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# Environmental Technology Verification Report

## Immunoassay Kit

EnviroLogix, Inc.  
PCB in Soil Tube Assay

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# Environmental Technology Verification Report

## Immunoassay Kit

### EnviroLogix, Inc. PCB in Soil Tube Assay

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Superfund Innovative Technology  
Evaluation Program



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## Notice

The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), and the U.S. Department of Energy's Environmental Management (EM) Program, funded and managed, through Interagency Agreement No. DW89937854 with Oak Ridge National Laboratory, the verification effort described herein. This report has been peer and administratively reviewed and has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.



**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**

Office of Research and Development  
Washington, D.C. 20460



**ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM  
VERIFICATION STATEMENT**

TECHNOLOGY TYPE:	<b>POLYCHLORINATED BIPHENYL (PCB) FIELD ANALYTICAL TECHNIQUES</b>
APPLICATION:	<b>MEASUREMENT OF PCBs IN SOILS AND SOLVENT EXTRACTS</b>
TECHNOLOGY NAME:	<b>PCB IN SOIL TUBE ASSAY</b>
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The U.S. Environmental Protection Agency (EPA) has created a program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the Environmental Technology Verification (ETV) Program is to further environmental protection by substantially accelerating the acceptance and use of improved and more cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of EnviroLogix Inc. PCB in Soil Tube Assay.

**PROGRAM OPERATION**

EPA, in partnership with recognized testing organizations, objectively and systematically evaluates the performance of innovative technologies. Together, with the full participation of the technology developer, they develop plans, conduct tests, collect and analyze data, and report findings. The evaluations are conducted according to a rigorous demonstration plan and established protocols for quality assurance. EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, with the support of the U.S. Department of Energy's (DOE) Environmental Management (EM) program, selected Oak Ridge National Laboratory (ORNL) as the testing organization for the performance verification of polychlorinated biphenyls (PCBs) field analytical techniques.

**DEMONSTRATION DESCRIPTION**

In July 1997, the performance of six PCB field analytical techniques was determined under field conditions. In September 1998, the performance of EnviroLogix Inc.'s PCB in Soil Tube Assay kit was evaluated similarly. Each technology was independently evaluated by comparing field analysis results with those obtained using approved reference methods. Performance evaluation (PE) samples were also used to assess independently the accuracy and comparability of each technology.

The demonstration was designed to detect and measure PCBs in soil and solvent extracts. For EnviroLogix, the demonstration was conducted at ORNL in Oak Ridge, Tennessee, from September 21 through 25, 1998. The study was conducted under two environmental conditions. The first site was outdoors, with naturally fluctuating temperatures and relative humidity conditions. The second site was inside a controlled environmental chamber, with generally cooler temperatures and lower relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed in this study. Solutions of PCBs were also analyzed to simulate extracted surface wipe samples. The results of the soil and extract analyses conducted under field conditions by the technology were compared with results from analyses of homogenous replicate samples conducted by conventional EPA SW-846 methodology in an approved

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reference laboratory. Details of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Immunoassay Kit, EnviroLogix Inc., PCB in Soil Tube Assay*, EPA/600/R-98/173.

## TECHNOLOGY DESCRIPTION

The EnviroLogix PCB in Soil Tube Assay applies the principles of enzyme linked immunosorbent assay (ELISA) to the determination of PCB. In such an assay, an enzyme has been chemically linked to a PCB molecule or PCB analog to create a labeled PCB reagent. The labeled PCB reagent (called a conjugate) is mixed with an extract of native sample containing the PCB contaminant. A portion of the mixture is applied to a surface (i.e., the inside of a test tube) to which an antibody specific for PCB has been affixed. The native PCB and PCB-enzyme conjugate compete for a limited number of antibody sites. After a period of time, the solution is washed away, and what remains is either PCB-antibody complexes or enzyme-PCB-antibody complexes attached to the test surface. The proportion of the two complexes on the test surface is determined by the amount of native PCB in the original sample. The enzyme present on the test surface is used to catalyze a color change reaction in a solution added to the test surface. Because the amount of enzyme present is inversely proportional to the concentration of native PCB contaminant, the amount of color development is inversely proportional to the concentration of PCB contaminant. The color development is quantified through the use of a hand-held photometer.

The EnviroLogix PCB in Soil Tube Assay is designed for semi-quantitative field screening for PCBs in soil. The kit is supplied with calibrators equivalent to 1 part per million (ppm) and 10 ppm PCB (Aroclor 1254) in soil. These calibrators are used to evaluate threshold levels of 1 and 10 ppm. A threshold level of 50 ppm can also be evaluated using the 10 ppm calibrator by preparing a 1:5 sample extract dilution into methanol. For the extract samples, the threshold levels are 0.4, 4, and 20  $\mu\text{g/mL}$ .

## VERIFICATION OF PERFORMANCE

The following performance characteristics of the PCB in Soil Tube Assay were observed:

**Throughput:** Throughput was 8 samples/hour under outdoor conditions and 7 samples/hour under chamber conditions for one operator. This rate included sample preparation and analysis.

**Ease of Use:** One operator analyzed samples during the demonstration. Minimal training (4 h) is required to operate the kit, provided the user has a fundamental understanding of basic chemical and field analytical techniques.

**Completeness:** The PCB in Soil Tube Assay generated results for all 232 PCB samples for a completeness of 100%.

**False positive/negative results:** All of the blank samples (soils and extracts) were reported as the lowest reporting interval, which included zero; therefore, the percentage of false positive results was 0%. The kit reported no false negative results for extracts, and 4% (7 of 192 samples) for soils.

**Precision:** The overall precision—based on the percentage of combined sample sets where all four replicates were reported as the same interval—was 56% for the PE soils, 68% for the environmental soils, and 75% for the extracts.

**Accuracy:** Accuracy was assessed using PE soil and extract samples. Accuracy, defined as the percentage of the PCB in Soil Tube Assay results that agreed with the accepted concentration, was 78% for PE soils and 92% for extracts. In general, the fraction of samples that was biased high was comparable (10% for PE soils and 0% for extracts) to the fraction that was biased low (13% for PE soils and 8% for extracts).

**Comparability:** Comparability, like accuracy, was defined as the percentage of results that agreed with, was above (i.e., biased high), or was below (i.e., biased low) the reference laboratory result. The percentage of samples that agreed with the reference laboratory results was 82% for all soils (PE and environmental). The fraction of samples that was biased

high was again comparable (12%) to the fraction that was biased low (7%). Extract results could not be compared because no reference laboratory data was generated for these samples.

**Regulatory Decision-making:** One objective of this demonstration was to assess the technology's ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils. For PE and environmental soil samples in the range of 40 to 60 ppm, 66% of the PCB in Soil Tube Assay results agreed with the reference laboratory, 32% were biased high, and 2% were biased low. The test kit results for this concentration range were different from what was observed for the entire data set in that the fraction of samples that were biased high was significantly higher (32% versus 12%).

**Data quality levels:** The performance of the test kit was characterized as unbiased, because most (78%) of the PCB in Soil Tube Assay results agreed with the certified PE values, but imprecise, because nearly half (44%) of the PE replicate results were not reported as the same interval. It should be noted that almost all of the imprecision occurred when the concentration of the sample was near one of the test kit's threshold values (i.e., 1, 10, or 50 ppm).

The results of the demonstration show that the PCB in Soil Tube Assay can provide useful, cost-effective data for environmental problem-solving and decision-making. Undoubtedly, it will be employed in a variety of applications, ranging from serving as a complement to data generated in a fixed analytical laboratory to generating data that will stand alone in the decision-making process. As with any technology selection, the user must determine if this technology is appropriate for the application and the project data quality objectives. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

Gary J. Foley, Ph.D.  
Director  
National Exposure Research Laboratory  
Office of Research and Development

**NOTICE:** EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable Federal, State, and Local requirements.





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## Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's natural resources. The National Exposure Research Laboratory (NERL) is EPA's center for the investigation of technical and management approaches for identifying and quantifying risks to human health and the environment. NERL's research goals are to (1) develop and evaluate technologies for the characterization and monitoring of air, soil, and water; (2) support regulatory and policy decisions; and (3) provide the science support needed to ensure effective implementation of environmental regulations and strategies.

EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This program is administered by NERL's Environmental Sciences Division in Las Vegas, Nevada.

The U.S. Department of Energy's (DOE's) Environmental Management (EM) program has entered into active partnership with EPA, providing cooperative technical management and funding support. DOE EM realizes that its goals for rapid and cost-effective cleanup hinge on the deployment of innovative environmental characterization and monitoring technologies. To this end, DOE EM shares the goals and objectives of the ETV.

Candidate technologies for these programs originate from the private sector and must be commercially ready. Through the ETV Program, developers are given the opportunity to conduct rigorous demonstrations of their technologies under realistic field conditions. By completing the evaluation and distributing the results, EPA establishes a baseline for acceptance and use of these technologies.

Gary J. Foley, Ph.D.  
Director  
National Exposure Research Laboratory  
Office of Research and Development



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## Abstract

In July 1997, the U.S. Environmental Protection Agency (EPA) conducted a demonstration of polychlorinated biphenyl (PCB) field analytical techniques. The purpose of this demonstration was to evaluate field analytical technologies capable of detecting and quantifying PCBs in soils and solvent extracts. The fundamental objectives of this demonstration were (1) to obtain technology performance information using environmental and quality control samples, (2) to determine how comparable the developer field analytical results were with conventional reference laboratory results, and (3) to report on the logistical operation of the technology. The demonstration design was subjected to extensive review and comment by EPA's National Exposure Research Laboratory (NERL) Environmental Sciences Division in Las Vegas, Nevada; Oak Ridge National Laboratory (ORNL); EPA Regional Offices; the U.S. Department of Energy (DOE); and the technology developers.

The demonstration study was conducted at ORNL under two sets of environmental conditions. The first site was outdoors, with naturally variable temperature and relative humidity conditions typical of eastern Tennessee in the summer. A second site was located inside a controlled environmental chamber having lower, and relatively stable, temperature and relative humidity conditions. The test samples analyzed during this demonstration were performance evaluation (PE) soil, environmental soil, and extract samples. Actual environmental soil samples, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed and ranged in concentration from 0.1 to 700 parts per million (ppm). Extract samples were used to simulate surface wipe samples, and were evaluated at concentrations of 0, 10, and 100 µg/mL. The reference laboratory method used to evaluate the comparability of data was EPA SW-846 Method 8081.

In September 1998, EnviroLogix's PCB in Soil Tube Assay was evaluated. Six other field analytical technologies were tested in July 1997: the L2000 PCB/Chloride Analyzer (Dexsil Corporation), the PCB Immunoassay Kit (Hach Company), the 4100 Vapor Detector (Electronic Sensor Technology), and three immunoassay kits—D TECH, EnviroGard, and RaPID Assay System (Strategic Diagnostics Inc.). The purpose of an Environmental Technology Verification Report (ETVR) is to document the demonstration activities, present demonstration data, and verify the performance of the technology. This ETVR presents information regarding the performance of EnviroLogix's PCB in Soil Tube Assay. Separate ETVRs have been published for the other technologies demonstrated.

The PCB in Soil Tube Assay is an immunoassay kit used to determine PCB concentrations as interval results. The test kit uses a competitive binding enzyme immunoassay to perform rapid testing for PCBs in soils and solutions at specified threshold values of 1, 10, and 50 ppm. The test kit is standardized using Aroclor 1254. The presence of PCBs is detected by a photometer based on a colored reaction in which the color development is inversely proportional to the concentration of PCB in the sample (e.g., the darker the color, the less analyte PCB is present in the sample). The kit provides no information on Aroclor identification.

The kit's quantitative results were based on the analysis of calibration standards with every batch of 12 to 17 samples. Because the test kit is an interval technique, method detection limits are not applicable. Precision, defined as the percentage of the sample sets where all four replicates were reported as the same interval range,

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was 56% for PE soils, 68% for environmental soils, and 75% for extracts. Accuracy, defined as the percentage of PCB in Soil Tube Assay results that agreed with the accepted concentration, was 78% for PE soils and 92% for extracts. In general, the percentage of samples that was biased high was comparable (10% for PE soils and 0% for extracts) to the percentage that was biased low (13% for PE soils and 8% for extracts). Comparability was defined similarly to accuracy, but the test kit result was compared with the reference laboratory result rather than with the accepted concentration to determine comparability. For all soil samples (PE and environmental), the percentage of samples that agreed with the reference laboratory results was 82%. The percentage of samples that was biased high was again comparable (12%) to the percentage that was biased low (7%). Comparability could not be assessed for extract samples because no reference laboratory data were generated for these samples.

The demonstration found that the PCB in Soil Tube Assay was simple to operate in the field, requiring about an hour for initial setup and preparation for sample analysis. Once the kit was operational, the sample throughput of the kit by a single analyst was 8 samples/hour under outdoor conditions and 7 samples/hour under chamber conditions. Minimal training (4 hours) is required to operate the test kit, provided the user has a fundamental understanding of basic chemical and field analytical techniques. The performance of the test kit was characterized as unbiased, because most (78%) of the PCB in Soil Tube Assay results agreed with the certified PE values, but imprecise, because nearly half (44%) of the PE replicate results were not reported as the same interval. It should be noted that almost all of the imprecision occurred when the concentration of the sample was near one of the test kit's threshold values (i.e., 1, 10, or 50 ppm). The test kit had no false positive results (i.e., a result in which the technology detects PCBs in the sample above the detection limit when there actually are no PCBs present), and 4% of the soil sample results were false negatives (i.e., the technology indicates that there are no PCBs present in the sample, when there actually are). For extract samples, the test kit had no false positive or false negative results.

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## List of Abbreviations and Acronyms

ASTM	American Society for Testing and Materials
BHC	benzenehexachloride
CCV	continuing calibration verification standard
CSCT	Consortium for Site Characterization Technology
DCB	decachlorobiphenyl
DOE	U.S. Department of Energy
DQO	data quality objective
ELISA	enzyme-linked immunosorbent assay
EM	Environmental Management (DOE)
EPA	U.S. Environmental Protection Agency
ERA	Environmental Resource Associates
ETTP	East Tennessee Technology Park
ETV	Environmental Technology Verification Program
ETVR	Environmental Technology Verification Report
EvTEC	Environmental Technology Evaluation Center
fn	false negative result
FN	false negative decision error rate
fp	false positive result
FP	false positive decision error rate
HEPA	high-efficiency particulate air
ID	identifier
LCS	laboratory control sample
LV	Las Vegas
MS	matrix spike

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MSD	matrix spike duplicate
n	number of samples
NRC	Nuclear Regulatory Commission
OD	optical density
ORNL	Oak Ridge National Laboratory
ORO	Oak Ridge Operations (DOE)
PARCC	precision, accuracy, representativeness, completeness, comparability
PCB	polychlorinated biphenyl
PE	performance evaluation
ppm	parts per million (equivalent units: mg/kg for soils and µg/mL for extracts)
Pr	probability
QA	quality assurance
QC	quality control
R <sup>2</sup>	coefficient of determination
RDL	reporting detection limit
RH	relative humidity
RFD	request for disposal
RPD	relative percentage difference
RSD	relative standard deviation (percent)
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	standard deviation
SITE	Superfund Innovative Technology Evaluation
SMO	Sample Management Office
SOP	standard operating procedure
SSM	synthetic soil matrix
TCMX	tetrachloro- <i>m</i> -xylene

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## Acknowledgments

The authors wish to acknowledge the support of all those who helped plan and conduct the demonstration, analyze the data, and prepare this report. In particular, we recognize the technical expertise of Mitchell Erickson (Environmental Measurements Laboratory), Viorica Lopez-Avila (Midwest Research Institute), and Robert F. O'Brien (Pacific Northwest National Laboratory), who were peer reviewers of this report. For internal peer review, we thank Stacy Barshick (ORNL); for technical support during the demonstration, Todd Skeen and Ralph Ilgner (ORNL); for site safety and health support, Kim Thomas, Marilyn Hanner, and Fred Smith (ORNL); for administrative support, Betty Maestas and Linda Plemmons (ORNL); for sample collection support, Wade Hollinger, Charlotte Schaefer, and Arlin Yeager (LMES), and Mike Rudacille and W. T. Wright (EET Corporation); for preliminary soil characterization support, Frank Gardner, John Zutman, and Bob Schlosser (ORNL, Grand Junction, Colo.); for sample management support, Angie McGee, Suzanne Johnson, and Mary Lane Moore (LMES); for providing performance evaluations samples, Michael Wilson (EPA's Office of Solid Waste and Emergency Response's Analytical Operations and Data Quality Center); and for technical guidance and project management of the demonstration, David Carden, Marty Atkins, and Regina Chung (DOE's Oak Ridge Operations Office), David Bottrell (DOE, Headquarters), Deana Crumbling (EPA's Technology Innovation Office), and Stephen Billets, Gary Robertson, and Eric Koglin (EPA's National Exposure Research Laboratory, Las Vegas, Nevada). The authors also acknowledge the participation of EnviroLogix, Inc., in particular, Jonathan Matt, who performed the analyses during the demonstration.

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## Section 1 Introduction

The performance evaluation of innovative and alternative environmental technologies is an integral part of the U.S. Environmental Protection Agency's (EPA's) mission. Early efforts focused on evaluating technologies that supported the implementation of the Clean Air and Clean Water Acts. In 1987, the Agency began to evaluate the cost and performance of remediation and monitoring technologies under the Superfund Innovative Technology Evaluation (SITE) program. This was in response to the mandate in the Superfund Amendments and Reauthorization Act (SARA) of 1986. In 1990, the U.S. Technology Policy was announced. This policy placed a renewed emphasis on "making the best use of technology in achieving the national goals of improved quality of life for all Americans, continued economic growth, and national security." In the spirit of the Technology Policy, the Agency began to direct a portion of its resources toward the promotion, recognition, acceptance, and use of U.S.-developed innovative environmental technologies both domestically and abroad.

The Environmental Technology Verification (ETV) Program was created by the Agency to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. The ETV Program capitalizes upon and applies the lessons that were learned in the implementation of the SITE Program to the verification of twelve categories of environmental technology: Drinking Water Systems, Pollution Prevention/Waste Treatment, Pollution Prevention/ Innovative Coatings and Coatings Equipment, Indoor Air Products, Air Pollution Control, Advanced Monitoring Systems, EvTEC (an independent, private-sector approach), Wet Weather Flow Technologies, Pollution Prevention/Metal Finishing, Source Water Protection Technologies, Site Characterization and Monitoring Technology [also referred to as the Consortium for Site Characterization Technology (CSCT)], and Climate Change Technologies. The performance verification contained in this report was based on the data collected during a demonstration of polychlorinated biphenyl (PCB) field analytical technologies. The demonstration was administered by CSCT.

For each pilot, EPA utilizes the expertise of partner "verification organizations" to design efficient procedures for conducting performance tests of environmental technologies. To date, EPA has partnered with federal laboratories and state, university, and private sector entities. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from all major stakeholder/customer groups associated with the technology area.

In July 1997, CSCT, in cooperation with the U.S. Department of Energy's (DOE's) Environmental Management (EM) Program, conducted a demonstration to verify the performance of six field analytical technologies for PCBs: the L2000 PCB/Chloride Analyzer (Dexsil Corporation), the PCB Immunoassay Kit



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(Hach Company), the 4100 Vapor Detector (Electronic Sensor Technology), and three immunoassay kits from Strategic Diagnostics, Inc.: D TECH, EnviroGard, and RaPID Assay System. Another technology, the EnviroLogix, Inc., PCB in Soil Tube Assay, was evaluated in September 1998. This environmental technology verification report (ETVR) presents the results of the demonstration study for the PCB in Soil Tube Assay. Separate ETVRs have been published for the other six technologies.

