July 16, 1997

Technology Demonstration Plan

Evaluation of Polychlorinated Biphenyl (PCB) Field Analytical Techniques

Sponsored by:

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

AND

U. S. Department of Energy
Cloverleaf Building, 19901 Germantown Road
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Prepared by:

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Oak Ridge, TN 37831-6120

APPROVAL SIGNATURES

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/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.

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_____

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Electronic Sensor Technology: _____

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Sentex Systems: _____

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Strategic Diagnostics, Inc.: _____

printed name

_____

signature/date

FORWARD

This work represents the technical and editorial contributions from a large number of U. S. Environmental Protection Agency (U. S. EPA) employees and others familiar with or interested in the demonstration and evaluation of innovative site characterization and monitoring technologies. The Characterization Research Division - Las Vegas (CRD-LV) first convened a body of experts--the Consortium Action Team--to define the elements of the guidance document. Subsequent discussions and meetings were held to revise and expand the contents to create the latest version (5.0) of the guidance document, which was used to prepare this demonstration plan. EPA staff from each of the ten Regions, the Office of Solid Waste and Emergency Response, and the Office of Research and Development participated in this process. This interdisciplinary, inter-programmatic team was convened to ensure that the demonstration procedures articulated are acceptable across the Agency. This was an important first step for gaining the acceptance of innovative technologies for use in characterizing and monitoring the environment.

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EXECUTIVE SUMMARY

The Consortium for Site Characterization Technology (CSCT), which is part of the Environmental Protection Agency's (EPA) Environmental Technology Verification (ETV) program, is a partnership between the EPA, and the Department of Energy (DOE) and Department of Defense (DoD) that offers valuable technical expertise to support the demonstration and verification of the performance of new and emerging technologies and access to a wide array of testing venues.

A goal of the Consortium is to facilitate the acceptance and use of cost-effective technologies applicable to a wide range of environmental problems. The Consortium will meet this goal by working with technology developers and other agencies in planning and conducting demonstrations, evaluating data generated in demonstrations and managing and disseminating information. The Consortium is not intended to become another technology testing organization that must touch every technology, but rather it is designed to support existing demonstration efforts or developer-driven demonstrations.

The purpose of the demonstration to be conducted in Oak Ridge is to evaluate field analytical technologies which are capable of detecting and quantitating polychlorinated biphenyls (PCBs). A fundamental objective of this demonstration is to evaluate how well the technologies can assist in regulatory decision-making processes for PCB-contaminated waste. The U. S. Environmental Protection Agency's National Exposure Research Laboratory, Characterization Research Division-Las Vegas, Nevada (EPA, NERL, CRD-LV) in collaboration with the U. S. Department of Energy's Environmental Management Program (DOE, EM) is sponsoring this project. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the demonstration. ORNL's role is to provide technical and administrative leadership and support in conducting the demonstration. Five technology developers will participate in this demonstration: Dexsil Corporation, Hach Company, Electronic Sensor Technology, Sentex Systems, Inc., and Strategic Diagnostics, Inc.

The demonstration of PCB field analytical techniques will be conducted at the Oak Ridge National Laboratory from July 22 through July 30. Usually two technology demonstrations are conducted at different geographic locations, to verify technology performance under different geologic and climatologic conditions. For this demonstration, we will leverage EPA and DOE resources by conducting the demonstration at one site (ORNL) and utilizing a controlled environmental atmosphere chamber. The soil samples evaluated during the demonstration will consist of: (1) environmental soil samples from the DOE's Oak Ridge Reservation, Paducah, and Portsmouth sites; (2) spiked environmental soil samples; and (3) purchased certified soil samples. The demonstration samples will be homogenized and split such that each developer and the fixed analytical laboratory (referred to as the reference lab) are supplied with equivalent samples. The technologies' ability to analyze surface wipe sample extracts will also be evaluated.

ABBREVIATIONS AND ACRONYMS

ANOVA	Analysis of Variance
CASD	Chemical and Analytical Sciences Division
CRD-LV	Characterization Research Division-Las Vegas
CSCT	Consortium for Site Characterization Technology
DoD	Department of Defense
DOE	U. S. Department of Energy
EPA	U. S. Environmental Protection Agency
ERA	Environmental Resource Associates
ESH&Q	Environmental Safety, Health, and Quality
GC	gas chromatography
FN	False Negative error rate
FP	False Positive error rate
IST	Ion Signature Technology
LCS	Laboratory Control Sample
LMER	Lockheed Martin Energy Research
LMES	Lockheed Martin Energy Systems
MS	mass spectrometry
MS/MSD	matrix spike/matrix spike duplicate
ORNL	Oak Ridge National Laboratory
ORNL-GJ	Oak Ridge National Laboratory, Grand Junction, Colorado
OSW	Office of Solid Waste
PCBs	polychlorinated biphenyls
PDS	performance demonstration sample
PE	performance evaluation
PPE	personal protective equipment
QA	Quality Assurance

QAPP	Quality Assurance Project Plan
QC	Quality Control
SDI	Strategic Diagnostics, Inc.
SMO	sample management office
SOW	statement of work
SVOCs	semivolatile organic compounds
VOCs	volatile organic compounds

1.0 INTRODUCTION

This chapter discusses the purpose of the demonstration and the demonstration plan, describes the elements of the demonstration plan, and provides an overview of the Consortium for Site Characterization Technology (CSCT) and the technology verification process.

1.1 Demonstration Objectives

The purpose of this demonstration is to evaluate field analytical technologies which are capable of detecting and quantitating polychlorinated biphenyls (PCBs). The U. S. Environmental Protection Agency's National Exposure Research Laboratory, Characterization Research Division-Las Vegas, Nevada (EPA, NERL, CRD-LV) in collaboration with the U. S. Department of Energy's Environmental Management Program (DOE, EM) will be sponsoring this project. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the demonstration. Some of the objectives proposed for this demonstration are designed to evaluate how well the technologies can assist in regulatory decision-making processes for PCB-contaminated waste.

Specifically, this plan defines the following elements of the demonstration:

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

1.2 What is the Consortium for Site Characterization Technology?

The Consortium for Site Characterization Technology (CSCT) is a partnership between the EPA, and the Department of Energy and Department of Defense. DoD and DOE have established programs and facilities (testing venues) for testing, demonstrating, and evaluating the performance of monitoring, measurement and site characterization technologies, among other technologies. As a partnership, the Consortium will offer valuable technical expertise to support the demonstration and verification of the performance of new and emerging technologies and will offer access to a wide array of testing venues.

A goal of the Consortium is to facilitate the acceptance and use of cost-effective technologies applicable to a wide range of environmental problems. The Consortium will meet this goal by working with technology developers and other agencies in planning and conducting demonstrations, evaluating data generated in demonstrations and managing and disseminating information. The Consortium is not intended to become another technology testing organization that must touch every technology, but rather it is designed to support existing demonstration efforts or developer-driven demonstrations. The Consortium does not offer any financial

support to those desiring to conduct a technology demonstration. The developer is expected to secure the appropriate resources to support their part of the technology verification process.

1.3 Technology Verification Process

The technology verification process established by the Consortium is intended to serve as a template for conducting technology demonstrations that will generate high quality data that the Agency can use to verify technology performance. The Consortium verification process is a model process that can help in moving innovative site characterization and monitoring technologies into routine use more quickly. After the completion of the selection process, the verification of a technology's performance involves five steps:

1. Development of a demonstration/test plan.
2. Execution of the demonstration.
3. Data reduction, analysis, and cost verification.
4. Report preparation
5. Information transfer.

Although the Agency is interested in any and all innovative site characterization and monitoring technologies, the Consortium resources, and those of the verification organization, are limited. Therefore, a major role of the Consortium is to identify the technology and data gaps that impede cost-effective and efficient environmental problem-solving and to communicate them to the developer community. This assessment identifies those technologies that meet the most pressing needs. The information that supports the assessment will be gathered from within EPA, other Federal agencies, states, tribes, and the user industries to ensure that the most pressing needs and gaps are addressed first.

1.4 Purpose of this Demonstration Plan

The purpose of the demonstration plan is to describe the procedures that will be used to verify the performance goals of a technology. This document incorporates the QA/QC elements needed to provide data of appropriate quality sufficient to reach a defensible position regarding the technology performance. This is not a method validation study, nor does it represent every environmental situation which may be acceptable for this technology. 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 demonstration.

2.0 DEMONSTRATION RESPONSIBILITIES AND COMMUNICATION

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

2.1 Demonstration Organization and Participants

Participants in this demonstration are listed in Table 1. The specific responsibilities of each demonstration participant are discussed in Section 2.3 This demonstration 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, Characterization Research Division - Las Vegas, Nevada (CRD-LV) and the U. S. Department of Energy’s Environmental Management

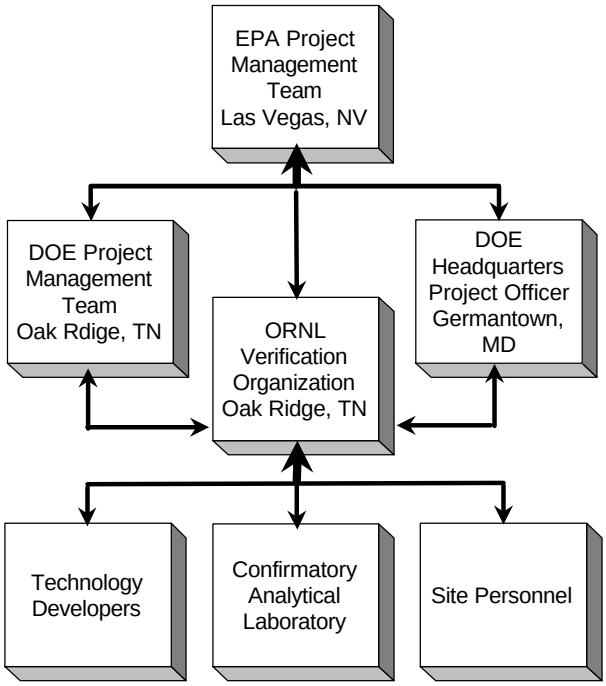
Program. CRD-LV and DOE's roles are to administer the CSCT Demonstration Program. ORNL's role is to provide technical and administrative leadership and support in conducting the demonstration. There are 5 technology developers participating in this demonstration: Dexsil Corporation (Dexsil), Hach Company (Hach), Electronic Sensor Technology (EST), Sentex Systems, Inc. (Sentex), and Strategic Diagnostics, Inc. (SDI).

Table 1 - Demonstration Participants
PCB Field Analytical Technology Demonstration

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 A. Jenkins, Ph.D. phone: (423) 576-8594 fax: (423) 576-7956 jenkinsra@ornl.gov **Technical Lead:** Amy B. Dindal phone: (423) 574-4863 fax: (423) 576-7956 dindalab@ornl.gov **Site Operations/ESH&Q:** Fred J. Smith phone: (423) 574-4945 fax: (423) 574-6721 smithfj@ornl.gov	verification organization
U. S. EPA Office of Research and Development National Exposure Research Laboratory Characterization Research Division P.O. Box 93478 Las Vegas, NV 89193-3478	**Project Officer:** Stephen Billets, Ph.D. phone: (702) 798-2232 fax: (702) 798-2261 email: billets-stephen@wpmail.las.epa.gov **QA Officer:** Gary Robertson phone: (702) 798-2215 fax: (702) 798-2261 email: robertson-gary@wpmail.las.epa.gov	EPA project management
U. S. DOE Cloverleaf Building 19901 Germantown Road Germantown, MD 20874	**Project Officer:** David Bottrell phone: (301) 903-7251 fax: (301) 903-7613 email: DAVID.BOTTRELL@em.doe.gov	DOE Headquarters project officer

U. S. DOE ORNL Site Office P.O. Box 2008 Bldg. 4500N, MS-6269 Oak Ridge, TN 37831-6269 Oak Ridge Operations Office Three Main Street Oak Ridge, TN 37830	**Program Coordinator:** Regina T. Chung phone: (423) 576-9902 fax: (423) 574-9275 email: chungr@ornl.gov **Technical Program Manager:** David Carden phone: (423) 576-9262 fax: (423) 576-6074 email: CARDENDM@oro.doe.gov	DOE/ORO project management team
Dexsil Corporation One Hamden Park Drive Hamden, CT 06517	Jack Mahon Wendy Schutt-Young phone: (208) 288-3509 fax: (208) 288-6523	technology developer
Hach Company 5600 Lindbergh Drive P.O. Box 539 Loveland, CO 80539	Ann Foster John Parsons phone: (970) 669-3050, x2266 fax: (970) 669-2932	technology developer
Electronic Sensor Technology 1077 Business Center Circle Newbury Park, CA 91320	Ed Staples phone: (805) 480-1994 fax: (805) 480-1984	technology developer
Sentex Systems, Inc. 553 Broad Avenue Ridgefield, NJ 07657	Marie Velasco phone: (201) 945-3694 fax: (201) 941-6064	technology developer
Strategic Diagnostics, Inc. 128 Sandy Drive Newark, DE 19713-1147	Craig Kostyshyn phone: (302) 456-6789 fax: (302) 456-6782	technology developer
Oak Ridge National Laboratory 2597 B-3/4 Road Grand Junction, CO 81503	Frank Gardner phone: (970) 248-6238 fax: (970) 248-6147	in-field support laboratory
LAS Laboratories 975 Kelly Johnson Drive Las Vegas, NV 89119	Mary Ford phone: (702) 361-3955 fax: (702) 361-8146	reference laboratory

Figure 1 - Organization Chart
PCB Field Analytical Technology Demonstration



Refer to Table 1 for the names of specific individuals involved in demonstration

2.2 Organization

In Figure 1 is presented an organizational chart depicting the lines of communication for the demonstration. Note that the double-arrow lines signify that each participant is encouraged to openly communicate with other members of the demonstration team.

2.3 Responsibilities

The following is a delineation of each participant's responsibilities for the demonstration. ORNL will perform the duties which are common to all of the developers. Henceforward, the term "developer" applies to each of the 5 technology developers that are participating in the PCB demonstration, namely Dexsil Corporation (Dexsil), Hach Company (Hach), Electronic Sensor Technology (EST), Sentex Systems, Inc. (Sentex), and Strategic Diagnostics, Inc. (SDI).

The Developer, in consultation with ORNL, DOE, and the EPA technical lead, is responsible for the following elements of this demonstration:

- Contribute to the design and preparation of the demonstration plan;
- Provide detailed procedures for using the technology;
- Prepare field-ready technology for demonstration;

- Operating and monitoring the technology during the demonstration;
- Documenting the developer's methodology and operation of the technology during the demonstration;
- Furnish data in a format that can be compared to reference values;
- Logistical, and other support, as required.

ORNL has responsibilities for:

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

ORNL, DOE, and EPA have coordination and oversight responsibilities for:

- Providing needed logistical support, establishing a communication network, and scheduling and coordinating the activities of all demonstration participants;
- Ensuring that appropriate sites are selected consistent with the objectives of the demonstration;
- Auditing the on-site sampling activities;
- Managing, evaluating, interpreting, and reporting on data generated by the demonstration; and,
- 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 demonstration, such as waste disposal.

3.0 TECHNOLOGY DESCRIPTION

This section provides a description of each PCB field analytical technology that is participating in the demonstration. The descriptions were provided by the technology developers, with minimal editing by ORNL.

3.1 Dexsil Corporation

GENERAL TECHNOLOGY DESCRIPTION

The L2000 PCB analyzer is a field portable instrument designed to quantify PCB concentration in soil, dielectric fluids and surface wipes. PCB in soil can be quantified over a range of 3 ppm to 2000 ppm with the ability to extend the range over 2000 ppm by reducing the sample size. Total time for analysis of soil is 10 minutes, dielectric fluid is 5 minutes and surface wipes is 12 minutes. Cost for analysis of soil is $8.00 to $10.00/sample, dielectric fluid is $3.75 to $5.00/sample and $12.00 to $16.00/sample for surface wipes. Initial cost investment for purchasing the system is $3500.

SOIL

SAMPLE PREPARATION

Sample preparation consists of extraction and dehalogenation of the PCB. The resulting chloride ions are then isolated in an aqueous buffered solution for analysis by the L2000 Analyzer.

Ten grams of soil is weighed into a polyethylene test tube. The soil is extracted with a premeasured, non-chlorinated solvent. The soil is allowed to settle and the supernatant is decanted onto a Florisil column. The solution is passed through the column where all the water and inorganic chloride is removed. Five milliliters is collected in a polyethylene reaction tube.

