 40-mL amber vials were fill with the homogenized soil for preliminary analytical characterization. A third sample was taken for total radiological activity screening. Paducah soil samples were collected at the site and shipped to ORNL for use in the verification test.

Figure 5-1. K-25 personnel collect a PCB sample from a 55-gallon drum.

5.1.2 Preliminary Soil Characterization

The two analytical samples taken of each field-homogenized soils were analyzed by ORNL-based Grand Junction, Colorado (ORNL-GJ) field team who performed a preliminary on-site analyses of the PCB-contaminated soils. In Appendix B is presented ORNL-GJ's analytical procedures. ORNL's Chemical and Analytical Sciences Division (CASD) also performed preliminary characterization of the PCB-contaminated soils using a similar procedure. The total PCB concentration was measured in each analytical sample to determine which samples would be used in the verification. Results from the total activity screening indicated that the soils were not considered radioactive.

Figure 5-2. K-25 sampling personnel sift through the collected soil to remove rocks and other large debris.

5.1.3 Sample Preparation for Verification Test

Aliquots of several of the environmental soils were analyzed and determined to be heterogeneous in PCB concentration. Because this is unsatisfactory for accurately comparing the performance of the field technology with the laboratory-based method, the environmental soils had to be homogenized prior to sample distribution. Each Portsmouth and Oak Ridge environmental soil sample was homogenized by first placing approximately 1500 g of soil in a glass Pyrex dish. The dish was then placed in a large oven set at 35°C, with the exhaust and blower fans turned on to circulate the air. After drying overnight, the soil was pulverized using a conventional blender and sieved using a 9-mesh screen (2 mm particle size). Last, the soil was thoroughly mixed using a spatula. A comparison of dried and undried soils showed that a minimal amount of PCBs (< 20%) was lost due to sample drying, making this procedure suitable for use in the preparation of the soil samples. The Paducah samples, because of their sandy characteristics, only required the sieving and mixing preparation steps. Multiple aliquots of each sample were analyzed using the analytical procedure described below to confirm the homogeneity of the samples with respect to PCB concentration.

To provide the vendors with soils contaminated at higher concentrations of PCBs, some of the environmental soils were spiked with additional PCBs. Spiked soils samples were prepared after the soil was first dried in a 35°C oven overnight. The dry soil was ground using a conventional blender and sieved through a 9-mesh screen (2 mm particle size). Approximately 1500 g of the sieved soil were spiked with a diethyl ether solution of PCBs at the desired concentration. The fortified soil was agitated using a mechanical shaker and then allowed to air-dry in a laboratory hood overnight. A minimum of four aliquots were analyzed using the analytical procedure described below to confirm the homogeneity of the soil with regard to the PCB concentration.

The procedure used to confirm the homogeneity of the soil samples entailed the extraction of 3 to 5 g of soil in a mixture of solvents (1 mL water, 4 mL methanol, and 5 mL hexane). After the soil/solvent mixture was agitated by a mechanical shaker, the hexane layer was removed and an aliquot was diluted for analysis. The hexane extract was analyzed on a Hewlett Packard 6890 gas chromatograph equipped with an electron capture detector and autosampler. The method used was a slightly modified version of EPA's SW-846 dual-column Method 8082 [3].

After analysis confirming homogeneity, the samples were split into jars for distribution. Each 4-oz sample jar contained approximately 20 g of soil. Four replicate splits of each soil sample were prepared for each participant. The samples were randomized in two fashions. First, the order in which the filled jars were distributed was randomized, such that the same developer did not always receive the first jar filled for a given sample set. Second, the order of analysis was randomized so that each developer analyzed the same set of samples, but in a different order. PE materials were labeled in the same manner, such that the PE samples are indistinguishable from other samples.

5.1.4 Sample Stability Study

In this study, the vendors will be analyzing the same samples that were used in the July 1997 verification of six PCB technologies and the September 1998 verification of one immunoassay technology. Soil samples are available for the verification because extra samples were prepared and stored since 1997. Prior to the 1998 and the 2000 study, ORNL performed chemical analyses of representative samples to verify that significant amounts of PCBs had not been lost due to storage. Duplicate analyses from each unique soil sample were performed. It was confirmed that no considerable losses in PCB concentration had occurred, and therefore, all soil samples (and the reference laboratory analyses performed by LAS Laboratories in 1997) will be utilized in the verification test.

5.2 Extract Samples

The extract samples were prepared by ORNL at two concentrations levels (10 and 100 μg/mL) in methanol. More detailed information is provided in Section 7. The concentrations were confirmed by ORNL's in-house laboratory. The samples were randomized and labeled similar to the soil samples (described in Section 5.1.3). The samples were stored at 4 ± 2° C until analyzed by the vendors.

5.3 Oil Samples

5.3.1 Sample Collection

Oil sample collection from active and in-active transformers occurred at ORNL in May and June 2000. Figure 5-3 is a picture of the transformer yard at ORNL's 7000 area where in-active transformers are stored. The transformers contain various levels of PCB-contamination in the oil, ranging from non-PCB classification (< 5 ppm) to PCB-containing (50 to 500 ppm). No transformers with oils containing > 500 ppm PCBs remain on the Oak Ridge site. Figure 5-4 shows an active transformer at Building 5507 which contains PCBs at regulatory levels (> 50 ppm). Samples were collected from this transformer for use in this study.

