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EPA/600/R-98/110
August 1998

Environmental Technology Verification Report

Immunoassay Kit

Hach Company
PCB Immunoassay Kit

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By

Amy B. Dindal
Charles K. Bayne, Ph.D.
Roger A. Jenkins, Ph.D.

Oak Ridge National Laboratory
Oak Ridge Tennessee 37831-6120

Stephen Billets, Ph.D.
Eric N. Koglin
U.S. Environmental Protection Agency
Environmental Sciences Division
National Exposure Research Laboratory
Las Vegas, Nevada 89193-3478

This demonstration was conducted in cooperation with
U.S. Department of Energy
David Bottrell, Project Officer
Cloverleaf Building, 19901 Germantown Road
Germantown, Maryland 20874

Superfund Innovative Technology
Evaluation Program

Notice

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

ETV

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**POLYCHLORINATED BIPHENYL (PCB) FIELD ANALYTICAL TECHNIQUES**
APPLICATION:	**MEASUREMENT OF PCBs IN SOILS**
TECHNOLOGY NAME:	**PCB IMMUNOASSAY KIT**
COMPANY:	**HACH COMPANY**
ADDRESS:	**PO BOX 389** **LOVELAND, CO 80539**
PHONE:	**1-800-227-4224**

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 the Hach Company PCB immunoassay kit.

PROGRAM OPERATION

The 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 Environmental Management program, selected Oak Ridge National Laboratory (ORNL) as the testing organization for the performance verification of polychlorinated biphenyl (PCB) field analytical techniques.

DEMONSTRATION DESCRIPTION

In July 1997, the performance of six PCB field analytical techniques was determined under field conditions. Each technology was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Performance evaluation (PE) samples were also used to independently assess the accuracy and comparability of each technology.

The demonstration was designed to detect and measure PCBs in soil. The demonstration was conducted at ORNL in Oak Ridge, Tennessee, from July 22 through July 29, 1997. 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 temperature and lower relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed in this study. The results of the soil analyses conducted under field conditions by the technology were compared with results from analyses of homogeneous replicate samples conducted by conventional EPA SW-846 methodology in an approved 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, Hach Company PCB Immunoassay Kit*, EPA/600/R-98/110.

TECHNOLOGY DESCRIPTION

The PCB immunoassay kit utilizes analyte-specific antibodies attached to the inside of plastic tubes to bind and remove PCBs selectively from complex sample matrices. The kit is a semi-quantitative screening method that indicates whether the PCB concentration is above or below the specified threshold values (1 ppm and/or 10 ppm). The kit has most applicability to establishing cleanup guidelines. To initiate the test, the sample (that may contain PCBs) and a reagent containing enzyme conjugate are added to the antibody-coated tubes. An enzyme conjugate consists of an enzyme to which an analyte is attached. Enzyme conjugates and PCBs competitively bind to the antibodies attached to the inside of the tube. Samples with higher levels of PCBs will have more antibody sites occupied by the analyte and fewer occupied by the enzyme conjugate molecules. After incubation, the sample and unbound enzyme conjugate are washed from the tube and color development reagents are added. The concentration of PCBs in a sample is determined by comparing the developed color intensity to that of a PCB standard. The PCB concentration is inversely proportional to the color development, where the lighter the color, the higher the sample PCB concentration.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the PCB immunoassay kit were observed:

Throughput: Throughput was 10 to 13 samples/hour under the outdoor conditions, and 7 to 10 samples/hour under the chamber conditions. These rates included preparation and analysis.

Ease of use: Two operators analyzed samples during the demonstration, but the technology can be run by a single operator. Minimal training (2 hours) is required to operate the PCB immunoassay kit, provided that the user has a basic knowledge of chemistry and lab techniques.

Completeness: The PCB immunoassay kit generated results for all 208 PCB samples for a completeness of 100%.

Blank results: PCBs were detected and reported as 1 to 10 ppm in three of the eight blank soil samples analyzed. Therefore, the percentage of false positive results was 38%. The PCB immunoassay kit reported 2% (4 of 192 samples) false negative results.

Precision: The overall precision, based on the percentage of combined sample sets where all four replicates were reported as the same interval, was 100% for the PE soils and 68% for the environmental soils.

Accuracy: Accuracy was assessed using PE soil samples. Accuracy, defined as the percentage of PCB immunoassay kit results that agreed with the accepted concentrations, was 90%, while the percentage that was biased high or low was 4 and 6%, respectively. All of the biased low results were at concentrations near the 10-ppm threshold value.

Comparability: Comparability, like accuracy, was defined as the percentage of samples that agreed with, was above (i.e., biased high), or was below (i.e., biased low) the reference laboratory results. The percentage of PE and environmental soil samples which agreed with the reference laboratory results was 85%, while the percentage that was biased high or low was 7 and 9%, respectively. In nearly all cases where the test kit result disagreed with the reference laboratory result, the concentration was near one of the kit's threshold values of 0, 1, or 10 ppm.

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, 98% of the PCB immunoassay kit results agreed with the reference laboratory in that the test kit reported PCB concentrations as greater than 10 ppm. In contrast, only 2% were biased low, while none of the samples were biased high. As tested, the PCB immunoassay kit's interval ranges would have limited application in determining whether a sample contained > 50 ppm of PCBs, only that the sample contained > 10 ppm of PCBs.

Data quality levels: The performance of the PCB immunoassay kit was characterized as unbiased and precise. In the format that was tested, the kit provided limited information. The kit would be more applicable to cleanup applications, where it could be utilized as a quick test to determine the status of cleanup activities. Hach is working to incorporate testing at additional threshold values.

The results of the demonstration show that the PCB immunoassay kit 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.

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

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 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). The reference laboratory method used to evaluate the comparability of data was EPA SW-846 Method 8081.

The field analytical technologies tested in this demonstration were 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 Hach Company's PCB immunoassay kit. Separate ETVRs have been published for the other technologies demonstrated.

The PCB immunoassay kit utilizes analyte-specific antibodies attached to the inside of plastic tubes to bind and remove PCBs selectively from complex sample matrices. The kit is a semi-quantitative screening method that indicates whether the PCB concentration is above or below the specified threshold values (1 ppm and/or 10 ppm). The kit has most applicability to establishing cleanup guidelines. The concentration of PCBs in a sample is determined by comparing the developed color intensity to that of a PCB standard. The PCB concentration is inversely proportional to the color development, where the lighter the color, the higher the sample PCB concentration. The PCB immunoassay kit provides no information on Aroclor identification.

The PCB immunoassay kit's semi-quantitative results were based on the analysis of a calibration standard at 1 ppm that was analyzed with every four samples. Because the PCB immunoassay kit was 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, was 100% for the PE soil samples and 68% for the environmental soil samples. Accuracy, defined as the percentage of PCB immunoassay kit results that agreed with the certified PE concentrations, was 90% for all PE soil samples. In general, the percentage of samples that was biased high (4%) was comparable to the percentage that was biased low (6%). All of the biased low results were at concentrations near the 10 ppm threshold value. Comparability was defined similarly to accuracy, but the PCB immunoassay kit results were compared to the reference laboratory results rather than the accepted concentrations. For all soil samples (PE and environmental), the percentage of PCB immunoassay kit results that agreed with the reference laboratory results was 85%, while the percentage that was biased high (7%) was again comparable to the percentage that was biased low (9%). In nearly all cases where the test kit result disagreed with the reference laboratory result, the concentration was near the one of the kit's threshold values of 0, 1, or 10 ppm.

The demonstration found that the PCB immunoassay kit was simple to operate in the field, requiring about one hour for initial setup and preparation for sample analysis. Once operational, the sample throughput of the PCB immunoassay kit was 7 to 10 samples per hour under chamber conditions and 10 to 13 samples per hour under outdoor conditions. Two operators analyzed samples during the demonstration, but the technology can be run by a single operator. Minimal training (2 hours) is required to operate the PCB immunoassay kit, provided the user has a fundamental understanding of basic chemical and field analytical techniques. The overall performance of the PCB immunoassay kit was characterized as unbiased and precise. The demonstration involved PCB concentrations ranging up to 700 ppm, yet the kit's current interval structure is focused on less than 10 ppm; therefore, in the format that was tested, the kit provided limited information. The kit could be applicable to cleanup applications, where it could be utilized as a quick test to determine the status of cleanup activities. Hach is working to expand the number of interval ranges that can be tested.

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List of Abbreviations and Acronyms

ASTM	American Society for Testing and Materials
BHC	benzenehexachloride
C	concentration at which the false positive error rate is specified
CCV	continuing calibration verification standard
CSCT	Consortium for Site Characterization Technology
DCB	decachlorobiphenyl
DOE	U.S. Department of Energy
DQO	data quality objectives
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
LMER	Lockheed Martin Energy Research
LMES	Lockheed Martin Energy Systems

LV	Las Vegas
MS	matrix spike
MSD	matrix spike duplicate
n	number of samples
NERL	National Exposure Research Laboratory (EPA)
NRC	Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
ORO	Oak Ridge Operations (DOE)
PARCC	precision, accuracy, representativeness, completeness, comparability
PCB	polychlorinated biphenyl
PE	performance evaluation
ppb	parts per billion
ppm	parts per million; equivalent units: mg/kg for soils and µg/mL for extracts
Pr	probability
QA	quality assurance
QC	quality control
R^2	coefficient of determination
RDL	reporting detection limit
RH	relative humidity
RFD	request for disposal
RPD	relative percent 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-m-xylene

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For more information on the PCB Field Analytical Technology Demonstration, contact

Eric N. Koglin
Project Technical Leader
Environmental Protection Agency
Environmental Sciences Division
National Exposure Research Laboratory
P.O. Box 93478
Las Vegas, Nevada 89193-3478
(702) 798-2432

For more information on Hach's PCB immunoassay kit, contact

James Welch
Hach Company
PO Box 389
5600 Lindbergh Drive
Loveland, Colorado 80539-0389
1-800-227-4224

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 (Hach Company), the 4100 Vapor Detector (Electronic Sensor Technology), and three immunoassay kits from Strategic Diagnostics Inc.: D TECH, EnviroGard, and RaPID Assay System. This environmental technology

verification report (ETVR) presents the results of the demonstration study for one PCB field analytical technology, Hach's PCB immunoassay kit. Separate ETVRs have been published for the other five 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; and

- 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; and

- 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)*.

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). Developers 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.

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, and may or may not be substantiated by the data presented in Section 5.

Principle

The Hach Immunoassay Test Kit for field PCB analysis applies the principles of enzyme-linked immunosorbent assay (ELISA) to the determination of PCB concentrations. 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 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.

In the case of the Hach Immunoassay Test Kit, the antibodies are fixed to the interior surface of a tube, and the color change is read with a small colorimeter. This method is a semi-quantitative screening method which indicates whether the PCB concentration is above or below 1 ppm and/or 10 ppm threshold values. This is accomplished by dilution of the sample extract. For each sample, two assays are performed. An aliquot of sample is prepared identically to an aliquot of 1 ppm calibration standard, and therefore represents a 1 ppm threshold value. An aliquot of the sample prepared for the 1 ppm threshold is diluted by a factor of ten, therefore representing a 10 ppm threshold value. The results from both sample assays are compared to the assay of a 1 ppm calibration standard. The sample is then determined to be above or below the threshold values of 1 and 10 ppm.

Test Kit Description

The Hach Immunoassay Test Kit for field analysis of PCB is designed for maximum convenience and is packaged in a durable polypropylene carrying case. Everything needed for the testing is supplied with the kit. Components are molded from durable plastic and are ideal for in field use where safety is a concern.