Both glass ampules are broken, introducing metallic sodium to the extract solution. 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 extract the newly formed chloride ions away from the oil. The aqueous layer is decanted off the oil then run through a filter into an analysis vial. The ion specific electrode is put into this aqueous solution where the millivolt potential of the chloride solution is measured and converted to ppm PCB.

Instrument Operation

Calibration: A one point calibration is run prior to analyzing the sample. The analyst simply selects calibration mode and inserts the electrode into a 50 ppm chloride solution supplied with the reagents. A start button is pushed and a "wait light" illuminates for approximately 30 seconds when a "read light" illuminates, the analyst calibrates the instrument by turning the calibration knob until the LCD reads 50 ppm. The instrument is now calibrated. Additional calibration is required when the recalibrate light illuminates approximately every 20 minutes or after the completion of 15 - 20 samples.

Analysis of Sample: The analyst can choose four different PCB setting; 1242, 1260, Askarel A (60% Aroclor

1260 plus 40% trichlorobenzene) and total chloride, depending on the site characterization profile. If the Aroclor is not known or if there is a mixture of Aroclors, the 1242 setting should be employed for the most conservative results. Total chloride setting is used if "odd" Aroclors (1221, 1248, etc.) are encountered or other chlorinated organics wished to be quantified. Simply divide the total chlorine results by the percent chlorine on the analyte times 100 and this will calculate out to ppm of the analyte. When the "read light" illuminates, in approximately 30 seconds, the concentration of PCB is read off the LCD.

The electrode is placed into the aqueous extract solution and the start button is pushed. The concentration of PCB is then read off the LCD in approximately 30 seconds when the "read light" illuminates.

Sample preparation and analysis take about ten minutes and one to 15 samples can be run concurrently.

SURFACE WIPES

Supplied with:

Chromatographic Grade Hexane in 2 mL sealed glass ampules.
Disposable PCB rated gloves.
Disposable forceps.
Goggles.
Gauze pads.
Reagents and vials.

A 1000* cm^2 area is wiped with chromatographic grade hexane. The PCB is then extracted from the gauze and five mls of extract is introduced into the sodium reaction tube. **Once in the reaction tube, the procedure is exactly the same as for the soil analysis.**

The PCB is measured as µg/100cm^2. Range 2 - 2000 µg/100 cm^2.

*1000 cm^2 area must be wiped to have the concentration for PCB at µg/100cm^2 when using the L2000 Analyzer.

3.2 Hach Corporation

General Description of how Immunoassay Technology Works - Immunoassay is a technique for detecting and measuring a target compound using an antibody which binds only to that substance. Antibodies recognize and latch onto a target substance even in a test tube. Increasingly, immunoassay is being applied to environmental analysis because of its selectivity, accuracy, speed, low limits of detection, economy and high through put. The antibodies in an immunoassay can zero-in on a target even within a complex sample matrix, often needing little or no sample preparation.

Basics Components of Immunoassay Technology - Immunoassay is not a biological technique, it is a physical assay whose chemical and physical reactions follow mathematically-based laws. Most types of immunoassay have these basic components:

- An *antigen*, or target substance to be analyzed (i.e. the analyte)
- An *antibody*, which binds specifically to the analyte
- A way to separate the *bound* from the free (unbound) analyte (called a separation method)

- A *label* or a *tag* on the antibody or the analyte enabling it to be recognized (The label may cause a color change or emit a signal. A test using an enzyme label is called an *enzyme immunoassay*, EIA.)
- *Standards* or *controls* containing known concentrations of analyte, provide the gauge for interpreting test results

The Antibody-antigen Reaction - Antibodies are a group of globular proteins (immunoglobulins) that help search out and destroy foreign substances that invade the body. Each antibody is custom-made in the immune system of vertebrates in response to an invader (or antigen). The word "antigen" is short for "antibody generating".

There are binding sites on the antibody that bind non-covalently to antigenic determinants, or epitopes, on the corresponding antigen. The close interaction of these complementary structures at the molecular levels explains how an antibody can bind so specifically to a particular antigen. The results of this binding is an antibody-antigen complex. The strength of the antibody-antigen bond is known as the *affinity constant*, and it can be mathematically calculated.

In immunoassay, the antibody seeks the analyte (antigen) or target substance to be analyzed. The antibody's affinity for the analyte determines the ultimate sensitivity of the test.

Production of Antibodies - The initial source of the antibodies used in immunoassay is an animal. It is often a rabbit, guinea pig, goat, mouse or other mammal that can be immunized with the antigen and produce antibodies of the desired characteristics.

Antibody Sensitivity - An immunoassay is only as good as its antibody. A *sensitive* antibody will detect even small amounts of analyte in the sample. A *specific* antibody will bind primarily (if not exclusively) to the target and ignore similar compounds.

Antibody sensitivity is measured in terms of *cross-reactivity*, or to what degree the antibody will bind to a substance other than its target. If an antibody binds equally to another compound, it is said to be 100% cross-reactive. Specificity is commonly measured by determining how much of another substance is necessary to create 50% reduction in the assay response.

Cross-reactivity may be a problem, if you are testing for one analyte in a matrix that may hold other similar compounds. But cross-reactivity is desirable if you are screening for a number of related compounds such as a family of pesticides. In this case, one cross-reactive antibody can do the work of several specific antibodies.

Summary Of the Hach Method - Samples, standards and reagents are added to test tubes coated with an antibody specific for PCBs. The concentration of PCBs in a sample is determined by comparing the developed color intensity to that of a PCB standard. The PCB concentration is inversely proportional to the color development; the lighter the color, the higher the PCB concentration.

This method is a semi-quantitative screening method which indicates whether the PCB concentration is above or below 1 ppm and/or 10 ppm threshold values. If the site has a clean-up guideline set to 10 ppm threshold, the analyst might choose to test at 10 ppm. If the presence of PCBs needs to be determined, the analyst may wish to use the 1 ppm threshold.

Physical Construction/Components of the Hach Immunoassay Kit for PCB Analysis - The Hach Immunoassay Test Kit for field analysis of PCB is designed for maximum convenience and is packaged in a durable polypropylene carrying case. The kit is rugged, easy to clean and built from the highest quality materials. Everything needed for the testing is supplied with the kit. Components are molded from durable plastic and are ideal for in field use where safety is a concern.

The kit includes: A Hach Pocket Colorimeter® instrument, designed for use with immunoassay-based analysis, four AAA batteries, reagents for five (5) PCB tests, labware required to run the analysis (including micro pipets, test tubes, test tube rack, reagent mixing bottles, portable scale) and instruction manual. The Hach Pocket Colorimeter® instrument supplied with the kit is a low cost, high quality filter photometer designed for single wavelength colorimetric measurement. The liquid crystal display provides a readout in counts.

General Environmental Requirements and Limitations - As part of good laboratory practice, it is best to familiarize yourself with the reagents in this procedure.

WARNING *The reagents used in the procedure may be hazardous if inappropriately handled or accidentally misused. Read all warnings on the Material Safety Data Sheets (MSDS) and reagent labels.*

It is always a good practice to wear safety glasses when handling chemicals. Follow instruction carefully. Rinse thoroughly if contact occurs. If Stop Solution or Soil Extractant Solution comes in contact with eyes, wash thoroughly for 15 minutes with cold water and seek immediate medical help.

The Soil Extractant contains methyl alcohol which is poisonous and flammable. Read MSDS sheet before using this reagent.

Weight - The shipping weight of the kit is 26.5 lb.

Transportability - The Hach Immunoassay Test Kit for field analysis of PCB is rugged and easy to use in the field. The case is designed to prevent kit components from shifting and breaking during transportation and use. Inserts prevent messy spills by keeping reagents stored in an upright position.

Ruggedness - The carrying case is built of durable polypropylene and will stand up to the harshest conditions. The case insert is also molded polypropylene and easy to clean.

Power Needed - Power is supplied by four AAA batteries (supplied with the kit). Typically, a set of batteries provides approximately 750 tests. Due to a battery-saving feature. incorporated into the software, the instrument will automatically shut off if no keystrokes are made for one minute.

Sample Matrices - The Hach Immunoassay PCB field analysis method instructions cover soil only. Hach does not supply a surface wipe technology, nor any other surface sampling technology for the measurement of PCB.

Sensitivity, Concentration Range and Aroclors - For concentration sensitivity, to assist in regulatory decision making, the instructions for the Hach Immunoassay PCB field analysis method currently cover making 1 and 10 ppm screening standards. Result interpretation is restricted to noting samples significantly above or below the standard used or approximately equal to the standard. Preparation of other standard concentrations, e.g. 2 or 50 ppm should be possible but is not covered in the method manual. Determining a sample between 2 and 50 ppm would require two screening tests and an additional dilution step, using both 2 and 50 ppm standards.

For the measurement of Aroclors and/or specific PCB compounds, please see the table on the next page. The method can not differentiate various PCBs. Sensitivity to specific chemicals varies, see table. If a sample contains only a specific known chemical, the standard concentration can probably be adjusted to reflect that compound's sensitivity and therefore make screening at selected thresholds possible.

PCBs are sold under the commercial name Aroclor. This method measures all commercial Aroclors and is sensitive to the most common Aroclors: 1248, 1254, and 1260. See the table on the next page for sensitivities to various Aroclors. Sensitivity to other halogenated compounds is generally less than 1% of the response to Aroclor 1260, making interference problems insignificant.

Sensitivity to Aroclor and other Compounds

Compound	Concentration necessary to give a positive result at 1 ppm threshold
Aroclor 1260	0.4 ppm
Aroclor 1254	0.4 ppm
Aroclor 1248	1 ppm
Aroclor 1242	2 ppm
Aroclor 1016	4 ppm
Aroclor 1232	4 ppm
Other Halogenated Compounds	
2,4,6-trichloro-*p-terphenyl*	>10,000 ppm
Halowax 1013	10,000 ppm
Halowax 1051	1,000 ppm
o,p -DDT	>10,000 ppm
2,4-D	10,000 ppm
Silvex	1,000 ppm
bifenox	1,000 ppm
tetradifon	100 ppm
Dicofop methyl	1,000 ppm
dichlorofenthion	10,000 ppm
trichloroethylene	>10,000
1,2,4-trichlorobenzene	10,000 ppm
2,4-dichloro-1-naphthol	50 ppm
2,4-dichlorophenyl benzene sulfonate	1,000 ppm
1-chloronaphthalene	>10,000 ppm
pentachlorobenzene	>10,000 ppm
hexachlorobenzene	>10,000 ppm
2,5-dichloroanaline	>10,000 ppm
Miscellaneous Compounds	
Toluene	>10,000 ppm
Naphthalene	>10,000 ppm
DIALA(R) Oil AX	>10,000 ppm
Envirotemp 200 fluid	>10,000 ppm

Diesel Fuel	>10,000 ppm
Gasoline	>10,000 ppm

Product validation studies indicate the test correctly identifies over 95% of samples that are spiked with PCBs at or above the chosen action (threshold) level.

Cost - The initial cost of the Hach Immunoassay Test Kit for field analysis of PCB is $955.40. The kit includes: A Hach Pocket Colorimeter® instrument designed for use with immunoassay-based analysis, reagents for five (5) PCB tests, labware required to run the analysis and instruction manual. The kit is supplied in a polypropylene carrying case. Standards are required, but not included in the reagent set or kit. The number of standards used will vary, but one ampule will possibly last for a day of testing. The PCB Standard Ampules, 350 µg/L, package of 5, cost $19.60. Replacement reagents are packaged five tests to a set and cost $175.40. Cost per test is $35.00 ($175.40/5= $35.08).

Training Requirements - The kit is supplied with detailed instructions to guide the user step by step through each procedure and interpretation of the results. The user does not have to be a trained chemist to get professional results with the Hach method.

Sample Handling/PCB in Soils Analysis Procedures By Hach Company

Immunoassay Overview

Hach immunoassay tests use analyte-specific antibodies attached to the inside of plastic tubes to selectively bind and remove analyte molecules from complex sample matrices. Samples that may contain the analyte molecule and a reagent containing enzyme conjugate molecules are added to the antibody tubes. An enzyme conjugate molecule is an analyte molecule that is attached to an enzyme. Enzyme conjugate molecules and analyte molecules bind to the antibodies attached to the inside of the tubes. Thus, the analyte and enzyme conjugate molecules compete for the antibody sites. So, samples with higher levels of analyte will have more antibody sites occupied by analyte molecules and fewer antibody sites occupied by the enzyme conjugate molecules after incubation.

After incubation, the sample and unbound enzyme conjugate are washed from the tube and color development reagents are added. Color development only occurs in the presence of enzyme conjugate molecules. The more enzyme conjugate molecules attached to the antibody on the tube, the more intense the resulting color. The more analyte molecules attached to the antibody on the tube, the less intense the resulting color. Hach immunoassay methods compare sample results with a standard to determine whether the analyte concentrations in the sample are greater or less than the threshold levels.

Measuring Hints

- Timing is critical; follow instructions carefully.
- Run duplicate tubes for each standard and sample.
- Handle the Antibody Tubes carefully. Scratching the inside or outside may cause erroneous results. Clean the outside of the tubes with a clean absorbent cloth or tissue before placing them into the instrument. Hold all dropper bottles vertical and direct the drops at the bottom of the tube.
- Antibody Tubes and Enzyme Conjugate are made in matched lots. Do not mix with other reagent lots.
- Paper towels, liquid waste container, and laboratory tissue are required, but not supplied with the kit.
- The tests provide semi-quantitative screening. They are designed to indicate whether the sample concentrations are above or below a specific threshold. The specific threshold is determined by the concentration of the standard used and dilution of sample extracts.
- The tests require about 30 minutes for complete analysis of one set of samples.

- The Soil Extractant contains methyl alcohol which is poisonous and flammable. Read Material Safety Data Sheet before using this reagent.
- Read the entire procedure before starting. Locate and identify all reagents, tubes and apparatus before analysis.

WARNING *The reagents used in the procedure may be hazardous if inappropriately handled or accidentally misused. Read all warnings on the Material Safety Data Sheets (MSDS) and reagent labels.*

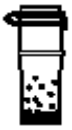
PCBs in Soil (Phase 1 - Soil Extraction)

1. Fill the extraction vial to the 0.75-oz line with Soil Extractant Solution. This is equivalent to adding 20 mL of the Soil Extractant. ***Note****: Read Measuring Hints Section before testing.*

2. Place a plastic weighing boat on the AccuLab balance. Zero the balance. *Note: Refer to the AccuLab Instructions for balance operation.*

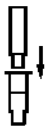
3. Weigh out 10 ±0.1 g of soil in a plastic weighing boat. Carefully pour the soil into the extraction vial.

4. Cap the extraction vial tightly and shake vigorously for 1 minute.



5. Allow to settle for 1 minute. Gently open the extraction vial.

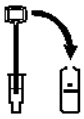
6. Using the disposable bulb pipet, withdraw 1.0-1.5 mL from the liquid (top) layer in the extraction vial. Transfer into the filtration barrel (the bottom part of the filtering assembly; the plunger inserts into it). Note: *not use more than 1.5 mL. The bulb is marked in 0.25-mL increments.*



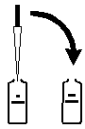
7. Insert the filtration plunger into the filtration barrel. Press firmly on the plunger until at least 0.5 mL of filtered sample is collected in the center of the plunger. ***Note:*** *The liquid is forced up through the filter. The liquid in the plunger is the sample extract. It may be necessary to place the filtration assembly on a table and press down on the plunger.*

PCBs in Soil (Phase 2 - Diluting Standards and Samples)

1. To prepare a 1 ppm threshold dilution, snap open a 1-ppm Dilution Ampule. Label the Dilution Ampule with appropriate sample information.

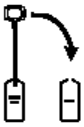


2. Using the WireTrol pipet, withdraw 100 µL (0.1 mL) of sample extract from the filtration plunger and add it to the 1-ppm Dilution Ampule. Swirl to mix. Discard the capillary tube. ***Note:*** *The lower line on the capillary tube is 100 µl.*



3. To prepare a 10-ppm threshold dilution, snap open a 10-ppm Dilution Ampule. Label the Dilution Ampule. Using a TenSette Pipet, withdraw 1.0 mL from the 1-ppm Dilution Ampule (Step 2) and add it to the 10-ppm Dilution Ampule. Swirl to mix.

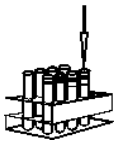
4. To prepare the standard, snap open a PCB Standard Ampule. Snap open a 1-ppm Dilution Ampule. Label the Dilution Ampule as "Standard".



5. Using the WireTrol pipet, withdraw 100 µL (0.1 mL) of the standard and add it to the 1-ppm Dilution Ampule. Swirl to mix. ***Note:*** Use the standard dilution prepared above for both 1-ppm and 10-ppm threshold. Do not further dilute the standard.