### **Technology Verification Process**

The technology verification process is intended to serve as a template for conducting technology demonstrations that will generate high-quality data that EPA can use to verify technology performance. Four key steps are inherent in the process:

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

### ***Needs Identification and Technology Selection***

The first aspect of the technology verification process is to determine technology needs of EPA and the regulated community. EPA, DOE, the U.S. Department of Defense, industry, and state agencies are asked to identify technology needs and interest in a technology. Once a technology need is established, a search is conducted to identify suitable technologies that will address this need. The technology search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searches of industry and trade publications, attendance at related conferences, and leads from technology developers. Characterization and monitoring technologies are evaluated against the following criteria:

- meets user needs;
- may be used in the field or in a mobile laboratory;
- is applicable to a variety of environmentally impacted sites;
- has high potential for resolving problems for which current methods are unsatisfactory;
- is cost competitive with current methods;
- performs better than current methods in areas such as data quality, sample preparation, or analytical turnaround time;

- 
- uses techniques that are easier and safer than current methods;
  - is a commercially available, field-ready technology.

### ***Demonstration Planning and Implementation***

After a technology has been selected, EPA, the verification organization, and the developer agree to the responsibilities for conducting the demonstration and evaluating the technology. The following tasks are undertaken at this time:

- identifying demonstration sites that will provide the appropriate physical or chemical environment, including contaminated media;
- identifying and defining the roles of demonstration participants, observers, and reviewers;
- determining logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network);
- arranging analytical and sampling support;
- preparing and implementing a demonstration plan that addresses the experimental design, sampling design, quality assurance/quality control (QA/QC), health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements.

### ***Report Preparation***

Innovative technologies are evaluated independently and, when possible, against conventional technologies. The field technologies are operated by the developers in the presence of independent technology observers. The technology observers are provided by EPA or a third-party group. Demonstration data are used to evaluate the capabilities, limitations, and field applications of each technology. Following the demonstration, all raw and reduced data used to evaluate each technology are compiled into a technology evaluation report, which is mandated by EPA as a record of the demonstration. A data summary and detailed evaluation of each technology are published in an ETVR.

### ***Information Distribution***

The goal of the information distribution strategy is to ensure that ETVRs are readily available to interested parties through traditional data distribution pathways, such as printed documents. Documents are also available on the World Wide Web through the ETV Web site (<http://www.epa.gov/etv>) and through a Web site supported by the EPA Office of Solid Waste and Emergency Response's Technology Innovation Office (<http://CLU-in.com>).

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**Demonstration Purpose**

The purpose of this demonstration was to obtain performance information for PCB field analytical technologies, to compare the results with conventional fixed-laboratory results, and to provide supplemental information (e.g., cost, sample throughput, and training requirements) regarding the operation of the technology. The demonstration was conducted under two climatic conditions. One set of activities was conducted outdoors, with naturally fluctuating temperatures and relative humidity conditions. A second set was conducted in a controlled environmental facility, with lower, relatively stable temperatures and relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were used in this study. PCB soil concentrations ranged from approximately 0.1 to 700 parts per million (ppm). The developer also analyzed 24 solutions of known PCB concentration that were used to simulate extracted wipe samples. The extract samples ranged in concentration from 0 to 100 µg/mL.

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## Section 2 Technology Description

### Objective

The objective of this section is to describe the technology being demonstrated, including the operating principles underlying the technology and the overall approach to its use. The information provided here is excerpted from that provided by the developer. Performance characteristics described in this section are specified by the developer; they may or may not be substantiated by the data presented in Section 5.

### Principle

The EnviroLogix PCB in Soil Tube Assay applies the principles of enzyme-linked immunosorbent assay (ELISA) to determine PCB concentration. In such an assay, an enzyme has been chemically linked to a PCB molecule or a PCB analog to create a labeled PCB reagent. The labeled PCB reagent (called a conjugate) is mixed with an extract of native sample containing the PCB contaminant. A portion of the mixture is applied to a surface (i.e., the inside of a test tube) to which an antibody specific for PCB has been affixed. The native PCB and PCB-enzyme conjugate compete for a limited number of antibody sites. After a period of time, the solution is washed away, and what remain are either PCB-antibody complexes or enzyme-PCB-antibody complexes attached to the test surface. The proportion of the two complexes on the test surface is determined by the amount of native PCB in the original sample. The enzyme present on the test surface is used to catalyze a color change reaction in a solution added to the test surface. Because the amount of enzyme present is inversely proportional to the concentration of native PCB contaminant, the amount of color development is inversely proportional to the concentration of PCB contaminant. The color development is quantified through the use of a hand-held photometer.

The EnviroLogix PCB in Soil Tube Kit is designed for semi-quantitative field-screening for PCBs in soil. The kit is supplied with calibrators equivalent to 1 part per million (ppm) and 10 ppm PCB (Aroclor 1254) in soil. These calibrators are used to evaluate threshold levels of 1 and 10 ppm. A threshold level of 50 ppm can also be evaluated using the 10-ppm calibrator by preparing a 1:5 sample extract dilution into methanol. For the extract samples, the threshold levels are 0.4, 4, and 20  $\mu\text{g/mL}$ .

### Applications and Advantages

The EnviroLogix PCB test kit can be used in a number of applications, including initial site characterization and mapping, real-time testing during remediation, and screening of negatives prior to gas chromatography confirmation. The test kit has a number of advantages:

- real-time progress monitoring while crews and equipment are on-site;
- clear, accurate pass/fail determinations at meaningful threshold values;
- meets site-specific calibration needs without a special kit;
- reduces wastes and costs.

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## Procedure

### *Materials*

The EnviroLogix PCB in Soil Tube Kit contains the following items:

- 40 antibody-coated test tubes
- 1 vial negative control
- 1 vial 1 ppm calibrator
- 1 vial 10 ppm calibrator
- 1 bottle of PCB-enzyme (horseradish peroxidase) conjugate
- 1 bottle of substrate
- 1 bottle of stop solution

The following items will need to be provided:

- EnviroLogix Soil Extraction Kit
- methanol (10 mL per sample)
- repeater pipettes
- (3) 12.5 mL combos-syringes
- positive displacement pipette
- marking pen
- timer
- distilled water
- portable photometer (Ariel Differential Photometer or equivalent)
- test tube rack

### *Extraction*

Five grams of soil are weighed into a soil extraction bottle containing two ball bearings. (The ball bearings are used to agitate the soil and may not be necessary for dry, sandy soils.) Then 10 mL of methanol are added to the bottle, and the bottle is capped tightly and shaken vigorously by hand for 2 min. After the contents are allowed to settle for 1 min, the extract is poured into the base of the Uniprep™ and the filter plunger is slowly pushed into the base until it stops at the bottom. To evaluate the samples relative to the 1 and 10 ppm calibrators, the filtered extract is poured into labeled 4-mL glass amber vials and capped tightly. To evaluate a 50 ppm threshold value, 800 µL of methanol are added to a 4-mL amber glass vial and then 200 µL of the sample extract are added. This is a 1:5 dilution.

### *Assay*

All reagents should be at room temperature before assay begins. The number of antibody-coated test tubes needed (up to 20) are removed from the kit and placed in the test tube rack; the tester should label one each for the negative control, for the two calibrators, and for each of the samples. After dispensing 500 µL of conjugate into each tube, dispensing down the side of the tubes with the syringe tip at an angle to prevent splashback, the tester adds 50 µL of sample to the tube(s) labeled for samples and 50 µL of negative control and calibrators to the appropriate tubes. The contents of the tubes are thoroughly mixed by moving the rack in a rapid circular motion for 20 to 30 seconds. After the tubes have been allowed to incubate for 10 min, the tube contents are

emptied into a suitable container. The tubes are then filled with distilled water, emptied, and shaken to remove any remaining drops. This wash process is repeated three times, after which the tubes are inverted and tapped on paper towels to remove excess water. Next, 500  $\mu\text{L}$  of substrate is added to each tube and the contents mixed thoroughly by moving the rack in a rapid circular motion for 20 to 30 seconds. The tubes are left to incubate for 10 min. **If blue color does not develop in the negative control tube, the assay is invalid and should be repeated.** Now, 500  $\mu\text{L}$  of stop solution is added to each tube. The tubes are read within 30 min of addition of the stop solution.

**Interpreting Results**

An Ariel Differential Photometer (or an equivalent) is used to measure the optical density of each tube's contents. The wavelength on the photometer should be set to 450 nanometers (nm). If the photometer has dual wavelength capability, 600, 630, or 650 nm should be used as the reference wavelength. If the photometer does not auto-zero on air, the instrument should be zeroed against 1 mL water in a blank tube. The optical density (OD) of each tube's contents is then measured and recorded. The information shown in Tables 2-1 and 2-2 is used to interpret the results.

The test kit results are reported as concentration ranges designated as intervals incorporating parenthesis/bracket notation. The parentheses indicate that the end-points of the concentration range are excluded, while brackets indicate that the end-points are included. As shown in Table 2-1, the interval [0, 1) indicates that the PCB concentration range is  $\geq 0$  and  $< 1$ . If the sample is  $> 10$  ppm, a 1:5 dilution of the sample can be prepared and assayed to determine if the concentration is  $> 50$  ppm. This diluted sample can be evaluated using the 10-ppm calibrator, as shown in Table 2-2.

**Table 2-1. Interpretation of photometer readings for undiluted samples**

Samples with OD values . . .	Contain . . .	And are reported as . . .
$>$ OD of 1-ppm calibrator	$< 1$ ppm PCB	[0, 1)
Between OD of 1-ppm and OD of 10-ppm calibrators	$1 \leq$ ppm PCB $< 10$	[1, 10)
$<$ OD of 10-ppm calibrator	$\geq 10$ ppm PCB	[10, $\infty$ )

**Table 2-2. Interpretation of photometer readings for diluted samples**

Diluted (1:5) samples with OD values . . .	Contain . . .	And are reported as . . .
$>$ OD of 10 ppm calibrator	$10 \leq$ ppm PCB $< 50$	[10, 50)
$<$ OD of 10 ppm calibrator	$\geq 50$ ppm PCB	[50, $\infty$ )

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**Precautions and Notes**

The following items should be noted about the test kit:

- All components should be stored at 4° to 8°C when not in use. Reagents must be allowed to come to ambient temperature before use. The components should not be used after the expiration date. It is important that the substrate solution not be exposed to direct sunlight during pipetting or while incubating in the test tubes.
- The stop solution is 1.0 N hydrochloric acid and should be handled with caution.
- It is recommended that positive results be confirmed by an alternate method (such as gas chromatography).

**Sensitivity and Cross-Reactivity**

The test kit can be calibrated with other Aroclors. Table 2-3 shows the degree of sensitivity with the other Aroclors. It should also be noted that at 1000 ppm, the following compounds had low cross-reactivity (i.e., did not result in a positive response) at the 1-ppm interpretation level: 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 3,4-dichlorophenol, 2,5-dichlorophenol, biphenyl, pentachlorophenol, and humic acid.

**Table 2-3. Sensitivity of PCB in Soil Tube Kit to various Aroclors**

Aroclor	Limit of Detection in Soil (ppm)
1242	1.7
1248	0.6
1254	0.3
1260	0.3

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## Section 3

### Site Description and Demonstration Design

#### Objective

This section describes the demonstration site, the experimental design for the verification test, and the sampling plan (sample types analyzed and the collection and preparation strategies). Included in this section are the results from the predemonstration study and a description of the deviations made from the original demonstration design.

#### Demonstration Site Description

##### *Site Name and Location*

The demonstration of PCB field analytical technologies was conducted at Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee. PCB-contaminated soils from three DOE sites (Oak Ridge; Paducah, Kentucky; and Piketon, Ohio) were used in this demonstration. The soil samples used in this study were brought to the demonstration testing location for evaluation of the field analytical technologies.

##### *Site History*

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

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

A population of more than 5000 drums containing PCB-contaminated soils was generated from 1986 to 1987 during the remediation of the East Drainage Ditch at the Portsmouth Gaseous Diffusion Plant. The ditch was reported to have three primary sources of potential contamination: (1) treated effluent from a radioactive liquid treatment facility, (2) runoff from a biodegradation plot where waste oil and sludge were disposed of, and (3) storm sewer discharges. In addition, waste oil was reportedly used for weed control in the ditch. Aside from



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PCB contamination, no other major hazardous contaminants were detected in these soils. Therefore, no EPA hazardous waste codes are assigned to this waste.

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

#### *Site Characteristics*

PCB-contaminated environmental soil samples from Oak Ridge, Portsmouth, and Paducah were collected from waste containers at storage repositories at ETPP and Paducah. Many of the soils contained interfering compounds such as oils, fuels, and other chlorinated compounds (e.g., trichloroethylene). Specific sample descriptions of the environmental soils used in this demonstration are given in Appendix A. In addition, each sample was characterized in terms of its soil composition, pH, and total organic carbon content. Those results are summarized in Appendix B.

Field demonstration activities occurred at two sites at ORNL: a natural outdoor environment (the outdoor site) and inside a controlled environmental atmosphere chamber (the chamber site). Figure 3-1 shows a schematic map of a section of ORNL indicating the demonstration area where the outdoor field activities occurred. Generally, the average September temperature for eastern Tennessee is 71 °F. Average temperatures during the testing periods ranged from 74 to 82 °F, as shown in Appendix C. Studies were also conducted inside a controlled environmental atmosphere chamber, hereafter referred to as the “chamber,” located in Building 5507 at ORNL. Demonstration studies inside the chamber were used to evaluate performance under environmental conditions that were markedly different from the ambient outdoor conditions at the time of the test. Average temperatures in the chamber during the testing periods ranged from 56 to 57 °F. The controlled experimental atmosphere facility consists of a room-size walk-in chamber 10 ft wide and 12 ft long with air processing equipment to control temperature and humidity. The chamber is equipped with an environmental control system, including reverse-osmosis water purification that supplies the chamber humidity control system. High-efficiency particulate air (HEPA) and activated charcoal filters are installed for recirculation and building exhaust filtration.

#### **Experimental Design**

The analytical challenge with PCB analysis is to quantify a complex mixture that may or may not resemble the original commercial product (i.e., Aroclor) because of environmental aging, and to report the result as a single number [1]. The primary objective of the verification test was to compare the performance of the field technology with laboratory-based measurements. Often, verification tests involve a direct one-to-one comparison of results from field-acquired samples. However, because sample heterogeneity can preclude replicate field or laboratory comparison, accuracy and precision data must often be derived from the analysis of QC and performance evaluation (PE) samples. In this study, replicates of all three sample types (QC, PE, and environmental soil) were analyzed. The ability to use environmental soils in the verification test was made

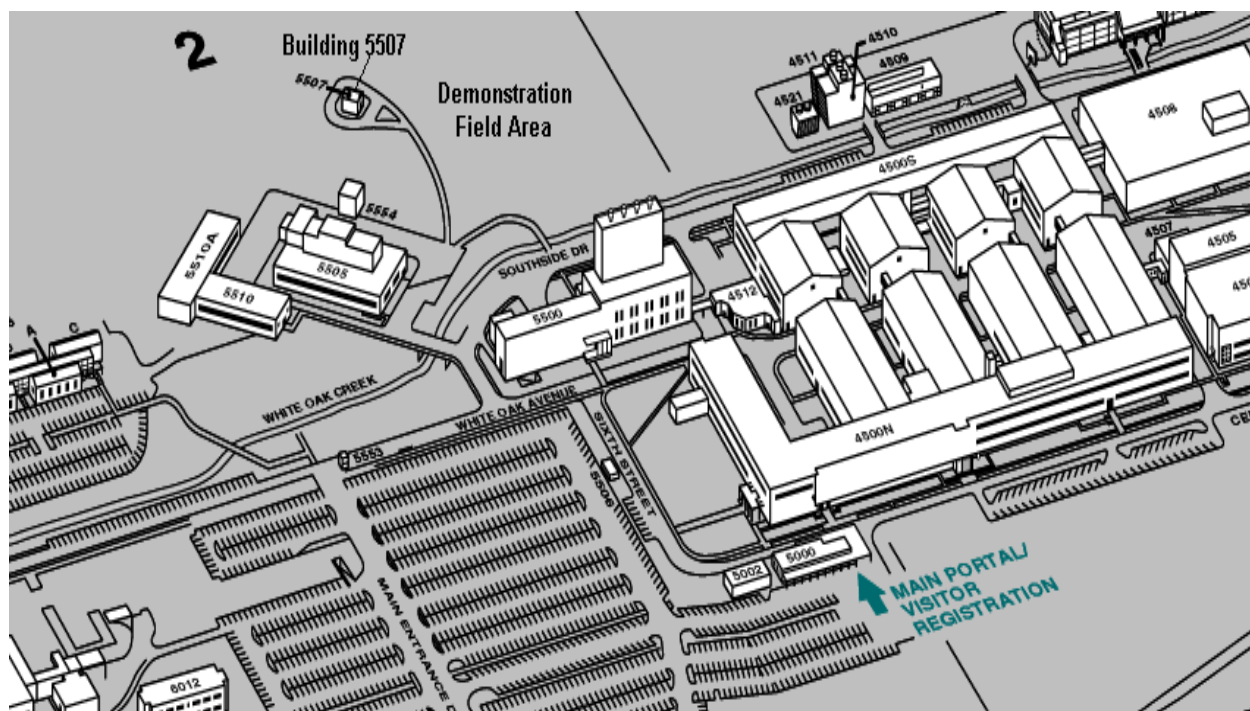


Figure 3-1. Schematic map of ORNL, indicating the demonstration area.

possible because the samples, collected from drums containing PCB-contaminated soils, could be thoroughly homogenized and characterized prior to the demonstration. This facet of the design, allowing additional precision data to be obtained on actual field-acquired samples, provided an added performance factor in the verification test.

Another objective of this demonstration was to evaluate the field technology's capability to support regulatory compliance decisions. For field methods to be used in these decisions, the technology must be capable of informing the user, with known precision and accuracy, that concentrations are greater than or less than an action level, such as 50 ppm for soil samples and 100  $\mu\text{g}/100\text{ cm}^2$  for wipe samples [2]. The samples selected for analysis in the demonstration study were chosen with this objective in mind.

The experimental design is summarized in Table 3-1. This design was approved by the developer prior to the start of the demonstration study. In total, the developer analyzed 208 soil samples, 104 each at both locations (outdoors and chamber). The 104 soil samples comprised 68 environmental samples (17 unique environmental samples prepared in quadruplicate) ranging in PCB concentration from 0.1 to 700 ppm and 36 PE soils (9 unique PE samples in quadruplicate) ranging in PCB concentration from 0 to 50 ppm. To determine the impact of different environmental conditions on the technology's performance, each batch of 104 samples contained five sets of quadruplicate soil samples from DOE's Paducah site. These were analyzed under both sets of environmental conditions (i.e., outdoor and chamber conditions). In addition, 12 extracts ranging in concentration from 0 to 100  $\mu\text{g}/\text{mL}$  were analyzed in each location (chamber and outdoors). All samples were analyzed without prior knowledge of sample type or concentration and were analyzed in a randomized order.

**Table 3-1. Summary of experimental design by sample type**

Concentration range	Sample ID <sup>a</sup>		Total # samples analyzed
	Outdoor site	Chamber site	
<i>PE materials</i>			
0	126	226	8
2.0 ppm	118	218	8
2.0 ppm	124	224	8
5.0 ppm	120	220	8
10.9 ppm	122	222	8
20.0 ppm	119	219	8
49.8 ppm	125	225	8
50.0 ppm	121	221	8
50.0 ppm	123	223	8
<i>Environmental soils</i>			
0.1–2.0 ppm	101, 107, 108, 109, 113, 114	201, 202, 206	36
2.1–20.0 ppm	102, 103, 104, 115	203, 207, 212, 213	32
20.1–50.0 ppm	111, 116	204, 208, 209, 214, 215	28
50.1–700.0 ppm	105, 106, 110, 112, 117	205, 210, 211, 216, 217	40
<i>Extracts</i>			
0	132	232	8
10 µg/mL	130	230	8
100 µg/mL	131	231	8
<b>Grand total</b>	<b>116</b>	<b>116</b>	<b>232<sup>b</sup></b>

<sup>a</sup> Each sample was analyzed in quadruplicate.