The kit includes a Hach Pocket Colorimeter® instrument designed for use with immunoassay-based analysis, four AAA batteries, reagents for five PCB tests, labware required to run the analysis (including micro pipets, test tubes, test tube rack, reagent mixing bottles, and portable scale) and instruction manual. The Hach Pocket

Colorimeter supplied with the kit is a low-cost, high-quality filter photometer designed for single-wavelength colorimetric measurement. The liquid crystal display provides a readout in counts.

Some features of Hach's PCB immunoassay kit are as follows:

- Weight—The shipping weight of the kit is 26.5 lb.
- Transportability—The carrying case of the Hach Immunoassay Test Kit for field analysis of PCB is designed to prevent kit components from shifting and breaking during transportation and use. Inserts prevent messy spills by keeping reagents stored in an upright position.
- Power needed—Power is supplied by four AAA batteries (supplied with the kit). Typically, a set of batteries provides approximately 750 tests. A battery-saving feature incorporated into the software will automatically shut off the instrument if no keystrokes are made for 1 min. Power for the portable balance is supplied by one 9-V battery.
- Sample matrices—The Hach immunoassay PCB field analysis method instructions cover soil only. Existing reagents can be modified to address surface wipe or water applications. This was not evaluated in this demonstration study.
- Speed of analysis—The Hach Immunoassay Test Kit for PCB allows on-site detection in less than 30 min.

Sensitivity, Concentration Range, and Aroclors

For concentration sensitivity, the instructions for the Hach immunoassay PCB field analysis method currently cover making 1 and 10 ppm threshold values. Result interpretation is restricted to noting samples significantly above, below, or approximately equal to the threshold values. For the measurement of Aroclors and/or specific PCB compounds, see Table 2-1. The method cannot differentiate various PCBs. Sensitivity to specific chemicals varies (see Table 2-1), and it is possible to evaluate the kit's usefulness at a selected threshold for a specific chemical in a specific matrix.

PCBs were sold under the commercial name Aroclor. This method measures all commercial Aroclors and is sensitive to the most common Aroclors: 1248, 1254, and 1260 (see Table 2-1). Sensitivity to other halogenated compounds is generally less than 1% of the response to Aroclor 1260, making interference problems insignificant. Product validation studies performed at Hach indicate that the test correctly identifies over 95% of samples that are spiked with PCBs at or above the chosen action (threshold) level.

Table 2-1. Sensitivity to Aroclors and other compounds

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

Procedure

Training Requirements

The kit is supplied with detailed instructions to guide the user step by step through each procedure and interpretation of the results. Although immunoassay kit methods are much simpler to use than many other methods, some skill and training is required to competently perform analyses. However, the user does not have to be a trained chemist to get professional results with the Hach method.

Method Overview

Hach immunoassay tests use analyte-specific antibodies attached to the inside of plastic tubes to selectively bind analyte molecules from extract solutions prepared from complex sample matrices. Sample extracts that contain the target analyte are mixed with a reagent containing enzyme-labeled conjugate, and the mixture is added to the antibody-coated tubes. The enzyme-labeled conjugate and the PCB from the sample compete to bind to the antibodies attached to the inside of the tubes. Samples with higher levels of analyte will have more antibody binding sites occupied by PCBs from the sample and fewer antibody sites occupied by the enzyme conjugate after room-temperature incubation.

After incubation, the sample and unbound enzyme conjugate are washed from the tube and color development reagents are added. Color development only occurs in the presence of enzyme conjugate. The more enzyme conjugate attached to the antibody on the tube, the more intense the resulting color. If more PCB is present in the sample being tested, more unlabeled PCB will outcompete the enzyme conjugate to bind to the antibody site, and the resulting color will be less intense. Hach immunoassay methods compare sample results with a standard to determine whether the analyte concentrations in the sample are greater or less than the threshold levels.

Method Phase 1 — Soil Extraction

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

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

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

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

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

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

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

Method Phase 2 — Diluting Standards and Samples

1. To prepare a sample to be compared to the 1 ppm threshold, snap open a 1 ppm Dilution Ampule. Label the Dilution Ampule with appropriate sample information.

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

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

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

5. Using the WireTrol pipet, withdraw 100 µL (0.1 mL) of the standard and add it to the 1 ppm Dilution Ampule. Swirl to mix. *Note:* The standard dilution prepared above is used to evaluate samples prepared at both the 1 ppm and 10 ppm thresholds. Do not further dilute the standard.

Method Phase 3 — Immunoassay

Note: Steps in this phase require exact timing.

1. Label one PCB Antibody Tube and one PCB Enzyme Conjugate Tube for each sample dilution ampule. Because the standard is to be analyzed in duplicate, label two PCB Antibody Tubes and two PCB Enzyme Conjugate Tubes as Standard #1 and Standard #2. *Note:* The PCB Antibody and PCB Enzyme Conjugate Tubes are matched lots. Mixing with other reagent lots will cause erroneous results. To confirm the sample results, the samples can also be analyzed in duplicate (see Deviations to Demonstration Plan in Section 3).

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

3. Begin a 10-min reaction period.

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

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

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

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

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

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

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

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

Method Phase 4 — Color Development

Note: Check reagent labels carefully! Reagents must be added in proper order.

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

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

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

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

Method Phase 5 — Color Measurement

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

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

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

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

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

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

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

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

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

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

11. See Table 2-2 to interpret results.

Table 2-2. Determining if samples are above PCB threshold values

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

Measuring Hints

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

- The Soil Extractant contains methyl alcohol, which is poisonous and flammable. Read Material Safety Data Sheet before using this reagent.

- Read the entire procedure before starting. Locate and identify all reagents, tubes, and apparatus before analysis.

Environmental Limits

- Store reagents at room temperature and out of direct sunlight (less than 80°F or 27°C).

- Keep aluminized pouch that contains PCB Antibody Tubes sealed when not in use.

- Operational temperature of the reagents is 40 to 90°F (5 to 32°C).

- Power to the Hach Pocket Colorimeter instrument is supplied by four AAA batteries (supplied with the kit).

- Dilution solution is provided in the kit.

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

A population of over 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 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 ETTP 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 summer temperature in eastern Tennessee is 75.6°F, with July and August temperatures averaging 79.1°F and 76.8°F, respectively. Average temperatures during the testing periods ranged from 79 to 85°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 55 to 70°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) due to 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 to 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

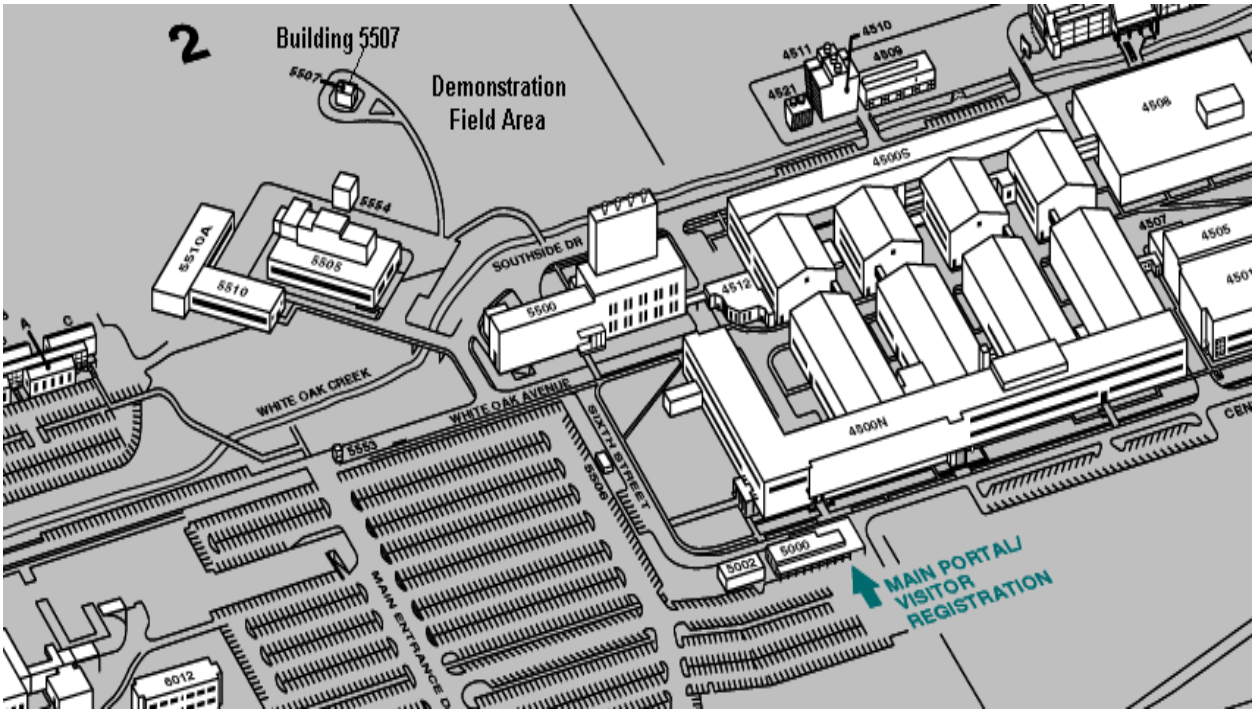

Figure 3-1. Schematic map of ORNL, indicating the demonstration area.

environmental soil) were analyzed. The ability to use environmental soils in the verification test was made 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 soil concentrations are greater than or less than 50 ppm, and that wipe samples are greater than or less than 100 µg/100 cm^2 [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 all participants prior to the start of the demonstration study. In total, the developers 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). For the developers participating in the extract sample portion (i.e., simulated wipe samples) of the demonstration, 12 extracts, ranging in concentration from 0 to 100 µg/mL, were analyzed in each

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

Concentration Range	Sample ID [a]		Total # Samples Analyzed
	Outdoor Site	Chamber Site	
PE Materials			
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
Environmental Soils			
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
Extracts			
0	129 [b]/132 [c]	229/232	8
10 µg/mL	127/130	227/230	8
100 µg/mL	128/131	228/231	8
Grand Total	**116**	**116**	**232** [d]

[a] Each sample ID was analyzed in quadruplicate.
[b] Extract prepared in iso-octane for Dexsil and the reference laboratory.
[c] Extract prepared in methanol for Electronic Sensor Technology, Strategic Diagnostics Inc., and the reference laboratory.
[d] All samples were analyzed in random order.

location (chamber and outdoors). All samples were analyzed without prior knowledge of sample type or concentration and were analyzed in a randomized order that was unique for each developer.

Environmental Conditions during Demonstration

As mentioned above, 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 was relatively uncomfortable during the July demonstration, with highs approaching 100°F and 90% relative humidity (RH). Daily average temperatures were around 85°F with 70% RH. While outside, the developers set up canopies to provide shade and protection from frequent late afternoon thundershowers.

In the indoor chamber tests, conditions were initially set to 55°F and 25% RH. An independent check of the conditions inside the chamber revealed that the temperature was closer to 68°F with a 38% RH on the first day of testing. A maintenance crew was called in to address the inconsistencies between the set and actual conditions. By the middle of the third day of testing, the chamber was operating properly at 55°F and 50% RH.

Appendix C contains a summary of the environmental conditions (temperature and relative humidity) during the demonstration. The Hach team worked outdoors July 25 and 28, 1997, and in the chamber on July 22 and 23, 1997.

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. Preprepared 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 (α, β, Δ, and δ-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 and were split for the developer and reference laboratory analyses as received.

Environmental Soil Samples

As noted in the site description above, 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.

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 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). Because different developers required the extract samples prepared in different solvents (e.g., methanol and iso-octane), the reference laboratory analyzed sets of extracts in both solvents. A total of 12 extracts were analyzed per site; these consisted of four replicates each of a blank and two concentration levels (10 and 100 µg/mL). Hach did not participate in the extract portion of the demonstration.