5.3.1 Sample Preparation

The oil samples did not require homogenization. The samples, contained in 4-oz glass jars, were split into 10-mL aliquots using a disposable plastic syringe. The samples were randomized and labeled similar to the soil samples (described in Section 5.1.3). As mentioned previously, most of the native concentrations of total PCBs in the environmentally-contaminated oil samples were less than 50 ppm. Several of the transformer oils were augmented with additional Aroclors (up to ~200 ppm), so that a larger dynamic range could be tested. To spike the samples, approximately 250 mL of oil was poured into a 1-L wide-mouth jar. A stir bar was added, and the jar was placed on a magnetic stirrer. With the oil being stirred, hexane solutions of known concentrations of Aroclors were added to increase the total PCB concentration. A single Aroclor was added to specific transformer oils. The specific augmented concentration levels are described in Section 7.

Figure 5-3. Transformer yard at ORNL.

Figure 5-4. Transformers at ORNL containing oils with PCB concentrations greater than 50 ppm.

6 REFERENCE LABORATORY ANALYSES

The verification process is based on the presence of a statistically validated data set against which the performance goals of the technology may be compared. The choice of an appropriate reference method and reference laboratory are critical to the success of the verification.

6.1 Methods Selection

The reference analytical method for PCBs in soil is EPA SW-846 Method 8082 [3]. The reference analytical method for PCBs in transformer oil is EPA 600/4-81-045 [4].

6.2 Reference Laboratory Selection

To assess the performance of the PCB field analytical technology, the data obtained using the technology will be compared to data obtained using conventional analytical methods. This decision is based on the experience of prospective laboratories with QA procedures, reporting requirements, and data quality parameters consistent with the goals of the Program. The laboratory must also demonstrate past proficiency with the method.

Because the PCB soil sample concentrations were statistically unchanged, the reference laboratory data generated in 1997 by LAS Laboratories will be used for comparison with the field analytical technology results. Because PCB oil analyses are being added new to this verification test, a new reference analytical laboratory, United Power Services Inc. (UPSI), Nashville, TN, was selected. The selection process and analytical methods are described below.

6.2.1 Analysis of PCB Soils - LAS Laboratories

At the time of the 1997 verification, Oak Ridge Sample Management Office (SMO) was tasked by DOE Oak Ridge Operations with maintaining a list of qualified laboratories to provide analytical services. In Appendix C are presented the standard operating procedures that SMO used to identify, qualify, and select analytical laboratories. The first procedure (LMES-ASO-AP-203, REV. 0) describes the process for selecting, adding and expelling commercial laboratories to the Lockheed Martin Energy Systems (LMES) Pricing Agreement. The second procedure (LMES-ASO-AP-210, REV. 0) defines the methodology used by Oak Ridge Sample Management Office personnel in processing statements of work (SOWs), processing purchase requisitions, and selecting commercial analytical laboratories. These activities for the procurement of commercial laboratory services were used to support projects sponsored by the DOE Oak Ridge Operations Office. The procedure served to ensure that as an operation of a DOE contractor, LMES SMO maintained an optimum level of technical and administrative oversight on each project, and SMO commercial procurement activities complied with federal acquisition laws and LMES procurement policy. Using the procedures listed in Appendix C, ORNL and SMO selected LAS Laboratories, in Las Vegas, NV, as the reference laboratory for the 1997 verification study. In Appendix D is presented the LAS standard operating procedure.

The SMO conducted on-site audits of LAS annually as part of the laboratory qualification program. At the time of selection, the most recent audit of LAS had occurred in February 1997. Results from this audit indicated that LAS was proficient in several areas, including program management, quality management, and training programs. No findings regarding PCB analytical procedure implementation were noted. A second on-site assessment of LAS occurred August 11–12, 1997, during the analysis of the verification study samples. This surveillance focused specifically on the procedures that were currently in use for the analysis of the verification samples. The audit, jointly conducted by the SMO, DOE-ORO, and EPA ESD-LV, verified that LAS was procedurally compliant. The audit team noted that LAS had excellent adherence to the analytical protocols and that the staff were knowledgeable of the requirements of the method. No findings impacting data quality were noted in the audit report.

6.2.2 Analysis of PCB Oils - United Power Services Inc.

Because LAS Laboratories was no longer in business in 2000, a new reference laboratory had to be selected for the analysis of the PCBs in oil. The industry standard test method for the determination of PCBs in transformer oils is EPA Method 600/4-81-045 [4]. A copy of the method is presented in Appendix E. A predemonstration study, described in Section 7, was used as a qualification activity for the lab. Additionally, an on-site audit of the laboratory will occur while the PCBs in oil samples are being analyzed.

7 VERIFICATION TEST DESIGN

This section discusses the objectives of the pre-demonstration study, the verification test, factors that must be considered to meet the performance objectives, and the information that ORNL and EPA will use to evaluate the results of the verification.

7.1 Pre-Demonstration Study

A pre-demonstration study is required by the SCMT pilot to allow the technology vendors to refine their technologies and revise their operating instructions, if necessary. The pre-demonstration also serves as a test of the reference laboratory. This analysis also allows an evaluation of matrix effects or interferences that may affect the verification. A failure to meet the performance goals at this point could indicate a lack of maturity of the technology and the verification would be canceled. This requirement has the following objectives:

- To allow the vendors to analyze samples that will be included in the verification in advance, and, if necessary, refine and calibrate their technologies and revise their operating instructions
- To allow an evaluation of any unanticipated matrix effects or interferences that may occur during the verification

For the pre-demonstration study, the vendors analyzed five PCB-contaminated soils (1 blank and 4 PEs) and/or six PCB-contaminated oils (1 blank, 1 spike, and 4 environmentally-contaminated); no extract samples were provided in the pre-demonstration study. PE samples were obtained from Environmental Resource Associates.