<sup>b</sup> All samples were analyzed in random order.

**Environmental Conditions during Demonstration**

As mentioned earlier, field activities were conducted both outdoors under natural environmental conditions and indoors in a controlled environmental atmosphere chamber to evaluate the effect of environmental conditions on technology performance. The weather outside during the September demonstration consisted of highs approaching 90°F and 90% relative humidity (RH). Daily average temperatures were around 78°F with 50%

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RH. While outside, the developer set up a canopy to provide shade and protection from late afternoon thundershowers. In the indoor chamber tests, conditions were set to 55°F and 50% RH. An independent check of the conditions inside the chamber indicated that the temperature ranged from 54 to 58°F, while relative humidities ranged from 44 to 55%. Appendix C contains a summary of the environmental conditions (temperature and relative humidity) during the demonstration.

### **Sample Descriptions**

PCBs ( $C_{12}H_{10-x}Cl_x$ ) are a class of compounds that are chlorine-substituted linked benzene rings. There are 209 possible PCB compounds (also known as congeners). PCBs were commercially produced as complex mixtures beginning in 1929 for use in transformers, capacitors, paints, pesticides, and inks [1]. Monsanto Corporation marketed products that were mixtures of 20 to 60 PCB congeners under the trade name Aroclor. Aroclor mixtures are identified by a number (e.g., Aroclor 1260) that represents the mixture's chlorine composition as a percentage (e.g., 60%).

### ***Performance Evaluation Materials***

Samples of Tennessee reference soil [3] served as the blanks. Pre-prepared certified PE samples were obtained from Environmental Resource Associates (ERA) of Arvada, Colorado, and the Analytical Operations and Data Quality Center of EPA's Office of Solid Waste and Emergency Response. The soils purchased from ERA had been prepared using ERA's semivolatile blank soil matrix. This matrix was a topsoil that had been dried, sieved, and homogenized. Particle size was approximately 60 mesh. The soil was approximately 40% clay. The samples acquired from EPA's Analytical Operations and Data Quality Center had been prepared using contaminated soils from various sites around the country in the following manner: The original soils had been homogenized and diluted with a synthetic soil matrix (SSM). The SSM had a known matrix of 6% gravel, 31% sand, and 43% silt/clay; the remaining 20% was topsoil. The dilution of the original soils was performed by mixing known amounts of contaminated soil with the SSM in a blender for no less than 12 h. The samples were also spiked with target pesticides ( $\alpha$ ,  $\beta$ ,  $\Delta$ , and  $\Delta$ -BHC, methoxychlor, and endrin ketone) to introduce some compounds that were likely to be present in an actual environmental soil. The hydrocarbon background from the original sample and the spiked pesticides produced a challenging matrix. The PE soils required no additional preparation by ORNL.

### ***Environmental Soil Samples***

As noted in the site description, PCB-contaminated environmental soil samples from Oak Ridge, Portsmouth, and Paducah were used in this demonstration. The soils were contaminated with PCBs as the result of spills and industrial processing activities at the various DOE facilities. Originally, the contaminated soils were excavated from dikes, drainage ditches, catch basins, and organic waste storage areas. The excavated soils were then packaged into waste containers and stored at the repositories in ETTP and Paducah in anticipation of disposal by incineration. The environmental soil samples used in this study were collected from these waste containers. Many of the soils contained interfering compounds such as oils, fuels, and other chlorinated compounds, while some contained multiple Aroclors. For more information on sampling locations and sample characteristics (soil composition, pH, and total organic carbon content), refer to Appendices A and B, respectively.

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### ***Extract Samples***

Traditionally, the amount of PCBs on a contaminated surface is determined by wiping the surface with a cotton pad saturated with hexane. The pad is then taken to the laboratory, extracted with additional hexane, and analyzed by gas chromatography. Unlike soil samples, which can be more readily homogenized and divided, equivalent wipe samples (i.e., contaminated surfaces or post-wipe pads) were not easily obtainable. Therefore, interference-free methanolic solutions of PCBs were analyzed to simulate an extracted surface wipe pad. Extract sample analyses provided evaluation data that relied primarily on the technology's performance rather than on elements critical to the entire method (i.e., sample collection and preparation). A total of 12 methanolic solutions were analyzed per site; these consisted of four replicates each of a blank and two concentration levels (10 and 100 µg/mL).

### **Sampling Plan**

#### ***Sample Collection***

Environmental soil samples were collected from April 17 through May 7, 1997. Portsmouth and Oak Ridge Reservation soils were collected from either storage boxes or 55-gal drums stored at ETTP. Briefly, the following procedure was used to collect the soil samples. Approximately 30 lb of soil were collected from the top of the drum or B-25 box using a scoop and placed in a plastic bag. The soil was sifted to remove rocks and other large debris, then poured into a plastic-lined 5-gal container. All samples were subjected to radiological screening and were determined to be nonradioactive. Similarly, soil samples were collected from 55-gal drums stored at Paducah and shipped to ORNL in lined 5-gal containers.

#### ***Sample Preparation, Labeling, and Distribution***

Aliquots of several of the environmental soils were analyzed and determined to be heterogeneous in PCB concentration. Because this is unsatisfactory for accurately comparing the performance of the field technology with the laboratory-based method, the environmental soils had to be homogenized prior to sample distribution. Each Portsmouth and Oak Ridge environmental soil sample was homogenized by first placing approximately 1500 g of soil in a glass Pyrex dish. The dish was then placed in a large oven set at 35°C, with the exhaust and blower fans turned on to circulate the air. After drying overnight, the soil was pulverized using a conventional blender and sieved using a 9-mesh screen (2 mm particle size). Last, the soil was thoroughly mixed using a spatula. A comparison of dried and undried soils showed that a minimal amount of PCBs (< 20%) was lost as the result of sample drying, making this procedure suitable for use in the preparation of the soil samples. The Paducah samples, because of their sandy characteristics, required only the sieving and mixing preparation steps. Extract sample preparation involved making solutions of PCBs in methanol at two concentration levels (10 and 100 µg/mL). Multiple aliquots of each sample were analyzed using the analytical procedure described below to confirm the homogeneity of the samples with respect to PCB concentration.

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



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The environmental soils were characterized at ORNL prior to the demonstration study. The procedure used to confirm the homogeneity of the soil samples entailed the extraction of 3 to 5 g of soil in a mixture of solvents (1 mL water, 4 mL methanol, and 5 mL hexane). After the soil/solvent mixture was agitated by a mechanical shaker, the hexane layer was removed and an aliquot was diluted for analysis. The hexane extract was analyzed on a Hewlett Packard 6890 gas chromatograph equipped with an electron capture detector and autosampler. The method used was a slightly modified version of EPA's SW-846 dual-column Method 8081 [4].

After analysis confirming homogeneity, the samples were split into jars for distribution. Each 4-oz sample jar contained approximately 20 g of soil. Four replicate splits of each soil sample were prepared for each developer. The samples were randomized in two fashions. First, the order in which the filled jars were distributed was randomized so that the same developer did not always receive the first jar filled for a given sample set. Second, the order of analysis was randomized so that each developer analyzed the same set of samples, but in a different order. The extract samples were split into 10-mL aliquots and placed into 2-oz jars. The extracts were stored in the refrigerator (at  $\leq 4^{\circ}\text{C}$ ) until released to the developers. Each sample jar had three labels: (1) developer order number, (2) sample identifier number, and (3) a PCB warning label. The developer order number corresponded to the order in which the developer was required to analyze the samples (e.g., 1001 through 1116). The sample identifier number was in the format of "xxxxyz," where "xxx" was the three-digit sample ID (e.g., 101) listed in Table 3-1, "y" was the replicate (e.g., 1 to 4), and "zz" was the aliquot order of each replicate (e.g., 01 to 11). For example, sample identifier 101101 corresponded to sample ID "101" (an Oak Ridge soil from RFD 40022, drum 02), "1" corresponded to the first replicate from that sample, and "01" corresponded to the first jar filled in that series.

Once the samples were prepared, they were stored at a central sample distribution center. During the demonstration study, the developer was sent to the distribution center to pick up his samples. Samples were distributed sequentially in batches of 12 to ensure that samples were analyzed in the order specified. Completion of chain-of-custody forms and scanning of bar code labels documented sample transfer activities. Some of the developers received information regarding the samples prior to analysis. This was provided at the request of the developer to simulate the type of information that would be available during actual field testing. EnviroLogix elected not to receive sample information prior to analysis. The developer returned the unused portions of the samples with the analytical results to the distribution center when testing was completed. The sample bar codes were scanned upon return to document sample throughput time.

Three complete sets of extra samples, called archive samples, were available for distribution in case the integrity of a sample was compromised. No archive samples were utilized over the course of the EnviroLogix demonstration.

### **Predemonstration Study and Results**

Ideally, environmental soil samples are sent to the developers prior to the demonstration study to allow them the opportunity to analyze representative samples in advance of the verification test. This gives developers the opportunity to refine and calibrate their technologies and revise their operating procedures on the basis of the predemonstration study results. The predemonstration study results can also be used as an indication that the selected technologies are of the appropriate level of maturity to participate in the demonstration study.

According to ORNL regulations, however, one of two conditions must exist in order to ship environmental soils that were once classified as mixed hazardous waste: either the recipient—in this case, the developer’s facilities—must have proper Nuclear Regulatory Commission (NRC) licensing to receive and analyze radiological materials; or the soils must be certified as entirely free of radioactivity, beyond the no-rad certification issued from radiological screening tests based on ORNL standards. Because the developer did not have proper NRC licensing, and proving that the soils were entirely free of radioactivity was prohibitive, PE soils were used for the predemonstration study. The developer also analyzed a solvent extract.

The predemonstration samples were sent to the developer on August 17, 1998. Predemonstration results were received by August 19, 1998. Table 3-2 summarizes the test kit’s results for the predemonstration samples. Results indicated that EnviroLogix’s PCB in Soil Tube Assay was ready for field evaluation.

**Table 3-2. Summary of PCB in Soil Tube Assay predemonstration results**

Sample	Certified concentration	Aroclor	Acceptance range <sup>a</sup>	Total PCB concentration (ppm)	
				Result #1	Result #2
1	0	n/a	n/a	[0, 1) <sup>b</sup>	[0, 1)
2	5.0 ppm	1254	2.1–6.2 ppm	[1, 10)	[1, 10)
3	49.8 ppm	1254, 1260	23.0–60.8 ppm	[50, ∞)	[50, ∞)
4	20.0 ppm	1248	11.4–32.4 ppm	[10, 50)	[10, 50)
5	10 µg/mL	1254	n/a	[4, 20)	[4, 20)

<sup>a</sup> Acceptance ranges were provided by the supplier of the performance evaluation material.

<sup>b</sup> The notation [0, 1) indicates that the sample concentration was ≥0 and <1. See Sections 2 and 5 for more information on interval reporting.

**Deviations from the Demonstration Plan**

A few deviations from the demonstration plan occurred. In Appendix B of the technology demonstration plan [5], the reference laboratory’s procedure states that no more than 10 samples will be analyzed with each analytical batch (excluding blanks, standards, QC samples, and dilutions). The analytical batch is also stated as 10 samples in the Quality Assurance Project Plan of the demonstration plan. The reference laboratory actually analyzed 20 samples per analytical batch. Because a 20-sample batch is recommended in SW-846 Method 8081, this deviation was deemed acceptable. In addition, the parentheses and bracket notations in Tables 3-1 and 3-2 are slightly different than what was used in the demonstration. See Tables 2-1 and 2-2 of this report for the correction notation.

During the demonstration study, EnviroLogix noted the following deviations from the procedure described in the technology demonstration plan [5] for the PCB in Soil Tube Assay:

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- Instead of two, one or no ball bearings were used for extraction. The purpose of the ball bearings is to aid in the extraction by agitating the soil. Since the soils were sandy and dry, the ball bearings were not necessary.
  - Multiple filtrations (usually 12 tubes at a time) were performed by pushing the plungers down on the UniPrep tubes simultaneously using a small board. The procedure in the demonstration plan describes the filtration as being performed individually.
  - Three washes of the coated tubes were performed instead of four.





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## Section 4

### Reference Laboratory Analytical Results and Evaluation

#### Objective and Approach

This section presents the evaluation of the PCB data generated by the reference laboratory. Evaluation of the results from the analysis of PE, environmental soil, and extract samples was based on precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters [6]. This section describes how the analytical data generated by the reference laboratory were used to establish a baseline performance for PCB analysis.

EnviroLogix demonstrated the PCB in Soil Tube Assay kit using the same samples that were used in the July 1997 demonstration of six PCB technologies. Soil samples were available for the EnviroLogix demonstration because extra samples were prepared and stored since 1997. ORNL performed chemical analyses of representative samples to verify that significant amounts of PCBs had not been lost due to storage for one year. Duplicate analyses from each unique soil sample were performed. It was confirmed that no considerable losses in PCB concentration had occurred. Therefore, all soil samples were utilized in the EnviroLogix demonstration, and the reference laboratory data described in this section was used for comparison with the PCB in Soil Tube Assay results. Because the original extract samples were prepared in methanol, new extract samples were prepared by ORNL. Therefore, no reference laboratory results are presented for these samples. Instead of a reference laboratory result, the EnviroLogix's result was compared to the nominal concentration value only. Confirmational analyses at ORNL indicated that the extracts were prepared to the nominal concentrations.

#### Reference Laboratory Selection

The Oak Ridge Sample Management Office (SMO) has been tasked by DOE Oak Ridge Operations (DOE-ORO) with maintaining a list of qualified laboratories to provide analytical services. The technology demonstration plan [5] contains the SMO's standard operating procedures (SOPs) for identifying, qualifying, and selecting analytical laboratories. Laboratories are qualified as acceptable analytical service providers for the SMO by meeting specific requirements. These requirements include providing pertinent documentation (such as QA and chemical hygiene plans), acceptance of the documents by the SMO, and satisfactory performance on an on-site prequalification audit of laboratory operations. All laboratory qualifications are approved by a laboratory selection board, composed of the SMO operations manager and appointees from all prime contractors that conduct business with the SMO.

All of the qualified laboratories were invited to bid on the demonstration study sample analysis. The lowest-cost bidder was LAS Laboratories, in Las Vegas, Nevada. A readiness review conducted by ORNL and the SMO confirmed the selection of LAS as the reference laboratory. Acceptance of the reference laboratory was finalized by satisfactory performance in the predemonstration study. The SMO contracted LAS to provide full data packages for the demonstration study sample analyses within 30 days of sample shipment.

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

### **Reference Laboratory Method**

The reference laboratory's analytical method, also presented in the technology demonstration plan [5], followed the guidelines established in EPA SW-846 Method 8081 [4]. According to LAS's SOP, PCBs were extracted from 30-g samples of soil by sonication in hexane. Each extract was then concentrated to a final volume that was further subjected to a sulfuric acid cleanup to remove potential interferences. The analytes were identified and quantified using a gas chromatograph equipped with dual electron-capture detectors. Each extract was analyzed on two different chromatographic columns with slightly different separation characteristics (primary column: RTX-1701, 30 m × 0.53 mm ID × 0.5 μm; confirmatory column: RTX-5, 30 m × 0.53 mm ID × 0.5 μm). PCBs were identified when peak patterns from a sample extract matched the patterns of standards for both columns. PCBs were quantified based on the initial calibration of the primary column.

### ***Calibration***

Method 8081 states that, because Aroclors 1016 and 1260 include many of the peaks represented in the other five Aroclor mixtures, it is only necessary to analyze two multilevel standards for these Aroclors to demonstrate the linearity of the detector response for PCBs. However, per LAS SOPs, five-point (0.1 to 4 ppm) initial calibration curves were generated for Aroclors 1016, 1248, 1254, and 1260 and the surrogate compounds [decachlorobiphenyl (DCB) and tetrachloro-*m*-xylene (TCMX)]. Single mid-level standards were analyzed for the other Aroclors (1221, 1232, and 1242) to aid in pattern recognition. All of the multi-point calibration data, fitted to quadratic models, met the QC requirement of having a coefficient of determination ( $R^2$ ) of 0.99 or better over the calibration range specified. The detection limits for soil samples were 0.033 ppm (μg/g) for all Aroclors except Aroclor 1221, which was 0.067 ppm. For extract samples, the detection limits were 0.010 ppm (μg/mL) for all Aroclors except Aroclor 1221, which was 0.020 ppm. Reporting detection limits were calculated based on the above detection limits, the actual sample weight, and the dilution factor.

### ***Sample Quantification***

For sample quantification, Aroclors were identified by comparing the samples' peak patterns and retention times with those of the respective standards. Peak height ratios, peak shapes, sample weathering, and general similarity in detector response were also considered in the identification. Aroclor quantifications were performed by selecting three to five representative peaks, confirming that the peaks were within the established retention time windows, integrating the selected peaks, quantifying the peaks based on the calibrations, and averaging the results to obtain a single concentration value for the multicomponent Aroclor. If mixtures of Aroclors were suspected to be present, the sample was typically quantified in terms of the most representative Aroclor pattern. If the identification of multiple Aroclors was definitive, total PCBs in the sample were

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calculated by summing the concentrations of both Aroclors. Aroclor concentrations were quantified within the concentration range of the calibration curve. If PCBs were detected and the concentrations were outside of the calibration range, the sample was diluted and reanalyzed until the concentration was within the calibration range. If no PCBs were detected, the result was reported as a non-detect (i.e., “ $\leq$  reporting detection limit”).

### ***Sample Receipt, Handling, and Holding Times***

The reference laboratory was scheduled to analyze a total of 256 PCB samples (208 soil samples, 24 iso-octane extract samples, and 24 methanol extract samples). Of these same samples, the developer was scheduled to analyze a total of 232 PCB samples (208 soil samples and 24 extract samples in solvent of choice). The samples were shipped to LAS at the start of the technology demonstration activities (July 22). Shipment was coordinated through the SMO. Completion of chain-of-custody forms documented sample transfer. The samples were shipped on ice in coolers to maintain  $<6^{\circ}\text{C}$  temperatures during shipment. Samples were shipped with custody seals to ensure sample integrity and to prevent tampering during transport.

Upon receipt of the samples, the reference laboratory checked the receipt temperature and conditions of the sample containers, assigned each sample a unique number, and logged each into its laboratory tracking system. All samples were received at the proper temperature and in good condition. Demonstration samples were divided into 11 analytical batches (with no more than 20 samples per batch). The samples were analyzed in an order specified by ORNL to ensure that the analysis of sample types was randomized. Analyses of QC samples, supplied by the reference laboratory to indicate method performance, were performed with each analytical batch of soils.

Prior to analysis, samples were stored in refrigerators kept at 4 to  $6^{\circ}\text{C}$  to maintain analyte integrity. The reference laboratory was required to analyze the extract samples and to extract the soil samples within 14 days of shipment from ORNL. Once the soils were extracted, the reference laboratory had an additional 40 days to analyze the soil extracts. Maximum holding times were not exceeded for any of the demonstration samples. The final reference laboratory data package for all samples was received at ORNL in 72 days, on October 1, 1997. The contractual obligation was 30 days.

The remainder of this section is devoted to summarizing the data generated by the reference laboratory and to assessing the analytical performance.

### **Quality Control Results**

#### ***Objective***

The purpose of this section is to provide an assessment of the data generated by the reference laboratory's QC procedures. The QC samples included continuing calibration verification standards (CCVs), instrument blanks, method blanks, surrogate spikes, laboratory control samples (LCSs), and matrix spike (MS) /duplicate matrix spike (MSD) samples. Each control type is described in more detail in the following text and in the technology demonstration plan [5]. Because extraction of these liquid samples was not required, calibration check standards and instrument blanks were the only control samples implemented for the extract samples. The reference laboratory's implementation of QC procedures was consistent with SW-846 guidance.