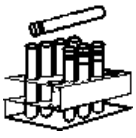
PCBs in Soil (Phase 3 - Immunoassay) Steps in this phase require exact timing.

1. Label two PCB Antibody Tubes for each dilution ampule. Likewise, label two PCB Enzyme Conjugate Tubes for each dilution ampule. ***Note:*** *The PCB Antibody and PCB Enzyme Conjugate Tubes are matched lots. Mixing with other reagent lots will cause erroneous results.*



2. Use a TenSette Pipet to add a 1.0-mL aliquot from each dilution ampule prepared (1- ppm or 10-ppm) to the bottom of each appropriately-labeled PCB Antibody Tube. Do this for each sample and standard. Use a new pipet tip for each solution.

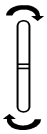
3. Begin a 10-minute reaction period.



4. At the end of the 10-minute reaction period, decant the solution from the Antibody Tubes into the respective Enzyme Conjugate Tubes.

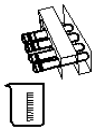
5. Invert and place the Antibody Tubes over the Enzyme Conjugate Tubes until they fit tightly onto the Enzyme Conjugate Tubes.

6. Begin a 5-minute reaction period. ***Note:*** *Immediately proceed with the next step while the timer counts down.*

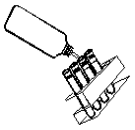


7. Immediately invert the solution repeatedly until the Antibody Tube has been filled four times and the enzyme conjugate has been dissolved. After the last inversion make sure that all of the solution is in the Antibody Tube and that it is upright.

8. Place the Antibody Tube in the rack and remove the Enzyme Conjugate Tube from the mouth of the Antibody Tube. Discard the used Enzyme Conjugate Tube.



9. After the 5-minute period, discard the contents of the PCB Antibody Tubes into an appropriate waste container.

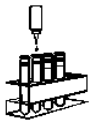


10. Wash each tube forcefully and thoroughly 4 times with Wash Solution. Empty the tubes into an appropriate waste container. Shake well to ensure most of the Wash Solution drains after each wash. ***Note:*** *Wash Solution is a harmless dilute detergent.*

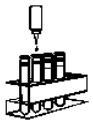
Go to Phase 4 Now

11. Continue to the next phase immediately. ***Note:*** *Ensure most of the Wash Solution is drained from the tubes by turning the tubes upside down and gently tapping them on a paper towel to drain. Some foam may be left from the Wash Solution; this will not affect results.*

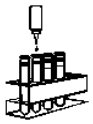
PCBs in Soil (Phase 4 - Color Development) Check reagent labels carefully! Reagents must be added in proper order.



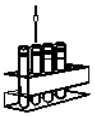
1. Add 5 drops of Solution A to each tube. Replace the bottle cap. ***Note****: Hold all reagent bottles vertically for accurate delivery, or erroneous results may occur.*



2. Begin a 2.5-minute reaction period and immediately add 5 drops of Solution B to each tube. Swirl to mix. Replace the bottle cap. ***Note:*** *Solution will turn blue in some or all of the tubes.*

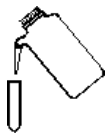


3. After exactly 2.5 minutes add 5 drops of Stop Solution to each tube. Replace the bottle cap. ***Note****: Blue solutions will turn yellow when Stop Solution is added.*

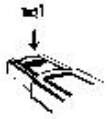


4. Using the TenSette Pipet and a new tip, add 0.5 mL of deionized water to each tube. Swirl to mix. ***Note:*** *PCB concentration is inversely proportional to color development; less color indicates higher PCB levels.*

PCBs in Soil (Phase 5 - Color Measurement)



1. Label and fill the Zeroing Tube with deionized water. Wipe the outside of all the tubes with a tissue to remove smudges and fingerprints.



2. Insert the Immunoassay Tube Adapter into the cell holder.

3. Insert the Zeroing Tube into the cell holder. Cover the Zeroing Tube with the instrument cap.

4. Press: ZERO. The instrument will turn on and the display will show - - -, followed by 0. ***Note:*** *Discard the Zeroing Tube after use.*

5. Insert the Standard #1 tube into the cell holder. Cover the tube with the instrument cap.

6. Press: READ. Record the count value displayed. Hold the adapter in place when removing the tube.

7. Repeat Steps 5 and 6 for the Standard #2 tube. ***Note:*** *If Standard #1 and #2 are more than 250 counts apart, repeat the test beginning at Phase 2 Standard Preparation.*

8. Insert the Sample #1 tube into the cell holder. Cover the tube with the instrument cap.

9. Press: READ. Record the count value displayed. Hold the adapter in place when removing the tube. ***Note:*** *Flashing 0 indicates analyte concentrations much greater than the standard. Flashing 990 indicates analyte concentration much less than the standard.*

10. Repeat Steps 8 and 9 for the Sample #2 tube.

See Table 1, Below

11. See Table 1 to interpret results.

Table 1 Determining if Samples are above PCB Threshold Values

If sample count is...	1 ppm Threshold	10 ppm Threshold
.. less than highest standard count	Sample PCB is greater than 1 ppm	Sample PCB is greater than 10 ppm
.. greater than highest standard count	Sample PCB is less than 1 ppm	Sample PCB is less than 10 ppm

FOR A COPY OF HACH'S PROCEDURE AT NO COST, PLEASE CALL 1-800-227-4224

Performance -Although semi-quantitative results are possible with the product, normal usage is as a screening tool in which standards are prepared and samples are screened as having more or less than the threshold value. The method provides instructions for preparing 1 and 10 ppm standards. The method indicates whether the PCB concentration is above or below 1 ppm and/or 10 ppm threshold values. For example, if the site has a clean-up guideline set to 10 ppm threshold, the analyst might choose to test at 10 ppm. If the presence of PCB needs to be determined, the analyst may wish to use the 1 ppm threshold.

Product validation studies indicate the test correctly identifies over 95% of samples that are spiked with PCBs at or above the chosen action (threshold) level.

Accuracy Check - To confirm results, use a reference analytical method chosen from standard methods approved by the USEPA (SW-846 Method 8080A, 8081A or 8082 or appropriate CLP method).

HISTORY OF THE TECHNOLOGY

Immunoassay is not a new technology, it was introduced in 1960 and has long been a preferred analytical technique in clinical chemistry and endocrinology labs. It has been used to detect a wide range of substances including hormones, drugs and viruses. Immunodiagnostics are so simple to run that test kits are now common in the home (i.e. test kits for pregnancy hormones).

TECHNOLOGY APPLICATION

Potential Users of the Technology - The Hach Immunoassay Method for field analysis of PCB is suited for environmental professionals, extension agencies, soil analysts, utilities and the natural gas pipeline industry. The kit is also ideal for use by analysts responsible for testing contaminated soils on-site, monitoring remediation sites and evaluating the progress of remediation.

Advantages of the Hach Immunoassay product for PCB

- The Hach immunoassay product is conveniently packaged into a portable kit and can be carried into the field for on-site analysis of PCB. A rugged carrying case contains everything necessary for analysis.
- The Hach immunoassay product uses standards to ensures accurate results. The procedure includes replicate standards for 1 and 10 ppm thresholds.
- The Hach immunoassay product detects low levels of PCB - down to 1 ppm.
- The Hach immunoassay product is very selective, no false negatives, even in complex samples.
- The Hach immunoassay product is economic, providing results at a fraction of the laboratory cost.
- The Hach immunoassay product can process large numbers of samples rapidly.

Benefits of Immunoassay Technology

- Immunoassay based tests are easily automated. Immunoassay produces a colored end-product that can be accurately and easily measured with a colorimeter or spectrophotometer.
- Immunoassay based test can easily be made portable. Immunoassay can be conveniently conducted in polystyrene test tubes and easily packaged into a portable kit.
- Immunoassay based tests are not significantly affected by the composition of the sample (soil or water) or the presence of other compounds.
- Immunoassay based tests are extremely specific.
- Immunoassay based tests are accurate and precise.
- Immunoassay reagents have a long self life.
- Immunoassay based tests are easy to use. They have simpler procedures than other methods for the analysis of PCB.
- Immunoassay is safer than other methods because it contains no radioactive materials.

Speed of Analysis - The Hach Immunoassay Test Kit for PCB is useful in the fast environmental monitoring of PCB in soil and allows on-site detection in less than 30 minutes. Using immunoassay technology, the kit eliminates the delay in waiting for results and high costs associated with other technologies.

LIMITS OF THE TECHNOLOGY

Interferences - Sensitivity to other halogenated compounds is generally less than 1% of the response to Aroclor 1260, making interference problems insignificant.

Environmental Limits - Temperature, Power Requirements, Water Needs
Store reagents at room temperature and out of direct sunlight (less than 80 ° F or 27 ° C).
Keep aluminized pouch that contains PCB Antibody Tubes sealed when not in use.
Operational temperature of the reagents is 40 to 90 ° F (5 to 32 ° C).
Power to the Hach Pocket Colorimeter® instrument is supplied by four AAA batteries (supplied with the kit).
Dilution solution is provided in the kit.

Upper Concentration Limits - This method is a semi-quantitative screening method which indicates whether the PCB concentration is above or below 1 ppm and/or 10 ppm threshold values.

Experience Requirements - Although much simpler than alternative methods, some skill and time is still required to complete an analysis. The user does not have to be a trained chemist to get professional results. The kit is supplied with detailed instructions to guide the user step by step through each procedure and interpretation of the results.

Limitations of Immunoassay for Field Analysis of PCB - The method provides semi-quantitative results.

Hach's test for PCB allows for the reporting of the data in terms of threshold ranges. The kit is supplied with one standard which represents a 1 ppm PCB level. Different sample dilution techniques allow samples to be evaluated at 1 and 10 ppm levels. The sample reading is compared to the standard reading and the results reported as either above or below the thresholds of 1 and/or 10 ppm as compared to the standard reading. Although our current kit is not structured to provide a measurement at 50 ppm threshold, theoretically it could be achieved if that is a need of ORNL.

Logistical Performance Goals for the Hach Method

- Ease of Operation
- Operator Training Requirements
- Sample Throughput
- Portability
- Operating Cost
  - Initials Investment
  - Cost of Consumables
- Ruggedness
- Special Requirements
  - Power
  - Apparatus
  - In-field Laboratory Set-up
- Safety of Technology
  - Use of Hazardous Reagents
- Ease of Interpretation of Results

Technical Performance Goals for the Hach Method

- Accuracy of the Technology as a screening technique
  - Relative to the Certified PDS result
  - Relative to the reference method
  - Relative to manufacturer's specifications (where applicable)
- Precision of the Technology as a screening technique
  - Relative to the Certified PDS result
  - Relative to the reference method
  - Relative to manufacturer's specifications (where applicable)
- Matrix Effects/Interferences
- Ability to operate in the Concentration Range - as specified by manufacturer
- Rate of False positives (fp) and false negatives (fn)
- Ability to perform at low concentration levels near 1 ppm
- Sample Size
- Waste generation
  - Non-Hazardous Waste
  - Consumable Waste
  - Hazardous Waste
- Overall QC sample performance, including blanks, performance demonstration samples
- Affect of environmental conditions on operation (controlled environmental atmosphere studies)

Rapid Screening for Polychlorinated Biphenyl and 2,3,7,8 Dioxin in Soil and Flyash Using a SAW/GC

Introduction

A handheld portable chromatography system equipped with a non-specific Surface Acoustic Wave (SAW) detector is used to speciate and quantify PCB and dioxin contamination in soil and flyash with a 10 second analysis time. The SAW detector is an integrating mass detector (micro-balance) with zero dead volume and the ability to quantify chromatography peaks at the picogram level and with peak widths measured in milliseconds. Measurement speed and accuracy make the instrument well suited to rapid screening of soil samples. Early separation of those soil samples below the regulatory level from those which require laboratory validation with a GC/MS reduces the cost associated with site characterization and monitoring. The SAW/GC screening procedure, when incorporated into EPA Methods (e.g. 8080), allows for pre-dilution's optimized to the limited dynamic range of a GC/MS laboratory instrument.

A sampling pump and loop trap are used to sample and inject analyte into a GC capillary column. Speciation is based upon retention time measurements using a temperature programmed DB-5 column. Quantification is based upon the frequency shift produced by analytes or PCB isomers as they exit the GC column. By focusing the effluent onto a specific area on the surface of a temperature controlled piezoelectric crystal, high sensitivity is achieved with a 10 second analysis time. The SAW/GC is able to selectively screen and quantify PCB levels for dioxins and Aroclor compounds in soil and flyash with ppb precision.

Two procedures for extracting PCBs from soil matrices are used. These procedures have been tested on the dioxin and Aroclor mixtures shown. The **first procedure** uses an open tubular direct desorption tube (OTDDT) held at approximately 200°C. The desorption tube is pre-packed with a soil sample and attached to the inlet of the SAW/GC. Heat is used to desorb vapors from a soil while the sampling pump of the SAW/GC collects the desorbed vapors. Total extraction by direct desorption is a fast and accurate method for soils with contamination levels below 250 ppb.

Analytes Tested
Aroclor 1221
Aroclor 1016
Aroclor 1248
Aroclor 1232
Aroclor 1242
Aroclor 1254
Aroclor 1260
Aroclor 1262
2,3,7,8 Dioxin

The **second procedure** is best suited to testing soil with contamination levels of 250 ppb or higher because of the sample dilution inherent in the method. A liquid extraction of the soil using a mixture of hexane, water, and methanol is first carried out and then a small amount of the liquid extract is injected into the SAW/GC inlet and the PCB content measured.

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These methods should be used by, or under the supervision of, analysts experienced in the use of sampling techniques and gas chromatography. The analysts should also be skilled in the interpretation of gas chromatograms and in the use of chromatography as a quantitative tool.

The accuracy of the SAW/GC PCB/dioxin soil screening method is based upon n-point calibrations using Standard solutions. Quality assurance measurements require GC validation using only standards certified by an independent laboratory. All spiking solutions, prior to their use in soil recovery analyses or calibration by direct injection, must first be validated by GC measurement.

Interference

Due to the universal detection capability of the SAW detector, other non-PCB compounds may co-elute with PCB standards. Any such compounds detected may be misidentified and quantified as a PCB. If the quantification level is above the alarm threshold, the method requires the soil sample to be laboratory tested and the SAW/GC screening measurement validated. It is implicit in a screening method that there are no false negatives and that all positive responses require laboratory validation.

Impurities from contaminants within the instrument or inlet train desorption tubing may interfere. Contamination by carryover can also occur whenever high-level and low-level samples are analyzed sequentially. To insure against interference, the screening method requires that acceptable (method) blanks be recorded before and after all measurements

Quality Control

The minimum required elements of quality control are as follows:

1. Initial Demonstration of Proficiency
2. Method Detection Limit Determination
3. Analysis of Blank Samples
4. Laboratory Control Sample Analysis

Expendable Materials

Laboratory Standard PCB-hexane solutions for field spikes and calibration. The concentration of the standards should provide nanogram quantities of PCB when injecting 1 to 10 μliters of standard solution. A supply of reagent grade hexane is required for method blanks.

A pre-mixed supply of hexane, methanol, and water is required for performing liquid soil extractions. Other expendable items include septa equipped vials and pipette filters for filtering soil extractions.

Weighing Balance

A weighing balance accurate to 0.1 mg is required to weigh the soil samples.

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Syringes

To create soil audit samples for recovery confirmation, spiking solutions and quality assurance calibrations, a standard chromatography syringe is used. Recommended is a 10 µliter syringe available from SGE, 10R-GT, Part No. 002250.

Soil Samples

For the direct thermal desorption method a soil sample collection consists of placing homogeneous samples (approximately 0.1-0.25 grams) from a source to be analyzed into pre-weighed 6 x ¼ inch glass tubes. For the liquid extraction method soil samples are placed in 4 mL glass vials with septa caps. Sampling spatula or other utensils which come into contact with the soil should be clean so as not to contaminate the sample. If the content of the soil is not to be measured immediately the ends of the glass tube are sealed with slip-on septa covers.

Procedure No. 1 - Direct Thermal Extraction (DTE)

The SAW/GC inlet sample port is glass lined stainless steel for sampling of vapors directly into the instrument. Total extraction from soil is performed using an open heated glass tube fitted with a glass-to-luer adapter attached directly to the inlet of the instrument. Calibration is performed using a syringe needle to inject laboratory standard solutions directly into the open tubular desorption tube.