The pre-demonstration samples were sent to the vendors on May 30, 2000. The results for the pre-demonstration sample analyses were provided to ORNL approximately two weeks after the receipt of the samples (June 16, 2000).

The soils distributed were all of known concentration, due to complications with shipping DOE waste materials, and also because there were no reference analyses being performed. The vendor pre-demonstration results were compared to the performance acceptance ranges. (The acceptance ranges, based on the analytical verification data, are guidelines established by the provider of the PE materials to gauge acceptable analytical results.) For the oils, the results were compared to the reference laboratory. All of the vendors participating in this verification test demonstrated through the pre-demonstration study that their technology is prepared for rigorous field testing.

Because a new reference laboratory was being selected for the PCBs in oil analyses, a more extensive pre-demonstration study was conducted with UPSI. Forty oil samples, including environmentally-contaminated samples, spikes, and replicates, were sent blindly to UPSI for analysis. UPSI produced results that were comparable to expected concentrations, reproducible on replicate samples, and accurate on spiked samples.

7.2 Objective of the Verification Test

The primary objectives of this verification are to evaluate the PCB field analytical technologies in the following areas: (1) how well each performs relative to conventional analytical methods, (2) PE results, and (3) the logistical and economic resources necessary to operate the technology. Secondary objectives for this verification are to evaluate the PCB field analytical technology in terms of its reliability, ruggedness, cost, range of usefulness, sample throughput, data quality, and ease of operation. Specifically, the verification process will evaluate the performance of the technology against the performance goals as stated in Section 3.

7.3. Summary of Verification Activities

The verification test will be held at ORNL (see Figure 7-1) from August 21 through September 1, 2000. The vendors have the option of analyzing soils (208 samples), extracts (24 samples), and/or oils (152 samples) contaminated with PCBs. The samples evaluated during the verification will consist of (1) environmental soil samples from the Oak Ridge Reservation, Paducah, and Portsmouth DOE sites, (2) spiked environmental soil samples, (3) purchased certified soil samples, (4) ORNL-prepared methanol extract samples, (5) transformer oils, (6) spiked transformer oils, and (7) purchased, certified oil samples. The verification soil and oil samples have been homogenized and split such that the vendor is supplied with equivalent samples analyzed by a fixed analytical laboratory (referred to as the reference lab). The field technology results for the extract samples will be compared to the nominal spike concentration. The experimental design approach is presented in Tables 7-1 through 7-6.

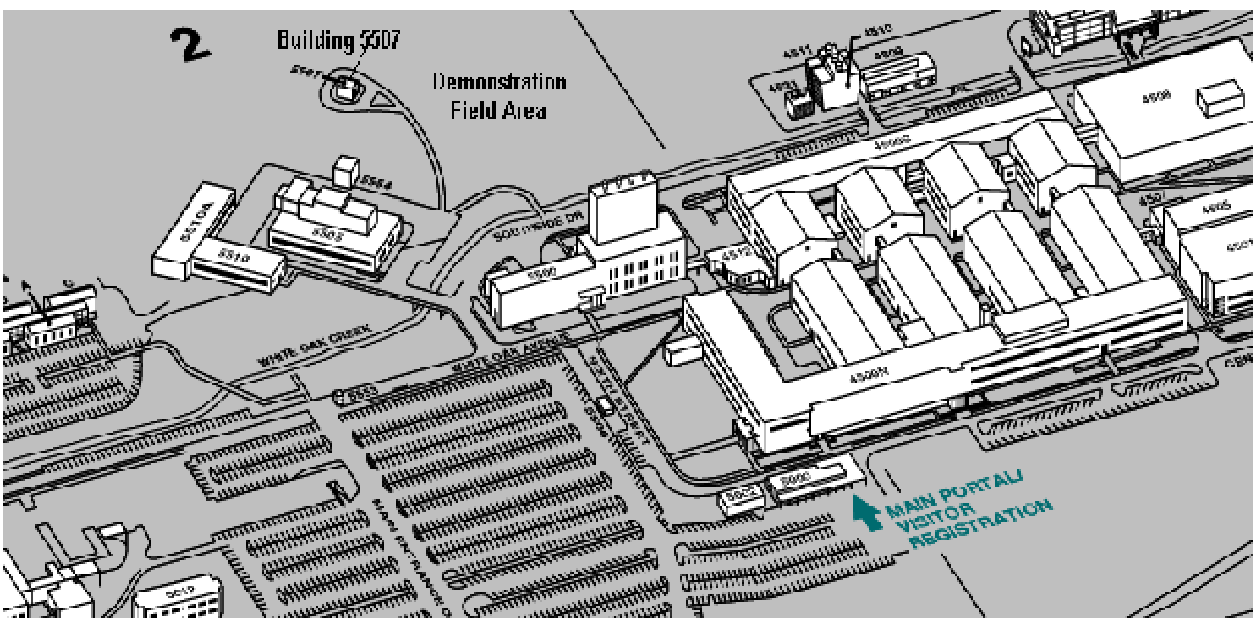

Figure 5-1. Field Verification Test site at ORNL, near Building 5507.