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### ***Continuing Calibration Verification Standard Results***

A CCV is a single calibration standard of known concentration, usually at the midpoint of the calibration range. This standard is evaluated as an unknown and is quantified against the initial calibration. The calculated concentration is then compared with the nominal concentration of the standard to determine whether the initial calibration is still valid. CCVs were analyzed with every 10 samples or at least every 12. The requirement for acceptance was a percentage difference of less than 15% for the CCV relative to the initial calibration. This QC requirement was met for all Aroclors and surrogates, except for one standard that had a 16% difference for DCB. These results indicated that the reference laboratory maintained instrument calibrations during the course of sample analysis.

### ***Instrument and Method Blank Results***

Instrument blanks (hexane) were analyzed prior to each CCV. The QC requirement was that instrument blanks must contain less than the reporting detection limit for any analyte. All instrument blanks were acceptable.

A method blank is an analyte-free soil matrix sample that is taken through the extraction process to verify that there are no laboratory sources of contamination. One method blank was analyzed for each analytical batch. The QC requirement was that method blanks must contain less than the reporting detection limit for any Aroclor. No PCBs were detected in any of the eleven method blanks that were analyzed. These results demonstrated that the reference laboratory was capable of maintaining sample integrity, and that it did not introduce PCB contamination to the samples during preparation.

### ***Surrogate Spike Results***

A surrogate is a compound that is chemically similar to the analyte group but is not expected to be present in the environmental sample. A surrogate is added to test the extraction and analysis methods to verify the ability to isolate, identify, and quantify a compound similar to the analyte(s) of interest without interfering with the determination. Two different surrogate compounds, DCB and TCMX, were used to bracket the retention time window anticipated in the Aroclor chromatograms. All soil samples, including QC samples, were spiked with surrogates at 0.030 ppm prior to extraction. Surrogate recoveries were deemed to be within QC requirements if the measured concentration fell within the QC acceptance limits that were established by past method performance. (For LAS this was 39 to 117% for DCB, and 66 to 128% for TCMX). The results were calculated using the following equation:

$$\text{percent recovery} = \frac{\text{measured amount}}{\text{actual amount}} \times 100\% \quad (4-1)$$

In all undiluted samples, both of the surrogates had percentage recoveries that were inside the acceptance limits. Surrogate recoveries in diluted samples were uninformative because the spike concentration (0.030 ppm, as specified by the method) was diluted below the instrument detection limits. The surrogate recovery results for undiluted samples indicated that there were no unusual matrix interferences or batch-processing errors for these samples.

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### ***Laboratory Control Sample Results***

An LCS is an aliquot of a clean soil that is spiked with known quantities of target analytes. The LCS is spiked with the same analytes and at the same concentrations as the MS. (MSs are described in the next section.) If the results of the MS analyses are questionable (i.e., indicating a potential matrix effect), the LCS results are used to verify that the laboratory can perform the analysis in a clean, representative matrix.

Aroclors 1016 and 1260 were spiked into the clean soil matrix at approximately 0.300 ppm, according to the reference laboratory's SOP. The QC requirements (defined as percent recovery) for the LCS analyses were performance-based acceptance limits that ranged from 50 to 158%. In all but 1 of the 11 LCSs analyzed, both Aroclor percent recoveries fell within the acceptance limits. Satisfactory recoveries for LCS verified that the reference laboratory performed the analyses properly in a clean matrix.

### ***Matrix Spike Results***

In contrast to an LCS, a MS sample is an actual environmental soil sample into which target analytes are spiked at known concentrations. MS samples are used to assess the efficiency of the extraction and analytical methods for real samples. This is accomplished by determining the amount of spiked analyte that is quantitatively recovered from the environmental soil. An MSD sample is spiked and analyzed to provide a measure of method precision. Ideally, to evaluate the MS/MSD results, the environmental soil is analyzed unspiked so that the background concentrations of the analyte in the sample are considered in the recovery calculation.

For the demonstration study samples, one MS and MSD pair was analyzed with each analytical batch. The MS samples were spiked under the same conditions and QC requirements as the LCS (50 to 158% acceptance limits), so that MS/MSD and LCS results could be readily compared. The QC requirement for MS and MSD samples was a relative percentage difference (RPD) of less than 30% between the MS/MSD pair. RPD is defined as

$$RPD = \frac{|MS \text{ recovery} - MSD \text{ recovery}|}{\text{average recovery}} \times 100\% \quad (4-2)$$

A total of 11 MS/MSD pairs were analyzed. Because the MS/MSD spiking technique was not always properly applied (e.g., a sample which contained 100 ppm of Aroclor 1254 was spiked ineffectively with 0.300 ppm of Aroclor 1260), many of the MS/MSD results were uninformative. For the samples that were spiked appropriately, all MS/MSD QC criteria were met.

### ***Conclusions of the Quality Control Results***

The reference laboratory results met performance acceptance requirements for all of the samples where proper QC procedures were implemented. Acceptable performance on QC samples indicated that the reference laboratory was capable of performing analyses properly.

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## Data Review and Validation

### *Objective*

The purpose of validating the reference laboratory data was to ensure usability for the purposes of comparison with the demonstration technologies. The data generated by the reference laboratory were used as a baseline to assess the performance of the technologies for PCB analysis. The reference laboratory data were independently validated by ORNL and SMO personnel, who conducted a thorough quality check and reviewed all sample data for technical completeness and correctness.

### *Corrected Results*

Approximately 8% of the results provided by the reference laboratory (20 of 256) were found to have correctable errors. So as not to bias the assessment of the technology's performance, errors in the reference laboratory data were corrected. These changes were made conservatively, based on the guidelines provided in the SW-846 Method 8081 for interpreting and calculating Aroclor results. The errors (see Appendix D, Table D-3) were categorized as transcription errors, calculation errors, and interpretation errors. The corrections listed in Table D-3 were made in the final data set that was used for comparison with the demonstration technologies.

### *Suspect Results*

Normally, one would not know if a single sample result was "suspect" unless (1) the sample was a PE sample, where the concentration is known, or (2) a result was reported and flagged as suspect for some obvious reason (e.g., no quantitative result was determined). The experimental design implemented in this demonstration study provided an additional indication of the abnormality of data through the inspection of the replicate results from a homogenous soil sample set (i.e., four replicates were analyzed for each sample ID).

Data sets were considered suspect if the standard deviation (SD) of the four replicates was greater than 30 ppm and the percent relative standard deviation (RSD) was greater than 50%. Five data sets (sample IDs 106, 205, 216, 217, 225) contained measurements that were considered suspect using this criteria, and the suspect data are summarized in Table 4-1. A number of procedural errors may have caused the suspect measurements (e.g., spiking heterogeneity, extraction efficiencies, dilution, etc.). In the following subsections for precision and accuracy, the data were evaluated with and without these suspect values to represent the best- and worst-case scenarios.



**Table 4-1. Suspect measurements within the reference laboratory data**

Criteria	Sample ID	PCB concentration (ppm)		Data usability
		Replicate results (ppm)	Suspect result(s) (ppm)	
SD > 30 ppm RSD > 50%	106	255.9, 269.9, 317.6	649.6	Performed data analysis with and without this value
	205	457.0, 483.3, 538.7	3305.0	
	216	47.0, 54.3, 64.0	151.6	
	217	542.8, 549.8, 886.7	1913.3	
	225	32.1, 36.5, 56.4	146.0	
Qualitative result	110	≤ reporting detection limits	≤66, ≤98, ≤99, ≤490	Used as special case for comparison with developer results
	112		≤66, ≤130, ≤200, ≤200	

Samples that did not fall into the above criteria, but were also considered suspect, were non-blank samples that could not be quantified and were reported as “≤ the reporting detection limit.” This was the case for environmental soil sample IDs 110 and 112. It is believed that the reference laboratory had trouble quantifying these soil samples because of the abundance of chemical interferences. These samples were diluted by orders of magnitude to reduce interferences, thereby diluting the PCB concentrations to levels that were lower than the instrument detection limits. With each dilution, the reporting detection limits values were adjusted for sample weight and dilution, which accounts for the higher reporting detection limits (up to 490 ppm). It is believed that these samples should have been subjected to additional pre-analytical cleanup to remove these interferences before quantification was attempted. Sample IDs 110 and 112 were collected from the same cleanup site (see Appendix B), so it is not surprising that similar difficulties were encountered with both sample sets. Because the results for sample IDs 110 and 112 were not quantitative, these data were compared with the technology data only on a special case basis.

**Data Assessment**

*Objective*

The purpose of this section is to provide an evaluation of the performance of the reference laboratory results through statistical analysis of the data. The reference laboratory analyzed 72 PE, 136 environmental soil, and 48 extract samples. All reference laboratory analyses were performed under the same environmental conditions. Therefore, site differentiation was not a factor in data assessment for the reference laboratory. For comparison with the technology data, however, the reference laboratory data are delineated into “outdoor site” and “chamber site” in the following subsections. For consistency with the technology review, results from both sites were also combined to determine the reference laboratory’s overall performance for precision and accuracy. This performance assessment was based on the raw data compiled in Appendix D. All statistical tests were performed at a 5% significance level.



**Precision**

The term “precision” describes the reproducibility of measurements under a given set of conditions. The SD of four replicate PCB measurements was used to quantify the precision for each sample ID. SD is an absolute measurement of precision, regardless of the PCB concentration. To express the reproducibility relative to the average PCB concentration, RSD is used to quantify precision, according to the following equation:

$$RSD = \frac{\text{Standard Deviation}}{\text{Average Concentration}} \times 100\% \quad (4-3)$$

**Performance Evaluation Samples**

The PE samples were homogenous soils containing certified concentrations of PCBs. Results for these samples represent the best estimate of precision for soil samples analyzed in the demonstration study. Table 4-2 summarizes the precision of the reference laboratory for the analysis of PE samples. One suspect measurement (sample ID 225, 146.0 ppm) was reported for the PE soil samples. The RSDs for the combined data ranged from 9 to 33% when the suspect measurement was excluded, and from 9 to 79% including the suspect measurement. The overall precision, determined by the mean RSD for all PE samples, was 21% for the worst case (including the suspect result) and 18% for the best case (excluding the suspect result).

**Table 4-2. Precision of the reference laboratory for PE soil samples**

Outdoor site				Chamber site				Combined sites		
Sample ID	Average concentration (ppm)	SD (ppm)	RSD (%)	Sample ID	Average concentration (ppm)	SD (ppm)	RSD (%)	Average concentration (ppm)	SD (ppm)	RSD (%)
126 <sup>a</sup>	0	n/a	n/a	226	0	n/a	n/a	0	n/a	n/a
118	1.6	0.6	39	218	2.6	0.2	6	2.1	0.7	33
124	1.7	0.2	13	224	1.7	0.5	29	1.7	0.4	21
120	5.0	1.0	20	220	5.8	1.8	31	5.4	1.4	26
122	11.1	0.9	8	222	12.8	0.3	3	11.9	1.1	9
119	20.1	3.4	17	219	23.3	6.1	26	21.7	4.9	23
125	37.9	6.9	18	225	41.7 <sup>b</sup>	12.9 <sup>b</sup>	31 <sup>b</sup>	39.5 <sup>c</sup>	9.2 <sup>c</sup>	23 <sup>c</sup>
121	54.6	3.4	6	221	44.9	11.3	25	49.8	9.3	19
123	60.1	4.6	8	223	55.8	7.7	14	58.0	6.3	11

<sup>a</sup> All PCB concentrations were reported as non-detects.

<sup>b</sup> Results excluding the suspect value (results including the suspect value: mean = 67.8 ppm, SD = 53.2 ppm, RSD = 79%).

<sup>c</sup> Results excluding the suspect value (results including the suspect value: mean = 52.8 ppm, SD = 38.6 ppm, RSD = 73%).

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### **Environmental Soil Samples**

The precision of the reference laboratory for the analysis of environmental soil samples is reported in Table 4-3. In this table, results including suspect measurements are presented in parentheses. Average concentrations were reported by the reference laboratory as ranging from 0.5 to 1196 ppm with RSDs that ranged from 7 to 118% when the suspect results were included. Excluding the suspect results, the highest average concentration decreased to 660 ppm, and the largest RSD decreased to 71%. Because the majority of the samples fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low concentrations (< 125 ppm) and high concentrations (> 125 ppm). For the low concentrations, the average RSD was 23% excluding the suspect value and 26% including the suspect value. These average RSDs were only slightly larger than the RSDs for the PE soil samples of comparable concentration (18% for best case and 21% for worst case). Five soil sample sets (sample IDs: 106, 117, 205, 211 and 217) were in the high-concentration category. The average precision for high concentrations was 56% for the worst case and 19% for the best case. The precision estimates for the low and high concentration ranges were comparable when the suspect values were excluded. This indicated that the reference laboratory's precision for the environmental soils was consistent (approximately 21% RSD) and was comparable to the PE soil samples when the suspect values were excluded.

The Paducah soils (indicated as bold sample IDs in Table 4-3) were analyzed by the technologies under both outdoor and chamber conditions to provide a measure of the effect that two different environmental conditions had on the technology's performance. Although this was not an issue for the reference laboratory (because all the samples were analyzed under laboratory conditions), the reference laboratory's results were delineated into the different site categories for comparison with the technologies. Sample IDs 113 and 201, 114 and 202, 115 and 203, 116 and 204, and 117 and 205 each represent a set of eight replicate samples of the same Paducah soil. The RSDs for four of the five Paducah pairs (excluding the suspect value for sample ID 205) ranged from 11 to 17%. The result from one pair (sample IDs 113 and 201) had an RSD of 42%, but the reported average concentration was near the reporting limits.

### **Extract Samples**

The extract samples, which were used to simulate surface wipe samples, were the simplest of all the demonstration samples to analyze because they required no extraction and were interference-free. Three types of extract samples were analyzed: solvent blanks, spikes of Aroclor 1248 at 10 µg/mL, and spikes of Aroclor 1016 at 100 µg/mL. The reference laboratory did not analyze the extract samples for the EnviroLogix demonstration.

### **Accuracy**

Accuracy represents the closeness of the reference laboratory's measured PCB concentrations to the accepted values. Accuracy was examined by comparing the measured PCB concentrations in the PE soils with the certified PE values and known spiked extract concentrations. Percent recovery was used to quantify the accuracy of the results. The optimum percent recovery value is 100%. Percent recovery values greater than 100% indicate results that are biased high, and values less than 100% indicate results that are biased low.

Table 4-3. Precision of the reference laboratory for environmental soil samples

Outdoor site				Chamber site			
Sample ID	Average concentration (ppm)	Standard deviation (ppm)	RSD (%)	Sample ID	Average concentration (ppm)	Standard deviation (ppm)	RSD (%)
101	0.5	0.1	16	206	1.9	0.9	49
102	2.0	0.3	16	207	18.8	3.5	19
103	2.3	0.6	27	208	30.5	7.9	26
104	9.4	4.0	43	209	40.2	28.5	71
105	59.4	16.5	28	210	88.6	25.6	29
106	281.0 (373.2) <sup>a</sup>	32.4 (186.2)	12 (50)	211	404.5	121.8	30
107	1.3	0.3	20	212	3.2	1.6	50
108	1.8	0.1	8	213	8.1	1.6	20
109	2.0	0.4	20	214	25.2	3.7	15
110	n/a <sup>b</sup>	n/a	n/a	215	26.7	3.2	12
111	38.7	4.3	11	216	55.1 (79.2)	8.5 (48.7)	15 (62)
112	n/a	n/a	n/a	217	659.8 (973.2)	196.6 (647.0)	30 (66)
<b>113<sup>c</sup></b>	1.1	0.6	55	<b>201</b>	0.9	0.2	24
<b>114</b>	1.3	0.3	20	<b>202</b>	1.4	0.2	12
<b>115</b>	14.8	1.8	12	<b>203</b>	13.9	1.7	12
<b>116</b>	41.3	5.9	14	<b>204</b>	44.3	2.9	7
<b>117</b>	383.9	55.2	14	<b>205</b>	493.0 (1196.0)	41.7 (1406.4)	8 (118)

<sup>a</sup> Data in parentheses include suspect values.

<sup>b</sup> N/a indicates that qualitative results only were reported for this sample.

<sup>c</sup> Bold sample IDs were matching Paducah sample pairs (i.e., 113/201, 114/202, 115/203, 116/204, 117/205).

The reference laboratory's performance for the PE samples is summarized in Table 4-4. Included in this table are the performance acceptance ranges and the certified PCB concentration values. The acceptance ranges, based on the analytical verification data, are guidelines established by the provider of the PE materials to gauge acceptable analytical results. As shown in Table 4-4, all of the average concentrations were within the acceptance ranges, with the exception of sample ID 218. The average result of sample ID 225 was outside of

the acceptance range only when the suspect result was included. All of the replicate measurements in sample ID 225 were biased slightly high. Average percent recoveries for the PE samples (excluding suspect values) ranged from 76 to 130%. Overall accuracy was estimated as the average recovery for all PE samples. The overall percent recovery was 105% as a worst case when the suspect value was included. Excluding the suspect value as a best case slightly lowered the overall percent recovery to 101%. A regression analysis [7] indicated that the reference laboratory's results overall were unbiased estimates of the PE sample concentrations.

**Table 4-4. Accuracy of the reference laboratory for PE soil samples**

Certified concentration (ppm) (acceptance range, ppm)	Outdoor site			Chamber site			Combined sites	
	Sample ID	Average conc (ppm)	Recovery (%)	Sample ID	Average conc (ppm)	Recovery (%)	Average conc (ppm)	Recovery (%)
0 <sup>a</sup> (n/a)	126	0	n/a	226	0	n/a	0	n/a
2.0 (0.7-2.2)	118	1.6	79	218	2.6	130	2.1	105
2.0 (0.9-2.5)	124	1.7	85	224	1.7	85	1.7	85
5.0 (2.1-6.2)	120	5.0	99	220	5.8	117	5.4	108
10.9 (4.0-12.8)	122	11.1	102	222	12.8	117	11.9	109
20.0 (11.4-32.4)	119	20.1	100	219	23.3	116	21.7	109
49.8 (23.0-60.8)	125	37.9	76	225	41.7 <sup>b</sup>	84 <sup>b</sup>	39.5 <sup>c</sup>	79 <sup>c</sup>
50.0 (19.7-63.0)	121	54.6	109	221	44.9	90	49.8	100
50.0 (11.9-75.9)	123	60.1	120	223	55.8	112	58.0	116

<sup>a</sup> All PCB concentrations reported as non-detects by the laboratory.

<sup>b</sup> Results excluding the suspect value (results including the suspect value: average = 67.8 ppm and recovery = 136%).

<sup>c</sup> Results excluding the suspect value (results including the suspect value: average = 52.8 ppm and recovery = 106%).

**Representativeness**

Representativeness expresses the degree to which sample data accurately and precisely represent the capability of the method. Representativeness of the method was assessed based on the data generated for clean-QC samples (i.e., method blanks and laboratory control samples) and PE samples. Based on the data assessment discussed in detail in various parts of this section, it was determined that the representativeness of the reference laboratory data was acceptable. In addition, acceptable performance on laboratory audits substantiated that

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the data set was representative of the capabilities of the method. In all cases, the performance of the reference laboratory met all requirements for both audits and QC analyses.

### ***Completeness***

Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result was not rejected). Usable results were obtained for 248 of the 256 samples submitted for analysis by the reference laboratory. Eight results (for sample IDs 110 and 112) were deemed incomplete and therefore not valid because the measurements were not quantitative. To calculate completeness, the total number of complete results were divided by the total number of samples submitted for analysis, and then multiplied by 100 to express as a percentage. The completeness of the reference laboratory was 97%, where a completeness of 95% or better is typically considered acceptable.

### ***Comparability***

Comparability refers to the confidence with which one data set can be compared with another. The demonstration study was designed to have a one-to-one, sample-by-sample comparison of the PCB results obtained by the reference laboratory and the PCB results obtained by the technology being evaluated. Based on thorough examination of the data and acceptable results on the PE samples, it was concluded that the reference laboratory's SOPs for extraction and analysis, and the data generated using these procedures, were of acceptable quality for comparison with the field technology results. Additional information on comparability was available because the experimental design incorporated randomized analysis of blind, replicate samples. Evaluation of the replicate data implicated some of the individual data points as suspect (see Table D-2). The reference laboratory's suspect data were compared with the technology data on a special-case basis, and exceptions were noted.