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 due to sample drying, making this procedure suitable for use in the preparation of the soil samples. The Paducah samples, because of their sandy characteristics, only required the sieving and mixing preparation steps. Extract sample preparation involved making solutions of PCBs in methanol and iso-octane 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 developers 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 a 35°C oven overnight. The dry soil was ground using a conventional blender and sieved through a 9-mesh screen (2 mm particle size). Approximately 1500 g of the sieved soil were spiked with a diethyl ether solution of PCBs at the desired concentration. The fortified soil was agitated using a mechanical shaker and then allowed to air-dry in a laboratory hood overnight. A minimum of four aliquots were analyzed using the analytical procedure described below to confirm the homogeneity of the soil with regard to the PCB concentration.

The 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, such that the same developer did not always receive the first jar filled for a given sample set. Second, the order of analysis was randomized so that each developer analyzed the same set of samples, but in a different order. The extract samples were split into 10-mL aliquots and placed into 2-oz jars. The extracts were stored in the refrigerator (at ≤4°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., Hach 1001 through Hach 1116). The sample identifier number was in the format of "xxxyzz," 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, developers were sent to the distribution center to pick up their 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 developers to simulate the type of information that would be available during actual field testing. Hach, however, did not receive any such information pertaining to the samples. The developers 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. Very few (<5) archive samples were utilized over the course of the demonstration.

Predemonstration Study

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. First, the recipient—in this case, the developer's facilities—must have proper Nuclear Regulatory Commission (NRC) licensing to receive and analyze radiological materials. Second, 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 none of the developers had proper NRC licensing and proving that the soils were entirely free of radioactivity was prohibitive, spiked samples of Tennessee reference soil were used for the predemonstration study. The developers had an opportunity to evaluate the Tennessee reference soils spiked with PCBs at concentrations similar to what would be used in the demonstration study. The developers also analyzed two performance evaluation samples and one solvent extract. The reference laboratory analyzed the same set of samples, which included two extracts samples, prepared in the two solvents (methanol and iso-octane) requested by the developers.

Predemonstration Sample Preparation

Two soil samples were prepared by ORNL using Tennessee reference soil [3]. The soil was a Captina silt loam from Roane County, Tennessee, that was slightly acidic (pH ~5) and low in organic carbons (~1.5%). The soil composition was 7.7% sand, 29.8% clay, and 62.5% silt. To prepare a spiked sample, the soil was first ground either using a mortar and pestle or a conventional blender. The soil was then sieved through a 16-mesh screen (1 mm particle size). Approximately 500 g of the sieved soil was spiked with a diethyl ether solution of PCBs at the desired concentration. The soil was agitated using a mechanical shaker, then allowed to air-dry overnight in a laboratory hood. A minimum of five aliquots were analyzed by gas chromatography using electron capture detection. The PCB concentration of the spiked samples was determined to be homogeneous. The remaining two soil samples used in the predemonstration study were performance evaluation materials acquired from ERA and EPA (see the section "Performance Evaluation Materials" above). In addition, a solvent extract was prepared by ORNL to simulate an extracted surface wipe sample. The extracts were prepared in two different solvents (iso-octane and methanol) to accommodate developer requests.

Predemonstration Results

The predemonstration samples were sent to the developers and the reference laboratory on June 2, 1997. Predemonstration results were received by June 26, 1997. Table 3-2 summarizes the the test kit's results for the predemonstration samples. Results indicated that Hach's PCB immunoassay kit was ready for field evaluation.

Table 3-2. Summary of Hach's PCB immunoassay kit predemonstration results

Sample Description	Matrix	Source	Hach		Reference Laboratory	
			Result (ppm)	Duplicate result (ppm)	Result (ppm)	Duplicate result (ppm)
2 ppm of Aroclor 1260	soil	ORNL	[1, 10] [a]	[1, 10]	2.2	2.3
100 ppm (total) of Aroclors 1254 and 1260	soil	ORNL	(10, ∞)	(10, ∞)	78.0	89.0
11 ppm of Aroclor 1260	soil	EPA	[1, 10]	[1, 10]	11.0	9.5
50 ppm of Aroclor 1254	soil	ERA	(10, ∞) [b]		37.0 [b]	
5 ppm of Aroclor 1242	extract	ORNL	n/a [c]	n/a [c]	4.7	4.9

[a] The notation [1, 10] indicates that the sample concentration was greater than or equal to 1 and less than or equal to 10. See Sections 2 and 5 for more information on interval reporting.
[b] Replicate was not analyzed due to lack of adequate sample for second analyses.
[c] Hach did not participate in the extract sample portion of the demonstration.

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.

Table 5 of the demonstration plan [5] delineates the environmental soils according to concentration. The classification was based on a preliminary analysis of the soils at ORNL. Table 3-1 of this report arranges the concentrations as characterized by the reference laboratory. The reference laboratory determined that five sample sets (sample IDs 102, 105, 110, 111, and 210) were in the next highest concentration range, differing from what was originally outlined in the demonstration plan. Also, the highest concentration determined by the reference laboratory was 700 ppm, while the preliminary analysis at ORNL found the highest concentration to be 500 ppm.

During the demonstration study, the Hach team made one modification to the procedure described in the technology demonstration plan [5]. This involved the number of antibody tubes used for the analysis of each sample at the 1 and 10 ppm threshold levels. The written procedure describes four tubes used for each sample—two replicates at the 1 and 10 ppm thresholds. If either of the two replicates tests positive, the

concentration is considered to be above that particular threshold value. During the demonstration, only one tube was analyzed at each threshold level, for a total of two antibody tubes per sample. While this change halved the number of tubes consumed, it removed the duplicate analysis on each sample, which provides a greater degree of caution.

Section 4
Reference Laboratory Analytical Results and Evaluation

Objective and Approach

The purpose of this section is to present 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.

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 (see Table 3-2). The SMO contracted LAS to provide full data packages for the demonstration study sample analyses within 30 days of sample shipment.

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 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., "≤ 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°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° 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/matrix spike duplicate (MS/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.

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 into 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:

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

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.

Laboratory Control Sample Results
A 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 matrix spike (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 one of the eleven 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 a laboratory control sample (LCS), an 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 percent difference (RPD) of less than 30% between the MS/MSD pair. RPD is defined as:

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

A total of eleven 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.

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 performance evaluation 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 and 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	3,305.0	
	216	47.0, 54.3, 64.0	151.6	
	217	542.8, 549.8, 886.7	1,913.3	
	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:

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

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

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 [a]	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 [b]	12.9 [b]	31 [b]	39.5 [c]	9.2 [c]	23 [c]
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

[a] All PCB concentrations were reported as non-detects.
[b] Results excluding the suspect value (results including the suspect value: mean = 67.8 ppm, SD = 53.2 ppm, and RSD =79%).
[c] Results excluding the suspect value (results including the suspect value: mean = 52.8 ppm, SD = 38.6 ppm, and RSD = 73%).

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).

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 1,196 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 soils 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 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.

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) [a]	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 [b]	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)
113 [c]	1.1	0.6	55	**201**	0.9	0.2	24
114	1.3	0.3	20	**202**	1.4	0.2	12
115	14.8	1.8	12	**203**	13.9	1.7	12
116	41.3	5.9	14	**204**	44.3	2.9	7
117	383.9	55.2	14	**205**	493.0 (1196.0)	41.7 (1406.4)	8 (118)

[a] Data in parenthesis include suspect values.
[b] n/a indicates that qualitative results only were reported for this sample.
[c] Bold sample IDs were matching Paducah sample pairs (i.e., 113/201, 114/202, 115/203, 116/204, 117/205).

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 1242 at 10 µg/mL, and spikes of Aroclor 1254 at 100 µg/mL. Identical extract samples were prepared in two solvents (iso-octane and methanol) to accommodate the developer's request. The reference laboratory analyzed both solvent sets. A Student's t-test [7, 8] was used to compare the reference laboratory's average PCB concentrations for the two different solvents and showed that no significant differences were observed at either concentration. Therefore, the reference laboratory results for the two extract solvents were combined. Additionally, all blank samples were quantified as non-detects by the reference laboratory.

Table 4-4 summarizes the reference laboratory results for the extract samples by site. RSDs for the four replicates for each sample ID ranged from 3 to 24%. For the combined data set (16 replicate measurements), the average RSD at the 10-µg/mL level was 19%, while the average RSD at the 100-µg/mL level was 8%. For the entire extract data set, an estimate of overall precision was 14%. The overall precision for the extract samples was comparable to the best-case precision for environmental soil samples (21%) and PE soil samples (18%).

Table 4-4. Precision of the reference laboratory for extract samples

Outdoor Site				Chamber Site				Combined Sites		
Sample ID	Average Conc (µg/mL)	SD (µg/mL)	RSD (%)	Sample ID	Average Conc (µg/mL)	SD (µg/mL)	RSD (%)	Average Conc (µg/mL)	SD (µg/mL)	RSD (%)
129 [a]	0	n/a	n/a	229	0	n/a	n/a	0	n/a	n/a
132 [a]	0	n/a	n/a	232	0	n/a	n/a			
127	10.9	0.4	4	227	9.6	0.8	8	10.4	1.9	19
130	12.1	2.9	24	230	8.9	1.4	16			
128	67.4	2.3	3	228	65.2	5.1	8	63.5	5.2	8
131	63.8	5.0	8	231	57.7	3.1	5			

[a] All PCB concentrations reported as non-detects by the laboratory.

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 (for PE soil and extract samples) 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.

Performance Evaluation Soil Samples

The reference laboratory's performance for the PE samples is summarized in Table 4-5. 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 4-5, 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 [9] indicated that the reference laboratory's results overall were unbiased estimates of the PE sample concentrations.

Table 4-5. 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 [a] (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 [b]	84 [b]	39.5 [c]	79 [c]
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

[a] All PCB concentrations reported as non-detects by the laboratory.
[b] Results excluding the suspect value (results including the suspect value: average = 67.8 ppm and recovery = 136%).
[c] Results excluding the suspect value (results including the suspect value: average = 52.8 ppm and Recovery = 106%).

Extract Samples

Percent recovery results for extract samples are summarized in Table 4-6 for the reference laboratory. The average percent recoveries for extract samples ranged from 58 to 121%. In terms of concentration levels, the average recovery at the 10-µg/mL level (for both solvents) was 104%, compared with 64% at the 100-µg/mL level. The reference laboratory classified all 16 samples spiked at 10 µg/mL as Aroclor 1016; however, these samples were actually spiked with Aroclor 1242. Despite this misclassification, the results did not appear to be biased. In contrast, the samples spiked at 100 µg/mL were correctly classified as Aroclor 1254 but were all biased low. Although these results suggested that Aroclor classification had little effect on the quantification of the extract samples, there was an obvious, consistent error introduced into the analysis of the 100-µg/mL samples to cause the low bias. For the entire extract data set, the overall percent recovery was 84%.

Table 4-6. Accuracy of the reference laboratory for extract samples

Spike Concentration (µg/mL)	Outdoor Site			Chamber Site			Combined Sites	
	Sample ID	Avg Conc (µg/mL)	Recovery (%)	Sample ID	Avg Conc (µg/mL)	Recovery (%)	Avg Conc (µg/mL)	Recovery (%)
0 [a]	129	0	n/a	229	0	n/a	0	n/a
0 [a]	132	0	n/a	232	0	n/a		
10	127	10.9	109	227	9.6	96	10.4	104
10	130	12.1	121	230	8.9	89		
100	128	67.4	67	228	65.2	65	63.5	64
100	131	63.8	64	231	57.7	58		

[a] All PCB concentrations reported as non-detects by the laboratory.