Table 7-1. Summary of Environmental Soil Sample Analyses (by Drum Number)

Target Concentration Range	Outdoor Site				Chamber Site			
	Oak Ridge#1	Oak Ridge#2	Paducah#1	Totals # Samples	Paducah#1	Portsmouth#1	Portsmouth#2	Total # Samples
0.1 - 2.0 ppm	40022-02 [a]	24375-01 40267-02 24375-02	97002-04 97002-01	28	97002-04 97002-01	7515-4096		12
2.1 - 20.0 ppm	40267-03 40267-01 40267-04		97002-03	16	97002-03	7515-1898	7515-2528 7515-3281	20
20.1 - 50.0 ppm		134555-03S	97002-02	12	97002-02	7515-1096 7515-2143	7515-1069 7515-0858	24
50.1 - 700 ppm	40267-01S [b] 24375-03	43275-01 43275-02	97002-02S	12	97002-02S	7515-0940 7515-0538S	7515-0538 7515-0538S	12
Total # samples	24	24	20	68	24	24	20	68
Grand Total	**136**							

[a] Four replicates will be analyzed for each drum number.
[b] "S" indicates that the sample is a matrix spiked environmental sample.

Table 7-2. Summary of Performance Evaluation Soil Samples

Sample	Concentration (ppm)	Number of Replicates	
		Outdoor Site	Chamber Site
Aroclor 1248 [a]	2	4	4
	20	4	4
Aroclor 1254 [a]	5	4	4
	50	4	4
Aroclor 1260 [a]	11	4	4
	50	4	4
Mixture of Aroclor 1254 and 1260 [b]	2 [c]	4	4
	50 [c]	4	4
Uncontaminated (blank) soil (Tennessee Reference Soil)	n/a	4	4
Total # samples		**36**	**36**
Grand Total		**72**	

[a] Provided by the EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center.
[b] Provided by Environmental Resource Associates.
[c] Total PCB concentration

Table 7-3. Summary of Extract Sample Analyses

Sample Concentration	Number of Replicates		Grand Total
	Outdoor Site	Chamber Site	
10 µg/mL	4	4	8
100 µg/mL	4	4	8
Methanol Blank	4	4	8
Total # samples	12	12	**24**

Table 7-4. Summary of Oil Sample Analyses

Target Concentration Range	Number of Samples [a]	
	Environmental	**PE**
blank	2	5
≤ 5.0 ppm	2	1
5.1 - 25.0 ppm	4	1
25.1 - 40.0 ppm	4	1
40.1 - 50.0 ppm	3	1
50.1 - 75.0 ppm	4	2
75.1 - 100.0 ppm	2	1
> 100 ppm	4	1
Total # samples, including 4 replicates each	100	52
Grand Total	**152**	

[a] Four replicates will be analyzed for each.

Table 7-5. Summary of Oil PE Samples

Nominal Concentration (ppm)	Aroclor	Ratio in Mixture
5	1254	n/a
25	1260	n/a
40	1254/1260	50/50
50	1254/1260	50/50
60	1254/1260	50/50
75	1260	n/a
100	1254	n/a
175	1254/1260	50/50

Table 7-6. Summary of Verification Analyses

Sample Type	Number of Samples
Environmental soil samples	136
PE soil samples	72
Extract samples	24
Environmental oil samples	100
PE oil samples	52
Grand Total	**384**

7.3.1 Environmental Conditions for the Test

Verification activities for the soils and extracts will occur at two sites at ORNL: a natural outdoor environment (the outdoor site) and inside a controlled environmental atmosphere chamber (the chamber site). Generally, the average August temperature for eastern Tennessee is 77°F. Studies will also be conducted inside a controlled environmental atmosphere chamber, hereafter referred to as the "chamber", located in Building 5507 at ORNL. The controlled experimental atmosphere facility consists of a room-size, walk-in chamber ten feet wide and twelve feet in length with air processing equipment to control temperature and humidity. Verification studies inside the chamber will be used to evaluate performance under environmental conditions that are markedly different from the ambient outdoor conditions at the time of the test. The temperatures in the chamber during the testing periods will be set at 55 °F. The temperature and relative humidity will be monitored at both sites during the testing.

7.4 Sample Distribution

ORNL will be responsible for sample distribution. The samples will be packaged in 4 ounce (120 mL) jars, as described in Section 6. All samples will be prepared for distribution at the start of the verification. The vendors will go to a sample distribution table located in Building 5507 to pick-up the samples. The samples will be distributed in batches of 12. Completion of chains-of-custody will document sample transfer.

7.5 Archive Samples

Archive samples which are replicates of the vendor samples will be retained by ORNL. An archive sample will be used during the verification if the integrity of a vendor's sample has been compromised. Additional unhomogenized material and unused archive samples will also be retained at ORNL at the completion of the verification, in case any questions arise where reanalysis is necessary.

7.6 Submission of Results

The vendor will provide the results to ORNL. The vendor will be responsible for reducing the raw data into a presentation format consistent with the evaluation requirements. At the end of the verification test, the vendor will submit all final results and raw data to ORNL. After the conclusion of the field activities, the vendors will have one week to review their data and make revisions to their results. These revisions will not involve re-analysis of any sample. The revisions will be limited to correcting for calculation and transcription errors.

7.7 Verification Performance Factors

The following are the logistical and technical performance verification factors that will be verified for each technology.

- Accuracy: closeness of technology result to known value;
- Precision: reproducibility of technology's results;
- Comparability: performance relative to reference laboratory;
- False positive results: number of blanks where PCBs detected;
- False negative results: number of contaminated samples that technology reported as non-detect;
- Sample throughput: number of samples/hour/number of analysts
- Application to regulatory-decision making: performance at regulatory decision-making levels for PCBs (50 ppm for soils and oils and 100 $\mu g/100cm^2$ for surface wipes).

These factors and the anticipated statistical analyses are further discussed in Section 8.

8 QUALITY ASSURANCE PROJECT PLAN (QAPP)

The QAPP for this verification test specifies procedures that will be used to ensure data quality and integrity. Careful adherence to these procedures will ensure that data generated from the verification will meet the desired performance objectives and will provide sound analytical results.