### **Summary of Observations**

Table 4-5 provides a summary of the performance of the reference laboratory for the analysis of all sample types used in the technology demonstration study. As shown in the table, the precision of the PE soils was comparable to that of the environmental soils. A weighted average, based on the number of samples, gave a best-case precision of 21% and a worst-case precision of 28% for all the soil data (PE and environmental). Evaluation of overall accuracy was based on samples with certified concentrations. The overall accuracy, based on percent recovery, for the PE samples (which ranged from 0 to 50 ppm) was 105% for the worst case (which included the suspect value) and 101% for the best case (which excluded the suspect value). These results indicated that the reference laboratory results were unbiased estimates of the certified PE concentrations. The reference laboratory correctly reported all blank samples as non-detects but had difficulty with two soil samples (IDs 110 and 112) that contained chemical interferences. Overall, it was concluded that the reference laboratory results were acceptable for comparison with the developer's technology.

**Table 4-5. Summary of the reference laboratory performance**

Sample matrix	Sample type	Number of samples	Precision (average % RSD)	Accuracy (average % recovery)
Blank	Soil	8	n/a <sup>a</sup>	All samples reported as non-detects
Environmental soil with interferences	Sample ID 110	4	n/a <sup>a</sup>	All samples reported as non-detects
	Sample ID 112	4		
Soil — Best case (excluding suspect data)	PE	63	18	101
	Environmental < 125 ppm > 125 ppm	107	23	n/a <sup>b</sup>
		17	19	n/a <sup>b</sup>
	Overall	187	21	101
Soil — Worst case (including suspect data)	PE	64	21	105
	Environmental < 125 ppm > 125 ppm	108	26	n/a <sup>b</sup>
		20	56	n/a <sup>b</sup>
	Overall	192	28	105

<sup>a</sup> Because the results were reported as non-detects, precision assessment is not applicable.

<sup>b</sup> Accuracy assessment calculated for samples of known concentration only.



## Section 5

### Technology Performance and Evaluation

#### Objective and Approach

This section presents the evaluation of the data generated by EnviroLogix's PCB in Soil Tube Assay. The technology's precision and accuracy are presented for the data generated in the demonstration study. In addition, an evaluation of comparability, through a one-to-one comparison with the reference laboratory data, is presented. An evaluation of other aspects of the technology (such as cost, sample throughput, hazardous waste generation, and logistical operation) is also presented in this section.

#### Interval Reporting

The PCB in Soil Tube Assay results were reported as concentration ranges that were designated as intervals incorporating parenthesis/bracket notation. The parentheses indicated that the end-points of the concentration range were excluded, while brackets indicated that the end-points were included. The reporting intervals for soils and extracts are presented in Table 5-1. Note that the intervals are different for the soils and extracts because the soils incorporate an extraction step into the procedure. As shown in the table, the interval [1, 10) indicates that the PCB concentration range is  $\geq 1$  and  $< 10$ .

**Table 5-1. PCB in Soil Tube Assay reporting intervals**

Interval	Soil concentration range	Interval	Extract concentration range
[0, 1)	$0 \leq \text{PCB ppm} < 1$	[0, 0.4)	$0 \leq \text{PCB ppm} < 0.4$
[1, 10)	$1 \leq \text{PCB ppm} < 10$	[0.4, 4)	$0.4 \leq \text{PCB ppm} < 4$
[10, 50)	$10 \leq \text{PCB ppm} < 50$	[4, 20)	$4 \leq \text{PCB ppm} < 20$
[50, $\infty$ )	$\text{PCB ppm} \geq 50$	[20, $\infty$ )	$\text{PCB ppm} \geq 20$

#### Data Assessment

##### *Objective*

The purpose of the data assessment section is to present the evaluation of the performance of EnviroLogix's PCB in Soil Tube Assay through a statistical analysis of the data. PARCC parameters were used to evaluate the test kit's ability to measure PCBs in PE soil, environmental soil, and extract samples. The developer analyzed splits of replicate samples that were also analyzed by the reference laboratory (72 PE soil samples and 136 environmental soil samples). See Section 4 for a more detailed analysis of the reference laboratory's results. Replicate samples were analyzed by the developer at two different sites (under outdoor conditions and inside an environmentally controlled chamber) to evaluate the effect of environmental conditions on the test



kit's performance; see Section 3 for further details on the different sites. Evaluation of the measurements made at each site indicated that there were no significant differences between the two data sets. Because environmental conditions did not appear to affect the results significantly, data from both sites were also combined for each parameter—precision and accuracy—to determine the test kit's overall performance. All statistical tests were performed at the 5% significance level. Appendix D contains the raw data that were used to assess the performance of the kit.

**Precision**

Precision is the reproducibility of measurements under a given set of conditions. The frequency with which the same interval was reported within a set of replicates was used to quantify precision. Examples of how the precision was classified are presented in Table 5-2. Reporting a higher number of replicates in the same interval for a given replicate set indicates higher precision. In other words, reporting all four replicate results as the same interval indicates the highest possible precision.

**Table 5-2. Classification of precision results**

<b>If the replicate results are . . .</b>	<b>. . . then the number reported in identical intervals are . . .</b>	<b>. . . and the precision classification is . . .</b>
[1, 10), [1, 10), [1, 10), [1, 10)	4	high
[0, 1), [1, 10), [1, 10), [1, 10)	3	medium
[0, 1), [1, 10), [1, 10), [10, 50)	2	low
[0, 1), [1, 10), [10, 50), [50, ∞)	0	none

**Performance Evaluation Samples**

Table 5-3 summarizes the precision information for the test kit's analysis of the PE samples. The PCB in Soil Tube Assay reported all four replicates as the same interval (i.e., high precision) for five of the eight PE sample sets under outdoor conditions, and four of the eight PE sample sets under the chamber conditions. Operating under the outdoor conditions, all eight replicate sets were classified as having either medium or high precision. Under the chamber conditions, medium to high precision was achieved for five of eight replicate sets, with the remaining three replicate sets classified as having low precision. A more detailed analysis of the data showed that the replicates classified as having medium to low precision were never more than one interval away from the most frequently reported interval. None of the replicate sets were classified with the lowest precision (i.e., none) under either set of environmental conditions. With the exception of sample ID 119, the sample sets with low to medium precision had concentrations that were near threshold values (i.e., 10 and 50 ppm), which caused the results to be split into two intervals. For example, for sample ID 222, which had a nominal concentration of 10.9 ppm, the technology reported two results in the [1, 10) interval and two results in the [10, 50) interval.

**Table 5-3. Precision of the PCB in Soil Tube Assay for PE soil samples**

Certified PE Conc. (ppm)	Outdoor site					Chamber site				
	Sample ID	precision				Sample ID	precision			
		none	high				none	high		
		Number of replicates reported in identical intervals					Number of replicates reported in identical intervals			
0 <sup>a</sup>	2	3	4	0 <sup>a</sup>	2	3	4			
0	126 <sup>b</sup>				x	226 <sup>b</sup>				x
2.0	118			x		218				x
2.0	124				x	224				x
5.0	120				x	220				x
10.9	122			x		222		x		
20.0	119			x		219				x
49.8	125				x	225		x		
50.0	121				x	221		x		
50.0	123				x	223			x	
<b>No. in each precision classification</b>		<b>0</b>	<b>0</b>	<b>3</b>	<b>5</b>		<b>0</b>	<b>3</b>	<b>1</b>	<b>4</b>

<sup>a</sup> Indicates that all four replicates were reported as different intervals.

<sup>b</sup> Blank data were not included in the determination of the overall precision.

**Environmental Soil Samples**

The PCB in Soil Tube Assay results for the replicate environmental soil sample measurements are presented in Table 5-4. Under the outdoor conditions, the highest precision classification (i.e., the same interval reported for all four replicates) was achieved for 12 of 17 replicate sets. Under the chamber conditions, 11 of 17 sample sets were classified as high-precision. None of the replicate sets were classified with the lowest precision (i.e., none) under either set of environmental conditions. Of the sample sets for which precision was classified as medium to low, only sample ID 203 had one replicate result that differed by more than one interval range.

Because most of the measurements fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low concentration (reference laboratory values < 125 ppm) and high concentration (reference laboratory values > 125 ppm). See Section 4 for the delineation of which sample IDs were in the low and high categories. For the low concentrations, 62% of the sample sets (18 of 29) were reported with

**Table 5-4. Precision of the PCB in Soil Tube Assay for environmental soil samples**

Outdoor site					Chamber site				
Sample ID	precision				Sample ID	precision			
	none			high		none			high
	Number of replicates reported in identical intervals					Number of replicates reported in identical intervals			
	0 <sup>a</sup>	2	3	4		0 <sup>a</sup>	2	3	4
101				x	206				x
102				x	207				x
103				x	208			x	
104			x		209				x
105				x	210				x
106				x	211				x
107		x			212				x
108				x	213		x		
109				x	214				x
110				x	215				x
111		x			216		x		
112				x	217				x
<b>113 <sup>b</sup></b>				x	<b>201</b>				x
<b>114</b>			x		<b>202</b>			x	
<b>115</b>				x	<b>203</b>			x	
<b>116</b>			x		<b>204</b>			x	
<b>117</b>				x	<b>205</b>				x
<b>No. in each precision classification</b>	<b>0</b>	<b>2</b>	<b>3</b>	<b>12</b>		<b>0</b>	<b>2</b>	<b>4</b>	<b>11</b>

<sup>a</sup> Indicates that all four replicates were reported as different intervals.

<sup>b</sup> Bold sample IDs were matching Paducah sample pairs (i.e., 113/201, 114/202, 115/203, 116/204, 117/205).

all four replicates in the same interval (i.e., highest possible precision). For the high concentration category, 100% of the sample sets (five of five) were reported with the highest possible precision.

The Paducah soils (indicated by bold sample IDs in Table 5-4) were analyzed at both sites to provide an assessment of the test kit’s performance under different environmental conditions. For these samples, the data generated under both environmental conditions were also combined to provide an overall assessment of precision. Sample IDs 113 and 201, 114 and 202, 115 and 203, 116 and 204, and 117 and 205 represented replicate Paducah soil sample sets; the 100 series were samples analyzed under the outdoor conditions, and the 200 series were samples analyzed inside the chamber. Additional statistical analysis was used to compare the effect of the two environmental conditions on the measurements. Results from this analysis showed that there were no significant differences in the data generated at each site. This indicated that these different environmental conditions did not affect the performance of the test kit.

**Extract Samples**

The PCB in Soil Tube Assay results for the replicate extract measurements are presented in Table 5-5. All three sample sets analyzed under the chamber conditions were reported with the highest possible precision (i.e., all four replicates were within the same interval). Two sample sets analyzed under the outdoor conditions achieved the highest precision, and the remaining sample set (sample ID 132) was reported with low precision (i.e., two replicates were reported in the same interval).

**Table 5-5. Precision of the PCB in Soil Tube Assay for extract samples**

Outdoor site					Chamber site				
Sample ID	precision				Sample ID	precision			
	none			high		none			high
	Number of replicates reported in identical intervals					Number of replicates reported in identical intervals			
	0 <sup>a</sup>	2	3	4		0 <sup>a</sup>	2	3	4
130 <sup>b</sup>				x	230 <sup>b</sup>				x
131				x	231				x
132		x			232				x
<b>No. in each precision classification</b>	<b>0</b>	<b>1</b>	<b>0</b>	<b>1</b>		<b>0</b>	<b>0</b>	<b>0</b>	<b>2</b>

<sup>a</sup> Indicates that all four replicates were reported as different intervals.

<sup>b</sup> Blank data were not included in the determination of the overall precision

**Precision Summary**

A summary of the test kit's overall precision is presented by sample type (PE, environmental soil, and extract samples) in Table 5-6. For PE and environmental soil samples, 56% and 68% of the samples, respectively, achieved the highest possible precision (i.e., all four sample replicates were reported as the same interval). For the extract samples, 75% of the samples achieved the highest precision.

**Accuracy**

Accuracy represents the closeness of the PCB in Soil Tube Assay's measured PCB concentrations to the certified values. Because the test kit produced interval results, accuracy was evaluated in terms of the percentage of samples that agreed with, were above (i.e., biased high), and were below (i.e., biased low) the certified value.

**Table 5-6. Overall precision of the PCB in Soil Tube Assay for all sample types**

Environmental site	Percentage of samples classified in each precision category											
	PE samples				Environmental soil samples				Extract samples			
	None	Low	Med	High	None	Low	Med	High	None	Low	Med	High
Outdoor site	0	0	38	63	0	12	18	71	0	50	0	50
Chamber site	0	38	13	50	0	12	24	65	0	0	0	100
Combined sites	0	19	25	56	0	12	21	68	0	25	0	75

**Performance Evaluation Soil Samples**

Table 5-7 contains a comparison between the PCB in Soil Tube Assay’s interval result and the corresponding certified PE value. The interval(s) listed under a particular column indicate how many of the four replicates were reported as that interval. For example, for sample ID 222, two replicates were reported as [1, 10), and two were reported as [10, 50). For sample ID 119, three are reported as [10, 50), and one is reported as [1, 10). The table also presents performance acceptance ranges for the PE results, which are the guidelines established by the provider of the PE materials to gauge acceptable analytical results. These ranges were not used to evaluate the test kit results because the acceptance ranges overlap several reporting intervals. However, in all but two individual analyses of PE samples, the reported interval result included a value within the range of acceptable results.

The data in Table 5-7 were used to derive the accuracy results that are presented in Table 5-8. Accuracy was based on a comparison of the certified PE value with the interval reported by the test kit. If the interval encompassed the certified PE value, the PCB in Soil Tube Assay result “agreed” with the certified value. If the test kit result was above the certified value, the result was classified as “biased high.” If the test kit result was below the certified value, the result was classified as “biased low.” For example, for sample ID 222, the certified value was 10.9 ppm (for Aroclor 1260). The comparison would be classified as “agreed” for the PCB in Soil Tube Assay interval result [10, 50) and as “biased low” for the interval result [1, 10).

Separate comparisons were made for the two environmental conditions to determine if ambient temperature and humidity had an effect on the technology performance. Statistical analysis showed that there was no significant difference between the results obtained by the test kit under the two different environmental conditions evaluated in this demonstration. Therefore, all PE sample results were combined to determine the overall percentage of agreement between the PCB in Soil Tube Assay results and the certified PE value. The overall percentage of agreement was 78%. A comparable number of results were biased high (10%) and biased low (13%). For most of the samples which did not agree with the certified value, the result was near a test kit threshold value. For example, six of the eight test kit results for sample IDs 125 and 225 were [50, ∞); the other two results were [10, 50). The nominal concentration for these PE sample was 49.8 ppm, so those reported [10, 50) would be in agreement with the certified value, while the results reported as [50, ∞) were biased high. It appears that the test kit generally reports the more conservative (i.e., higher)

**Table 5-7. PCB in Soil Tube Assay accuracy data for PE soil samples**

Certified conc. (ppm) (Acceptance range, ppm)	Outdoor site					Chamber site				
	Sample ID	# of replicates reported at each interval				Sample ID	# of replicates reported at each interval			
		1	2	3	4		1	2	3	4
0 (n/a)	126				[0, 1)	226				[0, 1)
2.0 (0.7-2.2)	118	[1, 10)		[0, 1)		218				[1, 10)
2.0 (0.9-2.5)	124				[1, 10)	224				[1, 10)
5.0 (2.1-6.2)	120				[1, 10)	220				[1, 10)
10.9 (4.0-12.8)	122	[50, ∞) <sup>a</sup>		[10, 50)		222		[1, 10) [10, 50)		
20.0 (11.4-32.4)	119	[1, 10) <sup>a</sup>		[10, 50)		219				[10, 50)
49.8 (23.0-60.8)	125				[50, ∞)	225		[10, 50) [50, ∞)		
50.0 (19.7-63.0)	121				[50, ∞)	221		[10, 50) [50, ∞)		
50.0 (11.9-75.9)	123				[50, ∞)	223	[10, 50)		[50, ∞)	

<sup>a</sup> Result not included in the acceptance range.

interval when the result is near the threshold value of 50 ppm (see Regulatory Decision-Making Applicability section for more information). Note that in a situation where the sample concentration is near the threshold value, the operator of the test kit can compare the optical density of the sample assay to that of the calibration assay and recognize that the sample concentration is near the threshold value.

**Extract Samples**

Table 5-9 contains a comparison between the PCB in Soil Tube Assay interval result and the corresponding spike concentration for the extract samples. The test kit’s percentage of agreement with the spike concentration of the extract samples is summarized in Table 5-10. Statistical analysis showed that environmental conditions had no significant effect upon the performance of the test kit. Therefore, the data sets generated under the outdoor and chamber conditions were combined. Overall, 22 of 24 extract samples (92%) agreed with the spike concentration. Two 100-ppm spiked samples (8%) were biased low relative to the spike concentration. None were biased high.

**Table 5-8. Evaluation of agreement between PCB in Soil Tube Assay's PE sample results and the certified PE values as a measure of accuracy**

Environmental site	Relative to certified values for performance evaluation samples			Number of samples
	Biased low	Agree	Biased high	
Outdoor site	11%	75%	14%	36
Chamber site	14%	81%	6%	36
Combined sites	13%	78%	10%	72

**Table 5-9. Accuracy of the PCB in Soil Tube Assay for extract samples**

Spike conc. (µg/mL)	Outdoor site					Chamber site				
	Sample ID	# of replicates reported at each interval				Sample ID	# of replicates reported at each interval			
		1	2	3	4		1	2	3	4
0	132				[0, 0.4)	232				[0, 0.4)
10	130				[4, 20)	230				[4, 20)
100	131		[20, ∞) [4, 20)			231				[20, ∞)

**Table 5-10. Evaluation of agreement between PCB in Soil Tube Assay's extract results and the spike concentration as a measure of accuracy**

Environmental site	Relative to spike concentration for extract samples			Number of samples
	Biased low	Agree	Biased high	
Outdoor site	17%	83%	0%	12
Chamber site	0%	100%	0%	12
Combined sites	8%	92%	0%	24

**False Positive/False Negative Results**

A false positive (fp) result [10] is one in which the technology detects PCBs in the sample above the detection limit when there actually are no PCBs present. A false negative (fn) result [8] is one in which the technology indicates that there are no PCBs present in the sample, when there actually are. Both fp and fn results are influenced by the method detection limit of the technology. All of the eight blank soil samples were reported as the lowest reporting interval, which included zero, so the fp result was 0%. Of the 192 non-blank soil samples analyzed, the PCB in Soil Tube Assay reported twelve in the lowest reporting interval (0 to 1 ppm).

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Five of the corresponding reference laboratory results fell into the test kit's reporting interval (0.5 ppm), and were therefore reported correctly. Seven results were false negatives. Therefore, 4% of the soil sample results were false negatives. For the eight extract samples, the kit reported all blanks as [0, 1), indicating no fp results. All other extract samples were reported as non-blanks; therefore, the fn result was 0%.

### ***Representativeness***

Representativeness expresses the degree to which the sample data accurately and precisely represent the capability of the technology. The performance data were accepted as being representative of the technology because the PCB in Soil Tube Assay was capable of analyzing diverse sample types (PE samples, simulated wipe extract samples, and actual field environmental samples) under multiple environmental conditions. When this technology is used, QC samples should be analyzed to assess the performance of the test kit under the testing conditions.

### ***Completeness***

Completeness is defined as the percentage of measurements that are judged to be useable (i.e., the result was not rejected). Valid results were obtained by the technology for all 232 samples. Therefore, completeness was 100%.