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 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 4-7 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 Table 4-7, the precision of the PE soils was comparable to 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). The extract samples had a smaller overall RSD of 14%. Evaluation of overall accuracy was based on samples with certified or known spiked concentrations (i.e., PE and extract samples). The overall accuracy, based on percent recovery, for the PE samples 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 measured values were unbiased estimates of the certified PE concentrations (for samples that contained $\leq$50 ppm of PCBs). Accuracy for the extract samples at 10 ppm was also unbiased, with an average percent recovery of 104%. However, the accuracy for the extract samples at 100 ppm was biased low, with an average recovery of 64%. Overall, the average percent recovery for all extract samples was 84%. The reference laboratory correctly reported all blank samples as non-detects, but had difficulty with two soil sample IDs (110 and 112) that contained chemical interferences. In general, the reference laboratory's completeness would be reduced, at the expense of an improvement in precision and accuracy, if the suspect measurements were excluded from the data analysis. Based on this analysis, it was concluded that the reference laboratory results were acceptable for comparison with the developer's technology.

Table 4-7. Summary of the reference laboratory performance

Sample Matrix	Sample Type	Number of Samples	Precision (Average % RSD)	Accuracy (Average % Recovery)
Blank	Soil Extract	8 16	n/a [a]	All samples were reported as non-detects.
Environmental soil with interferences	Sample ID 110 Sample ID 112	4 4	n/a [a]	All samples were reported as non-detects.
Soil Best Case (excluding suspect data)	PE Environmental < 125 ppm > 125 ppm overall	63 107 17 187	18 23 19 21	101 n/a [b] n/a [b] 101
Soil Worst Case (including suspect data)	PE Environmental < 125 ppm > 125 ppm overall	64 108 20 192	21 26 56 28	105 n/a [b] n/a [b] 105
Extract	10 ppm 100 ppm overall	16 16 32	19 8 14	104 64 84

[a] Because the results were reported as non-detects, precision assessment is not applicable.
[b] Accuracy assessment calculated for samples of known concentration only.

Section 5
Technology Performance and Evaluation

Objective and Approach

The purpose of this section is to present the evaluation of the data generated by the Hach PCB immunoassay kit. The technology's precision and accuracy performance 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 test kit results were reported as concentration ranges that were designated as intervals incorporating parentheses/bracket notation. The parentheses indicated that the end-points of the concentration range were excluded, while brackets indicated that the end-points were included. As shown in Table 5-1, the interval [0, 1) indicates that the PCB concentration range is greater than or equal to 0 and less than 1. All samples are reported as one of the three intervals listed in Table 5-1, and are not adjusted for Aroclor specificity.

Table 5-1. Hach PCB immunoassay kit reporting intervals

Interval	Concentration Range
[0, 1)	$0 \leq$ PCB ppm < 1
[1, 10]	$1 \leq$ PCB ppm ≤ 10
(10, ∞)	PCB ppm > 10

Data Assessment

Objective

The purpose of the data assessment section is to present the evaluation of the performance of the Hach PCB immunoassay kit through a statistical analysis of the data. PARCC parameters were used to evaluate the test kit's ability to measure PCBs in PE, 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 difference 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 test 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. Because there were only three possible intervals to be reported, at least two of the four replicates would be reported as the same interval.

Table 5-2. Classification of precision results

If the replicate results are...	...and the number reported in identical intervals are...	..then the precision classification is...
[0, 1), **[1, 10]**, **[1, 10]**, (10, ∞)	2	low
[0, 1), [**1, 10]**, **[1, 10]**, **[1, 10]**	3	medium
[1, 10], **[1, 10]**, **[1, 10]**, **[1, 10]**	4	high

Performance Evaluation Samples

Table 5-3 summarizes the precision information for the test kit's analysis of the PE samples. The test kit reported all four replicates as the same interval (i.e., high precision) for all eight non-blank PE sample sets under both the outdoor and chamber conditions. The blanks were reported with low and medium precision.

Environmental Soil Samples

Hach's test kit results for the replicate environmental soil sample measurements are presented in Table 5-4. Under the outdoor conditions, 12 of 17 replicate sets achieved the highest precision classification (i.e., the same interval was reported for all four replicates). Under the chamber conditions, 11 of 17 sample sets were reported with high precision. Of the sample sets where precision was classified as medium to low, none differed by more than one interval range.

Table 5-3. Precision of Hach's PCB immunoassay kit for PE soil samples

Certified PE Conc. (ppm)	Outdoor site				Chamber site			
	Sample ID	Precision low high			Sample ID	Precision low high		
		Number of replicates reported in identical intervals				Number of replicates reported in identical intervals		
		2	3	4		2	3	4
0	126 [a]	x			226 [a]		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
# in each precision classification		**1**	**0**	**8**		**0**	**1**	**8**

[a] Blank data were not included in the determination of the overall precision.

Because the majority of the measurements fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low (reference laboratory values < 125 ppm) and high concentrations (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, 66% of the sample sets were reported with all four replicates in the same interval (i.e., highest possible precision). For the high concentration category, 80% of the sample sets (4 of 5) 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, where 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

Table 5-4. Precision of Hach's PCB immunoassay kit for environmental soil samples

Outdoor site				Chamber site			
Sample ID	Precision low high Number of replicates reported in identical intervals			Sample ID	Precision low high Number of replicates reported in identical intervals		
	2	3	4		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
113 [a]			x	**201**	x		
114		x		**202**		x	
115		x		**203**			x
116			x	**204**		x	
117			x	**205**		x	
# in each precision classification	**1**	**4**	**12**		**1**	**5**	**11**

[a] Bold sample IDs were matching Paducah sample pairs (i.e., 113/201, 114/202, 115/203, 116/204, 117/205).

site. This indicated that these different environmental conditions did not impact the performance of the test kit. However, the test kit appeared to have slightly more difficulty with the Paducah samples relative to the other soil matrices that comprised the environmental soil samples.

Precision Summary

A summary of the test kit's overall precision is presented by sample type (PE and environmental soil) in Table 5-5. For PE and environmental soil samples, 100% and 68% of the samples, respectively, achieved the highest possible precision (i.e., all four samples replicates were reported as the same interval).

Table 5-5. Overall precision of the Hach PCB immunoassay kit for all sample types

Environmental Site	Percentage of samples classified in each precision category					
	PE Samples			Environmental Soil Samples		
	low	med	high	low	med	high
Outdoor Site	0	0	100	6	24	71
Chamber Site	0	0	100	6	29	65
Combined Sites	0	0	100	6	26	68

Accuracy

Accuracy represents the closeness of the test kit'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 which agreed with, were above (i.e., biased high), and were below the certified value (i.e., biased low).

Performance Evaluation Soil Samples

Table 5-6 contains a comparison between the test kit's interval result and the corresponding certified PE value. The interval(s) listed under a particular column indicates how many of the four replicates were reported as that interval. For example, for sample ID 126, two replicates were reported as [0, 1), and two were reported as [1, 10]. For sample ID 226, three are reported as [0, 1), and one is reported as [1, 10]. Note that performance acceptance ranges for the PE results, which are the guidelines established by the provider of the PE materials to gauge acceptable analytical results, are also presented in Table 5-6 for information. These ranges were not used to evaluate the test kit results because the acceptance ranges overlap several of the test kit's reporting intervals.

The data in Table 5-6 were used to derive the accuracy results presented in Table 5-7. 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 test kit 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 118, the certified value was 2.0 ppm (for Aroclor 1248). The comparison would be classified as "agreed" for the test kit's interval result [1, 10], as "biased high" for the interval result (10, ∞), or as "biased low" for the interval result [0, 1). 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

Table 5-6. Hach's PCB immunoassay kit accuracy 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) [1, 10]			226	[1, 10]		[0, 1)	
2.0 (0.7-2.2)	118				[1, 10]	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				(10, ∞)	222				[1, 10]
20.0 (11.4-32.4)	119				(10, ∞)	219				(10, ∞)
49.8 (23.0-60.8)	125				(10, ∞)	225				(10, ∞)
50.0 (19.7-63.0)	121				(10, ∞)	221				(10, ∞)
50.0 (11.9-75.9)	123				(10, ∞)	223				(10, ∞)

Table 5-7. Evaluation of agreement between Hach's PCB immunoassay kit'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	0%	94%	6%	36
Chamber Site	11%	86%	3%	36
Combined Sites	6%	90%	4%	72

different environmental conditions evaluated in this demonstration. Therefore, all PE sample results were combined to determine the overall percentage of agreement between test kit results and the certified PE value. The overall percentage of agreement was 90%. Of the sample results which disagreed, 4% were biased high,

which were three blank results reported as [1, 10]. The remaining 6% of the test kit results were biased low, which were the four replicates from sample ID 222 that were reported as [1, 10], where the nominal concentration was 10.9 ppm. Note that for sample ID 122, all four replicates were correctly reported as (10, ∞).

False Positive/False Negative results

A false positive (fp) result [10] is one in which the technology detects PCBs in the sample when there actually are none. A false negative (fn) result [10] 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. Of the eight blank soil samples, three were reported as [1, 10], so the fp result was 38%. Of the 192 non-blank soil samples analyzed, the test kit reported four in the lowest reporting interval (e.g., 0 to 1 ppm), but the corresponding reference laboratory results were greater than 1 ppm. Therefore, the fn result for the soil samples was 2%.

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 Hach's PCB immunoassay kit was capable of analyzing diverse sample types (PE samples and actual field environmental samples) under multiple environmental conditions. When using this technology, quality control samples should be analyzed to assess the performance of the Hach PCB immunoassay 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). Useable 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 test kit results and the reference laboratory results was performed for all soil samples. Accuracy was evaluated in terms of the percentage of samples which agreed with, were above (i.e., biased high), and were below (i.e., biased low) the certified value. For comparability, the kit's 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 5-8. The percentage of test kit results that agreed with the reference laboratory results was 85%. Approximately 7% were biased high, while approximately 9% were biased low relative to the results reported by the reference laboratory. In nearly all cases where the test kit result disagreed with the reference laboratory result, the concentration was near one of the kit's threshold values of 0, 1, or 10 ppm. For example, for sample ID 203, the reference laboratory's four replicate results were 12.4, 12.8, 14.0, and 16.2 ppm. The test kit reported all four results as [1, 10], which was classified as biased low. Note that Hach recommends either secondary confirmation or use of the more conservative interpretation for sample results that are near the threshold values.

Table 5-8. Evaluation of agreement between Hach's PCB immunoassay kit'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	4%	87%	9%	96
Chamber Site	13%	84%	4%	104
Combined Sites	9%	85%	7%	200

As discussed in the Precision section, the Paducah samples were analyzed at both environmental sites to evaluate the effect that environmental conditions had on performance. Results for these samples were more imprecise than the results for the other matrices (i.e., Oak Ridge and Portsmouth samples). Additional statistical tests on the Paducah sample results indicated that the test kits results were significantly different from the reference laboratory results under the chamber conditions. Because the disagreement with the reference laboratory results was significantly increased for these particular samples, the test kit's difficulty with the Paducah samples may be related to a matrix effect.

The soil data not included in previous comparability evaluations (because the replicate data for the reference laboratory were considered suspect) are shown in Table 5-9. Refer to Section 4, in particular Table 4-1, for more information on the reference laboratory's suspect measurements. The reference laboratory's suspect data were compared with the test kit's matching results. For sample IDs 110 and 112, the reference laboratory obtained qualitative results only. The test kit also had some difficulty with sample ID 110, producing results in two different intervals in contrast to sample ID 112, where all four replicates were reported as the same interval. For four of the five other suspect values for the reference laboratory data, the test kit generated results that agreed with the replicate means of the reference laboratory. One of the test kit results ([1, 10]) was biased low relative to the reference laboratory's replicate mean (493.0 ppm). These comparisons demonstrated that the test kit did not have difficulty with most of the samples that were troublesome for the reference laboratory.