8.1 Purpose and Scope

The primary purpose of this section is to outline steps that will be taken to ensure that data resulting from this verification is of known quality and that a sufficient number of critical measurements are taken. This section is written in compliance with the SCMT Quality Management Plan [5].

8.2 Quality Assurance Responsibilities

The implementation of the verification test plan must be consistent with the requirements of the study and routine operation of the technology. The ORNL technical lead is responsible for coordinating the preparation of the QAPP for this verification and for its approval by EPA and ORNL. The ORNL program manager will ensure that the QAPP is implemented during all verification activities. ORNL's QA specialist (QAS) will review and approve the QAPP and will provide QA oversight of the verification activities. The ORNL technical lead will be responsible for the reference laboratory data validation. The ORNL statistician will primarily be responsible for the reduction of the vendor and reference laboratory data. The EPA project manager and QA manager will review and approve this plan.

8.3 Field Operations

8.3.1 Site Training

Preliminary site training will be provided to all vendors on the first day of testing. This will be required before initiation of the field study. This training will be conducted by the ORNL program manager or his designee. It will entail an overview of the test site, safety information, emergency procedures, and logistical information regarding the verification test.

8.3.2 Communication and Documentation

Successful field operations require detailed planning and extensive communication. ORNL will communicate regularly with the verification participants to coordinate all field activities associated with this verification and to resolve any logistical, technical, or QA issues that may arise as the verification progresses. Pertinent vendor and ORNL field activities will be thoroughly documented. Field documentation will include field logbooks, photographs, field data sheets, and chain-of-custody forms.

The ORNL technical lead 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. Any deviations from the approved final verification test plan will be thoroughly documented in the field logbook and provided to the ORNL. Photographs will be taken with a digital camera.

8.4 Performance and System Audits

The following audits will be performed during this verification.

8.4.1 Technical Systems Audit

ORNL's QAS will perform a surveillance during verification testing to assess compliance with the demonstration plan.

8.4.2 Data quality audit of the reference laboratory

UPSI will be audited during the analyses of the oil samples. The audit will focus on adherence to method requirements and procedures, particularly in sample preparation, sample management, and quality control.

8.4.3 Surveillance of Technology Performance

During verification testing, ORNL staff will observe the operation of the field technology, such as observing the vendor operations, photo-documenting the demonstration activities, surveying calibration procedures, and reviewing sample data. The observations will be documented in a laboratory notebook or by completing a field audit form.

8.5 Quality Assurance Reports

QA reports provide 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 verification.

8.5.1 Status Reports

ORNL will regularly inform the EPA project manager of the status of the verification. Project progress, problems and associated corrective actions, and future scheduled activities associated with the verification test will be discussed. When problems occur, the vendor and ORNL will discuss them, estimate the type and degree of impact, describe the corrective actions taken to mitigate the impact and to prevent a recurrence of the problems, and discuss with EPA, as necessary. Major problems will be documented in the field logbook.

8.5.2 Audit Reports

Any QA audits or inspections that take place in the field while the verification test is being conducted will be formally reported by the auditors to the ORNL technical lead, who will forward them to the EPA project manager. Informal reporting of audit results will be reported immediately to EPA.

8.6 Corrective Actions

Routine corrective action may result from common monitoring activities, such as:

- Performance evaluation audits
- Technical systems audits
- Calibration procedures

If the problem identified is technical in nature, the individual vendors will be responsible for seeing that the problem is resolved. If the issue is one that is identified by ORNL or EPA, the identifying party will be responsible for seeing that the issue is properly resolved. All corrective actions will be documented. Any occurrence that causes discrepancies from the verification test plan will be noted in the technology verification report. The reference laboratory procedures (See Appendices D and E) describe the corrective action plan for not meeting minimum QC requirements.

8.7 Reference Laboratory Quality Control Checks

Quality control (QC) samples will be analyzed by UPSI to indicate whether or not the samples were analyzed properly. A summary of QC samples include: initial calibration, continuing calibration verification, and analysis of known samples (spiked Aroclors in isooctane and oil). This data will be reviewed by ORNL as part of the data validation process. Discrepancies will be noted in the data validation records.

Note that the LAS reference laboratory data for soil analyses has already been reviewed and validated by ORNL.

8.8 Data Management

The vendor, ORNL, and EPA each have distinct responsibilities for managing and analyzing verification data. ORNL is responsible for managing all the data and information generated during the verification test. The vendor is responsible for obtaining, reducing, interpreting, validating, and reporting the data associated with their technology's performance. These data should be reported on the chain-of-custody. Vendor results will be due to ORNL at the conclusion of a day's field activities. The vendor's final report will be due to ORNL one week after the verification. Any discrepancies between the originally reported result and the final result must be described. EPA and ORNL are responsible for analysis and verification of the data.

8.9 Data Reporting, Validation, and Analysis

To maintain good data quality, specific procedures will be followed during data reduction, review, and reporting. These procedures are detailed below.

8.9.1 Data Reporting

Data reduction refers to the process of converting the raw results into a concentration which will be used for evaluation of performance. The procedures to be used will be technology dependent, but the following is required for data reporting:

- The reported PCB concentration should be either total PCB concentration or PCB concentration by Aroclor. The result will be definitively labeled as such.
- The concentration units for oil and soil samples will be parts per million (i.e., ppm, as received) and µg/mL for extract samples.
- If no PCB is detected, the concentration should be reported as less than the reporting limits of the technology, with the reporting limits stated (e.g., < 0.5 ppm). If the technology reports interval results, a non-detect will be reported as the lowest interval (e.g., 0 to 0.5 ppm).

