♻EPA

Environmental Technology Verification Report

Immunoassay Kit

Strategic Diagnostics Inc. D TECH PCB Test Kit

ETV ETV ETV

Environmental Technology Verification Report

Immunoassay Kit

Strategic Diagnostics Inc.
D TECH PCB Test Kit

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

SITE

Superfund Innovative Technology
Evaluation Program

ornl

Notice

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

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

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

ET✔

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**POLYCHLORINATED BIPHENYL (PCB) FIELD ANALYTICAL TECHNIQUES**
APPLICATION:	**MEASUREMENT OF PCBs IN SOILS AND SOLVENT EXTRACTS**
TECHNOLOGY NAME:	**D TECH PCB TEST KIT**
COMPANY: ADDRESS:	**STRATEGIC DIAGNOSTICS INC.** **111 PENCADER DRIVE** **NEWARK, DE 19702-3322**
PHONE:	**(302) 456-6789**

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 Strategic Diagnostics Inc. (SDI) D TECH PCB test kit.

PROGRAM OPERATION

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

The demonstration was designed to detect and measure PCBs in soil and solvent extracts. The demonstration was conducted at the Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee, from July 22 through July 29. The study was conducted under two environmental conditions. The first site was outdoors, with naturally fluctuating temperatures and relative humidity conditions. The second site was inside a controlled environmental chamber, with generally cooler temperatures and lower relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed in this study. Solutions of PCBs were also analyzed to simulate extracted surface wipe

samples. The results of the soil and extract analyses conducted under field conditions by the technology were compared with results from analyses of 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, Strategic Diagnostics, Inc., D TECH PCB Test Kit,* EPA/600/R-98/112.

TECHNOLOGY DESCRIPTION

The D TECH PCB test kit is designed to provide quick, semi-quantitative, and reliable test results for making environmental decisions. The D TECH kit utilizes immunoassay technology to detect trace amounts of PCBs in environmental samples. This test specifically detects Aroclors 1254, 1260, and 1262 equally; reacts well with Aroclors 1242, 1248, and 1268; reacts moderately with Aroclors 1232 and 1016; and shows little reactivity to Aroclor 1221. The test is calibrated for Aroclor 1254 and has conversions for Aroclors 1242 and 1248. The D TECH PCB test kit uses latex particles as the solid support component of the assay. With this immunoassay system, antibodies are immobilized on the thousands of latex particles that are free to interact and react with the sample solution. To initiate the test, a sample solution (soil extract) is added to a dry mixture of antibody-coated latex particles and enzyme conjugate. A competitive reaction for binding sites on the antibodies then occurs between the analyte PCBs (in the sample solution) and the added enzyme conjugate. When the reaction is complete, the particles are collected on the membrane surface of a collection cup and briefly washed. The test is completed by adding a color developing solution to the surface of the collection cup. As with the other immunoassay systems, the enzyme conjugate produces a color change reaction which can then be detected and measured. The darker the color, the less analyte PCB is present in the sample. Measurement of the test results can be completed instrumentally with a reflectance meter or visually with the included color card.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the D TECH PCB test kit were observed:

Throughput: Throughput was 11 samples/hour in the chamber and 15 samples/hour outdoors. This rate included sample preparation and analysis.

Ease of Use: Three operators analyzed samples during the demonstration, but the technology can be run by a single operator. Minimal training (2 to 4 hours) is required to operate the D TECH kit, provided the user has a fundamental understanding of basic chemical and field analytical techniques.

Completeness: The D TECH kit generated results for all 232 PCB samples, for a completeness of 100%.

Blank results: PCBs were detected above the lowest reporting interval for five of the eight blank soil samples. Therefore, the percentage of false positive results was 62%. Two false positive results (25%) were reported for the extract samples. The D TECH kit reported 0.5% false negative results for soils and 0% for extracts.

Precision: The overall precision, based on the percentage of combined sample sets where all four replicates were reported as the same interval, was 44% for the PE soils, 21% for the environmental soils, and 25% for the extracts.

Accuracy: Accuracy was assessed using PE soil and extract samples. Accuracy, defined as the percentage of D TECH results that agreed with the accepted concentrations, was 56% for PE soils and 42% for extracts. The percentage of samples that was biased high was greater (28%) than the percentage that was biased low (17%) for the PE soil samples, while the percentage that was biased high or low was comparable (29%) for the extract samples.

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 samples that agreed with

the reference laboratory results was 53% for all soils (PE and environmental) and 42% for extracts. The percentage of samples that was biased high was greater (28%) than the percentage that was biased low (20%) for the soil samples, while the percentage that was biased high or low was again comparable (29%) for the extract samples.

Regulatory decision-making: One objective of this demonstration was to assess the technology's ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils and 100 μg/100cm^2 for surface wipes. For PE and environmental soil samples in the range of 40 to 60 ppm, 61% of the D TECH results agreed with the reference laboratory. By contrast, 7% were biased high, while 32% were biased low. For the extract samples representing surface wipe sample concentrations of 100 μg/100 cm^2 and 1000 μg/100 cm^2 (assuming a 100 cm^2 wipe sample), 42% of the D TECH results agreed with the extract spike concentration. In comparison, the percentage of extract samples biased high or low was comparable (29%).