GC Analysis

1. Take Blank samples before and after each analytical run. Monitor the blank for background levels or carryover. Continue blanks until the levels are below preset minimums. Each sample tube is weighed and pre-screened before loading with soil.
2. The instrument should be used with the SAW/GC Method and instrument settings for which the calibration was performed. Use of any other method requires the generation of a new calibration curve. The operator must save all chromatograms (SAV-ALL=ON), including blanks and calibration checks performed with liquid standards.
3. After loading tube with approximately 250 mg of soil, attach luer adapter to one end of sample tube. Attach the sample tube to the luer inlet fitting of the SAW/GC.
4. Slide heater jacket, pre-heated to 200°C, over the sample tube and immediately initiate soil sampling with sample time set to 30 seconds. Repeat 30 second soil sampling at 1 minute intervals until analyte concentration readings are less then 10% of initial sample values. Record the concentration mass, in nanograms, for each sample measurement, N_i, as well as the total of all sample measurements, N_T.
5. Measure the weight of the sample tube packed with soil. Subtract the weight of the empty tube and designate the result as W_{SOIL} in grams..

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Procedure No. 2 - Liquid Extraction and Injection

This method is well suited to analysis of soils with high concentrations of PCBs. First the PCBs are extracted from the soil using a mixture of hexane, methanol, and water.

1. Add a weighed amount of soil (0.25-1 gram) to 1 mL of solution, shake until soil is well dispersed, and let stand until hexane solute is clearly seen to separate and float on top of methanol-water layer with soil sediment resting on bottom of vial.
2. Extract approximately 0.25 mL of the hexane and use a disposable pipette filter to transfer into a clean vial and seal with septa cap.

Sampling of the extract solution is performed using an open tubular thermal desorption tube packed with glass wool. The tube is fitted with a glass-to-luer adapter which attaches directly to the inlet of the instrument. Calibration is performed using a syringe needle to inject laboratory standard solutions directly into the open tubular desorption tube.

GC Analysis

1. Take Blank samples before and after each analytical run. Monitor the blank for background levels or carryover. Continue blanks until the levels are below preset minimums. Each sample tube is weighed and pre-screened before loading with soil.
2. The instrument should be used with the SAW/GC Method and instrument settings for which the calibration was performed. Use of any other method requires the generation of a new calibration curve. The operator must save all chromatograms (SAV-ALL=ON), including blanks and calibration checks performed with liquid standards.
3. With the heater jacket removed and the extraction tube at room temperature inject a measured amount of extract into the tube. Initiate analysis runs with the SAW/GC to remove volatile compounds and until liquid can no longer be seen in the glass tube
4. Slide heater jacket, pre-heated to 200°C, over the sample tube and immediately initiate sampling with sample time set to 30 seconds. Repeat 30 second sampling at 1 minute intervals until analyte concentration readings are less then 10% of initial sample values. Record the concentration mass, in nanograms, for each sample measurement, N_i, as well as the total of all sample measurements, N_T.

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Calculations

Windows 95, SAW/GC system software (Version 4.0), and Excel and is required to operate the system, log data, and provide measurement documentation. With the system software, three calibration options are provided. The operator may select individual compound peaks and calibrate based upon the measured signal in Hz and the standard input in nanograms. Alternately the operator may select to use either the total area of all peaks over a specified range of retention times, or the sum of a set of 'tagged' peaks specified in a calibration file, to determine a response factor in terms of a standard input.

Soil contamination is expressed in either ppm (mg/kg), ppb(µg/kg), or ppt (ng/kg). To calculate soil contamination perform the following calculation:

$$Conc_{SOIL} = \frac{\sum_i N_i}{W_{SOIL}} = \frac{N_T}{W_{SOIL}}$$

For liquid extractions the above result must be multiplied by the ratio of the total amount of hexane solution divided by the amount of solution extract injected (dilution ratio).

Instrument Calibration Procedure

A calibration curve and the response factors must be entered into the Peak File software dialog screen, before analysis can begin. If the instrument has been previously calibrated in the lab, only a single mid level calibration check for each analyte is required. If the value of the check is within 30% of the lab value, then the response factor is confirmed. If the value is greater than 30%, then the instrument must be re-calibrated.

Check instrument status. Measure the instrument sample flow using the mass flow meter. Record the sample flow and enter the value in the Peak File software dialog screen under sample flow in ccm (cc/min) units.

Run an instrument blank. Assure that the background is below 10 ppb for any compounds in the peak file. The blank should be a method injection into an empty desorption tube.

Create a calibration standard solution. Fill a 4 mL vial with an appropriate amount of standard solution and an appropriate amount of solute so that a concentration (nanograms/µliter) which is mid-level to the desired measurement range, is achieved. Seal the vial with a new septa lid.

To define the instrument response factor, SF (in Hz/picogram), a liquid injection into the desorption tube with a known standard is made. The instrument reading, F_m, in measurement units of frequency (Hz=Hertz) and the total amount of analyte injected, M_a, in picograms defines the response factor:

$$SF = \frac{F_m}{M_a}$$

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Note: If the proper scale factor is entered into the peak file dialog screen, the software will display PCB or dioxin measurement in picograms or nanograms in the peak window. An example using a 1 µliter injection with a solution of 10 nanogram/µliter 2,3,7,8 dioxin is shown in **Figure 1.**

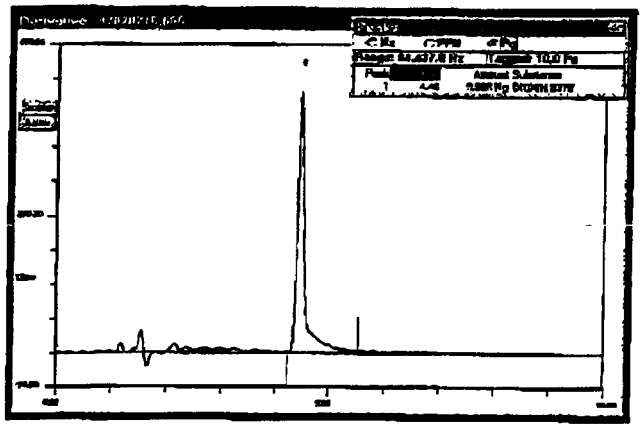

Figure 1- Calibration with 10 nanograms of 2,3,7,8 dioxin.

Peak File Setup

Confirm the retention time windows for each component to be analyzed. Make three injections of the component and calculate the standard deviation of the retention time of each component. The average retention time and response factor for each analyte is entered into the peak recognition file.

PCB Aroclor mixtures typically contain 15 or more isomers as shown in **Figure 2.** In this case the system software provides the operator with the ability to use either the sum of peaks over a retention time range or the sum of a selected peaks, as the basis for calibration. A single average response factor for the sum of the peaks within the mixture is used to calculate the concentration of the Aroclor mixture.

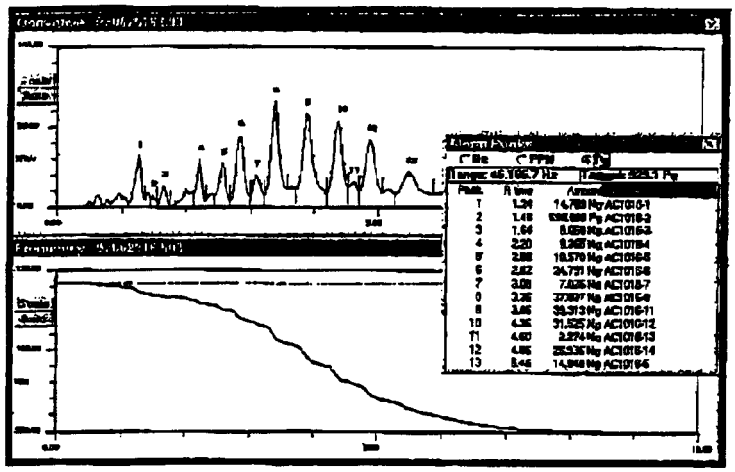

Figure 2- Calibration with Aroclor 1016.

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Aroclor Pattern Recognition

Commercial Aroclor mixtures of PCB isomers are commonly found at environmental sites and their composition and vapor signature can readily be recognized by a trained operator. Five different Aroclor vapor signatures in vertically offset chromatograms are shown in **Figure 3**.

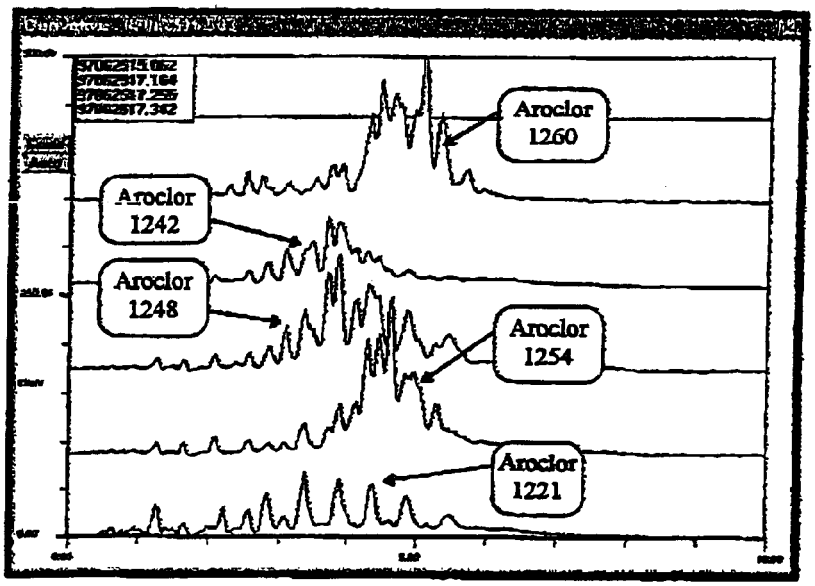

Figure 3- Vapor signatures of several Aroclor mixtures.

By creating peak identification files for the Aroclor mixtures, the pattern recognition process can be quantified and the relative degree of fit for an unknown set of PCB peak retention time determined. Data logging to Excel spread sheets using different peak recognition file patterns for the raw data, provides documentation and archival of all SAW/GC measurements.

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3.4 Sentex Systems, Inc.

INTRODUCTION

Several industries widely used PCBs as dielectric and heat transferring fluids, flame retardants and plasticizers. PCBs have been a major environmental concern because of their widespread use in the past and its very stable molecular structure. Although there are 209 known PCB isomers, only 90 isomers are commercially available for laboratory use. In the United States, the most prevalent PCBs used were the Aroclors manufactured by Monsanto. These Aroclor PCBs exhibit characteristic patterns, which became the basis for their identification. Approved EPA Methods 508, 608, and 8080 use pattern recognition to tentatively identify the Aroclor PCBS. Quantitation is accomplished either by summing all the areas or by selecting three to five major peaks of the sample and comparing them to the corresponding peaks of the Aroclor PCB standards. This method , however, is carried out in the laboratory and consumes a considerable amount of time. The Scentograph Plus II can be used for the on-site screening of samples for PCBs. It is a fast way of determining the presence or absence of the Aroclor PCBs at relatively low concentration levels. Solvents, such as the Trichlorobenzenes and Tetrachlorobenzenes, which are typically present in Aroclor PCB mixes, may interfere in the qualitative and quantitative analysis of the samples.

METHODOLOGY

A procedure based on approved EPA methods will be performed to isothermally analyze Aroclor PCBs within fifteen minutes, utilizing a Scentograph Plus II Portable GC (PGC) with a short capillary column and an ECD. The carrier gas used was helium. at a head column pressure of 12 PSI. The column, an MXT-5 from Restek (7.5 meters x 0.53 m ID x 3 mm film thickness), was operated at 170 C. Samples (2 µL) are manually introduced into the heated injection port.

The Scentograph Plus II is a complete analytical system. The GC includes a sample injection system, a precisely controlled column oven, detection system, gas flow regulation and electronics. The data management system is provided by a detachable lap-top computer (PC). The program that operates the GC collects, stores and processes data, which is saved to the hard disk drive. Data can be collected on the hard drive or on floppy disks. The Sentex operating program is user-friendly, and can be easily operated by a person with no previous computer experience.

Prior to field analysis, the Scentograph Plus II can be calibrated with all the different Aroclor PCBs. The generated chromatograms of the standards are stored in memory. A special feature of the Sentex program allows the stored chromatograms to be grouped into sets. After each sample analysis, the set of calibration chromatograms can be recalled and the sample chromatogram will be superimposed against the chromatograms of the different standards in the set one at a time. This facilitates fast pattern recognition and qualitative results. For increased accuracy, once an Aroclor PCB is identified in the sample, the corresponding standard of a chosen concentration is analyzed and the sample is quantified against this standard.

The ECD is very sensitive to functional groups that are electronegative, such as the halogens and the oxygenated compounds. Therefore, solvents typically found with the Aroclor PCBs, such as the Trichlorobenzenes and Tetrachlorobenzenes, can interfere in the analysis of samples for PCBs.

The PGC requires the same preventive maintenance as the laboratory GC for it to perform efficiently and accurately. The detection limit for PCB is comparable to those obtained by laboratory instruments, 0.2 ppm. Using sample preparation similar to those used in laboratories, i.e. extraction or dilutions, water, oil and soil samples can be analyzed. The limiting factor in the use of the PGC in the field is the power source. The PGC is equipped with lead-acid batteries, and will normally last eight hours at a temperature of 70 C. However, at a temperature

of 170 C, the batteries will last approximately 2 hours. This can easily be extended with the use of an external battery pack.

PROCEDURE

PCB in Soil Extraction Procedure

I. Materials

- High purity water
- Pesticide grade methanol
- Pesticide grade
- Anhydrous sodium sulfate, granulated
- Glass wool
- Syringes, 500 mL & 1 mL, airtight
- Vials, 2 mL & 4 mL, screw cap
- Disposable pasteur pipet
- Pipet bulb
- Top loader balance

II. Procedure

- Weigh 800 mg of soil into a 4 mL vial.
- Add to the sample:
  200 mL water
  800 mL methanol
  1000 mL n-hexane
- Cap the vial.
- Shake vigorously for 1 minute.
- Pipet out the top layer (hexane) through a pasteur pipet packed with anhydrous sodium sulfate into a 2 mL vial.
- Cap and label.

Reference:

Field Measurement of PCB c in Soil and Sediment Using a Portable Gas Chromatograph, T.M. Spitter, Proceedings of the 4th National Conference on Management of Hazardous Waste Sites, Washington, D.C., Oct. 31 - Nov 2, 1983.

PCB on SURFACES Extraction Procedure

I. Materials

- Pesticide grade n-hexane
- Syringes, 10 mL, airtight
- Vials, 2.0 and 20 mL, screw cap
- Kim wipe
- Ruler

II. Procedure

• Measure a 100 cm2 area.
• Wet the kim wipe with n-hexane.
• Wipe the surface area of interest.
• Place the wipe in a 20 mL vial.
• Add 10 mL of n-hexane to the vial containing the wipe.
• Let is stand for 15 minutes prior to analysis.
• Transfer an aliquot into a 2 mL vial, cap and label.

This is a modified procedure adopted from the NJ sampling guide.

3.5 Strategic Diagnostics, Inc.

Environmental Immunoassays

Immunoassay technology is a widely accepted and utilized analytical tool in clinical diagnostics, with over a billion clinical tests performed annually in the United States. Immunoassay technology has recently been applied to environmental contaminants, providing environmental professionals with a fast, low-cost, sensitive, accurate and simple method for the field detection and quantification of environmental contaminants such as toxic organics and pesticides. The use of immunoassay testing in environmental applications is growing rapidly, with applications in the hazwaste/remediation, food/agriculture and water quality fields.

Immunoassay testing permits rapid and simple field and on-site analysis. It does not require the use of complex equipment or instrumentation (such as GC/MS or HPLC) and enables non-laboratory field technicians to perform accurate analyses for select analytes at sub-part per million concentrations. Typical users of immunoassay testing systems for site assessment and remediation applications include the field personnel for consulting/engineering, remedial contracting, and oversight agency organizations. Immunoassays are used as field screening tools to expedite field decisions in site assessments and remediations, to facilitate the use of innovative and "fast track" project management approaches which accelerate project completion, and to dramatically reduce overall project and analytical costs.