### ***Comparability***

Comparability refers to the confidence with which one data set can be compared to another. A one-to-one sample comparison of the PCB in Soil Tube Assay results and the reference laboratory results was performed for all soil samples. Accuracy was evaluated in terms of the percentage of samples that agreed with, were above (i.e., biased high), and were below (i.e., biased low) the certified value. For comparability, the test kit results were compared with the results generated by the reference laboratory, including both environmental soils and PE samples. Sample IDs 110 and 112 were excluded because the reference laboratory did not generate quantitative results for these samples. The results are summarized in Table 5-11. The percentage of PCB in Soil Tube Assay results that agreed with the reference laboratory results was 81%. Approximately 12% were biased high, and approximately 7% were biased low relative to the results reported by the reference laboratory. For the extract samples, the test kit's results could not be compared with the reference laboratory's results because no reference laboratory data were generated for these samples.

The soil data not included in previous comparability evaluations (because the replicate data for the reference laboratory were considered suspect) are shown in Table 5-12. Refer to Section 4, especially Table 4-1, for more information on the reference laboratory's suspect measurements. The reference laboratory's suspect data were compared with the PCB in Soil Tube Assay's matching results. For sample IDs 110 and 112, the reference laboratory obtained qualitative results only. The test kit appeared to have little difficulty with these samples, as all four replicates were reported as the same interval. For the other five suspect values for the reference laboratory data, the test kit generated results that agreed with the replicate means of the reference laboratory. The only exceptions were sample IDs 216 and 225, where the



**Table 5-11. Evaluation of agreement between PCB in Soil Tube Assay’s soil results and the reference laboratory’s results as a measure of comparability**

Environmental site	Relative to reference laboratory results for soil samples			Number of samples
	Biased low	Agree	Biased high	
Outdoor site	7%	78%	15%	96
Chamber site	6%	85%	10%	104
Combined sites	7%	81%	12%	200

reference laboratory results were near the kit’s threshold value of 50 ppm. These comparisons demonstrated that the PCB in Soil Tube Assay did not have difficulty with most of the samples that were troublesome for the reference laboratory.

**Summary of PARCC Parameters**

Table 5-13 summarizes the test kit’s performance for precision, accuracy, and comparability. Precision was assessed by the percentage of replicate samples where the highest precision was achieved (i.e., all four replicates were reported as the same interval), which was 56% for the PE samples, 62% for the environmental soils, and 75% for the extract samples. The test kit’s performance was based on agreement and disagreement with the certified PE values (accuracy) and the reference laboratory results (comparability). Overall, the test kit’s performance was similar for all samples because the percentages of agreement and disagreement were not significantly different for each sample type. The percentage in agreement ranged from 78 to 92, the percentage biased high was 10 to 13, and the percentage biased low was 6 to 13.

**Regulatory Decision-Making Applicability**

One objective of this demonstration was to assess the technology’s ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils. To assess this ability, the test kit’s performance for PE and environmental soil samples ranging in concentration from 40 to 60 ppm (as determined by the paired reference laboratory analyses) can be used. For this concentration range, the test kit’s results agreed with the reference laboratory’s results 66% of the time. Results were biased high 32% of the time, and 2% of the results were biased low. No false negatives were observed. The test kit results for this concentration range were different from what was observed for the entire data set, in that the percentage of samples that were biased high was significantly higher (32% versus 12%). This indicates that the test kit results appear to have a higher percentage of results that are reported more conservatively (i.e., reported in a higher interval range than the actual result) for this concentration range than for the entire concentration range of the samples analyzed (0 to 700 ppm).

**Table 5-12. Comparison of the PCB in Soil Tube Assay results with the reference laboratory's suspect measurements**

Sample ID	Reference laboratory		PCB in Soil Tube Assay	
	Suspect measurement (ppm)	Replicate mean <sup>a</sup> (ppm)	Suspect-matching result (ppm)	No. of replicates reported as this interval
110	≤RDL <sup>b</sup>	≤RDL <sup>b</sup>	[10, 50)	4
112	≤RDL <sup>b</sup>	≤RDL <sup>b</sup>	[50, ∞)	4
106	649.6	281.0	[50, ∞)	4
205	3,305.0	493.0	[50, ∞)	4
216	151.6	55.1	[10, 50)	2
217	1,913.3	659.8	[50, ∞)	4
225	146.0	41.7	[50, ∞)	2

<sup>a</sup> Mean result excluding the suspect measurement.

<sup>b</sup> Measurement reported qualitatively as less than or equal to the reporting detection limit (RDL) for all replicates (See Table D-1).

**Table 5-13. PCB in Soil Tube Assay performance for precision, accuracy, and comparability**

Sample type	Precision <sup>a</sup>	Accuracy <sup>b</sup>			Comparability <sup>c</sup>		
	% high precision	% biased low	% agreed	% biased high	% biased low	% agreed	% biased high
PE	56	13	78	10	8	82	10
Environmental soil	68	n/a <sup>b</sup>	n/a	n/a	6	81	13
Extract	75	8	92	0	n/a <sup>c</sup>	n/a	n/a

<sup>a</sup> Percentage of sample sets that achieved highest precision (i.e., all four replicates were reported as the same interval).

<sup>b</sup> PCB in Soil Tube Assay result vs certified value. Note that accuracy cannot be assessed for environmental soils.

<sup>c</sup> PCB in Soil Tube Assay result vs reference laboratory result. Comparability cannot be assessed for extract samples.

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## **Additional Performance Factors**

### ***Sample Throughput***

Sample throughput is representative of the estimated amount of time required to extract the PCBs, to perform appropriate reactions, and to analyze the sample. Operating under the outdoor conditions, one analyst from EnviroLogix had a sample throughput rate of about eight samples per hour. Working in the chamber, the analyst obtained a lower rate, around seven samples per hour. This increased sample throughput under the outdoor conditions may be attributed to the analysis order: because the EnviroLogix analyst performed sample analyses under the chamber conditions first, he may have gained valuable experience that was applied during the analysis of the outdoor samples. Alternatively, EnviroLogix may have had more difficulty with the sample matrices that were analyzed only under the indoor conditions.

### ***Cost Assessment***

The purpose of this economic analysis is to provide an estimation of the range of costs for an analysis of PCB-contaminated soil samples using the PCB in Soil Tube Assay and a conventional analytical reference laboratory method. The analysis was based on the results and experience gained from this demonstration, costs provided by EnviroLogix, and representative costs provided by the reference analytical laboratories that offered to analyze these samples. To account for the variability in cost data and assumptions, the economic analysis was presented as a list of cost elements and a range of costs for sample analysis using the test kit and by the reference laboratory.

Several factors affected the cost of analysis. Where possible, these factors were addressed so that decision-makers can independently complete a site-specific economic analysis to suit their needs. The following categories are considered in the estimate:

- sample shipment costs,
- labor costs,
- equipment costs, and
- waste disposal costs.

Each of these cost factors is defined and discussed and serves as the basis for the estimated cost ranges presented in Table 5-14. Costs for sample acquisition and pre-analytical sample preparation, which are tasks common to both methods, were not included here. This analysis assumed that the individuals performing the analyses were fully trained to operate the technology. EnviroLogix provides free assistance, on an as-needed basis, through its technical service department. It also offers a free one-day training session on the kit at its facility in Portland, Maine. Training at the user's facility is handled on a case-by-case basis.

**Table 5-14. Estimated analytical costs for PCB soil samples**

<b>PCB in Soil Tube Assay EnviroLogix Inc.</b>		<b>EPA SW-846 Method 8081 Reference laboratory</b>	
<b>Sample throughput rate: 8 samples/hour (outdoors) 7 samples/hour (chamber)</b>		<b>Typical turnaround time: 14–30 days Actual turnaround time: 72 days</b>	
Cost category	Cost (\$)	Cost category	Cost (\$)
<b>Sample shipment</b>	0	<b>Sample shipment</b>	
		Labor	100–200
		Overnight shipping charges	50–150
<b>Labor</b>		<b>Labor</b>	
Mobilization/demobilization	250–400	Mobilization/demobilization	Included <sup>a</sup>
Travel	15–1000 per analyst	Travel	Included
Per diem	0–150 per day per analyst	Per diem	Included
Rate	30–75 per hour per analyst	Rate	44–239 per sample
<b>Equipment</b>		<b>Equipment</b>	
Mobilization/demobilization	0–150	Mobilization/demobilization	Included
Purchase field lab kit	965–1975	Purchase field lab kit	Included
Reagents/supplies	18 per sample	Reagents/supplies	Included
<b>Waste disposal</b>	135–1790	<b>Waste disposal</b>	Included

<sup>a</sup> “Included” indicates that the cost is included in the labor rate.

**PCB in Soil Tube Assay Costs**

- **Sample shipment costs.** Because the samples were analyzed on site, no sample shipment charges were associated with the cost of operating the test kit.
- **Labor costs.** Labor costs included mobilization/demobilization, travel, per diem, and on-site labor.
  - Labor mobilization/demobilization: This cost element included the time for one person to prepare for and travel to each site. The estimate ranged from 5 to 8 hours, at a rate of \$50 per hour.
  - Travel: This element was the cost for the analyst(s) to travel to the site. If the analyst is located near the site, the cost of commuting to the site (estimated to be 50 miles at \$0.30 per mile) would be minimal (\$15). The estimated cost of an analyst traveling to the site for this demonstration (\$1000) included the cost of airline travel and rental car fees.
  - Per diem: This cost element included food, lodging, and incidental expenses and was estimated ranging from zero (for a local site) to \$150 per day per analyst
  - Rate: The cost of the on-site labor was estimated at a rate of \$30 to \$75 per hour, depending on the required expertise level of the analyst. This cost element included the labor involved with the entire analytical process comprising sample preparation, sample management, analysis, and reporting.

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- **Equipment costs.** Equipment costs included mobilization/demobilization, purchase of equipment, and the reagents and other consumable supplies necessary to complete the analysis.
    - Equipment mobilization/demobilization: This included the cost of shipping the equipment to the test site. If the site is local, the cost would be zero. For this demonstration, the cost of shipping equipment and supplies was estimated at \$150.
    - Purchase of field test lab: At the time of the demonstration, EnviroLogix offered a field laboratory that would include all of the equipment necessary to complete the analyses for a cost of \$1975. The field laboratory includes a Microman M-25 positive displacement pipettor, an Eppendorf Repeater pipettor, Acculab pocket balance, Artel Differential Photometer, wash bottle, and black suitcase. The field lab can also be purchased without the photometer for \$965 (photometer alone is \$1250).
    - Reagents/supplies: These items are consumable and are purchased on a per-sample basis. At the time of the demonstration, the cost of the reagents and supplies needed to prepare and analyze PCB soil samples using the test kit was \$18 per sample. This cost included the sample preparation/ extraction supplies (including solvent), assay supplies, and consumable reagents.
  - **Waste disposal costs.** Waste disposal costs are estimated based on the 1997 regulations for disposal of PCB-contaminated waste. The test kit generated approximately 43 lb of vials containing soils and liquid solvents (classified as solid PCB waste suitable for disposal by incineration) and approximately 43 lb of other solid PCB waste (used and unused soil, gloves, paper towels, ampules, etc.). The cost of disposing of PCB solid waste by incineration at a commercial facility was estimated at \$1.50 per pound. The cost for solid PCB waste disposal at ETTP was estimated at \$18/lb. The test kit also generated approximately 21 lb of liquid waste. The cost for liquid PCB waste disposal at a commercial facility was estimated at \$0.25/lb, while the cost at ETTP was estimated at \$11/lb.

#### Reference Laboratory Costs

- **Sample shipment costs.** Sample shipment costs to the reference laboratory included overnight shipping charges, as well as labor charges associated with the various organizations involved in the shipping process.
  - Overnight shipping: The overnight express shipping service cost was estimated to be \$50 for one 50-lb cooler of samples.
  - Labor: This cost element included all of the tasks associated with the shipment of the samples to the reference laboratory. Tasks included packing the shipping coolers, completing the chain-of-custody documentation, and completing the shipping forms. Because the samples contained PCBs, the coolers were inspected by qualified personnel to ensure acceptance with the U.S. Department of Transportation's shipping regulations for PCBs. The estimate to complete this task ranged from 2 to 4 hours at \$50 per hour.
- **Labor, equipment, and waste disposal costs.** The labor bids from commercial analytical reference laboratories who offered to perform the PCB analysis for this demonstration ranged from \$44 per sample to \$239 per sample. The bid was dependent on many factors, including the perceived difficulty of the sample matrix, the current workload of the laboratory, and the competitiveness of the market. In this case, the wide variation in bids may also be related to the cost of PCB waste disposal in a particular laboratory's state. LAS Laboratories was awarded the contract to complete the analysis as

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the lowest qualified bidder (\$44 per sample). This rate was a fully loaded analytical cost that included labor, equipment, waste disposal, and report preparation.

#### **Cost Assessment Summary**

An overall cost estimate for the PCB in Soil Tube Assay vs the reference laboratory was not made because of the extent of variation in the different cost factors, as outlined in Table 5-14. The overall costs for the application of each technology will be based on the number of samples requiring analysis, the sample type, and the site location and characteristics. Decision-making factors, such as turnaround time for results, must also be weighed against the cost estimate to determine the value of the field technology vs the reference laboratory.

#### **General Observations**

The following are general observations regarding the field operation and performance of the test kit:

- The system was light, easily transportable, and rugged. It took less than an hour for the EnviroLogix analyst to prepare to analyze samples on the first day of testing. While working at the outdoor site, the analyst completely disassembled his work station, bringing everything inside at the close of each day. It took the analyst less than an hour each morning to prepare for sample analyses.
- The technology was operated by a single person.
- Operators generally require 4 hours of training. They should have a basic knowledge of field analytical techniques.
- Each batch of samples was analyzed with 1 and 10 ppm standards and a negative control. (A batch generally consisted of 12 to 17 samples). This controlled for changing environmental conditions (i.e., temperature and humidity), and other causes of batch-to-batch variation.
- Data processing and interpretation was minimal. The results were quantified relative to the two calibration standards and reported in terms of intervals. The photometer's optical density readings were recorded in a laboratory notebook.
- All reagents were allowed to come to ambient temperature before use. It is recommended that all of the reagents in the test kit be stored under refrigerated conditions. When work was being done outdoors, the analyst returned the reagents and calibrators to the refrigerator at the end of the day. During the chamber work, the reagents were stored in the chamber overnight because of the low temperature (around 57°F). New reagents and calibrators were used for each site (i.e., outdoor and chamber conditions).
- The measurement system (photometer) was recharged nightly. The analyst used the photometer for up to 10 hours without recharging. The manufacturer specifies that 500 measurements can be made on a single battery charge.
- The test kit generated approximately 43 lb of vials containing soils and liquid solvents (classified as solid PCB waste suitable for disposal by incineration) and approximately 43 lb

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of other solid PCB waste (used and unused soil, gloves, paper towels, ampules, etc.). The test kit also generated approximately 21 lb of liquid waste (aqueous with trace methanol).

### **Performance Summary**

A summary of the performance characteristics of EnviroLogix's PCB in Soil Tube Assay, presented previously in this section, is shown in Table 5-15. The performance of the test kit was characterized as unbiased, because most (78%) of the PCB in Soil Tube Assay results agreed with the certified PE values, but imprecise, because nearly half (44%) of the PE replicate results were not reported as the same interval. It should also be noted that almost all of the imprecision occurred when the concentration of the sample was near one of the test kit's threshold values (i.e., 1, 10, or 50 ppm). The test kit had no fp and 4% of the soil sample results were fn. For extract samples, the test kit had no fp or fn results.

**Table 5-15. Performance summary for the PCB in Soil Tube Assay**

Feature/parameter	Performance summary								
Blank results	<b>Soils:</b> Correctly reported all 8 samples as [0,1) ppm <b>Extracts:</b> Correctly reported all 8 samples as [0,1) ppm								
Precision	<b>Percentage of combined sample sets where all four replicates were reported as the same interval</b> PE soils: 56% Environmental soils: 68% Extracts: 75%								
Accuracy	<table border="0" style="width: 100%;"> <tr> <td style="width: 50%;"><b>PE soils</b></td> <td style="width: 50%;"><b>Extracts</b></td> </tr> <tr> <td>agreed = 78%</td> <td>agreed = 92%</td> </tr> <tr> <td>biased high = 10%</td> <td>biased high = 0%</td> </tr> <tr> <td>biased low = 13%</td> <td>biased low = 8%</td> </tr> </table>	<b>PE soils</b>	<b>Extracts</b>	agreed = 78%	agreed = 92%	biased high = 10%	biased high = 0%	biased low = 13%	biased low = 8%
<b>PE soils</b>	<b>Extracts</b>								
agreed = 78%	agreed = 92%								
biased high = 10%	biased high = 0%								
biased low = 13%	biased low = 8%								
False positive results	<b>Blank soils:</b> 0% (0 of 8 samples) <b>Blank extracts:</b> 0% (0 of 8 samples)								
False negative results	<b>PE and environmental soils:</b> 4% (7 of 192 samples) <b>Spiked extracts:</b> 0% (0 of 16 samples)								
Comparison with reference laboratory results	<b>PE and environmental soils</b> agreed = 82% biased high = 12% biased low = 7%								
Regulatory decision-making applicability	<b>PE and environmental soils (40 to 60 ppm)</b> agreed = 66% biased high = 32% biased low = 2%								
Sample throughput	7 samples/hour (chamber) 8 samples/hour (outdoors)								
Power requirements	Photometer with rechargeable battery								
Operator requirements	Basic knowledge of chemical techniques; 4 hours technology-specific training								
Cost	Incremental: \$18 per sample Field lab: \$1975 (\$965 without photometer); \$1250 photometer alone								
Hazardous waste generation	40 lb of solid/liquid (classified as solid PCB waste suitable for disposal by incineration) 40 lb of solid (used gloves, pipettes, paper towels, etc.) 20 lb of liquid waste (aqueous with trace methanol)								





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## Section 6

### Technology Update and Representative Applications

#### Objective

The purpose of this section is to allow the developer to provide information regarding new developments with its technology since the demonstration activities. In addition, the developer has provided a list of representative applications in which its technology has been or is currently being used.

#### Technology Update

At the time of the demonstration, the PCB in Soil Tube Assay was just beginning commercialization, so no changes to the technology were anticipated by EnviroLogix in the near future.

#### Representative Applications

A typical application of the PCB in Soil Tube Assay would be at a Superfund site where EPA and the site contractor were mapping the site for PCB contamination. The PCB immunoassay kit would be used to determine where the samples should be taken and which samples should be sent off-site to the fixed analytical laboratory for evaluation. In October 1998, EnviroLogix participated in such a study at a small Superfund site in Kingston, New Hampshire. Other customers of the PCB immunoassay kit are the U.S. Army Corp of Engineers, General Electric, Metcalf and Eddy, and the Roy F. Weston Corporation.

#### Data Quality Objective Example

This application of EnviroLogix's PCB in Soil Tube Assay is based on data quality objective (DQO) methods for project planning advocated by the American Society for Testing and Materials [9, 10] and EPA [11]. ORNL derived a DQO example from the performance results in Section 5. This example, which is presented in Appendix E, illustrates the use of the performance data for the test kit from the ETV demonstration in the DQO process to select the number of samples to characterize the FP and FN error rates for the decision rule.