8.9.2 Data Validation

Validation determines the quality of the results relative to the end use of the data. ORNL will be responsible for validating the reference laboratory data. (Note that the vendor is responsible for validating its own data prior to final submission.) Several aspects of the data (listed below) will be reviewed. The findings of the review will be documented in the validation records. As appropriate, the ETVR will describe instances of failure to meet quality objectives and the potential impact on data quality.

8.9.2.1 Completeness of Laboratory Records

This qualitative review ensures that all of the samples that were sent to the laboratory were analyzed, and that all of the applicable records and relevant results are included in the data package.

8.9.2.2 Holding Times

For oil, the method requirement is that the samples be prepared within 14 days of receipt and analyzed within 40 days of preparation. Adherence to this requirement will be reviewed for all samples.

8.9.2.3 Correctness of Data

So as not to bias the assessment of the technology's performance, errors in the reference laboratory data will be corrected as necessary. Corrections may be made to data that has transcription errors, calculation errors, and interpretation errors. These changes will be made conservatively, and will be based on the guidelines provided in the method used. The changes will be justified and documented in the validation records.

8.9.2.4 Correlation Between Replicates

Normally, one would not know if a single sample result was "suspect" unless (a) the sample was a spiked sample, where the concentration is known or (b) a result was reported and flagged by the reference laboratory as suspect for some obvious reason (e.g., no quantitative result was determined). The experimental design implemented in this verification study will provide an additional indication of the abnormality of data through the inspection of the replicate results from homogenous sample sets. Criteria has been established to determine if data is suspect. Data sets will be considered suspect if the percent relative standard deviation for replicate samples was greater than 50%, because this criteria would indicate imprecision. These data would be flagged so as not to bias the assessment of the technology's performance. Precision and accuracy evaluations may be made with and without these suspect values to represent the best and worst case scenarios. If both the reference laboratory and the vendor(s) report erratic results, the data may be discarded if it is suspected that the erratic results are due to a sampling error.

8.9.2.5 Evaluation of QC Results

As stated in Section 8.7, QC samples will be analyzed by the reference laboratory with every batch of samples to indicate whether or not the samples were analyzed properly. Performance on these samples will be reviewed and major findings will be noted in the validation records.

8.9.2.6 Evaluation of Spiked Sample Data

Spiked samples are homogenous samples containing known concentrations of analyte(s). The performance of the reference laboratory will be evaluated relative to the spiked samples. Results for these samples represent the best estimate of accuracy and precision for verification testing.

8.9.3 Data Analysis

This section contains a list of the five primary performance factors to be evaluated for both the field technology and the reference laboratory.

8.9.3.1 Precision

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. Environmental applications often involve situations where "measurements of the same materials" can take on a number of interpretations. In environmental applications, precision is often best specified as a percentage of contaminant concentration. The following lists several possible interpretations of precision for environmental applications.

1) The precision involved in repeated measurements of the same sample without adjusting the test equipment.

2) The precision involved in repeated measurements of the same sample after reset, repositioning, or re-calibration of the test equipment or when using different equipment of the same technology.

3) The precision of measurements due to spatial variability of soil samples from adjacent locations.

4) The precision characteristics of a specific technology in determining contamination at a specific site or at an arbitrary site.

In general, users of the technology will want to be assured that measurement variability in 1) and 2) is small. Measurement variability due to spatial variability described in 3) is likely to be site specific and is minimized in this verification by using homogeneous samples. The measurement variability discussed in 4) is perhaps of most interest as it includes measurement variability resulting from possible differences in the design activities and effects of environmental conditions such as temperature that would vary from one site characterization to another as well as site and technology specific sources.

The strength of this verification's experimental design is that since an equal number of replicates will be performed for every sample at every concentration level, an equal number of precision comparisons can be made. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance.

Precision for this verification will be estimated by the variance, or standard deviation from the measured data. If “n” PCB concentration measurements are represented by Y_1, Y_2, ..., Y_n, the estimated variance about their average value “$\bar{Y}$” is calculated by:

$$S^2 = \frac{1}{n-1} \sum_{k=1}^{n} (Y_k - \bar{Y})^2 \quad .$$

The standard deviation is the square root of S^2 and implies that the uncertainty is independent of the PCB concentration values. To express the reproducibility relative to the average PCB concentration, percent relative standard deviation (RSD) is used to quantify precision, according to the following equation:

RSD = (standard deviation / average concentration) x 100%

Replicate samples at each PCB concentration can be used to establish the relationship between the uncertainty and the average PCB concentration. RSD cannot be calculated for PCB concentration results reported as interval data. To assess precision, the frequency of results reported as the same interval will be determined.

8.9.3.2 Accuracy

Accuracy is a measure of how close, on average, the measured PCB concentrations are to the true values or to an accepted value. Accuracy for the PCB verification will be relative to a spiked PCB concentration in the performance evaluation samples, computed as percent recovery using the equation:

$$\textit{percent recovery} = (\textit{measured amount} / \textit{spiked amount}) \times 100\%$$

The optimum percent recovery value is 100%. Percent recovery values greater than 100% indicate results that are biased high, and values less than 100% indicate results that are biased low. Percent recovery will be used to assess the accuracy of the reference laboratory and of technologies which report quantitative results. For technologies which produce interval results, accuracy will be evaluated in terms of the percentage of samples which agree with, are above (i.e., biased high), and are below the certified values (i.e., biased low).