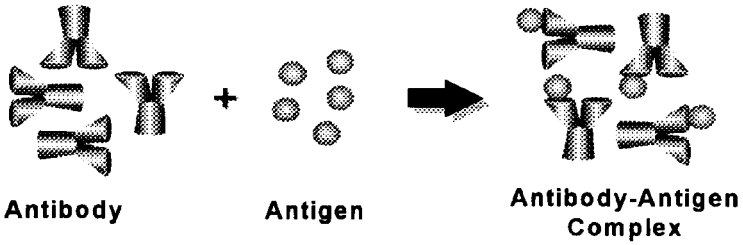
Features & Benefits of Immunoassay Test Kits

- ⇒ ***Rapid***
- ⇒ ***Easy-to-Use***
- ⇒ ***Accurate***
- ⇒ ***Reliable***
- ⇒ ***Economical***
- ⇒ ***EPA Accepted: Validated as EPA SW-846 Method 4020***

Immunoassay Technology

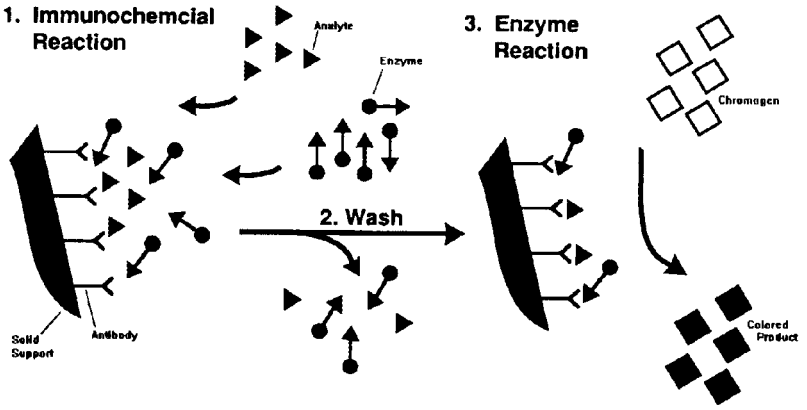
ELISA based analytical systems utilize biologically generated "immunoglobulin" proteins, called antibodies, capable of reacting with specific target compounds, called antigens, to form antigen-antibody complexes (Figure 1).

Figure 1.



In a competitive ELISA immunoassay, sample to be tested is combined with an enzyme labeled analog of the analyte and the analyte specific antibody (Figure 2.). Both the unlabeled (sample) analyte and the enzyme labeled analyte analog compete for a limited number of antibody sites and bind to the antibodies in direct proportion to their relative concentrations in the reaction. After an incubation period, the antibody with the labeled and unlabeled analyte bound to it are separated from the unbound substances. Color producing reagents are then added to the antibody labeled-analog complex and allowed to develop color during an incubation step. This color development may then be terminated with a stopping reagent.

Figure 2.



The immunochemical reaction of ELISA's contribute high analytical selectivity due to the extraordinary discriminatory capability of antibodies, and high sensitivity because of the powerful catalytic capability of the enzymes.

The four main components of environmental immunoassays are:

1. Antibody
2. Enzyme Conjugate (Linked Antigen)
3. Solid Phase
4. Reporter System

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The use of different solid phase materials and types enable the development of assays with different features and performance characteristics each best suited to particular testing applications and situations. Four different types (in four product lines) of immunoassay field test kits for the analysis of PCB's are commercially available using three different solid phases. For the purposes of the 1997 EPA/DOE/ORNL evaluation, three different test kit "formats" (product lines) will be evaluated. Information on data type, matrix applications, minimum detection levels, and decision support capability of the three test kit formats is presented in Tables 1 to 3.

Table 1. PCB Immunoassay Test Kit Formats, Data Type, and Application Matrices

SDI Immunoassay Based PCB Test Kits for 1997 EPA/DOE/ORNL Demonstration

	Product Name & Technology		
	D TECH latex particle EIA	**EnviroGard** coated tube EIA	**RaPID Assay** magnetic particle EIA
Data Type:	• qualitative at action level(s) • semi-quantitative	• qualitative at action level(s) • semi-quantitative	• qualitative at action level(s) • semi-quantitative • quantitative
Matrix			
Soil	+	+	+
Water		+	+
Wipe	+	+	+

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Table 2. PCB Immunoassay Test Kit Format Detection Limits for PCB's.

	Product Name & Technology		
	D TECH latex particle EIA	**EnviroGard** coated tube EIA	**RaPID Assay** magnetic particle EIA
Data Type:	• qualitative at action level(s) • semi-quantitative	• qualitative at action level(s) • semi-quantitative	• qualitative at action level(s) • semi-quantitative • quantitative
Matrix	**Detection Limits**		
Soil	**0.5 ppm**	**1.0 ppm**	**0.5 ppm**
Water		**0.1 ppb**	**0.5 ppb**
Wipe	**10 *u* g / 100 cm^2**	**10 *u* g / 100 cm^2**	**5 *u* g / 100 cm^2**
Concrete / Cable			**0.5 ppm**

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Table 3. PCB Immunoassay Test Kit Format Decision Support Capability.

Sample & Matrix	Product Name & Technology		
	D TECH latex particle EIA	EnviroGard coated tube EIA	RaPID Assay magnetic particle EIA
> 50 ppm Soil	Yes w/ dilution	Yes	Yes
> 2 ppm but < 50 ppm Soil	Yes w/ dilution	Yes w/ dilution	Yes
< 2 ppm Soil	Yes w/ dilution	Yes w/ dilution	Yes
< 10 ug / 100 cm^2 Wipe	Yes	Yes	Yes

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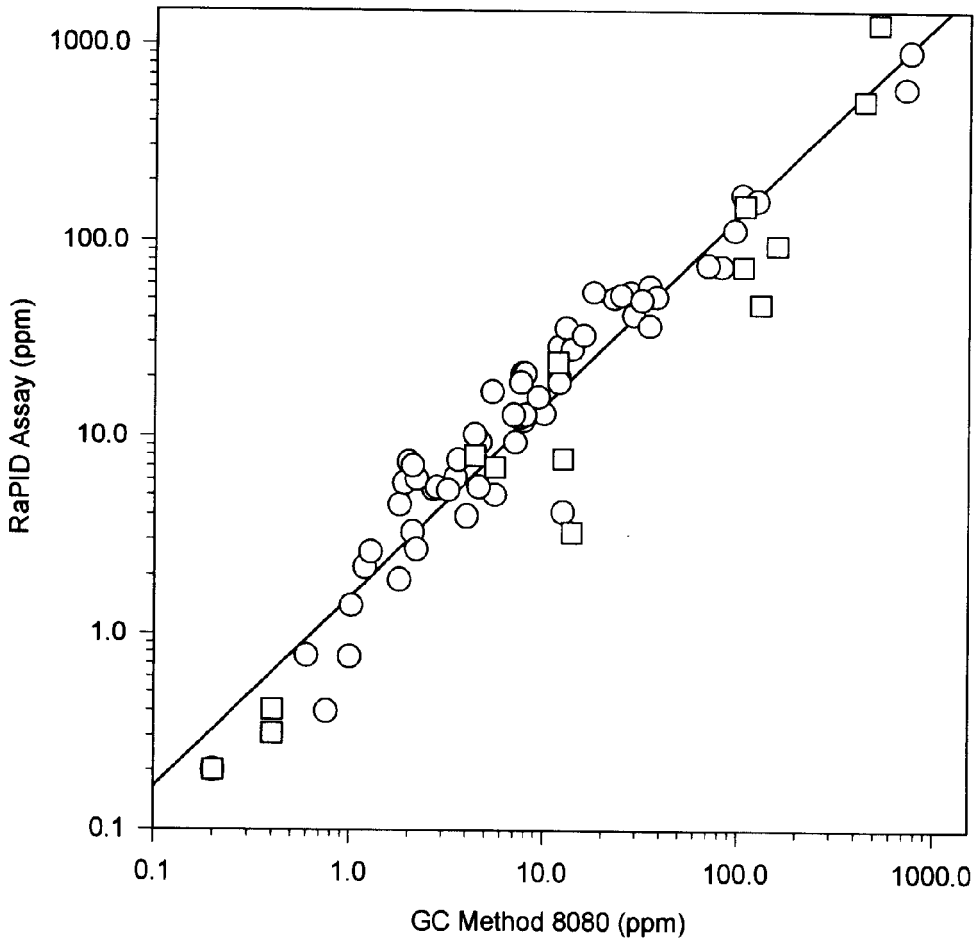
Correlational data for the magnetic particle immunoassay test kit (Ohmicron RaPID Assay) versus conventional SW-846 Method 8080 GC analysis is presented in Figure 3 below.

Figure 3. Magnetic Particle PCB Test Kit soil sample data vs. Method 8080.



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Additional Information

For additional information on PCB environmental immunoassay test kits, and test kit for other environmental compounds of concern, please contact **Strategic Diagnostics Inc.** at phone: (302) 456-6789 and fax: (302) 456-6782. Formed with the 1996 mergers of Strategic Diagnostics, EnSys, and Ohmicron Environmental Diagnostics, SDI provides to the environmental marketplace a single source for the best known and respected immunoassay testing products.

3.6 Demonstration Performance Goals

This section discusses the logistical and technical performances goals for the demonstration . Any method/instrument specification that is evaluated will be defensible by scientific data.

Logistical Performance Goals:

A. Ease of operation

B. Operator training requirements

C. Sample throughput

D. Portability

E. Operating costs

F. Ruggedness

G. Special requirements (e.g., power)

Technical Performance Goals:

A. Accuracy of the technology
   1. Relative to the certified performance evaluation sample results
   2. Relative to the reference method
   3. Relative to manufacturer's specifications (where applicable)

B. Precision of the technology
   1. Relative to the certified performance evaluation results
   2. Relative to the reference method
   3. Relative to manufacturer's specifications (where applicable)

C. Rate of false positives (fp) and false negatives (fn)

D. Ability to perform at low concentration levels (near Method Detection Limit, if known)

E. Waste generation

F. Affect of environmental conditions on operation (controlled environmental atmosphere studies)

4.0 DEMONSTRATION SITE DESCRIPTIONS

This section discusses the history and characteristics of the demonstration site.

4.1 Site Name and Location

The demonstration of PCB field analytical techniques will be conducted at the Oak Ridge National Laboratory (ORNL), which is managed by Lockheed Martin Energy Research Corporation, Oak Ridge, Tennessee. Field activities will occur at two sites at ORNL: the area west of Building 5507(Site #1) and inside a controlled environmental atmosphere chamber (Site #2) which is located in Building 5507.

4.2 Site History

Oak Ridge, Tennessee, is located a short distance from Gatlinburg and the Great Smoky Mountains National Park. Recreation areas include Big South Fork and several Tennessee Valley Authority rivers and dams. A new highway extension allows easier access to the airport, now within 20 miles of the three Oak Ridge facilities. The city of Oak Ridge is home to the American Museum of Science and Energy, the University of Tennessee Arboretum, Oak Ridge Associated Universities, and several hotels and restaurants to accommodate area visitors. Figure 2 is a route map of the Oak Ridge/Knoxville area.

Oak Ridge National Laboratory, a U.S. Department of Energy facility managed by Lockheed Martin Energy Research Corporation, took root in an isolated East Tennessee valley during the Manhattan Project, the secret World War II race to develop the atomic bomb. When the war ended, ORNL turned its attention away from nuclear weaponry and toward the development of nuclear power and the production of radioisotopes for medicine and other peaceful purposes. Lockheed Martin Energy Systems manages two facilities in Oak Ridge, Tennessee--Y-12 and K-25 -- as well as programs at both the Paducah, Kentucky facility and the Portsmouth plant in Piketon, Ohio. The Oak Ridge Reservation includes 35,000 acres.

The Oak Ridge Gaseous Diffusion Plant at the K-25 Site, now known as the East Tennessee Technology Park, serves as the center of operations for Lockheed Martin Energy Systems' Environmental Management and Enrichment Facilities programs. K-25 is the repository for PCB contaminated materials from several DOE facilities, including the Oak Ridge Reservation, Paducah, and Portsmouth sites. PCB contaminated material for evaluation during the demonstration will be furnished by K-25 site personnel. Three PCB-contaminated soil matrices, i.e., soil from three DOE sites, will be evaluated during the demonstration. This demonstration will take advantage of the repository of waste from different sites and availability of the controlled environmental atmosphere chamber to simulate geological and climatological differences in lieu of conducting the demonstration at multiple sites.

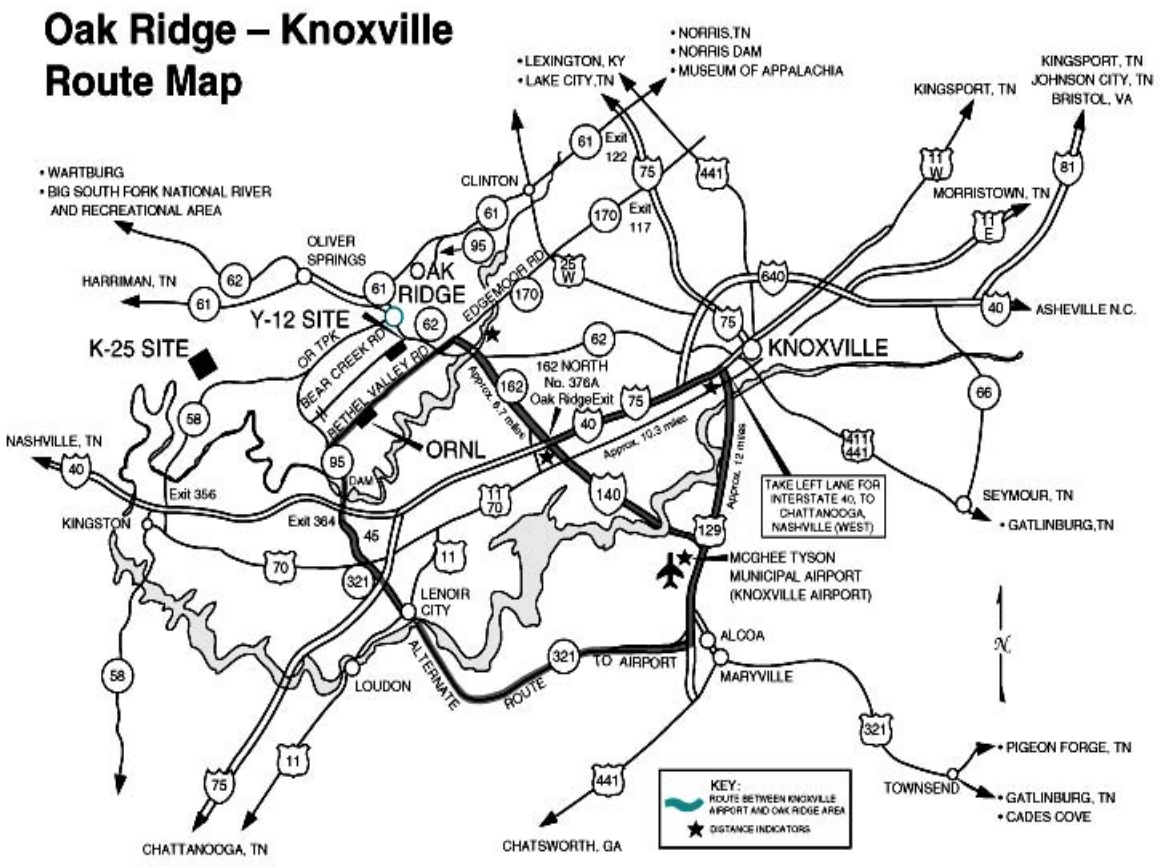

FIGURE 2: Route map of Oak Ridge and Knoxville, showing the ORNL, K-25 and Y-12 sites of Lockheed Martin plants.

4.3 Site Characteristics

Site #1 field activities will occur outside Building 5507. Figure 3 shows the field area where the demonstration will be conducted. Field studies at Site #2 will be conducted inside a controlled environmental atmosphere chamber, hereafter referred to as the "chamber". Figure 4 is a photograph of the chamber. 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 for temperature, humidity, and slightly subambient pressure control at air circulation flow rates up to five hundred cubic feet per minute. This chamber is an extension of work conducted for almost twenty years involving the generation, sampling, and characterization of controlled experimental atmospheres. The chamber is equipped with an environmental control system including reverse osmosis water purification supplying the chamber humidity control system. High-efficiency particulate air (HEPA) and activated charcoal filters are installed for recirculation and building exhaust filtration. Facility support systems include several smaller chambers for generating and diluting primary atmospheres; aerosol and vapor generation equipment; and instrumental monitors. Analytical instrument performance tests will take place

FIGURE 3: Field area where demonstration will be conducted. The structure in the corner of the picture is Building 5507.

FIGURE 4: Controlled Environmental Atmosphere ("chamber") facility at Building 5507.

entirely inside the CEA chamber to test performance in a climate which is different from the ambient outdoor conditions.

4.4 Soil Sample Descriptions

4.4.1 Oak Ridge Reservation, Portsmouth, and Paducah Soils

In Table 2 is presented a summary of the Oak Ridge Reservation, Portsmouth, and Paducah soils which will be evaluated as part of the PCB technology demonstration.