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## Section 7 References

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- [10] American Society for Testing and Materials (ASTM), *Standard Practice for Generation of Environmental Data Related to Waste Management Activities Development of Data Quality Objectives*, D5792-95, 1997.
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**Appendix A**  
**Description of Environmental Soil Samples**



**Table A-1. Summary of soil sample descriptions**

Location	Request for Disposal (RFD) #	Drum #	Description
Oak Ridge	40022	02	Soil from spill cleanup at the Y-12 Plant in Oak Ridge, Tennessee. This soil is PCB-contaminated soil excavated in 1992.
Oak Ridge	40267	01 02 03 04	Soil from the Elza Gate area, a DOE Formerly Utilized Sites Remedial Action Program site in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1992.
Oak Ridge	24375	01 02 03	Catch-basin sediment from the K-711 area (old Powerhouse Area) at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated storm drain sediment that was excavated in 1991.
Oak Ridge	43275	01 02	Soil from the K-25 Building area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil that was excavated in 1993.
Oak Ridge	134555	03	Soil from the K-707 area at the DOE East Tennessee Technology Park (formerly known as Oak Ridge Gaseous Diffusion Plant) in Oak Ridge, Tennessee. This soil is PCB-contaminated soil from a dike spillage that was excavated in 1995.
Paducah	97002	01 02 03 04	Soil from the DOE Paducah Gaseous Diffusion Plant in Kentucky. This soil is PCB-contaminated soil from a spill cleanup at the C-746-R (Organic Waste Storage Area) that was excavated in 1989.
Portsmouth	7515	858 1069 1096 1898 2143 2528 3281 538 940 4096	Soil from the DOE Portsmouth Gaseous Diffusion Plant in Ohio. This soil is PCB-contaminated soil from a probable PCB oil spill into the East Drainage Ditch that was excavated in 1986.
Tennessee Reference Soil	n/a	n/a	Captina silt loam from Roane County, Tennessee; used as a blank in this study (i.e., not contaminated with PCBs)





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**Appendix B**  
**Characterization of Environmental Soil Samples**



**Table B-1. Summary of environmental soil characterization**

Location	Sample ID	RFD Drum # <sup>a</sup>	Composition			Total Organic Carbon (mg/kg)	pH
			% gravel	% sand	% silt + clay		
Oak Ridge	101	40022-02	0	91.8	8.2	5384	7.12
	102	40267-03	0.5	99.3	0.2	13170	7.30
	103	40267-01	0.2	96.7	3.1	13503	7.21
	104	40267-04	0.6	98.2	1.2	15723	7.07
	105	40267-01S <sup>b</sup>	0.5	94.8	4.7	14533	7.28
	106	24375-03	0.5	87.8	11.7	19643	7.36
	107	24375-01	2.5	92.5	5.0	1196	7.26
	108	40267-02	0.4	94.2	5.4	9007	7.30
	109	24375-02	0.3	93.1	6.6	1116	7.48
	110	43275-01	0	89.2	10.8	14250	7.57
	111	134555-03S <sup>b</sup>	0.5	88.1	11.4	10422	7.41
	112	43275-02	0.1	91.4	8.5	38907	7.66
	126, 226	non-PCB soil	0	85.6	14.4	9249	7.33
Paducah	113, 201	97002-04	0	92.4	7.6	1296	7.71
	114, 202	97002-01	0.2	87.6	12.2	6097	7.64
	115, 203	97002-03	0.1	83.6	16.3	3649	7.59
	116, 204	97002-02	0.4	93.7	5.8	4075	7.43
	117, 205	97002-02S <sup>b</sup>					
Portsmouth	206	7515-4096	0	87.1	12.9	3465	7.72
	207	7515-1898	0.2	78.0	21.8	3721	7.66
	208	7515-1096	0.4	74.4	25.2	3856	7.77
	209	7515-2143	0	74.3	25.7	10687	7.71
	210	7515-0940	0.3	73.0	26.7	7345	7.78
	216	7515-0538	0.5	73.3	26.3	1328	7.78
	211	7515-0538S <sup>b</sup>					
	217	7515-0538S <sup>b</sup>					
	212	7515-2528	0.5	70.4	29.1	5231	7.92
	213	7515-3281	0.5	72.6	26.8	5862	7.67
	214	7515-0858	0	65.8	34.2	6776	7.85
	215	7515-1069	1.3	75.0	23.7	4875	7.56

<sup>a</sup> Request for disposal drum number (see Table A-1).

<sup>b</sup> "S" indicates that the environmental soil was spiked with additional PCBs.



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**Appendix C**  
**Temperature and Relative Humidity Conditions**

**Table C-1. Average temperature and relative humidity conditions during testing periods**

Date	Outdoor site		Chamber site	
	Average temperature (°F)	Average relative humidity (%)	Average temperature (°F)	Average relative humidity (%)
9/21/98	<i>a</i>	<i>a</i>	57	46
9/22/98	<i>a</i>	<i>a</i>	57	46
9/23/98	82	43	56	52
9/24/98	78	56	<i>a</i>	<i>a</i>
9/25/98	74	59	<i>a</i>	<i>a</i>

<sup>a</sup> The developer was working at the other site on this day.

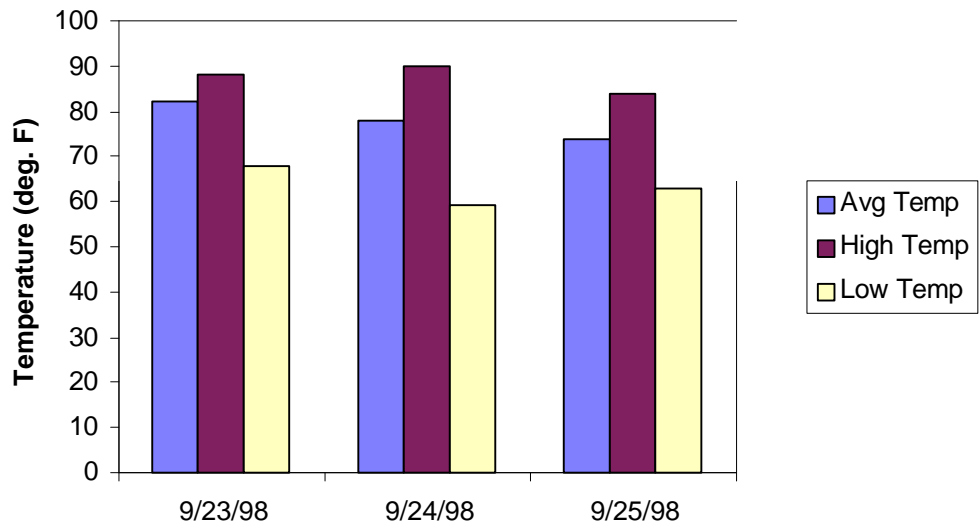


Figure C-1. Summary of temperature conditions for outdoor site.

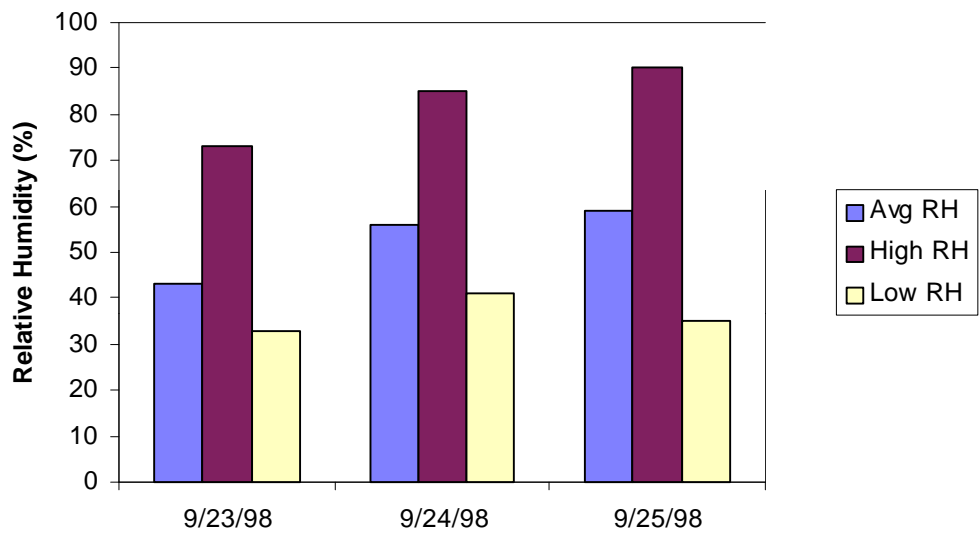


Figure C-2. Summary of relative humidity conditions for outdoor site.

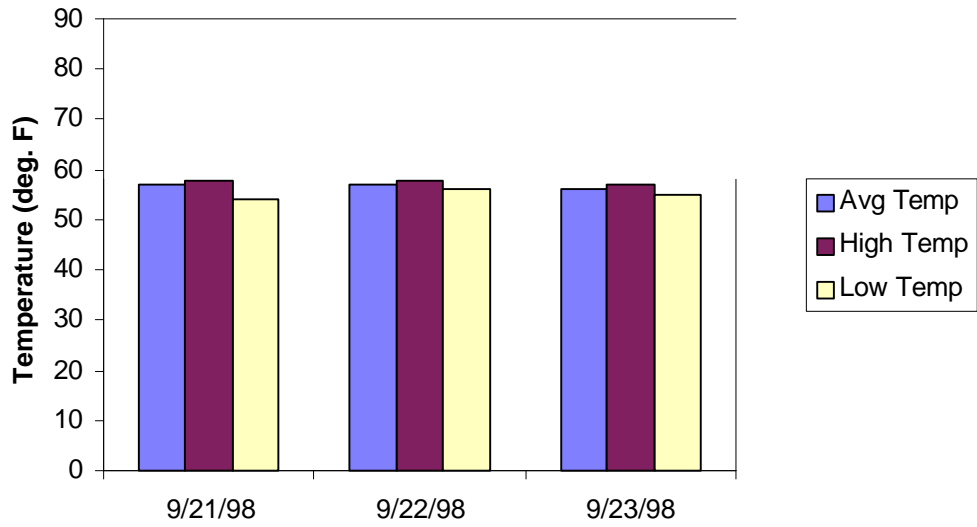


Figure C-3. Summary of temperature conditions for chamber site.

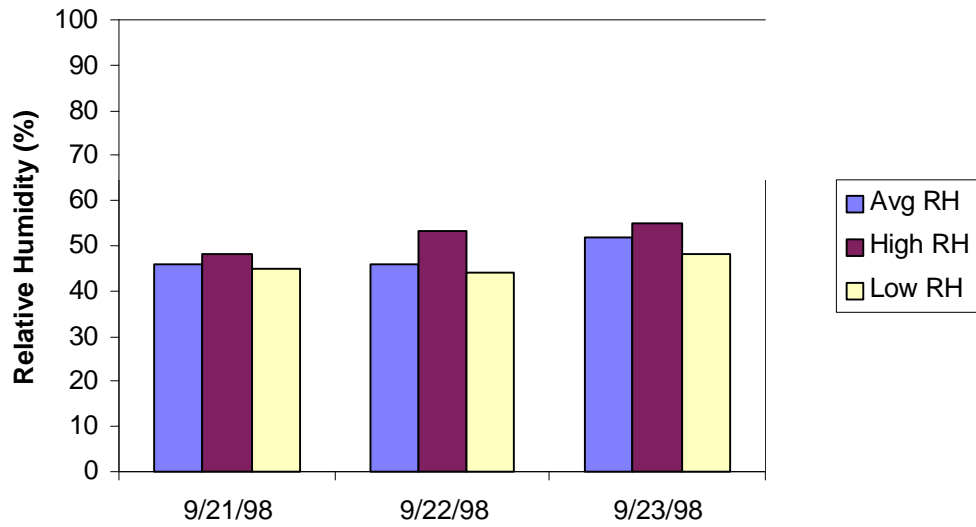


Figure C-4. Summary of relative humidity conditions for chamber site.



**Appendix D**  
**PCB in Soil Tube Assay**  
**PCB Technology Demonstration Sample Data**

**Legend for Appendix D Tables**

<b>Table Heading</b>	<b>Definition</b>
Obs	Observation
Sample ID	Sample identification 101 to 126 = outdoor site soil samples 127 to 130 = outdoor site extract samples 201 to 226 = chamber site soil samples 227 to 230 = chamber site extract samples
Rep	Replicate of sample ID (1 through 4)
EnviroLogix Result	PCB in Soil Tube Assay's measured PCB concentration (ppm)
Ref Lab Result	LAS reference laboratory measured PCB concentration (ppm) Values with "≤" are samples that the reference laboratory reported as "≤ reporting detection limit"
Reference Aroclor	Aroclor(s) identified by the reference laboratory
Type	Sample = environmental soil 1016, 1248, 1254, 1260 = Aroclor in PE soil samples or extract spiking solutions Blank = non-PCB-contaminated sample
Spike	Nominal spike concentrations (µg/mL) of extract solutions prepared by ORNL
Order	Order of sample analysis by EnviroLogix (started with 2001–2116, then 1001–1116)

Table D-1. PCB in Soil Tube Assay technology demonstration soil sample data

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
1	101	1	[0,1)	0.6	1254	Sample	1048
2	101	2	[0,1)	0.4	1254	Sample	1085
3	101	3	[0,1)	0.5	1254	Sample	1018
4	101	4	[0,1)	0.5	1254	Sample	1043
5	102	1	[1,10)	2.2	1254	Sample	1070
6	102	2	[1,10)	2.1	1254	Sample	1033
7	102	3	[1,10)	1.7	1260	Sample	1067
8	102	4	[1,10)	2.5	1260	Sample	1042
9	103	1	[1,10)	3.0	1254	Sample	1104
10	103	2	[1,10)	2.4	1254	Sample	1022
11	103	3	[1,10)	2.0	1260	Sample	1034
12	103	4	[1,10)	1.6	1260	Sample	1031
13	104	1	[10,50)	6.8	1260	Sample	1076
14	104	2	[10,50)	6.0	1254	Sample	1092
15	104	3	[1,10)	14.8	1254	Sample	1019
16	104	4	[10,50)	9.9	1254	Sample	1053
17	105	1	[50,∞)	49.7	1260	Sample	1086
18	105	2	[50,∞)	84.1	1260	Sample	1082
19	105	3	[50,∞)	50.6	1260	Sample	1098
20	105	4	[50,∞)	53.2	1260	Sample	1060
21	106	1	[50,∞)	269.6	1254	Sample	1007
22	106	2	[50,∞)	255.9	1254	Sample	1061
23	106	3	[50,∞)	317.6	1254	Sample	1071
24	106	4	[50,∞)	649.6	1254	Sample	1017
25	107	1	[0,1)	1.0	1254	Sample	1062
26	107	2	[1,10)	1.6	1254	Sample	1078
27	107	3	[0,1)	1.2	1254	Sample	1021
28	107	4	[1,10)	1.2	1254	Sample	1102
29	108	1	[1,10)	1.7	1254	Sample	1032
30	108	2	[1,10)	2.0	1254	Sample	1103
31	108	3	[1,10)	1.7	1254	Sample	1072
32	108	4	[1,10)	1.9	1254	Sample	1080
33	109	1	[1,10)	1.5	1254	Sample	1016
34	109	2	[1,10)	2.1	1254	Sample	1058
35	109	3	[1,10)	1.8	1254	Sample	1056
36	109	4	[1,10)	2.4	1254	Sample	1083
37	110	1	[10,50)	≤490.0	Non-Detect	Sample	1054
38	110	2	[10,50)	≤99.0	Non-Detect	Sample	1009
39	110	3	[10,50)	≤66.0	Non-Detect	Sample	1039
40	110	4	[10,50)	≤98.0	Non-Detect	Sample	1026
41	111	1	[50,∞)	44.5	1254	Sample	1020
42	111	2	[10,50)	36.0	1254	Sample	1025
43	111	3	[50,∞)	39.3	1254	Sample	1059
44	111	4	[10,50)	35.1	1254	Sample	1050

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
45	112	1	[50,∞)	≤66.0	Non-Detect	Sample	1073
46	112	2	[50,∞)	≤200.0	Non-Detect	Sample	1088
47	112	3	[50,∞)	≤130.0	Non-Detect	Sample	1064
48	112	4	[50,∞)	≤200.0	Non-Detect	Sample	1081
49	113	1	[1,10)	0.7	1260	Sample	1066
50	113	2	[1,10)	1.1	1260	Sample	1052
51	113	3	[1,10)	0.6	1260	Sample	1044
52	113	4	[1,10)	1.9	1248/1260	Sample	1075
53	114	1	[1,10)	1.1	1260	Sample	1093
54	114	2	[1,10)	1.2	1260	Sample	1100
55	114	3	[1,10)	1.3	1260	Sample	1012
56	114	4	[0,1)	1.7	1260	Sample	1023
57	115	1	[10,50)	14.9	1248	Sample	1084
58	115	2	[10,50)	12.4	1016	Sample	1028
59	115	3	[10,50)	15.0	1248	Sample	1001
60	115	4	[10,50)	16.9	1248	Sample	1038
61	116	1	[10,50)	41.4	1248	Sample	1015
62	116	2	[10,50)	41.2	1016	Sample	1077
63	116	3	[10,50)	48.5	1248	Sample	1011
64	116	4	[50,∞)	34.0	1016	Sample	1003
65	117	1	[50,∞)	431.6	1016	Sample	1094
66	117	2	[50,∞)	406.3	1016	Sample	1014
67	117	3	[50,∞)	304.7	1016	Sample	1040
68	117	4	[50,∞)	392.8	1016	Sample	1002
69	118	1	[0,1)	2.1	1248	1248	1037
70	118	2	[1,10)	1.9	1016	1248	1036
71	118	3	[0,1)	0.7	1248	1248	1069
72	118	4	[0,1)	1.6	1248	1248	1027
73	119	1	[1,10)	21.2	1016	1248	1049
74	119	2	[10,50)	17.2	1248	1248	1024
75	119	3	[10,50)	17.4	1248	1248	1005
76	119	4	[10,50)	24.4	1248	1248	1090
77	120	1	[1,10)	4.5	1254	1254	1057
78	120	2	[1,10)	4.0	1254	1254	1099
79	120	3	[1,10)	6.3	1254	1254	1091
80	120	4	[1,10)	5.0	1254	1254	1045
81	121	1	[50,∞)	58.7	1254	1254	1087
82	121	2	[50,∞)	55.7	1254	1254	1101
83	121	3	[50,∞)	53.2	1254	1254	1063
84	121	4	[50,∞)	50.9	1254	1254	1047
85	122	1	[10,50)	12.2	1260	1260	1013
86	122	2	[50,∞)	10.9	1260	1260	1089
87	122	3	[10,50)	11.3	1260	1260	1041
88	122	4	[10,50)	10.0	1260	1260	1095

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
89	123	1	[50,∞)	59.2	1260	1260	1004
90	123	2	[50,∞)	56.9	1260	1260	1079
91	123	3	[50,∞)	66.8	1260	1260	1008
92	123	4	[50,∞)	57.5	1260	1260	1006
93	124	1	[1,10)	1.8	1254	1254/1260	1055
94	124	2	[1,10)	1.4	1260	1254/1260	1030
95	124	3	[1,10)	1.9	1254	1254/1260	1096
96	124	4	[1,10)	1.8	1254	1254/1260	1065
97	125	1	[50,∞)	32.0	1254	1254/1260	1097
98	125	2	[50,∞)	41.3	1254	1254/1260	1051
99	125	3	[50,∞)	46.0	1254	1254/1260	1029
100	125	4	[50,∞)	32.2	1260	1254/1260	1046
101	126	1	[0,1)	≤0.1	Non-Detect	Blank	1035
102	126	2	[0,1)	≤0.1	Non-Detect	Blank	1010
103	126	3	[0,1)	≤0.2	Non-Detect	Blank	1074
104	126	4	[0,1)	≤1.3	Non-Detect	Blank	1068
105	201	1	[1,10)	1.0	1016/1260	Sample	2031
106	201	2	[1,10)	1.0	1016/1260	Sample	2035
107	201	3	[1,10)	1.1	1016/1260	Sample	2089
108	201	4	[1,10)	0.6	1260	Sample	2034
109	202	1	[1,10)	1.4	1260	Sample	2104
110	202	2	[1,10)	1.6	1260	Sample	2027
111	202	3	[1,10)	1.2	1260	Sample	2011
112	202	4	[0,1)	1.5	1260	Sample	2061
113	203	1	[10,50)	14.0	1248	Sample	2015
114	203	2	[10,50)	12.8	1248	Sample	2068
115	203	3	[0,1)	16.2	1248	Sample	2010
116	203	4	[10,50)	12.4	1248	Sample	2064
117	204	1	[10,50)	43.1	1248	Sample	2022
118	204	2	[10,50)	45.3	1248	Sample	2050
119	204	3	[50,∞)	41.0	1248	Sample	2102
120	204	4	[10,50)	47.7	1248	Sample	2095
121	205	1	[50,∞)	3305.0	1016/1260	Sample	2076
122	205	2	[50,∞)	538.7	1016	Sample	2065
123	205	3	[50,∞)	457.0	1016	Sample	2075
124	205	4	[50,∞)	483.3	1016	Sample	2016
125	206	1	[1,10)	2.9	1260	Sample	2001
126	206	2	[1,10)	1.1	1260	Sample	2014
127	206	3	[1,10)	1.1	1016/1260	Sample	2086
128	206	4	[1,10)	2.5	1260	Sample	2090
129	207	1	[10,50)	17.8	1260	Sample	2058
130	207	2	[10,50)	14.3	1260	Sample	2047
131	207	3	[10,50)	21.6	1260	Sample	2002
132	207	4	[10,50)	21.6	1254	Sample	2094