Inaccuracies or biases are the result of systematic differences between measured and true values. These biases may be due to limited calibration range, systematic errors, standards preparation, storage and homogeneity of the soil samples either at the PCB verification or at the reference laboratory. Consequently every effort will be made by ORNL, the technology vendors and the reference laboratory to identify specific sources of inaccuracies. The verification includes blanks, replicates, and performance evaluation samples that should provide substantiating evidence to support this partitioning of sources of bias when results become available.

8.9.3.3 False Positive/False Negative Results

A false positive (fp) result is one in which the technology detects PCBs in the sample when there actually are none [6]. A false negative (fn) result is one in which the technology indicates that no PCBs are present in the sample, when there actually are [6]. The evaluation of fp and fn results is influenced by the actual concentration in the sample and includes an assessment of the reporting limits of the technology. False positive results will be assessed in two ways. First, the results will be assessed relative to the blanks (i.e., the technology reports a detected value when the sample is a blank). Second, the results will be assessed on environmental and spiked samples where the analyte was not detected by the reference laboratory (i.e., the reference laboratory reports a nondetect and the field technology reports a detection). False negative results, also assessed for environmental and spiked samples, indicate the frequency that the technology reported a nondetect (i.e., < reporting limits) and the reference laboratory reported a detection. The reporting limit will be considered in the evaluation. For example, if the reference laboratory reported a result as 0.9 ppm, and the technology's paired result was reported as below reporting limits (<1 ppm), the technology's result will be considered correct and not a false negative result.

8.9.3.4 Comparability

Comparability refers to how well the field technology and reference laboratory data agree. The difference between accuracy and comparability is that whereas accuracy is judged relative to a known value, comparability is judged relative to the results of a standard or reference procedure, which may or may not report the results accurately. A one-to-one sample comparison of the technology results and the reference laboratory results will be performed in the ETVR.

A correlation coefficient quantifies the linear relationship between two measurements [7]. The correlation coefficient is denoted by the letter *r;* its value ranges from –1 to +1, where 0 indicates the absence of any linear relationship. The value $r = -1$ indicates a perfect negative linear relation (one measurement decreases as the second measurement increases); the value $r = +1$ indicates a perfect positive linear relation (one measurement increases as the second measurement increases). The slope of the linear regression line, denoted by the letter *m*, is related to *r*. Whereas *r* represents the linear association between the vendor and reference laboratory concentrations, *m* quantifies the amount of change in the vendor's measurements relative to the reference laboratory's measurements. A value of +1 for the slope indicates perfect agreement. Values greater than 1 indicate that the vendor results are generally higher than the reference laboratory, while values less than 1 indicate that the vendor results are usually lower than the reference laboratory.

In addition, a direct comparison between the field technology and reference laboratory data will be performed by evaluating the percent difference (%D) between the measured concentrations, defined as

$$\%D = ([\textit{field technology}] - [\textit{ref lab}]) / (\textit{ref lab}) \times 100\%.$$

8.9.3.5 Completeness

Completeness refers to the amount of data collected from a measurement process expressed as a percentage of the data that would be obtained using an ideal process under ideal conditions. The completeness objective for data generated during this verification is 95% or better.

There are many instances which might cause the sample analysis to be incomplete. Some of these are:

- Instrument failure
- Calibration requirements not being met
- Evaluated analyte levels in the method blank

9 HEALTH AND SAFETY PLAN

This section describes the specific health and safety procedures that will be used during the field work at the Oak Ridge National Laboratory.

9.1 Contact Information

The ORNL project manager will be Roger Jenkins, (865) 576-8594.
The ORNL technical lead will be Amy Dindal, (865) 574-4863.
The ES&H Coordinator will be Fred Smith, (865) 574-4945.
The Environmental Protection Officer will be Kim Jeskie, (865) 574-4947.
The Laboratory Shift Superintendent number is (865) 574-6606.
The Emergency Communications Center number is (865) 574-6646.
IN CASE OF ANY EMERGENCY, DIAL 9-1-1.

Emergency phone numbers will be posted at the test site.

9.2 Health and Safety Plan Enforcement

ORNL project manager, ORNL technical lead, and the ES&H Coordinator will be responsible for enforcing the health and safety plan. ORNL project manager will ultimately be responsible for ensuring that all verification participants abide by the requirements of this HASP. ORNL technical lead will oversee and direct field activities and is also responsible for ensuring compliance with this HASP.

9.3 Site Access

Visitors will be badged and escorted at all times by ORNL personnel. Visitors will follow standard ORNL safety and health policies and practices. Site training will be provided to the vendors prior to testing.

9.4 Waste Generation

All hazardous waste generated by the technology vendors will be properly disposed of by the Environmental Protection Officer. The technology vendors will assist with this process by providing accurate records of the waste contents and approximate concentrations.

9.5 Hazard Evaluation

PCBs will be the most prevalent chemical hazard at the verification test. PCBs are:

- Nonflammable liquids;
- Carcinogenic;
- Viscous liquids with a mild, hydrocarbon odor.

Some possible health effects from exposure to PCBs are: (1) irritation to the eyes and skin, possibly forming an acne condition; and (2) liver damage. If PCBs contact the skin, immediately wash the contaminated skin with soap and water. If PCBs penetrate the clothing, immediately remove the clothing and wash the skin with soap and water. Get medical attention promptly.

PCBs issues and hazards will be controlled per ORNL procedures (Oak Ridge Reservation Polychlorinated Biphenyl Federal Facilities Compliance Agreement, ORR-PCB-FFCA, and ORNL-EP-P04, Management of Polychlorinated Biphenyls). These procedures can be found on ORNL's interval web site.

Other hazards associated with this verification test include worker exposure to volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and additional physical hazards associated with the technology's equipment. Plastic ground covers will be placed underneath each technology set-up, in order to collect any spills of soil or solvent. Ground covers will be replaced as necessary.