Summary of PARCC Parameters

Table 5-10 summarizes the test kit's performance for precision, accuracy, and comparability. The percentage of replicate samples where the highest precision was achieved (i.e., all four replicates were reported as the same interval) was 100% for the PE samples and 68% for the environmental soil samples. The test kit's agreement and disagreement with certified values were based on the certified PE values (i.e., accuracy) and the reference laboratory results (i.e., comparability). Overall, the test kit's performance was similar for all samples, because the percentages of agreement and disagreement were not significantly different for PE and environmental samples. The percentage in agreement ranged from 83 to 90%, the percentage biased high was 4 to 7%, and the percentage biased low was 6 to 10%.

Table 5-9. Comparison of the Hach's PCB immunoassay kit results with the reference laboratory's suspect measurements

Sample ID	Reference Laboratory		Hach's PCB Immunoassay kit	
	Suspect Measurement (ppm)	Replicate Mean[a] (ppm)	Suspect Matching Result (ppm)	Number of Replicates Reported as This Interval
110	≤RDL [b]	≤RDL [b]	[1,10] (10, ∞)	3 1
112	≤RDL [b]	≤RDL [b]	(10, ∞)	4
106	649.6	281.0	(10, ∞)	4
205	3,305.0	493.0	[1,10]	1
216	151.6	55.1	(10, ∞)	4
217	1,913.3	659.8	(10, ∞)	4
225	146.0	41.7	(10, ∞)	4

[a] Mean result excluding the suspect measurement.
[b] Measurement reported qualitatively as less than or equal to the reporting detection limit for all replicates.

Table 5-10. Hach PCB immunoassay kit performance for precision, accuracy, and comparability

Sample Type	Precision [a]	Accuracy [b]			Comparability [c]		
	% high precision	% biased low	% agreed	% biased high	% biased low	% agreed	% biased high
PE	100	6	90	4	6	89	6
Environmental Soil	68	n/a [b]	n/a	n/a	10	83	7

[a] Percentage of sample sets that achieved highest precision (i.e., all four replicates were reported as the same interval.
[b] Hach result versus certified value; accuracy cannot be assessed for environmental soils.
[c] Hach result versus reference laboratory result.

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, 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 98% of the time in that the test kit reported PCB concentrations as greater than 10 ppm. One result was biased low (2%). No biased high results were possible for this concentration range, because the test kit's highest reporting interval was (10, ∞). As tested, the test kit's interval ranges would have limited application in determining whether a sample contained 50 ppm or more of PCB, only that the sample contained more than 10 ppm of PCB. In the format that was tested, Hach's kit would be applicable to cleanup applications, where the kit could be utilized as a quick test to determine if cleanup activities could stop or if more cleanup was needed. Hach is working to incorporate testing at the 50-ppm level.

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, the Hach team's sample throughput rate ranged from 10 to 13 samples per hour. Working in the chamber, the rate was lower, around 7 to 10 samples per hour. This increased sample throughput under the outdoor conditions may be attributed to the analysis order; because Hach analyzed samples under the chamber conditions first, they may have gained valuable experience that was applied during the analysis of the outdoor samples.

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 Hach PCB test kit and using a conventional analytical reference laboratory method. The analysis was based on the results and experience gained from this demonstration, costs provided by Hach, and representative costs provided by the reference analytical laboratories who 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 by the Hach 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,
- waste disposal costs.

Each of these cost factors is defined and discussed below and serves as the basis for the estimated cost ranges presented in Table 5-11. This analysis assumed that the individuals performing the analyses were fully trained to operate the technology. Hach does not offer a specific training course on the use of the Hach kit, but does provide free assistance, on an as-needed basis, through its technical service department. Costs for sample acquisition and pre-analytical sample preparation, which are tasks common to both methods, were not included here.

Hach PCB Immunoassay Kit Costs

Because the samples were analyzed on-site, no sample shipment charges were associated with the cost of operating the Hach test kit. Labor costs included mobilization/demobilization, travel, per diem, and on-

Table 5-11. Estimated analytical costs for PCB soil samples

PCB Immunoassay Kit Hach Company Sample throughput rate: 10 - 13 samples per hour (outdoors) 7 - 10 samples per hour (chamber)		EPA SW-846 Method 8080/8081/8082 Reference Laboratory Typical turn-around time: 14 - 30 days	
Cost Category	**Cost ($)**	**Cost Category**	**Cost ($)**
Sample Shipment	0	**Sample Shipment**	
		Labor	100 - 200
		Overnight shipping charges	50 - 150
Labor		**Labor**	
Mobilization/demobilization	250 - 400	Mobilization/demobilization	included[a]
Travel	15 - 1,000 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
Equipment		**Equipment**	
Mobilization/demobilization	0 - 150	Mobilization/demobilization	included
Kit purchase price	955	Rental/purchase of system	included
Reagents/supplies	35 per sample	Reagents/supplies	included
Waste Disposal	75 - 1,060	**Waste Disposal**	included

[a] "Included" indicates that the cost is included in the labor rate.

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 ($1,000) 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 $0 (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.

Equipment costs included mobilization/demobilization, rental fees or 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: At the time of the demonstration, the cost of purchasing the Hach test kit was $955. The kit included: a Hach Pocket Colorimeter instrument designed for use with immunoassay-based analysis; reagents for five PCB tests; labware required to run the analysis; and instruction manual. The kit was supplied in a polypropylene carrying case.

- 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 $35 per sample. This cost included the sample preparation supplies, assay supplies, and consumable reagents. Standard Ampules were also available for $19.60 for a package of five.

Waste disposal costs are estimated based on the 1997 regulations for disposal of PCB-contaminated waste. Using the test kit during the demonstration, Hach generated approximately 20 lb of vials containing soils and liquid solvents (classified as incinerable solid PCB waste) and approximately 20 lb of other solid PCB waste (i.e., used and unused soil, gloves, paper towels, ampules, etc.). The cost of disposing PCB solid waste by incineration at a commercial facility was estimated at $1.50 /lb. The cost for solid PCB waste disposal at ETTP was estimated at $18/lb. The test kit also generated approximately 19 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 to the reference laboratory included the overnight shipping charges, as well as labor charges associated with the various organizations involved in the shipping process.

- 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 compliance with the U.S. Department of Transportation's shipping regulations for PCBs. The estimate for completing this task was 2 to 4 hours at $50 per hour.

- Overnight shipping: The overnight express shipping service cost was estimated to be $50 for one 50-lb cooler of samples.

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 work load of the laboratory, and the competitiveness of the market. In this case, the wide range of 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 the lowest qualified bidder ($44 per sample). This rate was a fully loaded analytical cost, including equipment, labor, waste disposal, and report preparation.

Cost Assessment Summary

An overall cost estimate for Hach's PCB immunoassay kit versus the reference laboratory was not made because of the extent of variation in the cost factors, as outlined in Table 5-11. 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 turn-around time for results, must also be weighed against the cost estimate to determine the value of the field technology versus the reference laboratory.

General Observations

The following are general observations regarding the field operation and performance of Hach's PCB immunoassay kit:

- The system was light, easily transportable, and rugged. It took about one hour for the Hach team to prepare to analyze samples on the first day of testing. While working at the outdoor site, the Hach team completely disassembled their work station bringing everything inside at the close of each day. It took the Hach team less than one hour each morning to prepare for sample analyses.

- Two operators were used for the demonstration because of the number of samples and working conditions, but the technology can be operated by a single person.

- Operators generally require two hours of training and should have a basic knowledge of field analytical techniques.

- The Hach team calibrated the pocket colorimeter often, analyzing a 1 ppm standard in duplicate with every batch of four samples. This was done to account for changing environmental conditions (i.e., temperature and humidity).

- Data processing and interpretation was minimal. The results were reported in terms of intervals, relative to the calibration standard. No raw data were recorded, other than the interval result.

- The measurement system (pocket colorimeter) was battery-operated.

- The Hach PCB immunoassay kit generated approximately 20 lb of vials containing soils and liquid solvents (classified as incinerable solid PCB waste) and approximately 20 lb of other solid PCB waste (i.e., used and unused soil, gloves, paper towels, ampules, etc.). The test kit also generated approximately 19 lb of liquid waste (aqueous with trace methanol).

Performance Summary

A summary of the performance characteristics of the Hach PCB immunoassay kit, presented previously in this chapter, is shown in Table 5-12. The performance of Hach's PCB immunoassay kit was characterized as unbiased, because nearly all (90%) of the test kit results agreed with the certified PE values, and as precise, because 100% of the PE replicate results were reported as the same interval. The test kit had three false positive results (38%) and four false negative results (2%).

Table 5-12. Performance summary for the Hach PCB immunoassay kit

Feature/Parameter	Performance Summary
Blank Soil Results	Correctly reported 5 of 8 samples as [0,1) ppm; 3 samples reported as [1, 10]
Precision	**Percentage of combined PE sample sets where all four replicates were reported as the same interval** PE Soils: 100% Environmental Soils: 68%
Accuracy	**PE Soils** agreed = 90% biased high = 4% biased low = 6%
False Positive Results	**Blanks** 38% (3 of 8 samples)
False Negative Results	**PE and Environmental Soils** 2% (4 of 192 samples)
Comparison with Reference Laboratory Results	**PE and Environmental Soils** agreed = 85% biased high = 7% biased low = 9%
Regulatory Decision-Making Applicability	**PE and Environmental Soils (40 to 60 ppm)** agreed = 98% biased low = 2%
Sample Throughput	7-10 samples/hour (chamber) 10-13 samples/hour (outdoors)
Power Requirements	Battery-operated pocket colorimeter (four AAA); provides approximately 750 tests Battery-operated portable balance (one 9-V)
Operator Requirements	Basic knowledge of chemical techniques; 2 hours technology-specific training
Cost	Incremental: $35 per sample Instrumental: $955 (purchase)
Hazardous Waste Generation	~ 20 lb of solid/liquid (classified as incinerable solid) ~ 20 lb of solid (used gloves, pipettes, paper towels, etc.) ~ 19 lb of liquid waste (aqueous with trace methanol)

Section 6
Technology Update and Representative Applications

Objective

In this section, Hach describes new technology developments that are planned. The developer has also provided a list of representative applications where their PCB immunoassay kit has been or is currently being utilized. The data quality objective example described briefly below (and detailed in Appendix E) was derived by ORNL from the performance results that are summarized in Section 5.

Technology Update

The addition of a 50 ppm threshold level is anticipated for the near future. This update will simply incorporate either a different dilution scheme or a different calibrator. This update will provide the ability to test at levels other than 1 and 10 ppm.

Representative Applications

Potential Users of the Technology

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

Actual Users of the Technology

A query of Hach customers who have purchased the PCB kit over the past year shows that the kit has been purchased for use in the following industries: refuse systems, utilities, research and development testing, vocational schools, engineering services, and environmental consulting. The kit is used by customers within the United States (75%) and overseas (25%). Below are three examples of customers currently using the product.

> HZW Environmental Consultants: HZW is a consulting firm that does phase 1 and phase 2 testing. Each new job determines the need for testing. All PCB tests are run in the field and all tests are run per customer demand. HZW personnel have said that the tests were very easy to use and that they were great for the field. They said that after they ran tests with the Hach kit, they sent selective samples to a lab for confirmation and got the same results. They feel that Hach's PCB test provides them and their clients with immediate and accurate results.

Mill Service Inc.: Mill Service Inc. uses the PCB kit to test a waste stream of soil at a treatment disposal site. It is required to run the tests by the Pennsylvania Department of Environmental Protection. Personnel run about 5 or 6 samples every other day. They find the tests to be extremely easy to use and cost effective. They run tests because they need same-day results; if they had to send them to a lab, it would cost much more and take longer to receive the test results.