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
133	208	1	[10,50)	42.0	1260	Sample	2004
134	208	2	[10,50)	27.7	1016/1260	Sample	2018
135	208	3	[50,∞)	24.0	1254	Sample	2101
136	208	4	[10,50)	28.4	1260	Sample	2062
137	209	1	[50,∞)	32.7	1260	Sample	2063
138	209	2	[50,∞)	79.3	1260	Sample	2059
139	209	3	[50,∞)	11.0	1260	Sample	2078
140	209	4	[50,∞)	37.9	1260	Sample	2005
141	210	1	[50,∞)	123.2	1260	Sample	2049
142	210	2	[50,∞)	61.5	1260	Sample	2033
143	210	3	[50,∞)	84.1	1260	Sample	2081
144	210	4	[50,∞)	85.5	1260	Sample	2096
145	211	1	[50,∞)	387.8	1254	Sample	2083
146	211	2	[50,∞)	581.4	1254	Sample	2025
147	211	3	[50,∞)	330.0	1254	Sample	2009
148	211	4	[50,∞)	318.7	1254	Sample	2013
149	212	1	[1,10)	3.8	1260	Sample	2053
150	212	2	[1,10)	3.9	1260	Sample	2017
151	212	3	[1,10)	4.3	1260	Sample	2060
152	212	4	[1,10)	0.8	1260	Sample	2030
153	213	1	[1,10)	6.9	1260	Sample	2045
154	213	2	[10,50)	7.3	1260	Sample	2079
155	213	3	[1,10)	7.8	1260	Sample	2056
156	213	4	[10,50)	10.5	1260	Sample	2003
157	214	1	[10,50)	26.0	1260	Sample	2057
158	214	2	[10,50)	25.6	1260	Sample	2077
159	214	3	[10,50)	29.1	1260	Sample	2024
160	214	4	[10,50)	20.2	1260	Sample	2019
161	215	1	[10,50)	25.1	1260	Sample	2020
162	215	2	[10,50)	24.1	1260	Sample	2007
163	215	3	[10,50)	26.2	1260	Sample	2023
164	215	4	[10,50)	31.2	1016/1260	Sample	2038
165	216	1	[10,50)	151.6	1260	Sample	2088
166	216	2	[10,50)	47.0	1260	Sample	2042
167	216	3	[50,∞)	54.3	1260	Sample	2026
168	216	4	[50,∞)	64.0	1260	Sample	2052
169	217	1	[50,∞)	886.7	1254	Sample	2048
170	217	2	[50,∞)	549.8	1254	Sample	2046
171	217	3	[50,∞)	542.8	1254	Sample	2099
172	217	4	[50,∞)	1913.3	1016/1260	Sample	2039
173	218	1	[1,10)	2.8	1248	1248	2029
174	218	2	[1,10)	2.4	1248	1248	2036
175	218	3	[1,10)	2.6	1248	1248	2071
176	218	4	[1,10)	2.6	1248	1248	2012

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
177	219	1	[10,50)	22.4	1248	1248	2044
178	219	2	[10,50)	26.0	1016	1248	2037
179	219	3	[10,50)	29.4	1248	1248	2069
180	219	4	[10,50)	15.2	1248	1248	2097
181	220	1	[1,10)	8.5	1254	1254	2006
182	220	2	[1,10)	4.9	1254	1254	2054
183	220	3	[1,10)	4.7	1254	1254	2043
184	220	4	[1,10)	5.2	1254	1254	2021
185	221	1	[10,50)	32.0	1016/1260	1254	2087
186	221	2	[10,50)	44.1	1016/1260	1254	2070
187	221	3	[50,∞)	43.8	1254	1254	2073
188	221	4	[50,∞)	59.6	1254	1254	2092
189	222	1	[10,50)	13.2	1260	1260	2028
190	222	2	[10,50)	12.4	1260	1260	2032
191	222	3	[1,10)	12.7	1260	1260	2008
192	222	4	[1,10)	12.7	1260	1260	2091
193	223	1	[50,∞)	56.6	1260	1260	2103
194	223	2	[10,50)	50.3	1260	1260	2072
195	223	3	[50,∞)	49.9	1260	1260	2080
196	223	4	[50,∞)	66.4	1260	1260	2084
197	224	1	[1,10)	2.2	1254	1254/1260	2082
198	224	2	[1,10)	1.2	1260	1254/1260	2066
199	224	3	[1,10)	1.4	1260	1254/1260	2067
200	224	4	[1,10)	2.1	1254	1254/1260	2055
201	225	1	[50,∞)	56.4	1260	1254/1260	2040
202	225	2	[10,50)	36.5	1016/1260	1254/1260	2085
203	225	3	[10,50)	32.1	1260	1254/1260	2041
204	225	4	[50,∞)	146.0	1254	1254/1260	2100
205	226	1	[0,1)	≤0.1	Non-Detect	Blank	2093
206	226	2	[0,1)	≤0.8	Non-Detect	Blank	2074
207	226	3	[0,1)	≤0.1	Non-Detect	Blank	2098
208	226	4	[0,1)	≤0.1	Non-Detect	Blank	2051

Table D-2. PCB in Soil Tube Assay technology demonstration extract sample data

Obs	Sample ID	Rep	EnviroLogix Result (ppm)	Spike <sup>a</sup> (µg/mL)	Type	Order
1	130	1	[4, 20)	10	1248	1108
2	130	2	[4, 20)	10	1248	1116
3	130	3	[4, 20)	10	1248	1114
4	130	4	[4, 20)	10	1248	1115
5	131	1	[4, 20)	100	1016	1111
6	131	2	[4, 20)	100	1016	1110
7	131	3	[20, ∞)	100	1016	1109
8	131	4	[20, ∞)	100	1016	1107
9	132	1	[0, 0.4)	0	Blank	1106
10	132	2	[0, 0.4)	0	Blank	1105
11	132	3	[0, 0.4)	0	Blank	1112
12	132	4	[0, 0.4)	0	Blank	1113
13	230	1	[4, 20)	10	1248	2110
14	230	2	[4, 20)	10	1248	2115
15	230	3	[4, 20)	10	1248	2107
16	230	4	[4, 20)	10	1248	2109
17	231	1	[20, ∞)	100	1016	2105
18	231	2	[20, ∞)	100	1016	2114
19	231	3	[20, ∞)	100	1016	2116
20	231	4	[20, ∞)	100	1016	2111
21	232	1	[0, 0.4)	0	Blank	2106
22	232	2	[0, 0.4)	0	Blank	2112
23	232	3	[0, 0.4)	0	Blank	2108
24	232	4	[0, 0.4)	0	Blank	2113

<sup>a</sup> Nominal spike concentration of the extract sample prepared by ORNL.



Table D-3. Corrected reference laboratory data

Error	Sample ID	Reported Result (ppm)	Corrected Result (ppm)
Transcription	106	≤490	255.9
	130	5.6	10.3
	205	32,000	3,305.0
	207	180	17.8
	210	160	123.2
Calculation	118	3.6	2.1
	119	4.3	17.4
	209	2.3	37.9
	214	43.0	26.0
	219	29.0	22.4
Interpretation	101 <sup>a</sup>	≤0.7	0.5
	101 <sup>a</sup>	≤0.7	0.6
	107	≤1.3	1.2
	109	18.0	1.5
	113 <sup>b</sup>	≤0.9	0.6
	113 <sup>b</sup>	≤1.0	0.7
	119	18.0	21.2
	127	7.2	10.9
	201	≤ 1.0	0.6
	219	21.0	26.0

<sup>a</sup> Two of four measurements in sample ID 101 were corrected.

<sup>b</sup> Two of four measurements in sample ID 113 were corrected.



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**Appendix E**  
**Data Quality Objective Example**



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### Disclaimer

The following hypothetical example serves to demonstrate how the information provided in this report may be used in the data quality objectives (DQO) process. This example serves to illustrate the application of quantitative DQOs to a decision process, but it cannot attempt to provide a thorough education in this topic. Please refer to other educational or technical resources for further details. In addition, since the focus of this report is on the analytical technology, this example makes the simplifying assumption that the contents of these drums is homogeneous. In the real world, this assumption is seldom valid, and matrix heterogeneity constitutes a source of considerable uncertainty that must be adequately evaluated if the overall certainty of a site decision is to be quantified.

### Background and Problem Statement

An industrial company discovered a land area contaminated with PCBs from an unknown source. The contaminated soil was excavated into waste drums. The drums are to be treated as Resources Conservation and Recovery Act (RCRA) waste if the PCB concentration of their contents is  $<50$  ppm and as Toxic Substances Control Act (TSCA) waste if their PCB concentration is  $\geq 50$  ppm. The company's DQO team was considering the use of EnviroLogix's PCB in Soil Tube Assay kit to measure the PCB concentration in each drum. The plan was to randomly collect soil samples from each drum and test them with the kit to determine if the measured concentrations fell within one of four intervals:  $[0, 1)$ ,  $[1, 10)$ ,  $[10, 50)$ , or  $[50, \infty)$ . (Recall that this notation describes the concentration ranges  $0 \text{ ppm} \leq \text{PCB} < 1 \text{ ppm}$ ,  $1 \text{ ppm} \leq \text{PCB} < 10 \text{ ppm}$ ,  $10 \text{ ppm} \leq \text{PCB} < 50 \text{ ppm}$  and  $\text{PCB} \geq 50 \text{ ppm}$ , as used in Section 5.) The DQO team decided that a drum would be processed as TSCA waste if any of the test kit results indicated a concentration in the intervals  $[50, \infty)$ ; otherwise, the drum would be processed as RCRA waste. In agreement with the regulator, the DQO team determined that a decision rule for processing the waste would be based on the number of samples with PCB concentrations in the intervals  $[50, \infty)$ .

#### General Decision Rule

If all of the PCB sample results show concentrations  $< [50, \infty)$ , then process the soil drum by RCRA methods.

If any of the PCB sample results are in the intervals  $[50, \infty)$  then process the soil drum by TSCA methods.

### DQO Goals

Section 1.2 of EPA's *Guidance for Data Quality Assessment* states: "The true condition that occurs with the more severe decision error . . . should be defined as the null hypothesis."<sup>1</sup> The DQO team decided that the more severe decision error would be for a drum to be erroneously processed as RCRA waste if the drum's PCB concentration actually exceeded the 50 ppm limit. Therefore, the null hypothesis is constructed to assume that a drum's true PCB concentration exceeds the 50 ppm limit; as a "hot" drum, it would be processed as TSCA

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1. U.S. Environmental Protection Agency, *Guidance for Data Quality Assessment*, EPA QA/G-9; EPA/600/R-96/084, EPA, Washington, D.C., July 1996.

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waste. Drums would be processed as RCRA waste only if the null hypothesis is rejected and it is concluded that the “true” average PCB concentration is less than 50 ppm.

With the null hypothesis defined in this way, a false positive decision is made when it is concluded that a drum contains <50 ppm PCBs (i.e., the null hypothesis is rejected), when actually the drum is hot (i.e., the null hypothesis is true). The team required that the error rate for processing a hot drum as RCRA waste (i.e., FP = the false positive error rate for the decision) should be 5% or less. This error rate is expressed either as a percentage or as a probability. Therefore, a sufficient number of samples must be taken from each drum so that the false positive decision error rate (FP) is 0.05 (or less) if the true drum concentration is  $\geq 0$  ppm. This scenario represents a maximum risk of 5% for processing a drum containing 50 ppm or more of PCBs as RCRA waste.

The DQO team did not want to process an excessive number of drums as TSCA waste if the average PCB concentration was <50 ppm because of the expense. In this situation, a false negative decision is made when it is concluded that a drum is hot (i.e., the null hypothesis is not rejected), when in actuality, the drum contains soil with <50 ppm PCBs (i.e., the null hypothesis is actually false). After considering the guidelines presented in Section 1.1 of EPA’s *Guidance for Data Quality Assessment*, the DQO team recommended that the false negative decision error rate (FN) be 0.10 if the true drum concentration was <50 ppm. That is, there would be a 10% chance of processing a drum as TSCA waste (e.g., FN = Pr[False Processing a Drum as TSCA waste]) if the true PCB concentration for a drum was <50 ppm.

**Permissible FP and FN Error Rates and Critical Decision Point**

FP: Pr[Drum is RCRA waste]  $\leq$  0.05 when true PCB concentration  $\geq$  50 ppm

FN: Pr[Drum is TSCA waste]  $\leq$  0.10 when true PCB concentration < 50 ppm

**Use of Technology Performance Information to Implement the Decision Rule**

Technology performance information is used to evaluate whether a particular analytical technology can produce data of sufficient quality to support the site decision. Because the DQO team is considering the use of the EnviroLogix PCB kit, the performance of this technology [as reported in this Environmental Technology Verification (ETV) report] was used to assess its applicability to this project. The question arises: How many samples are needed from a single drum to permit a statistically valid decision at the specified certainty? Recall that the simplifying assumption was made that the PCB distribution throughout the soil within a single drum is homogeneous, and thus, matrix heterogeneity will not contribute to overall variability. The only variability to be considered in this example, therefore, is the variability in performance of the EnviroLogix test kit’s analytical method, which is determined by precision and accuracy studies.

**Determining the Number of Samples**

The number of samples needed to satisfy the FP and FN requirements depends on the misclassification error rates of the PCB test kit. Two types of misclassifications have to be considered:

1. underestimating the PCB concentration—i.e., classifying a sample concentration to be <50 ppm when the true PCB concentration is ≥50 ppm
2. overestimating the PCB concentration—i.e., classifying a sample concentration to be ≥50 ppm when the PCB concentration is <50 ppm.

The DQO team compared the experimental conditions for the EnviroLogix ETV demonstration with their expected project conditions for variables such as soil type and ambient conditions. The team determined that the relevant variables would be sufficiently similar so that misclassification rates determined from the ETV demonstration would be adequately predictive of rates for their project. The DQO team then used the ETV data to prepare Table E-1 to assess the misclassification rates around the 50 ppm action level using all performance evaluation (PE) and environmental soil samples. Table E-1 summarizes a sample-by-sample comparison of the reference lab results with the results generated by the EnviroLogix kit (see Appendix D).

**Table E-1. Comparison of PCB in Soil Tube Assay results with the reference laboratory at 50 ppm level**

Reference laboratory results	PCB in Soil Tube Assay results		Number of samples (PE + environmental soils)
	[0, 1) or [1, 10) or [10, 50)	[50, ∞)	
Greater or equal to 50 ppm	2	43	45
Less than 50 ppm	139	16	155

Table E-1 can be used to estimate the two types of misclassifications as

$$P_U = \text{Pr} [\text{Underestimating the PCB concentration}] = 2/45 = 0.044 \text{ when reference values } \geq 50 \text{ ppm,}$$

$$P_O = \text{Pr} [\text{Overestimating the PCB concentration}] = 16/155 = 0.103 \text{ when reference values } < 50 \text{ ppm.}$$

The probability distribution of classifying the number of soil samples in different concentration intervals follows a binomial probability distribution.<sup>2</sup> This probability distribution and the requirements for FP and FN can be used to determine the number of samples to meet the DQO goals. The FP for the decision rule is related to  $P_U$  by

$$FP = \text{Pr}[\text{All EnviroLogix results } < 50 \text{ ppm for } PCB \geq 50 \text{ ppm}] = (P_U)^n \quad . \quad (\text{E-1})$$

The FP error rate decreases as the sample size increases. The sample size is solved as

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<sup>2</sup> Lothar Sachs, *Applied Statistics: A Handbook of Techniques*, 2nd ed., Springer-Verlag, New York, 1984.

$$n = \frac{\text{Log}(FP)}{\text{Log}(P_U)} \quad . \quad (\text{E-2})$$

where

- $n$  = number of samples from a drum to be measured
- $FP$  = false positive decision error rate (e.g.,  $FP = 0.05$ )
- $P_U$  = probability of underestimating the PCB concentration

$$n = \frac{\text{Log}(0.05)}{\text{Log}(0.044)} = \frac{-1.301}{-1.357} = 0.96 \quad 1 \quad .$$

The sample size was rounded up to the next integer, an operation that will decrease the FP for the decision rule. The DQO team would have to analyze only one sample from each drum to meet the decision rule's FP requirement. The FN for the decision rule is related to  $P_o$  by

$$FN = Pr[\text{Some of EnviroLogix results} \geq 50 \text{ ppm for PCB} < 50 \text{ ppm}] = 1 - (1 - P_o)^n \quad . \quad (\text{E-3})$$

The error rate of a false negative decision actually increases with increasing sample size because the chance that the kit will overestimate a concentration increases with continued testing. The sample size required to meet the FN requirement is

$$n = \frac{\text{Log}(1 - FN)}{\text{Log}(1 - P_o)} \quad , \quad (\text{E-4})$$

where

- $n$  = number of samples from a drum to be measured
- $FN$  = false negative decision error rate (e.g.,  $FN = 0.10$ )
- $P_o$  = probability of overestimating a PCB concentration

$$n = \frac{\text{Log}(1 - 0.10)}{\text{Log}(1 - 0.103)} = \frac{-0.046}{-0.047} = 0.98 \quad 1 \quad .$$



The sample size must be rounded up to  $n = 1$  (fractions of a sample analysis are not possible). When  $n = 1$ , the value of FN = 10.3% which is only slightly higher than the DQO team's goal of 10% FN. This situation occurs because the 10.3% overestimation error rate of the kit is nearly equal to 10%. If additional samples were taken, FN would increase (e.g., for  $n = 2$ , FN = 19.5%) and might not meet the DQO team's goal. The only way to reduce the FN in this scenario is to use an analytical technology with a lower overestimation error rate.

The DQO team in this example decided that the sampling procedure would be to randomly select one soil sample from each drum and test the sample with the EnviroLogix's PCB in Soil Tube Assay kit. (Recall that for the purposes of this example, the soil in the drum has been assumed to be homogeneous.) The DQO team would process the drum as RCRA waste if the EnviroLogix result was less than 50 ppm, and process the drum as TSCA waste if the EnviroLogix result was greater than 50 ppm. The DQO team's goals of a 5% FP and a 10% FN would be closely met by this sampling plan.

**Decision Rule for 5% FP and 10% FN**

If one randomly selected soil sample has a PCB test result reported in an interval less than  $[50, \infty)$ , then process the soil drum as RCRA waste.

If one randomly selected soil sample has a PCB test result in the interval  $[50, \infty)$ , then process the soil drum as TSCA waste.

**Alternative FP Parameter**

The following statement describes how changing the FP requirement from 5% to 0.1% would affect the decision rule. Using  $FP = 0.001$ , the calculated sample sizes would be  $n = 2.2$ , which is rounded up to 3. The FN would be 28%. The higher FN occurs because any of the three samples have a 10.3% chance of being overestimated, and if only one is overestimated, the drum is processed as TSCA waste. The decision rule for the lower FP requirement would be as shown.

**Decision Rule for FP = 0.1% and FN = 28%**

If all three randomly selected soil samples have PCB test results reported in intervals less than 50 ppm, then process the soil as RCRA waste.

If any of the three randomly selected soil samples have a PCB test result in the interval  $[50, \infty)$ , then process the soil drum TSCA waste.