TABLE 2 - Summary of Soil Sample Descriptions

Site	Request for Disposal No.	Drums (PCB Range)	Description
Oak Ridge	24375	1,2,3 (0.8 - 220.9 ppm)	Catch basin sediment from the K-711 area (old power house area) at the DOE East Tennessee Technology Park (formerly known as Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB contaminated storm drain sediment that was excavated in 1991.
Oak Ridge	40022	2 (0.3 ppm)	Soil from spill clean up at the Y-12 plant in Oak Ridge Tennessee. This soil is PCB contaminated soil excavated in 1992.
Oak Ridge	40267	1,2,3,4 (1.3 - 6.2 ppm)	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	43275	1,2 (35.1 - 173.7 ppm)	Soil from the K-25 Building area at the DOE East Tennessee Technology Park (formerly known as Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB contaminated soil that was excavated in 1993.
Oak Ridge	134555	3 (0.2 ppm)	Soil from the K-707 area at the DOE East Tennessee Technology Park (formerly known as Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB contaminated soil from a dike spillage with rinse aid (#2 Diesel Fuel /Flushing /Transformer) that was excavated in 1995.

Paducah	LDR97002	1,2,3,4 (0.9-39.3 ppm)	Soil from the DOE Gaseous Diffusion Plant in Paducah, 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	858,1029,1069, 1096,1898,2143, 2403,2528,3281, 4096. (0.9 - 46.1 ppm)	Soil from the DOE Gaseous Diffusion Plant in Portsmouth, Ohio. This soil is PCB contaminated soil from a probable PCB oil spill into the East Drainage Ditch that was excavated in 1986.

4.4.2 Tennessee Reference Soil

The soil is a Captina silt loam from Roane County, Tennessee that 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. This soil will be used as a spiking matrix and the uncontaminated (blank) soil. To prepare a spiked sample, the soil was first ground either using a mortar and pestle or a conventional blender. The soil was then sieved through a screen which was 16 mesh, or 1 mm particle size. A solution of PCBs in diethyl ether was then added to the soil. The spiked soil was throughly mixed and allowed to air dry.

4.5 Surface Sample Descriptions

It is extremely difficult to "split" a PCB-contaminated surface such that the developers and the reference laboratory would have equivalent samples to analyze. Therefore, solutions of PCBs will be analyzed to simulate an extracted surface wipe pad. This process will focus on evaluating the analytical performance of the technology, rather than the acquisition of the sample.

5.0 CONFIRMATORY PROCESS

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 demonstration.

5.1 Method Selection

The reference analytical method will be EPA SW-846 Method 8081.

5.2 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.

Oak Ridge Sample Management Office (SMO) has been tasked by DOE Oak Ridge Operations with maintaining a list of qualified laboratories to provide analytical services. In Appendix A are presented SMO's standard operating procedures for identifying, qualifying, and selecting analytical laboratories. The first procedure

(LMES-ASO-AP-203, REV. 0) describes the process for selecting, adding and expelling commercial laboratories to the 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 are to support projects sponsored by the DOE Oak Ridge Operations Office . The procedure serves to ensure that as an operation of a DOE contractor, LMES SMO maintains an optimum level of technical and administrative oversight on each project, and SMO commercial procurement activities comply with federal acquisition laws and LMES procurement policy.

Using the procedures listed in Appendix A, ORNL and SMO has selected LAS Laboratories, in Las Vegas, NV, as the reference laboratory. In Appendix B is presented the LAS standard operating procedure.

5.3 Contingency Laboratory Selection

A contingency laboratory would be used to support the data from the reference laboratory if preliminary results differ significantly from those obtained by the technology in the field. DataChem Laboratories, in Salt Lake City, Utah, will be the contingency laboratory. Like LAS, DataChem was also selected using the procedures in Appendix A.

5.4 In-Field Support Laboratory

ORNL-based Grand Junction, Colorado (ORNL-GJ) field team served as the in-field support laboratory for the preliminary on-site analyses of the PCB-contaminated soils. In Appendix C 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 the same basic procedure.

5.5 Special QC Requirements

In order to increase the likelihood that high quality data will be obtained, an enhanced QC strategy will be required. Standard reference materials, double blind standards, matrix spiked soils, and special performance evaluation materials will be utilized.

5.6 Laboratory Audit

SMO conducts annual on-site audits of LAS and DataChem laboratories as part of the lab certification program. The most recent audits of the labs were performed in February 1997. It is likely that an audit of LAS will occur during the time period in which the field samples are being analyzed (possibly the week of August 11, 1997). The audit would address the QC procedures and document any changes to the analysis process. Most likely, SMO and EPA-LV will jointly conduct the audit.

5.7 Statistical Analysis of Results

PCB concentration measurements will be compared with the reference values, where possible. Deviations that are statistically different than zero will represent the bias for the method. The variation of the concentration measurements after they have been adjusted for the experimental factors will represent the precision of the method. Table 3 suggests some reference resources for the experimental design and statistical methods that will be used for the PCB verification demonstration.

5.7.1 Methods for Data Reduction and Adjustments

During any experiment, unusual measurements may occur either as random events or deterministic causes. It is important that the developer note and record any problems with each PCB measurement. This information will be used to decide if an unusual measurement was a gross measurement/ recording error or a problem with the homogeneity of the soil matrix. Graphical representation will be used to examine the data by histograms/frequency plots, stem-and-leaf plots, box-and-whisker plots, and scatter plots (See *Guidance for Data Quality Assessment*, Section 2.3, 4.4). These plots will be use to identify any unusual values and data distributional problems.

Identification of unusual measurements doesn't mean that they are automatically set aside. The statistical analysis can be performed with or without the suspected measurements to see if there are any changes in the conclusions of the demonstration experiment. The unusual measurements may also indicate that the distribution of data is not the assumed normal distribution. The deviations from the statistical analysis model can be examined (e.g., the Shapiro-Wilk test or the Kolmogorov-Smirnov test) to check if the normality assumption is reasonable. Two approaches may be used if the normality assumption is not appropriate. The first approach is to use a mathematical function to transform the data to an approximate normal distribution. Frequently, the logarithm or square root of the measured values performs this transformation. The second approach is to use statistical analysis methods that do not depend on the data distribution. These statistical analysis methods are call nonparametric methods (e.g., Median test, Wilcoxon, Kruskal-Wallis test, etc.)

5.7.2 Methods of Statistical Analysis

The data from the demonstration experiments will be used to test the statistical hypotheses about the PCB concentration population parameters:

H_0: Is the expected PCB concentration from a developer's method equal to the expected PCB concentration from the reference method?

H_0: Is the expected PCB concentration from a developer's method equal to the certified PCB concentration in the performance demonstration sample?

H_0: For field samples, is the variance of the PCB measurements from a developer's method equal to the variance of the PCB measurements from the reference method?

H_0: For performance demonstration samples, is the variance of the PCB measurements from a developer's method equal to the variance of the PCB measurements from the reference method?

The experimental design matches each developer's sample with a reference laboratory sample. This pairing of samples allows each concentration value measured by a developer's technology to be compared to a concentration value measured by the reference laboratory. The differences between the two concentration measurements (PCB DIFFERENCE) can be used to test the proposed hypotheses using the statistical methods of Analysis of Variance (ANOVA). We would conclude that no differences between the developer's method and the reference laboratory method can be detected if the expected value of PCB Difference is zero. The ANOVA model for each site (field or weather chamber) would contain the terms for SOIL type (Oak Ridge 1&2, Paducah 1, and Portsmouth 1 & 2), CONCENTRATION level (0.1-2.0, 2.1-20.0, 20.0-50.0, and 50.1-500.0), and the interaction of SOIL × CONCENTRATION. For example, an ANOVA model for analyzing data from each site has the form:

PCB DIFFERENCE = MEAN + SOIL + CONC + SOIL × CONC + EXPERIMENTAL ERROR.

This ANOVA model would be used to test significant effects due to soil type, concentration levels, and their interaction. The EXPERIMENTAL ERROR would be assumed to be a normally distributed random variable with an expected value of zero and constant variance. Replicate concentration measurements can be used to test applicability of competing ANOVA models.

Additional examination of the experimental factors can be made using multiple comparison tests that would indicate the similarities and differences between the developer's and reference laboratory measurement methods. Nonparametric statistical methods will be used if the approximating data distributional assumptions are not supported.

The nonparametric Wilcoxon signed rank test is particularly useful because of the pairing of developer's and reference laboratory samples. This test assumes the difference between the concentrations are continuous, mutually independent, and their distribution is symmetric. The Wilcoxon test is designed to test whether the developer's and reference laboratory measurements have the same median.

The ANOVA and Wilcoxon statistical tests are useful for analyzing continuous measurements but several of the technologies give interval or qualitative measurements. For these qualitative data, the statistical analyses will estimate the false positive and false negative error rates relative to the reference laboratory measurements. Confidence intervals will indicate the uncertainty of these error rates.

The SAS® System [*1,2,3*] or SAS will be used for the statistical analysis of data collected from the PCB verification demonstration. This software package is an integrated system of software providing complete data access, management, analysis, and presentation. SAS has more than 20 years of development history originating at North Carolina State University and implemented and improved by SAS Institute, Inc. All system components have been beta test by numerous institutions.

Table 3 Reference Sources for Statistical Analysis Methods [*1-11*]

Design/Test	Reference	Chapters/Pages
Factorial/Hierarchial Designs	Sachs Snedecor & Cochran	563-564 Chapter 12, 285-289
Analysis of Variance (ANOVA)	Draper & Smith Searle	Chapter 9 Chapters 4-7
Kolmogorov-Smirnov	Guidance for DQA Conover Kanji Sachs SAS	4.2-7 293-306 67 330-332 [*1*] 627-628
Median Test	Kanji Conover	78-79, 83-84 167-174
Wilcoxon	Guidance for DQA Kanji	3.3-10 -- 3.3-13 112

5.73 Reported Results

Evaluations of the precision and accuracy will be reported to evaluate the PCB field technology:

1. Precision will be based on the estimated variance or standard deviation of replicate PCB concentration measurements. Estimated variance (i.e., S^2) will be reported as a function of concentration [$g(S^2) = f(C)$]. The "g" function can be the identity function, square root function, or the logarithm function. The "f" function can be linear or exponential. Regression analysis methods will be used to estimate the functional forms and their coefficients. This precision relationship can be used for planning data quality objectives (DQOs) for remediation projects.

   Precision of the developer's method will be compared with the precision of the reference laboratory. Significant differences between the two measurements will be reported.

   Precision for qualitative data will be reported as false positive or false negative error rates relative to the reference laboratory. Confidence intervals will be reported to indicate the uncertainty of these error rates.

2. Accuracy will be quantified relative to the reference laboratory as a bias measurement. A bias measurement will be based on the difference between concentration measurements made by the developer's method and concentration measurements made by the reference laboratory. Statistical tests (i.e., ANOVA and Wilcoxon) will be used to identify any of the experimental design parameters that may cause biases significantly different than zero.

5.7.4 Reference Sources

1. SAS Institute Inc., SAS® Procedures Guide, Version 6, Third Edition, Cary, NC: SAS Institute Inc., 1990.

2. SAS/STAT® User's Guide, Version 6, Fourth Edition, Volume 1, Cary, NC: SAS Institute Inc., 1989.

3. SAS/STAT® User's Guide, Version 6, Fourth Edition, Volume 2, Cary, NC: SAS Institute Inc., 1989.

4. Conover, W. J. (1971). Practical Nonparametric Statistics, John Wiley & Sons Inc., New York.

5. Draper, N. R. and H. Smith. (1981). Applied Regression Analysis, Second Edition, John Wiley & Sons Inc., New York.

6. Guidance for Data Quality Assessment. (1996). EPA QA/G-9, United States Environmental Protection Agency Quality Assurance Division, Washington, DC.

7. Kanji, Gopal K. (1993). 100 Statistical Tests, Sage Publications, London.

8. Sachs, Lothar. (1984). Applied Statistics: A Handbook of Techniques, Second Edition, Springer-Verlag, New York.

9. Searle, S. R. (1971). Linear Models, John Wiley & Sons Inc., New York.

10. Shapiro, S. S. And Wilk, M. B. (1965). "An Analysis of Variance Test for Normality (complete samples)," *Biometrika*, 52, 591-611.

11. Snedecor, G. W. and William G. Cochran. (1967). Statistical Methods, The Iowa State University Press, Ames, Iowa.

6.0 PCB SOIL SAMPLE COLLECTION

6.1 Sample Collection Plan

In Appendix D is presented the sample collection plan. The sample collection plan for this demonstration 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 demonstration purpose and objectives.

6.1.1 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 5 is a photo of the Analytical Services Organization's sampling team acquiring some PCB soil samples from a 55-gallon drum.

FIGURE 5: K-25 personnel acquire a PCB soil sample 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 shows the samplers performing this procedure.

FIGURE 6: K-25 sampling personnel sift through the collected soil to remove rocks and other large debris.

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 demonstration.

6.2 Preliminary Soil Characterization

The two analytical samples taken of the homogenized soil were analyzed using the procedure described in Appendix C. The analyses were performed by ORNL-GJ and ORNL/CASD. The total PCB concentration was measured in each analytical sample to determine which samples would be used in the demonstration. Results from the total activity screening indicated that the soils were not considered radioactive.

6.3 Predemonstration Sample Preparation, Distribution, and Analysis

A predemonstration sampling and analysis event is required to allow the technology developers to refine their technologies and revise their operating instructions, if necessary. This sampling also allows an evaluation of matrix effects or interferences that may affect the demonstration. A failure to meet the performance goals at this point could indicate a lack of maturity of the technology and the demonstration would be canceled.

This sampling requirement has the following objectives:

- To allow the developers to analyze samples that will be included in the demonstration 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 demonstration

For the predemonstration study, the technology developers each analyzed five samples: 2 spiked soils, 2 performance evaluation materials, and 1 solvent extract. The reference laboratory (LAS Laboratories) analyzed the same set of samples, but 2 solvent extracts were analyzed. This was necessary because SDI requested their extract to be prepared in methanol rather than hexane.

6.3.1 Predemonstration Sample Preparation

Two soil samples were prepared using Tennessee reference soil (reference: ORNL/TM-12128, "Stability of Volatile Organics in Environmental Soil Samples"). The soil is a Captina silt loam from Roane County, Tennessee that 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. To prepare a spiked sample, the soil was first ground either using a mortar and pestle or a conventional blender. The soil was then sieved through a screen which was 16 mesh, or 1 mm particle size. Approximately 500 g of the sieved soil was spiked with a diethyl ether solution of PCBs at the desired concentration. The soil was agitated using a mechanical shaker, then allowed to air-dry overnight. At least five aliquots of the soil were analyzed by gas chromatography with electron capture detection (using the method described in Appendix B) . The spiked soils were determined to be homogeneous.

The remaining two soil samples utilized in the pre-demonstration study were acquired from external sources, for use as performance evaluation materials. One soil was purchased from Environmental Resource Associates (ERA, Arvada, Colorado) as a certified PCB standard. These custom standards 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. Soils were also acquired from the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center. These soils were prepared using contaminated soils from U. S. EPA regional sites. The original soils were homogenized and diluted with a synthetic soil matrix (SSM). The SSM was a known matrix of 31% sand, 6% gravel, 28% silt, 20% top soil, 5% montmorillonite clay, and 10% kaolinite clay. The dilution of the original soils was performed by mixing known amounts of contaminated soil with the SSM in a V-blender for no less than 12 hours. The samples were also spiked with target pesticides. The hydrocarbon background from the original sample and the spiked pesticides produced a challenging matrix.

A solvent extract was prepared by ORNL to simulate an extracted surface wipe sample, due to the difficulty in "splitting" an environmental wipe sample. The extracts were prepared in two different solvents (hexane and methanol) to accommodate developer requests.

6.3.2 Predemonstration Sample Distribution

The predemonstration samples were sent to the developers and the reference laboratory on June 2, 1997. In Appendix E are presented the pre-demonstration study instructions. Results of the predemonstration sample analyses by the developers were made available to ORNL approximately two weeks after the receipt of the samples (June 21, 1997).

6.3.3 Predemonstration Sample Analysis Results

Predemonstration results from the reference laboratory and the technology developers were received by June 26, 1997. The results indicated the technology were mature and ready for rigorous field evaluations.

6.4 Sample Preparation for Demonstration

The PCB soil samples will be homogenized (dried, sieved, and thoroughly mixed) prior to sample splitting. Each split will consist of approximately 20 g of sample, which will be placed in 4 ounce glass jars. The PCB surface sample extracts will be prepared in hexane, except for Strategic Diagnostic's samples, which will be prepared in methanol. The extracts will be stored in the refrigerator (4 C) until released to the developers.