Wilson Environmental Labs: This environmental laboratory runs PCB tests at the request of its customers. It does not run PCB tests on a regular basis; only three jobs have required them in the past year. Staff do not consider themselves experts at PCB testing, and since they perform the test infrequently, it is important to them to have a test that is easy to use and accurate. They reported that they have found the Hach test kit to be cost effective and easy to use.

Data Quality Objective Example

This application of Hach's PCB immunoassay kit is based on data quality objective (DQO) methods for project planning advocated by ASTM [11, 12] and EPA [13]. The example (given in Appendix E) illustrates the use of Hach's performance data from the ETV demonstration in the DQO process to select the number of samples to characterize the decision rule's false positive and false negative error rates.

Section 7
References

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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)

Appendix B
Characterization of Environmental Soil Samples

Table B-1. Summary of environmental soil characterization

Location	Sample ID	RFD Drum # [a]	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 [b]	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 [b]	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, 204 117, 205	97002-02 97002-02S [b]	0.4	93.7	5.8	4075	7.43
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
	216 211 217	7515-0538 7515-0538S [b] 7515-0538S [b]	0.5	73.3	26.3	1328	7.78
	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

[a] Request for disposal drum number (see Table A-1).
[b] "S" indicates that the environmental soil was spiked with additional PCBs.

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 (%)
7/22/97	85	62	70 [a]	38 [a]
7/23/97	85	70	60 [a]	58 [a]
7/24/97	85	67	58	66
7/25/97	80	70	56	54
7/26/97	85	55	57	51
7/27/97	80	75	55	49
7/28/97	79	88	57	52
7/29/97	**b**	**b**	55	50

[a] The chamber was not operating properly on this day. See discussion in Section 3.
[b] No developers were working outdoors on this day.

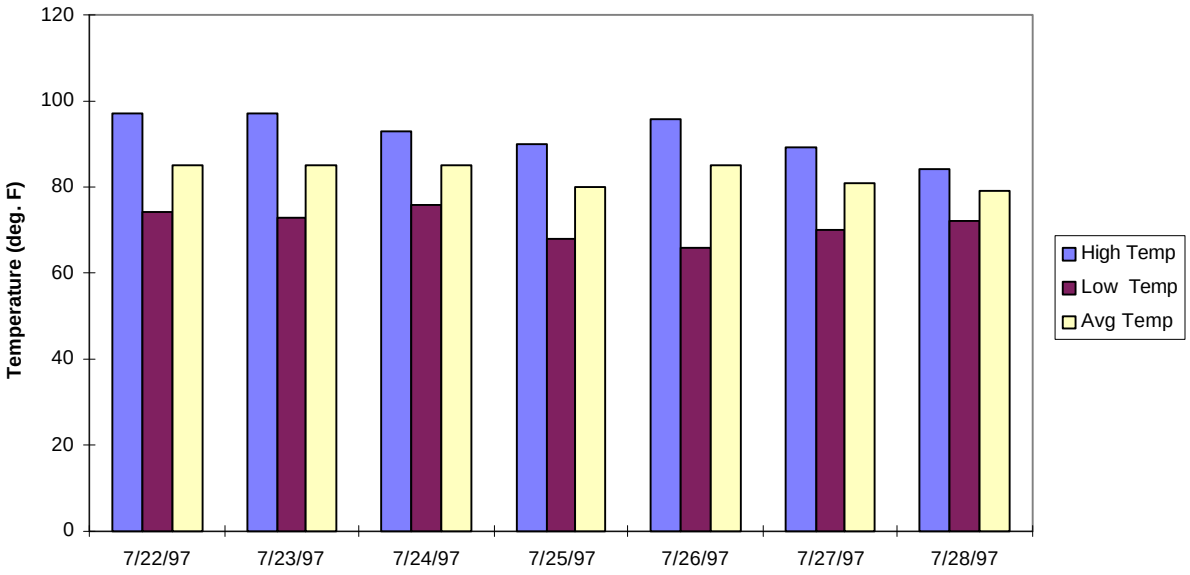

Figure C-1. Summary of temperature conditions for outdoor site.

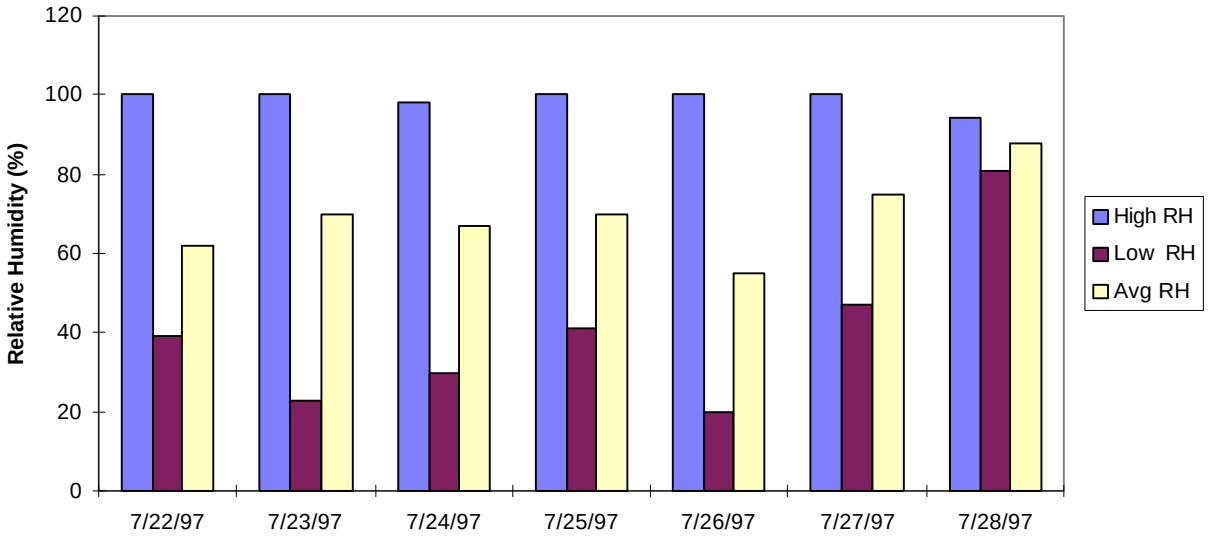

Figure C-2. Summary of relative humidity conditions for the outdoor site.

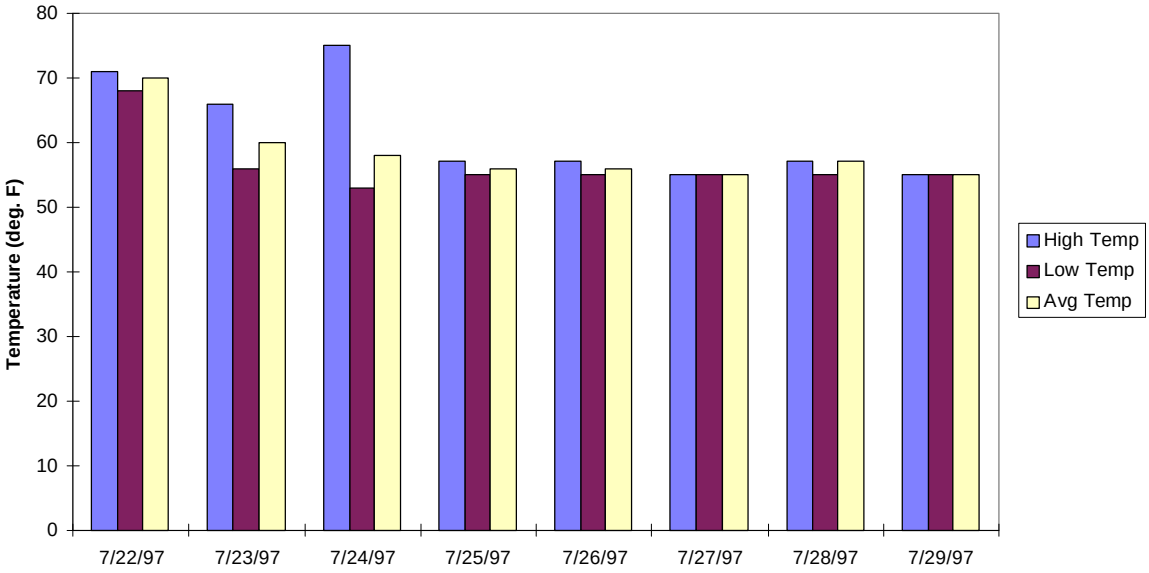

Figure C-3. Summary of temperature conditions for chamber site.

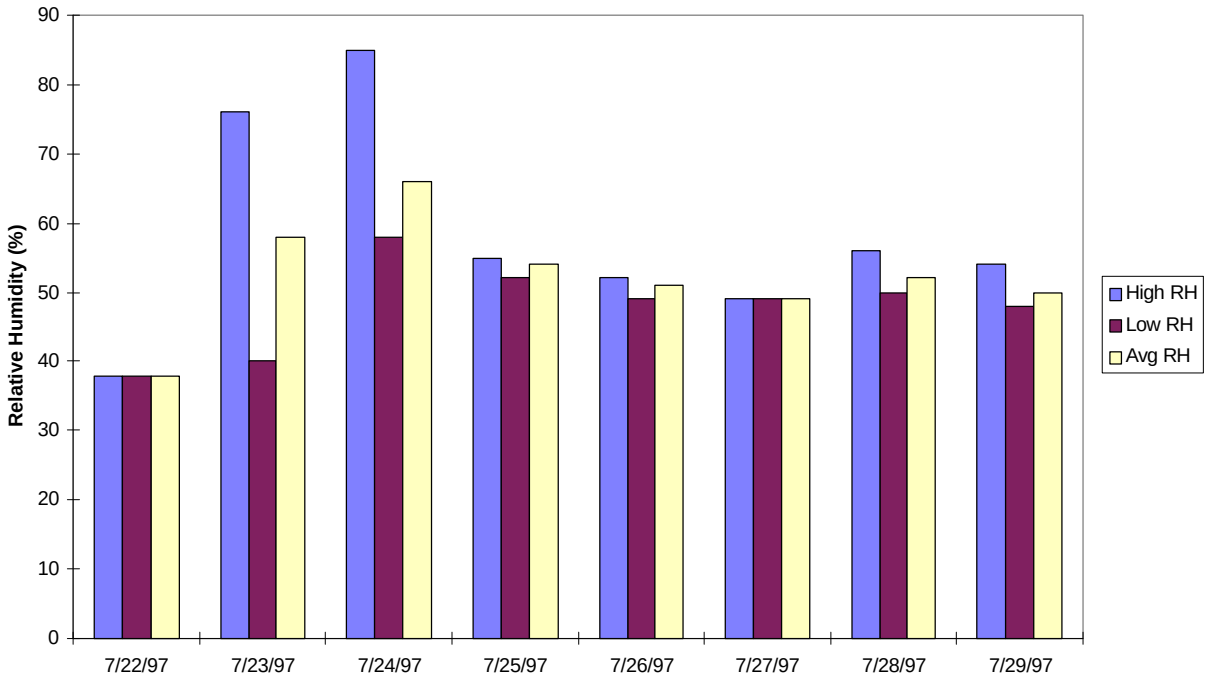

Figure C-4. Summary of relative humidity conditions for chamber site.