Exposure to VOCs and SVOCs during field activities may occur through inhalations or ingestion. The most likely exposure to VOCs and SVOCs during the verification test will be through dermal contact. Dermal contact with contaminated soil will be prevented through the use of personal protective equipment (PPE), such as gloves. The technology vendors must provide their own PPE. Although unlikely to be necessary, visitors will be provided with PPE if warranted.

9.6 Personal Protection

Personal Protective Equipment (PPE) shall be appropriate to protect against known and potential health hazards encountered during routine operation of the technology systems. For this verification, Level D PPE is required. Level D provides minimal protection against chemical hazards. It consists only as a work uniform, with gloves worn, where necessary. <u>Level D PPE will be supplied by the individual technology vendor</u>. ORNL will provide visitors with PPE if necessary. If site conditions or the results of Industrial Hygiene surveillance indicates that additional hazards are present, ORNL may recommened different or additional PPE to the vendors. The following is the list of protective equipment <u>required</u> for verification operations:

- Appropriate work clothes (no shorts or open-toed shoes);
- Safety glasses.

9.7 Physical Hazards

Physical hazards associated with field activities present a potential threat to on-site personnel. Dangers are posed by unseen obstacles, noise, heat, and poor illumination. Injuries may results from the following:

- Accidents due to slipping, tripping, or falling
- Improper lifting techniques
- Moving or rotating equipment
- Improperly maintained equipment

Injuries resulting from physical hazards can be avoided by adopting safe work practices and by using caution when working with machinery.

9.8 Fire

The following specific actions will be taken to reduce the potential for fire during site activities:

- No smoking within 20 feet of the site.
- Fire extinguishers will be maintained on-site.
- All personnel will be trained on the location of the portable fire extinguishers.

9.9 Mechanical, Electrical, Noise Hazards

Some technology-specific hazards may be identified once the vendors set-up their equipment. Proper hazards controls (i.e., guarding or markings) or PPE (i.e., ear plugs for noise hazards) will be implemented as necessary.

Electrical cables represent a potential tripping hazards. When practical, cables will be placed in areas of low pedestrian travel. If necessary, in high pedestrian travel areas, covers will be installed over cables.

9.10 Unstable/Uneven Terrain

The terrain around Building 5507 is uneven and bumpy. Site personnel shall be aware of uneven terrain to avoid slips, trips, and falls.

9.11 Inclement Weather

The verification test will occur the latter part of August. The possibility of inclement weather (particularly rain and thundershowers) exists. The vendors should be prepared to deal with a possible inclement weather situation.

Operating temperatures in the chamber could be as low as 50°F. Vendors should be prepared to work in those temperatures.

9.12 Heat Stress

Since the verification test will occur in August, the possibility of a heat-related injury during field work is possible. Heat stress symptoms include heat cramps, heat exhaustion, and heat stroke. Heat stroke is the most serious condition and can be life-threatening. To combat heat-related injuries, ORNL will:

- Provide water to all verification participants;
- Establish a work regimen that will provide adequate rest periods;
- Provide access to air-conditioned buildings;
- Notify all workers of health hazards and the importance of adequate rest.

Some symptoms of heat-related injuries are pale clammy skin, sweating, headache, weakness, dizziness, and nausea. Signs of heat stroke include dry, hot, red skin, chills, and confusion. In the case of a suspected heat-related injury, try to cool the person down and contact medical help.

9.13 Insect and Other Animal Stings and Bites

Building 5507 is located in a relatively secluded part of the Laboratory (see Figure 7-1). A potential for insect and other animal stings or bites exists during the technology verification. Insect repellent may be used to minimize insect bite hazards. In the event of snake or other large animal bite, the injury should be immobilized and immediately reported to medical personnel.

9.14 Medical Support

A complete medical facility is located on-site in Building 4500 North. Medical help can be summoned from any laboratory phone by dialing 9-1-1. The 911 system automatically contacts the Lab Emergency Response Center and Emergency Communications Center, and Medical. Pulling a fire alarm box will summon the fire department and the laboratory shift superintendent's office.

9.15 Environmental Surveillance

The Environmental Protection Officer will be responsible for surveying the site before, during, and after the verification test. Appropriate personnel will be on-hand to assist all verification participants to deal with any health or safety concerns.

9.16 Safe Work Practices

Each vendor will provide the required training and equipment for their personnel to meet safe operating practice and procedures. The individual technology vendor and their company are ultimately responsible for the safety of their workers.

The following safe work practices will be implemented at the site for worker safety:

- Eating, drinking, chewing tobacco, and smoking will be permitted only in designated areas;
- Wash facilities will be utilized by all personnel before eating, drinking, or toilet facility use;
- PPE requirements (See Section 9.6) will be followed.

9.17 Complaints

All complaints should be filed with the ORNL technical lead. All complaints will be treated on an individual basis and be dealt with accordingly.

9.18 Radiological Hazards

The PCB-contaminated samples that will be used in this verification test have been analyzed and found not to be radioactive. However, if an issue concerning radioactivity would occur during the verification ORNL-radiation procedures will be applied, where applicable.

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[3] EPA (U.S. Environmental Protection Agency). 1996. "Method 8082: Polychlorinated Biphenyls (PCBs) by Gas Chromatography." In *Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods, Update II.* SW-846. U.S. Environmental Protection Agency, Washington, D.C., December.

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[6] Berger, W., H. McCarty, and R-K. Smith. 1996. *Environmental Laboratory Data Evaluation* Genium Publishing Corp., Schenectady, N.Y.

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