The field soil samples will be characterized in terms of composition (% sand, silt, clay, etc.), total organic carbon, and pH. This data will be reported in the technology verification report.

The sample extracts will be prepared in iso-octane for Dexsil and Sentex, and in methanol for SDI and EST. Hach will not participate in this portion of the demonstration, since they do not market a surface sample analysis technology.

6.5 Sample Labeling for Demonstration

The samples will be labeled with the appropriate PCB label. Each jar will also be labeled with a sample name that corresponds to the developer. For example, aliquots of drum 40022-02 would be labeled Dexsil-1, Hach-1, Sentex-1, etc.. Replicate samples from drum 40022-02 will be assigned unique (but not sequential) sample numbers. PE materials will labeled in the same manner, such that the PE sample numbers will be indistinguishable from any other identifier. The order of analysis will be randomized and set for each developer.

6.6 Pre-Analysis Sample Information

Some of the technology developers will receive information regarding the samples, such as Aroclor identification, prior to analysis. This will be given at the request of the developer, in order to simulate the type of information that would be available during actual field testing. Any information that is provided to the developer will be documented as such in the technology verification report.

7.0 DEMONSTRATION DESIGN

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

7.1 Objectives

The primary objectives of this demonstration are to evaluate the PCB field analytical technologies in the following areas: (1) how well each performs relative to conventional analytical methods, (2) the impacts of sample matrix variations on performance, (3) the affect that environmental conditions have on performance, (4) quality control results, and (5) the logistical and economic resources necessary to operate the technology. Secondary objectives for this demonstration are to evaluate each PCB field analytical technique in terms of its reliability, ruggedness, cost, range of usefulness, data quality, and ease of operation. Where possible, the performance will be compared to the performance of conventional analytical methods used in performing similar site characterization activities. The verification process will also evaluate the performance of the technology against the performance goals as stated in Section 3.7. The experimental design will provide replicate data to test competing approximating models for the statistical analysis (see Section 5.7).

7.2 Experimental Factors

This section discusses factors that will be considered in the design and implementation of the demonstration. These factors include accuracy, precision, portability, ease of operation, ruggedness, health and safety issues, sample throughput, and sample matrix effects.

7.2.1 Qualitative Factors

Some factors, while important, are difficult or impossible to quantify. These are considered qualitative factors: ease of operation, operator training requirements, portability, ruggedness, and special requirements.

7.2.2 Quantitative Factors

Many factors in this demonstration can be quantified by various means, including the following: accuracy, precision, false positives (FP) error rate, false negative (FN) error rate, ability to perform at low concentration levels, waste generation, affect of environmental conditions on operation (controlled environmental atmosphere studies), sample throughput, and operating costs. These quantitative factors will be used to assess the technology performance by comparison to reference laboratory data, where possible.

7.3 Experimental Design

7.3.1 Glossary of Terms

Chamber - room-size controlled environmental atmosphere facility at ORNL that can accommodate 2-3 developers at a time. The developers will demonstrate their technologies inside the chamber under temperature and relative humidity conditions that are different from the ambient conditions. The chamber will be set at 55 F and 25% relative humidity. This will be a cost effective approach to simulate demonstrating the technologies at a second site.

PE/QC Sample - certified soil sample containing known concentrations of PCBs. The soils will consist of ones purchased from Environmental Resource Associates and obtained from the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center.

Reference Laboratory - an analytical laboratory that will perform EPA SW-846 (method 8080 or 8082) analyses of the PCB samples for comparison with developer field results. LAS Laboratories (Las Vegas, NV) is the reference laboratory.

Site #1 - area west of Building 5507 at Oak Ridge National Laboratory

Site #2 - in the chamber located in Building 5507 at Oak Ridge National Laboratory. The chamber settings will be 55 F and 55% relative humidity.

Soil Sample - an environmental soil sample from Oak Ridge, Paducah, and Portsmouth sites. Field samples will range from more simple, single Aroclor samples to more challenging mixtures of Aroclors with high oil and hydrocarbon contamination.

Spike - a soil sample that is a matrix spiked field sample

Surface Sample - a solvent extract containing known concentrations of PCBs. This will simulate a surface wipe sample that was collected and extracted.

7.3.2 Summary of Demonstration Activities

The demonstration is scheduled to be held at ORNL from July 22 through July 30. The soil samples evaluated during the demonstration consist of (1) environmental soil samples from the Oak Ridge Reservation, Paducah, and Portsmouth DOE sites; (2) spiked environmental soil samples; and (3) purchased certified soil samples. The demonstration samples will be homogenized and split such that each developer and the fixed analytical laboratory (referred to as the reference lab) are supplied with equivalent samples. The technologies' ability to analyze surface wipe sample extracts will also be evaluated.

Some features of the approach are presented in Table 4. The experimental design approach is presented in Tables 5 through 8. From the data collected during the field demonstration activities, each technology will be individually evaluated and compared to the reference laboratory's results. Each report, which will be prepared by ORNL, will assess the technology, at a minimum, in terms of its accuracy, precision, and false positive/negative error rate for qualitative data. A verification statement will be issued by EPA for each technology as a summary of the findings of the demonstration. It will describe how well the technology achieved the performance goals that the demonstration was designed to evaluate.

TABLE 4

Experimental Design Features
Properties: 17 unique samples per site; acquire more data on fewer samples; statistically rich approach
Replicates: equal number for all soil types, solvent extracts, and concentration levels
Accuracy: equal number of comparisons with the reference laboratory for all soil types, solvent extracts, and concentration levels
Precision: estimated for all soil types, solvent extracts, and concentration levels
Data Analysis: simplified statistics due to consistency with number of replicates

TABLE 5
SUMMARY OF SOIL SAMPLE ANALYSES (by Drum Number)

	Site #1 (in field)				**Site #2** (in chamber)			
Soil Type	Oak Ridge#1	Oak Ridge#2	Paducah#1	Totals # Samples	Paducah#1	Portsmouth#1	Portsmouth#2	Total # Samples
Target Conc. Range								
0.1 - 2.0 ppm	40022-02 [a] 40267-03	24375-01 40267-02 24375-02	97002-04 97002-01	28	97002-04 97002-01	7515-4096		12
2.1 - 20.0 ppm	40267-01 40267-04	134555-03S	97002-03	16	97002-03	7515-1898	7515-2528 7515-3281	20
20.1 - 50.0 ppm	40267-01S [b]	43275-01	97002-02	12	97002-02	7515-1096 7515-2143 7515-0940	7515-1069 7515-0858	24
50.1 - 500 ppm	24375-03	43275-02	97002-02S	12	97002-02S	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 sample drum number listed.*
[b] *"S" indicates that the sample is a matrix spiked field sample.*

TABLE 6

PERFORMANCE EVALUATION/QUALITY CONTROL (PE/QC) SOIL SAMPLES[a]

Sample	Concentration	Replicates
Aroclor 1248 [b]	2 ppm	4
	20 ppm	4
Aroclor 1254 [b]	5 ppm	4
	50 ppm	4
Aroclor 1260 [b]	11 ppm	4
	50 ppm	4
Mixture of 2 Aroclors [c]	2 ppm [d]	4
	50 ppm [d]	4
Uncontaminated soil (method blank) (Tennessee Reference Soil)	n/a	4
Total # samples		**36**

[a] *The same set of PE/QC samples will be analyzed at both sites.*

[b] *Provided by the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center.*

[c] *Provided by Environmental Resource Associates.*

[d] *Total PCB concentration.*

TABLE 7

SUMMARY OF SURFACE SAMPLE ANALYSES[a]

Sample Concentration	SITE #1 (in field)	SITE #2 (in chamber)	Grand Total
10 µg/mL	4 replicates	4 replicates	
100 µg/mL	4 replicates	4 replicates	
Blank	4 replicates	4 replicates	
Total # samples	12	12	**24**

[a] *Surface samples will be prepared as solvent (either iso-octane or methanol) extracts by ORNL.*

TABLE 8
SUMMARY OF DEMONSTRATION ANALYSES

Sample Type	Number of analyses	
	Site # 1 (in field)	**Site #2 (in chamber)**
Field Soil samples	68	68
PE/QC samples	36	36
Surface sample extracts	12 [a]	12 [a]
Grand Totals	**116**	**116**

[a] *The reference laboratory will analyze two sets of surface sample extracts, since two solvents (methanol and iso-octane) will be used to accommodate developer needs.*

7.4 Field Data

The technology will be operated by the developer, who will provide the results to ORNL**.** The developer will be responsible for reducing the raw data into a presentation format consistent with the evaluation requirements. The developer will submit all QC data and a description of how this data may be used to validate the field data.

7.4.1 Field Audit

The EPA, DOE, and/or ORNL will conduct audits of all field activities. This activity will document any deviations from the demonstration plan, use of QC materials, operational details, and other factors associated with an evaluation of the field technology. This audit report will be included as part of the Quality Assurance Project Plan (Section 8.0).

7.5 Demonstration Schedule

Demonstration activities will occur from July 22 through July 30**.** Developers are to arrive at ORNL for a briefing on the afternoon of July 21. Visitor's Day will be July 24. In Table 9 is presented the schedule for when the developers will be working at each site.

TABLE 9 - Site Schedule

MON	TUES	WED	THURS	FRI	SAT	SUN
July 21	July 22	July 23	July 24	July 25	July 26	July 27
Developer Briefing	Site 1:Group #2 Site 2:Group #1	Site 1:Group #2 Site 2:Group #1	*Visitors Day*	Site 1:Group #2 Site 2:Group #1	Site 1:Group #1 Site 2:Group #2	Site 1:Group #1 Site 2:Group #2
July 28	July 29	July 30				
Site 1:Group #1 Site 2:Group #2	additional time to be used as needed					

Group #1 : Hach and Strategic Diagnostics
Group #2: Electronic Sensor Technology, Dexsil, and Sentex

Site #1: in field
Site #2: in chamber

7.6 Field Operations

This section will describe the logistical requirements associated with sample collection and technology operation. This phase of the demonstration requires close communication between the developer, ORNL, DOE, and EPA. Preliminary site training (on July 21) will be required before initiation of field study. Successful field operations require detailed planning and extensive communication. The implementation of the demonstration must be consistent with the requirements of the study and routine operation of the technology.

7.6.1 Communication and Documentation

ORNL 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 developer/ORNL field activities will be thoroughly documented. Field documentation will include field logbooks, photographs, field data sheets, and chain-of-custody forms.

ORNL 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 and in the field logbook. Any deviations from the approved final demonstration plan will be thoroughly documented in the field logbook and provided to the ORNL**.**

The developer will obtain all equipment needed for field work associated with this demonstration. The activity may be coordinated with ORNL, where necessary.

7.6.2 Sample Distribution

ORNL will be responsible for sample distribution. The samples will be packaged in 4 ounce (120 mL) jars, as described in Section 6.4. All samples will be labeled and prepared for distribution at the start of the demonstration. Developers will go to a sample distribution table located in Building 5507 to pick-up their samples. Completion of chains-of-custody will document sample transfer.

7.6.2.1 Laboratory Samples

All of the PCB samples will be shipped to LAS Laboratories at the start of the demonstration activities (July 21). Shipment will be coordinated through ORNL's Sample Management Office. Completion of chains-of-custody will document sample transfer. The samples will be shipped in coolers.

7.6.2.2 Field Samples

Developers will go to a sample distribution table located in Building 5507 to pick-up their samples. Completion of chains-of-custody will document sample transfer.

7.6.2.3 Archive Samples

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

8.0 QUALITY ASSURANCE PROJECT PLAN (QAPP)

The QAPP for this demonstration specifies procedures that will be used to ensure data quality and integrity. Careful adherence to these procedures will ensure that data generated from the demonstration 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 by operators of the PCB field analytical technology and by the reference laboratory to ensure that data resulting from this demonstration is of known quality and that a sufficient number of critical measurements are taken. EPA considers the demonstration to be classified as a Category II project. This section of the demonstration plan addresses the key elements that are required for Category II projects prepared according to guidelines in the EPA guidance documents "Preparation Aids for the Development of Category II Quality Assurance Project Plans" (Simes 1991), "Preparing Perfect Project Plans (1989), and the Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (Stanley and Verner 1983).

8.2 Quality Assurance Responsibilities

The developer project manager is responsible for coordinating the preparation of the QAPP for this demonstration and for its approval by the EPA project manager and ORNL. The developer project manager will ensure that the QAPP is implemented during all demonstration activities. The developer QA manager for the demonstration will review and approve the QAPP and will provide QA oversight of all demonstration activities. The QA audit function will be the responsibility of the EPA.

Samples will be analyzed on site by the PCB field analytical technology and off site by the reference laboratory using EPA-approved methods. Primary responsibility for ensuring that sampling activities comply with the requirements of the sampling collection procedures will rest with the EPA technical lead and ORNL field team leader. QA/QC activities for the PCB field analytical technology will include those activities recommended by developer and those required by the EPA or ORNL to assure the demonstration will provide data of the necessary quality.

QA/QC activities for the reference laboratory analysis of samples will be the responsibility of the reference laboratory supervisor. If problems arise or any data appear unusual, they will be thoroughly documented and corrective actions will be implemented as specified in this section. The QA/QC measurements made by the reference laboratory are dictated by the analytical methods being used.

8.3 Data Quality Indicators

The data obtained during the demonstration must be of sufficient quality for conclusions to be drawn on the PCB field analytical technology. 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: representativeness, completeness, comparability, accuracy, and precision.

Data generated by the PCB field analytical technology will be compared to the data generated from LAS Laboratories. High quality, well documented reference laboratory results are essential for meeting the purpose and objectives of this demonstration. LAS Laboratories data will be validated by ORNL prior to comparison with the

technology developer data. The following indicators of data quality will be closely evaluated to determine the performance of the technology when measured against data generated by the reference laboratory.

8.3.1 Representativeness

Representative samples, in general, are samples that contain a reasonable cross-section of the "population" over which they are to be used to make inferences. The population for demonstrations analyzed as part of this project includes a variety of media and contaminants that the innovative technologies are developed to accommodate.

This demonstration will evaluate the technologies under multiple conditions, while leveraging resources by: (1) conducting the demonstration at one site and utilizing a controlled environmental atmosphere to simulate temperature and humidity conditions in another part of the country; (2) evaluating PCB-contaminated soil samples from three different DOE sites, namely Portsmouth, Paducah, and the Oak Ridge Reservation; and (3) studying a wide range of PCB concentrations (0 to 500 ppm).

8.3.2 Comparability

Comparability is a quality parameter determined for the most part in the planning stages of the demonstration, often on the basis of prior knowledge of the innovative technologies' performance capabilities. First, the innovative technology must be comparable in some way to a reference or baseline method for the demonstration to be worthwhile. The study has been designed such that it is a statistically-rich approach that allows for an equal number of comparisons for every soil type and concentration level. Therefore, direct comparisons can be made with the reference laboratory results. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance.

8.3.3 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 demonstration is 95%.

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

- Instrument failure
- QC (MS/MSD or LCS) recovery outside of performance range
- Calibration requirements not being met
- Evaluated analyte levels in the method blank

The reference laboratory's SOP (See Appendix B) describes the corrective action plan for addresses such problems. The reference laboratory is responsible for qualifying data that is not valid.

8.3.4 Accuracy

Accuracy is a measure of how close, on average, values of the innovative technology are to the true values. Inaccuracies or biases are the result of systematic differences between these values. When comparing the innovative technology to a reference technology difficulties can arise. In some cases biases can be attributed to the innovative technology. These biases are often the result of poor calibration. Other possible sources of bias include systematic errors in standards preparation, biases introduced in the sample extraction, storage and shipping

processes and biases resulting from setup-related differences at the reference laboratory. Only the former of these sources is likely to be incurred by users of the innovative technologies. Most of the remaining sources represent inaccuracy that might be avoided through use of the innovative technology. Consequently every effort should be made by ORNL, the developers and the reference laboratory to identify specific sources of accuracy. The design of blanks, replicates and performance assessment samples should provide substantiating evidence to support this partitioning of sources of inaccuracy when results become available.

The strength of this demonstration's experimental design is that since an equal number of replicates will be performed for every samples at every concentration level, an equal number of accuracy comparisons can be made. However, enough replicates and quality control samples will be analyzed to independently assess each technology's performance. Section 5.7.2 (Methods of Statistical Analysis) provides the basic principles on how the accuracy of the technologies will be assessed.

8.3.5 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 involved in measurements of materials taken 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 imprecision in 1) and 2) is small. The interpretation of precision described in 3) is likely to be too site specific to be of general interest. The imprecision discussed in 4) is perhaps of most interest as it includes imprecision 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. If available, this information would provide the potential user with an estimate of how close a site characterization using this technology would come to providing the true site contaminate levels. Unfortunately, it is unlikely that the demonstrations will be extensive enough to provide much information on how this estimate would be provided.