Appendix D
Hach's PCB Immunoassay Kit
Technology Demonstration Sample Data

Legend for Appendix D Tables

Table Heading	Definition
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)
Hach Result	Hach's measured PCB concentration range (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 1242, 1248, 1254, 1260 = Aroclor in PE samples Blank = non-PCB-contaminated sample
Order	Order of sample analysis (started with 2001–2116, then 1001–1116)

Table D-1. Hach's PCB technology demonstration soil sample data

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
1	101	1	[0,1)	0.6	1254	Sample	1081
2	101	2	[1.0,10.0]	0.4	1254	Sample	1036
3	101	3	[0,1)	0.5	1254	Sample	1034
4	101	4	[1.0,10.0]	0.5	1254	Sample	1093
5	102	1	[1.0,10.0]	2.2	1254	Sample	1042
6	102	2	[1.0,10.0]	2.1	1254	Sample	1048
7	102	3	[1.0,10.0]	1.7	1260	Sample	1096
8	102	4	[1.0,10.0]	2.5	1260	Sample	1040
9	103	1	[1.0,10.0]	3.0	1254	Sample	1026
10	103	2	[1.0,10.0]	2.4	1254	Sample	1067
11	103	3	[1.0,10.0]	2.0	1260	Sample	1045
12	103	4	[1.0,10.0]	1.6	1260	Sample	1086
13	104	1	(10.0,∞)	6.8	1260	Sample	1017
14	104	2	[1.0,10.0]	6.0	1254	Sample	1082
15	104	3	(10.0,∞)	14.8	1254	Sample	1059
16	104	4	(10.0,∞)	9.9	1254	Sample	1051
17	105	1	(10.0,∞)	49.7	1260	Sample	1089
18	105	2	(10.0,∞)	84.1	1260	Sample	1037
19	105	3	(10.0,∞)	50.6	1260	Sample	1098
20	105	4	(10.0,∞)	53.2	1260	Sample	1030
21	106	1	(10.0,∞)	269.6	1254	Sample	1039
22	106	2	(10.0,∞)	255.9	1254	Sample	1001
23	106	3	(10.0,∞)	317.6	1254	Sample	1065
24	106	4	(10.0,∞)	649.6	1254	Sample	1032
25	107	1	[1.0,10.0]	1.0	1254	Sample	1049
26	107	2	[1.0,10.0]	1.6	1254	Sample	1084
27	107	3	[1.0,10.0]	1.2	1254	Sample	1023
28	107	4	[1.0,10.0]	1.2	1254	Sample	1078
29	108	1	[1.0,10.0]	1.7	1254	Sample	1069
30	108	2	[1.0,10.0]	2.0	1254	Sample	1055
31	108	3	[1.0,10.0]	1.7	1254	Sample	1102
32	108	4	[1.0,10.0]	1.9	1254	Sample	1033
33	109	1	[1.0,10.0]	1.5	1254	Sample	1101
34	109	2	[1.0,10.0]	2.1	1254	Sample	1007
35	109	3	[1.0,10.0]	1.8	1254	Sample	1044
36	109	4	[1.0,10.0]	2.4	1254	Sample	1097
37	110	1	(10.0,∞)	≤490	Non-Detect	Sample	1077
38	110	2	(10.0,∞)	≤99	Non-Detect	Sample	1063
39	110	3	(10.0,∞)	≤66	Non-Detect	Sample	1083
40	110	4	[1.0,10.0]	≤98	Non-Detect	Sample	1100

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
41	111	1	(10.0,∞)	44.5	1254	Sample	1015
42	111	2	(10.0,∞)	36.0	1254	Sample	1006
43	111	3	(10.0,∞)	39.3	1254	Sample	1002
44	111	4	(10.0,∞)	35.1	1254	Sample	1035
45	112	1	(10.0,∞)	≤66	Non-Detect	Sample	1064
46	112	2	(10.0,∞)	≤200	Non-Detect	Sample	1027
47	112	3	(10.0,∞)	≤130	Non-Detect	Sample	1080
48	112	4	(10.0,∞)	≤200	Non-Detect	Sample	1068
49	113	1	[1.0,10.0]	0.7	1260	Sample	1054
50	113	2	[1.0,10.0]	1.1	1260	Sample	1061
51	113	3	[1.0,10.0]	0.6	1260	Sample	1071
52	113	4	[1.0,10.0]	1.9	1248/1260	Sample	1060
53	114	1	[1.0,10.0]	1.1	1260	Sample	1028
54	114	2	[0,1)	1.2	1260	Sample	1099
55	114	3	[1.0,10.0]	1.3	1260	Sample	1085
56	114	4	[1.0,10.0]	1.7	1260	Sample	1047
57	115	1	[1.0,10.0]	14.9	1248	Sample	1014
58	115	2	(10.0,∞)	12.4	1016	Sample	1021
59	115	3	[1.0,10.0]	15.0	1248	Sample	1062
60	115	4	[1.0,10.0]	16.9	1248	Sample	1010
61	116	1	(10.0,∞)	41.4	1248	Sample	1076
62	116	2	(10.0,∞)	41.2	1016	Sample	1012
63	116	3	(10.0,∞)	48.5	1248	Sample	1003
64	116	4	(10.0,∞)	34.0	1016	Sample	1104
65	117	1	(10.0,∞)	431.6	1016	Sample	1019
66	117	2	(10.0,∞)	406.3	1016	Sample	1009
67	117	3	(10.0,∞)	304.7	1016	Sample	1074
68	117	4	(10.0,∞)	392.8	1016	Sample	1016
69	118	1	[1.0,10.0]	2.1	1248	1248	1066
70	118	2	[1.0,10.0]	1.9	1016	1248	1073
71	118	3	[1.0,10.0]	0.7	1248	1248	1094
72	118	4	[1.0,10.0]	1.6	1248	1248	1031
73	119	1	(10.0,∞)	21.2	1016	1248	1052
74	119	2	(10.0,∞)	17.2	1248	1248	1008
75	119	3	(10.0,∞)	17.4	1248	1248	1092
76	119	4	(10.0,∞)	24.4	1248	1248	1072
77	120	1	[1.0,10.0]	4.5	1254	1254	1041
78	120	2	[1.0,10.0]	4.0	1254	1254	1046
79	120	3	[1.0,10.0]	6.3	1254	1254	1053
80	120	4	[1.0,10.0]	5.0	1254	1254	1025

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
81	121	1	(10.0,∞)	58.7	1254	1254	1020
82	121	2	(10.0,∞)	55.7	1254	1254	1050
83	121	3	(10.0,∞)	53.2	1254	1254	1011
84	121	4	(10.0,∞)	50.9	1254	1254	1057
85	122	1	(10.0,∞)	12.2	1260	1260	1091
86	122	2	(10.0,∞)	10.9	1260	1260	1103
87	122	3	(10.0,∞)	11.3	1260	1260	1095
88	122	4	(10.0,∞)	10.0	1260	1260	1018
89	123	1	(10.0,∞)	59.2	1260	1260	1090
90	123	2	(10.0,∞)	56.9	1260	1260	1022
91	123	3	(10.0,∞)	66.8	1260	1260	1038
92	123	4	(10.0,∞)	57.5	1260	1260	1024
93	124	1	[1.0,10.0]	1.8	1254	1254/1260	1088
94	124	2	[1.0,10.0]	1.4	1260	1254/1260	1043
95	124	3	[1.0,10.0]	1.9	1254	1254/1260	1087
96	124	4	[1.0,10.0]	1.8	1254	1254/1260	1075
97	125	1	(10.0,∞)	32.0	1254	1254/1260	1005
98	125	2	(10.0,∞)	41.3	1254	1254/1260	1029
99	125	3	(10.0,∞)	46.0	1254	1254/1260	1056
100	125	4	(10.0,∞)	32.2	1260	1254/1260	1070
101	126	1	[0,1)	≤0.1	Non-Detect	Blank	1013
102	126	2	[0,1)	≤0.1	Non-Detect	Blank	1079
103	126	3	[1.0,10.0]	≤0.2	Non-Detect	Blank	1004
104	126	4	[1.0,10.0]	≤1.3	Non-Detect	Blank	1058
105	201	1	[1.0,10.0]	1.0	1016/1260	Sample	2057
106	201	2	[0,1)	1.0	1016/1260	Sample	2027
107	201	3	[0,1)	1.1	1016/1260	Sample	2069
108	201	4	[1.0,10.0]	0.6	1260	Sample	2012
109	202	1	[1.0,10.0]	1.4	1260	Sample	2097
110	202	2	[1.0,10.0]	1.6	1260	Sample	2029
111	202	3	[0,1)	1.2	1260	Sample	2009
112	202	4	[1.0,10.0]	1.5	1260	Sample	2040
113	203	1	[1.0,10.0]	14.0	1248	Sample	2076
114	203	2	[1.0,10.0]	12.8	1248	Sample	2043
115	203	3	[1.0,10.0]	16.2	1248	Sample	2061
116	203	4	[1.0,10.0]	12.4	1248	Sample	2060
117	204	1	(10.0,∞)	43.1	1248	Sample	2011
118	204	2	[1.0,10.0]	45.3	1248	Sample	2086
119	204	3	(10.0,∞)	41.0	1248	Sample	2064
120	204	4	(10.0,∞)	47.7	1248	Sample	2047

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
121	205	1	[1.0,10.0]	3305.0	1016	Sample	2010
122	205	2	(10.0,∞)	538.7	1016	Sample	2034
123	205	3	(10.0,∞)	457.0	1016	Sample	2026
124	205	4	(10.0,∞)	483.3	1016	Sample	2088
125	206	1	[1.0,10.0]	2.9	1260	Sample	2053
126	206	2	[1.0,10.0]	1.1	1260	Sample	2007
127	206	3	[1.0,10.0]	1.1	1016/1260	Sample	2104
128	206	4	[1.0,10.0]	2.5	1260	Sample	2085
129	207	1	(10.0,∞)	17.8	1260	Sample	2099
130	207	2	(10.0,∞)	14.3	1260	Sample	2041
131	207	3	(10.0,∞)	21.6	1260	Sample	2048
132	207	4	(10.0,∞)	21.6	1254	Sample	2033
133	208	1	(10.0,∞)	42.0	1260	Sample	2042
134	208	2	(10.0,∞)	27.7	1016/1260	Sample	2005
135	208	3	(10.0,∞)	24.0	1254	Sample	2035
136	208	4	(10.0,∞)	28.4	1260	Sample	2015
137	209	1	(10.0,∞)	32.7	1260	Sample	2093
138	209	2	(10.0,∞)	79.3	1260	Sample	2082
139	209	3	(10.0,∞)	11.0	1260	Sample	2103
140	209	4	(10.0,∞)	37.9	1260	Sample	2096
141	210	1	(10.0,∞)	123.2	1260	Sample	2101
142	210	2	(10.0,∞)	61.5	1260	Sample	2025
143	210	3	(10.0,∞)	84.1	1260	Sample	2046
144	210	4	(10.0,∞)	85.5	1260	Sample	2030
145	211	1	(10.0,∞)	387.8	1254	Sample	2075
146	211	2	(10.0,∞)	581.4	1254	Sample	2079
147	211	3	(10.0,∞)	330.0	1254	Sample	2039
148	211	4	(10.0,∞)	318.7	1254	Sample	2037
149	212	1	[1.0,10.0]	3.8	1260	Sample	2089
150	212	2	(10.0,∞)	3.9	1260	Sample	2095
151	212	3	[1.0,10.0]	4.3	1260	Sample	2098
152	212	4	[1.0,10.0]	0.8	1260	Sample	2081
153	213	1	[1.0,10.0]	6.9	1260	Sample	2016
154	213	2	[1.0,10.0]	7.3	1260	Sample	2080
155	213	3	[1.0,10.0]	7.8	1260	Sample	2068
156	213	4	(10.0,∞)	10.5	1260	Sample	2014
157	214	1	(10.0,∞)	26.0	1260	Sample	2023
158	214	2	(10.0,∞)	25.6	1260	Sample	2008
159	214	3	(10.0,∞)	29.1	1260	Sample	2013
160	214	4	(10.0,∞)	20.2	1260	Sample	2045

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
161	215	1	(10.0,∞)	25.1	1260	Sample	2024
162	215	2	(10.0,∞)	24.1	1260	Sample	2066
163	215	3	(10.0,∞)	26.2	1260	Sample	2051
164	215	4	(10.0,∞)	31.2	1016/1260	Sample	2031
165	216	1	(10.0,∞)	151.6	1260	Sample	2059
166	216	2	(10.0,∞)	47.0	1260	Sample	2094
167	216	3	(10.0,∞)	54.3	1260	Sample	2002
168	216	4	(10.0,∞)	64.0	1260	Sample	2022
169	217	1	(10.0,∞)	886.7	1254	Sample	2072
170	217	2	(10.0,∞)	549.8	1254	Sample	2020
171	217	3	(10.0,∞)	542.8	1254	Sample	2078
172	217	4	(10.0,∞)	1913.3	1016/1260	Sample	2017
173	218	1	[1.0,10.0]	2.8	1248	1248	2032
174	218	2	[1.0,10.0]	2.4	1248	1248	2058
175	218	3	[1.0,10.0]	2.6	1248	1248	2044
176	218	4	[1.0,10.0]	2.6	1248	1248	2084
177	219	1	(10.0,∞)	22.4	1248	1248	2003
178	219	2	(10.0,∞)	26.0	1016	1248	2021
179	219	3	(10.0,∞)	29.4	1248	1248	2052
180	219	4	(10.0,∞)	15.2	1248	1248	2050
181	220	1	[1.0,10.0]	8.5	1254	1254	2036
182	220	2	[1.0,10.0]	4.9	1254	1254	2063
183	220	3	[1.0,10.0]	4.7	1254	1254	2019
184	220	4	[1.0,10.0]	5.2	1254	1254	2065
185	221	1	(10.0,∞)	32.0	1016/1260	1254	2077
186	221	2	(10.0,∞)	44.1	1016/1260	1254	2018
187	221	3	(10.0,∞)	43.8	1254	1254	2102
188	221	4	(10.0,∞)	59.6	1254	1254	2067
189	222	1	[1.0,10.0]	13.2	1260	1260	2073
190	222	2	[1.0,10.0]	12.4	1260	1260	2062
191	222	3	[1.0,10.0]	12.7	1260	1260	2004
192	222	4	[1.0,10.0]	12.7	1260	1260	2091
193	223	1	(10.0,∞)	56.6	1260	1260	2049
194	223	2	(10.0,∞)	50.3	1260	1260	2056
195	223	3	(10.0,∞)	49.9	1260	1260	2090
196	223	4	(10.0,∞)	66.4	1260	1260	2092
197	224	1	[1.0,10.0]	2.2	1254	1254/1260	2074
198	224	2	[1.0,10.0]	1.2	1260	1254/1260	2001
199	224	3	[1.0,10.0]	1.4	1260	1254/1260	2038
200	224	4	[1.0,10.0]	2.1	1254	1254/1260	2028

Obs	Sample ID	Rep	Hach Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
201	225	1	(10.0,∞)	56.4	1260	1254/1260	2100
202	225	2	(10.0,∞)	36.5	1016/1260	1254/1260	2054
203	225	3	(10.0,∞)	32.1	1260	1254/1260	2083
204	225	4	(10.0,∞)	146.0	1254	1254/1260	2006
205	226	1	[1.0,10.0]	≤0.1	Non-Detect	Blank	2055
206	226	2	[0,1)	≤0.8	Non-Detect	Blank	2087
207	226	3	[0,1)	≤0.1	Non-Detect	Blank	2070
208	226	4	[0,1)	≤0.1	Non-Detect	Blank	2071

Table D-2. 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[a]	≤0.7	0.5
	101[a]	≤0.7	0.6
	107	≤1.3	1.2
	109	18.0	1.5
	113[b]	≤0.9	0.6
	113[b]	≤1.0	0.7
	119	18.0	21.2
	127	7.2	10.9
	201	≤ 1.0	0.6
	219	21.0	26.0

[a] Two of four measurements in Sample ID 101 were corrected.
[b] Two of four measurements in Sample ID 113 were corrected.

Appendix E
Data Quality Objective Example

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 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 will be homogeneous. In the "real world," however, this assumption is seldom valid, and matrix heterogeneity constitutes a source of considerable uncertainty which 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. Preliminary evaluation determined that a number of PCB drums had to be incinerated to reduce or eliminate the PCB contamination. The incinerated soil was placed in drums for disposal in a landfill. However, a final check of each drum was required to verify for the regulator that the appropriate level of cleanup had been achieved. The regulator required that no drum have more than 2 ppm of PCB. The company's DQO team was considering the use of Hach's PCB kit to measure the PCB concentration in each drum. Soil samples would be randomly selected from each drum and tested with Hach's PCB kit to determine if the concentration was in one of the three intervals [0,1), [1,10], or (10,∞). Recall that this notation describes the concentration ranges 0 ppm ≤ PCB < 1 ppm, 1 ppm ≤ PCB ≤10 ppm, and PCB > 10 ppm, as used in Section 5. The DQO team decided that a drum would be reprocessed by incineration if any of Hach's results indicated a concentration in the intervals [1,10] or (10,∞). In agreement with regulators, the DQO team determined that a decision rule for disposal would be based on the number of samples with PCB concentrations in the intervals [1,10], and (10,∞).

General Decision Rule

If all of the PCB sample results show concentrations in [0, 1), then send the soil drum to the landfill.

If any of the PCB sample results are different than [0, 1) then reprocess the soil drum by incineration.

DQO Goals

EPA's *Guidance for Data Quality Assessment* [13] states in Sect. 1.2: "The true condition that occurs with the more severe decision error . . . should be defined as the null hypothesis." The DQO team decided that the more severe decision error would be for a drum to be erroneously sent to a landfill if the drum's PCB concentration actually exceeded the 2 ppm limit. Therefore, the null hypothesis is constructed to assume that the a drum's true PCB concentration exceeds the 2 ppm limit; and as a "hot" drum, it should be sent to the incinerator. Drums would be sent to the landfill only if the null hypothesis is rejected and it is concluded that the "true" average PCB concentration is less than 2 ppm.

With the null hypothesis defined in this way, a false positive decision is made when it is concluded that a drum contains less than 2 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 sending a "hot" drum to the

landfill (i.e., the false positive error rate for the decision) could not be more than 5%. 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 2 ppm. This scenario represents a 5% chance of sending a drum containing 2 ppm or more of PCBs to the landfill. The Hach interval boundary of 1 ppm can be used as a conservative estimate of the 2 ppm criterion.

The DQO team did not want to reprocess an excessive number of drums by incineration if the drum PCB concentration was less than 2 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 less than 2 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 [13], the DQO team recommended that the false negative decision error rate (FN) for the decision rule be 0.10 if the true drum concentration was less than 1 ppm. That is, there would be a 10% chance of reprocessing a drum by incineration if the true PCB concentration for a drum was less than 1 ppm.

Permissible FP and FN Error Rates and Critical Decision Point

FP: Pr[Take Drum to Landfill] ≤ 0.05 when true PCB concentration 1 ppm

FN: Pr[Reprocess Drum in Incinerator] ≤ 0.10 when true PCB concentration < 1 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 Hach's PCB kit, the performance of this technology (as reported in this 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, then, to be considered in this example is the variability in performance of the Hach 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 Hach's PCB kit. Two types of misclassifications have to be considered (1) underestimating the PCB concentration—classifying a sample concentration in [0, 1) when the true PCB concentration is greater or equal to 1ppm, and (2) overestimating the PCB concentration—classifying a sample concentration in [1, 10] or (10, ∞) when the PCB concentration is less than 1 ppm. The ETV demonstration results on performance evaluation soil samples and on environmental soil samples indicated the error rates for the two types of misclassifications to be P_U = Pr[Underestimating the PCB concentration] = 0.022 and P_O = Pr[Overestimating the PCB concentration] = 0.588.

The probability distribution of classifying the number of soil samples in different concentration intervals follows a binomial probability distribution [7, pg. 162-170]. 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

$$FP = Pr[\ All\ Hach's\ results < 1\ ppm\ for\ PCB \quad 1\ ppm\ ] = (P_U)^n \tag{E-1}$$

The FP error rate decreases as the sample size increases. Rearranging to solve for sample size, *n*, Equation E-1 becomes

$$n = \frac{Log(FP)}{Log(P_U)} \tag{E-2}$$

where

n	=	number of samples from a drum to be measured,
FP	=	false positive decision error rate (e.g., FP = 0.05), and
P_U	=	probability of underestimating the PCB concentration (e.g., P_U = 0.022).

Incorporating the appropriate values for the Hach PCB immunoassay kit into Equation E-2 gives

$$n = \frac{Log(0.05)}{Log(0.022)} = \frac{-1.301}{-1.658} = 0.785 \quad 1 \quad .$$

The DQO team would have to take one sample to meet the FP requirement. The FN for the decision rule is related to P_O by

$$FN = Pr[\ Some\ of\ Hach's\ results \quad 1\ ppm\ for\ PCB < 1\ ppm\ ] = 1 - (1 - P_O)^n \tag{E-3}$$

The probability of a false negative decision (FN = sending a drum for reprocessing) actually increases with increasing sample size because the chance of the kit overestimating a result increases with continued testing. The sample size required to meet the FN requirement is

$$n = \frac{Log(1 - FN)}{Log(1 - P_O)} \tag{E-4}$$

where

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

$$n = \frac{Log(1 - 0.10)}{Log(1 - 0.588)} = \frac{-0.046}{-0.385} = 0.119 \quad 1.$$

The sample size must be rounded up to $n = 1$. When $n = 1$, and the above equation is solved for FN, it is found that the DQO team cannot meet their goal of 10% FN, and would have to accept an FN of 59%. This situation occurs because of the 59% overestimation error rate of the kit. If a decision about a drum is based on a single sample, and that sample has a 59% chance of being overestimated, there is consequently a 59% chance that the drum will be unnecessarily sent for reprocessing through the incinerator (which was the definition of FN). Although this amount of conservatism may be desirable in some situations, in others it may not be. The only way to reduce the FN in this kind of scenario is to use an analytical technology with a lower overestimation error rate. The DQO team in our example decided that the sampling procedure would be to randomly select one soil sample from each drum and test the sample with Hach's PCB kit.

The DQO team would send the drum to the landfill if the result was less than 1 ppm and send the drum to be reprocessed by incineration if the result was greater than 1 ppm. To meet a 5% FP requirement, the DQO team would have to accept the FN of 59%.

Decision Rule for 5% FP and 59% FN

If one randomly selected soil sample has a PCB test result reported as the interval [0, 1) then send the soil drum to the landfill.

If one randomly selected soil sample has a PCB test result different than [0, 1) then reprocess the soil drum by incineration.

Alternative FP Parameter

The following 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 $n = 1.8$ 2. For this case, the sample size for FP would be rounded up to $n = 2$. The FN would be 83% which is larger than the FN specified by the DQO team. The higher FN occurs because each of the two samples has a 59% chance of being overestimated, and therefore there is an 83% chance that one of the two samples from a drum will be overestimated. Even if only one is overestimated, the drum is sent for reprocessing. The decision rule for the lower FP would be as shown below.

Decision Rule for FP = 0.1% and FN = 83%

If two randomly selected soil samples have PCB test results reported as the interval [0, 1) then send the soil drum to the landfill.

If either of the two randomly selected soil samples have PCB test results different than [0, 1) then reprocess the soil drum by incineration.