The strength of this demonstration'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. Section 5.7.2 (Methods of Statistical Analysis) provides the basic principles on how the accuracy of the technologies will be assessed.

8.4 Calibration Procedures and Quality Control Checks

This section describes the calibration procedures and method-specific QC requirements that apply to both the technology and the reference analyses. It also contains a discussion of the corrective action to be taken if the QC parameters fall outside of the evaluation criteria.

8.4.1 Initial Calibration Procedures

Initial calibration for each technology will be performed according to the developer's recommendation (see technology descriptions, Section 3.0). The reference laboratory's initial calibration procedure is described in Appendix B.

8.4.2 Continuing Calibration Procedures

Continuing calibration for each technology will be performed according to the developer's recommendation (see technology descriptions, Section 3.0). The reference laboratory's continuing calibration procedure is described in Appendix B.

8.4.3 Method Blanks

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing, and is carried through the complete sample preparation and analytical procedures. Four method blanks will be included as part of the PE/QC program (see Table 8).

8.4.4 Spike Samples

The spiked soil samples used in this demonstration will be matrix spiked field samples. To prepare a spiked sample, the soil is first ground either using a mortar and pestle or a conventional blender. (Real samples will be oven-dried prior to grinding.) The soil is then sieved through a screen which was 16 mesh, or 1 mm particle size. The sieved soil is spiked with a diethyl ether solution of PCBs at the desired concentration. The soil is agitated using a mechanical shaker, then allowed to air-dry overnight. Several spiked samples are incorporated into the experimental design (see Table 7).

LAS Laboratories will also prepare and analyze matrix spike /matrix spike duplicate samples (MS/MSD) samples with every analytical batch. (The analytical batch can include no more than ten samples, excluding blanks, standards, spikes, and dilutions.) Aroclor 1260 is the matrix spike analyte.

8.4.5 Laboratory Control Samples

Laboratory control samples are samples of known composition that are analyzed periodically to assure that the analytical system is in control. These are analyzed just like a regular sample. One LCS will be analyzed per analytical batch. LAS will use purchased certified LCS standards.

8.4.6 Performance Evaluation Materials

Performance evaluation (PE) samples will be submitted to the reference laboratory and to the PCB field analytical technology for analysis. The certified concentrations of the PE samples will be used to evaluate the PCB field analytical technology and the LAS's method performance. The PCB field analytical technology will analyze

the PE samples periodically during the demonstration. PE samples will be obtained from Environmental Resource Associates and the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center.

8.4.7 Replicate Samples

All of the samples (real, PE/QC, blank, extracts) will be analyzed in quadruplicate so that the precision of the technologies and reference laboratory can be determined independently and compared.

8.5 Data Reduction, Review, and Reporting

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

8.5.1 Data Reduction

Data reduction refers to the process of converting the raw results from the technology into a concentration or other data format which will be used in the comparison. The procedures to be used will be technology dependent, but the final result format will be comparable to the reference lab results and the other developers. The actual comparisons will be performed by ORNL. The following is required for data reduction:

Concentrations: The report PCB concentration should be total PCB concentration in parts per million (i.e., ppm or µg/g, as received) for soil samples and µg/mL for surface samples. The PCB concentrations should be reported to 1 decimal places (e.g., 0.2 ppm or 100.7 ppm).

Nondetect Concentrations: If no PCB is detected, the concentration should be reported as 0 ppm. If PCB concentrations are found below detection limits, the actual concentrations should be reported and identify as " < Detection Limit" with the detection limit value reported. The method for establishing detection limits should be given.

Interval Data: PCB concentrations reported as interval results (i.e. Concentration Threshold Value or Concentration > Threshold Value) should select Threshold Values that are associated with regulatory requirements (e.g., 2 ppm, 50 ppm).

8.5.2 Data Review

The operator will verify the completeness of the appropriate data forms and the completeness and correctness of data acquisition and reduction. The field team observer 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 be examined by the individual operators and DOE, EPA, and ORNL observers. The individual operators will verify that all instrument systems are in control and that QA objectives for accuracy, completeness, and method detection limits 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 reference 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.

8.5.3 Data Reporting

This section contains a list of the data to be reported by both the technology and the reference method. At a minimum, the data tabulation will list the results for each sample and include reporting units, sample numbers, results, and data qualifiers. (A sample results form will be provided for completion by the developers.) All QC information such as calibrations, blanks and reference samples will also be included with the raw analytical data. All data should be reported in hardcopy and electronically in a common spreadsheet or database format.

Developer results will be due to ORNL at the conclusion of a day's field activities. The developer's final report will be due to ORNL one week after the conclusion of the demonstration. Any discrepancies between the originally reported result and the final result must be described. Reference laboratory data is due to ORNL within 28 calendar days of sample receipt.

8.6 Calculation of Data Quality Indicators

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. Precision for the PCB verification demonstration 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 "$\overline{Y}$" is calculated by:

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

The standard deviation is the square root of S^2 and implies that the uncertainty is independent of the PCB concentration values. The percent relative error,

$\%RE = 100\% \times \frac{S}{\overline{Y}}$, is an alternative expression of precision and implies that the uncertainty increases linearly with the average PCB concentration values. Replicate samples at each PCB concentration can be used to establish the relationship between the uncertainty and the average PCB concentration.

Precision measurements can not be calculated for PCB concentration results reported as interval data (i.e. Concentration Threshold Value or Concentration > Threshold Value). The paired data for developer's measurements and reference laboratory's measurements are used to calculate the false positive error rate (FP) and false negative error rate (FN) by:

$$FP = \frac{(\textit{Number of Developer's Concentrations} > \textit{Threshold})}{(\textit{Number of Reference Concentrations} \leq \textit{Threshold})} \quad ,$$

and

$$FN = \frac{(\textit{Number of Developer's Concentrations} \leq \textit{Threshold})}{(\textit{Number of Reference Concentrations} > \textit{Threshold})} \quad .$$

Accuracy is a measure of how close, on average, the measured PCB concentrations are to the true values or to an accepted reference value. Accuracy for the PCB verification demonstration will be relative to a standard PCB concentration in the case of performance evaluation samples or to a reference value measured by a reference laboratory. 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 demonstration or at the reference laboratory. Consequently every effort will be made by ORNL, the technology developers and the reference laboratory to identify specific sources of inaccuracies. The demonstration includes blanks, replicates and performance evaluation samples that should provide substantiating evidence to support this partitioning of sources of inaccuracies when results become available.

Bias represents a constant error as opposed to a random error. Bias is estimated by the difference between the PCB measured concentration and the accepted value (performance evaluation value or reference laboratory value). The soil samples will be allocated so that measurements by the PCB technologies are paired with those made by the reference laboratory. If "n" PCB concentration measurements are represented by Y_1, Y_2, ..., Y_n for a PCB measurement technology and by R_1, R_2, ..., R_n for the reference laboratory, the estimated average bias is calculated by:

$$Bias = \frac{1}{n} \sum_{k=1}^{n} (Y_k - R_k) \quad .$$

Hypothesis test for testing if the bias is significantly different than zero will consider the appropriate uncertainty values.

Analysis of PCB surface samples will report percent recovery values relative to the spiking concentrations. The percent recovery will be calculated from average PCB concentration measured by the developer's technology, "$\overline{Y}$" , and compare it to the average PCB concentration measured to the reference average value "$\overline{R}$" by:

$$\% \ Recovery = 100\% \times \frac{\overline{Y}}{\overline{R}} \quad .$$

8.7 Performance and System Audits

The following audits will be performed during this demonstration. These audits will determine if this demonstration plan is being implemented as intended.

8.7.1 Performance Audit

Performance evaluation (PE) samples will be submitted to the reference laboratory and to the PCB field analytical technology for analysis. The certified concentrations of the PE samples will be used to evaluate the PCB field analytical technology and the LAS's method performance. The PCB field analytical technology will analyze

the PE samples periodically during the demonstration. PE samples will be obtained from Environmental Resource Associates and the U. S. EPA's Office of Solid Waste and Emergency Response's Analytical Operations Center.

8.7.2 On-Site System Audits

On-site system audits for sampling activities, field operations, and laboratories will be conducted as requested by the EPA project manager. These audits will be performed by the EPA Project Manager, DOE, and/or ORNL.

8.8 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 demonstration.

8.8.1 Status Reports

Through brief morning meetings on each day of the demonstration, the developers and ORNL will regularly inform the EPA and DOE project managers of the status of the project. They should discuss project progress, problems and associated corrective actions, and future scheduled activities associated with the demonstration. When problems occur, the developer and ORNL will discuss them with EPA and/or DOE, estimate the type and degree of impact, and describe the corrective actions taken to mitigate the impact and to prevent a recurrence of the problems.

8.8.2 Audit Reports

Any QA audits or inspections that take place in the field or at the reference laboratory while the demonstration is being conducted will be formally reported by the auditors to EPA and DOE project managers who will forward them to the developer, ORNL QC Manager, and the ORNL project manager for appropriate actions. Informal reporting of audit results will be reported immediately to EPA and DOE.

8.9 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 operators will be responsible for seeing that the problem is resolved. If the issue is one that is identified by ORNL, DOE, 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 demonstration plan will be noted in the technology verification report. The reference laboratory's SOP (See Appendix B) describes the corrective action plan for not meeting minimum QC requirements.

9.0 DATA MANAGEMENT AND ASSESSMENT

The developer, ORNL, DOE, and EPA each have distinct responsibilities for managing and analyzing demonstration data. ORNL is responsible for managing all the data and information generated during the

demonstration. The developer is responsible for furnishing those records generated by the technology operator. EPA, DOE, and ORNL are responsible for analysis and verification of the data.

There are a variety of pieces of data and information that will be generated during a demonstration. Each piece of data or information identified for collection in the demonstration plan will need to be provided to ORNL.

Innovative Technology Data: The developer is responsible for obtaining, reducing, interpreting, validating, and reporting the data associated with his technology's performance. These data should be reported in hard copy and electronic format (e.g., spreadsheet). Developer results will be due to ORNL at the conclusion of a day's field activities. The developer's final report will be due to ORNL one week after the demonstration. Any discrepancies between the originally reported result and the final result must be described.

Reference Laboratory Analyses: The raw data and the validated data must be provided to ORNL. These data should be provided in hard copy and in electronic format. As with the data generated by the innovative technology, the electronic copy of the laboratory data should be provided in a spreadsheet. In addition to the sample results, all QA/QC summary forms for the reference analyses must be provided. Reference laboratory data is due to ORNL within 28 calendar days of sample receipt.

Other items that must be provided include:

- field notebooks;
- photographs, slides and videotapes (copies);
- results from the use of other field analytical methods;
- profiles or traces

10.0 HEALTH AND SAFETY PLAN

10.1 Introduction

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

10.2 Contact Information

The ORNL project manager will be Roger Jenkins, (423) 576-8594.
The Field Site Supervisor will be Amy Dindal, (423) 574-4863.
The Site Health and Safety Officer will be Fred Smith, (423) 574-4945.
The ORNL Office of Safety and Health Protection Director is Ann Shirley, (423) 576-8262.
The ORNL PCB Site Coordinator is Jade Thomas, (423) 241-6043.
The Laboratory Shift Superintendent number is (423) 574-6606.
The Emergency Communications Center number is (423) 574-6646.
IN CASE OF ANY EMERGENCY, DIAL 9-1-1.

Note: To call any on-site number, dial the last five digits (e.g., 6-9584).

10.3 Health and Safety Plan Enforcement

ORNL project manager, field site supervisor, and site health and safety officer will be responsible for enforcing the health and safety plan. ORNL project manager will ultimately be responsible for ensuring that all demonstration participants abide by the requirements of this HASP. ORNL field site supervisor will oversee and direct field activities and is responsible for ensuring compliance with this HASP.

10.4 Site Background

The demonstration of PCB field analytical techniques will be conducted at the Oak Ridge National Laboratory (ORNL), which is managed by Lockheed Martin Energy Research Corporation, Oak Ridge, Tennessee. Oak Ridge is located a short distance from Gatlinburg and the Great Smoky Mountains National Park. Recreation areas include Big South Fork and several Tennessee Valley Authority rivers and dams. A new highway extension allows easier access to the airport, now within 20 miles of the three Oak Ridge facilities. The city of Oak Ridge is home to the American Museum of Science and Energy, the University of Tennessee Arboretum, Oak Ridge Associated Universities, and several hotels and restaurants to accommodate area visitors.

Field activities will occur at two sites at ORNL: the area west of Building 5507(Site #1) and inside a controlled environmental atmosphere chamber (Site #2) which is located in Building 5507. Building 5507 is located in a relatively secluded part of the Laboratory (see Figure 3). 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 for temperature, humidity, and slightly subambient pressure control at air circulation flow rates up to five hundred cubic feet per minute.

10.5 Visitors

Visitors will be badged and escorted at all times by ORNL personnel. Visitors will follow standard ORNL safety and health policies and practices.

10.6 Demonstration-Specific Hazard Evaluation

The proposed demonstration activities have been evaluated by ORNL radiation protection personnel. No radiation protection hazards have been identified. PCBs issues and hazards will be controlled per ORNL procedures (Oak Ridge Reservation Polychlorinated Biphenyl Federal Facilities Compliance Agreement, ORR-PCB-FFCA). The Lockheed Martin Energy Research Corporation procedure, "EPP 3.1 Management of Polychlorinated Biphenyls" will also be followed and can be found at the following web site address: http://www-internal.ornl.gov/~p2w/oecd/epp3.htm.

The hazards associated with this demonstration 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.

All hazardous waste generated by the technology developers will be properly disposed of by CASD's environmental protection officer (Kim Thomas). The technology developers will assist with this process by providing accurate records of the waste contents and approximate concentrations.

10.7 Training Requirements

All technology developers must be badged and escorted by ORNL personnel at all times. The developers will be escorted in lieu of additional site-specific training.

10.8 Exposure Pathways

Exposure to VOCs and SVOCs during field activities may occur through inhalations or ingestion. The most likely exposure to VOCs and SVOCs during the demonstration 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 developers must provide their own PPE. Although unlikely to be necessary, visitors will be provided with PPE if warranted.

10.9 Health Effects

PCBs will be the most prevalent chemical hazards at the demonstration. PCBs are:

Nonflammable liquids
Carcinogenic
Strong oxidizers
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.

10.10 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.

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.

Mechanical, Electrical, Noise Hazards

Some technology-specific hazards may be identified once the developers 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.

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.

Inclement Weather

The demonstration will occur the latter part of July. The possibility of inclement weather (particulary rain and thundershowers) exists. The developers should be prepared to deal with a possible inclement weather situation.

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

Heat Stress

Since the demonstration will occur in July, the possibility of a heat-related injury during field work is great. 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 demonstration 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.

Insect and Other Animal Stings and Bites

A potential for insect and other animal stings or bites exists during the technology demonstration. inspect 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.

10.11 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.

Levels of Protection

For this demonstration, 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.

Protective Equipment and Clothing

Because the anticipated hazard level is low, field and chamber work will be performed using Level D protection. Level D PPE will be supplied by the individual technology developer. ORNL will provide visitors with PPE if necessary. If site conditions or the results of Industrial Hygiene monitoring indicates that additional hazards are present, PPE levels will be reconsidered.

The following is the list of protective equipment necessary for demonstration operations:

Appropriate work clothes (no shorts or open-toed shoes)
Disposable outer gloves.

Medical Support

A complete medical facility is located on-site in Building 4500 North. Medical help can be summons 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 summons the fire department and the laboratory shift superintendent's office.

Environmental Surveillance

The ORNL PCB Site coordinator will be responsible for surveying the site before, during, and after the demonstration. Appropriate personnel will be on-hand to assist all demonstration participants to deal with any health or safety concerns.

10.12 Site Control

Site Control Zones

Access to the demonstration site will be unrestricted, but controlled. Any visitors to the site must be accompanied by a member of the ORNL demonstration field team.

Safe Work Practices

Each company will provide the required training and equipment for their personnel to meet safe operating practice and procedures. The individual technology developer 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 10.11) will be followed.

Complaints

All complaints should be filed with the ORNL Field Site Supervisor (Amy Dindal). All complaints will be treated on an individual basis and be dealt with accordingly.

10.13 Radiological Hazards

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

Appendices A, B, C, D, and E
Are Procedures and

Are Not Included in his Document

Contact Amy B. Dindal at Oak Ridge National Laboratory
If procedures are needed
423-574-4863
dindalab@ornl.gov