Data quality levels: The performance of the D TECH PCB test kit was characterized as biased, with approximately 50% of the D TECH results agreeing with the accepted values (in terms of accuracy), and imprecise, with consistently less than 50% replicate sample results reported as the same interval.

The results of the demonstration show that the D TECH PCB test 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

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). Extract samples were used to simulate surface wipe samples, and were evaluated at concentrations ranging from 0 to 100 µg/mL. 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 SDI's D TECH PCB test kit. Separate ETVRs have been published for the other technologies demonstrated.

The D TECH kit utilizes immunoassay technology to detect trace amounts of PCBs, determined as interval threshold values, in environmental samples. This test specifically detects Aroclors 1254, 1260, and 1262 equally; reacts well with Aroclors 1242, 1248, and 1268; reacts moderately with Aroclors 1232 and 1016; and shows little reactivity to Aroclor 1221. The D TECH PCB test kit uses latex particles, onto which antibodies are immobilized, as the solid support component of the assay. The test is calibrated for Aroclor 1254 and has conversions for Aroclors 1242 and 1248. As with the other immunoassay systems, the added enzyme conjugate produces a color change reaction that can then be detected and measured. The darker the color, the less analyte PCB is present in the sample. Measurement of the test results can be completed instrumentally with a reflectance meter or visually with the included color card. The D TECH kit provides no information on Aroclor identification.

The D TECH's quantitative results were based on the analysis of threshold standards for Aroclor 1254 that were analyzed with every four samples. Because the D TECH kit is an interval technique, method detection limits are not applicable. Precision, defined as the percentage of the sample sets where all four replicates were reported as the same interval range, was 44% for the PE soils, 21% for the environmental soils, and 25% for the extracts. Accuracy, defined as the percentage of D TECH results that agreed with the accepted concentrations, was 56% for PE soils and 42% for extracts. In general, the percentage that was biased high was greater (28%) than the percentage that was biased low (17%) for the PE soil samples, while the percentage that was biased high or low was comparable (29%) for the extract samples. Comparability was defined in a way similar to accuracy, but the D TECH results were compared to the reference laboratory results rather than to the certified concentrations. For all soil samples (PE and environmental), the percentage of D TECH results that agreed with the reference laboratory results was 53%, while the percentage that was biased high (28%) was slightly greater than the percentage that was biased low (20%).

The demonstration found that the D TECH kit was simple to operate in the field, requiring about one hour for initial setup and preparation for sample analysis. Once operational, the D TECH kit delivered a sample throughput of 11 samples/hour under chamber conditions and 15 samples/hour under outdoor conditions. Three operators analyzed samples during the demonstration, but the technology can be run by a single operator. Minimal training (2 to 4 hours) is required to operate the D TECH kit, provided the user has a fundamental understanding of basic chemical and field analytical techniques. The overall performance of the D TECH PCB test kit was characterized as biased, with approximately 50% of the D TECH results agreeing with the accepted values (in terms of accuracy), and imprecise, with consistently less than 50% replicate sample results reported as the same interval.

Table of Contents

List of Figures

List of Tables

List of Abbreviations and Acronyms

AL   action level

ANOVA   analysis of variance

ASTM   American Society for Testing and Materials

BHC   benzenehexachloride

C   concentration at which the false positive error rate is specified

CASD   Chemical and Analytical Sciences Division (ORNL)

CCV   continuing calibration verification standard

CSCT   Consortium for Site Characterization Technology

DCB   decachlorobiphenyl

DOE   U.S. Department of Energy

DQO   data quality objective

ELISA   enzyme-linked immunosorbent assay

EM   Environmental Management (DOE)

EPA   U.S. Environmental Protection Agency

ERA   Environmental Resource Associates

EST   Electronic Sensor Technology

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
GC	gas chromatography
HEPA	high-efficiency particulate air
ID	identifier
LCS	laboratory control sample
LMER	Lockheed Martin Energy Research
LMES	Lockheed Martin Energy Systems
LV	Las Vegas
MDL	method detection limit
MS	matrix spike
MSD	matrix spike duplicate
n	number of samples
NERL	National Exposure Research Laboratory (EPA)
NCEPI	National Center for Environmental Publications and Information
NRC	Nuclear Regulatory Commission
ORD	Office of Research and Development (EPA)
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)
RT	regulatory threshold
S^2	variance for the measurement
SARA	Superfund Amendments and Reauthorization Act of 1986
SD	standard deviation
SDI	Strategic Diagnostics Inc.
SITE	Superfund Innovative Technology Evaluation
SMO	sample management office
SOP	standard operating procedure
SSM	synthetic soil matrix
TCMX	tetrachloro-m-xylene
TSCA	Toxic Substance Control Act
$Z_{1\text{-}p}$	the $(1-p)$th percentile for the standard normal distribution
%D	percent difference

Acknowledgments

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.

1

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, SDI's D TECH PCB test 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
This section describes 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, which may or may not be substantiated by the data presented in Section 5.

Principle
The D TECH PCB test kit applies the principles of enzyme-linked immunosorbent assay (ELISA) to the determination of PCBs. 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 remain are either PCB-antibody complexes or enzyme-PCB- antibody complexes attached to the test surface. The proportion of the two complexes on the test surface is determined by the amount of native PCB in the original sample. The enzyme present on the test surface is used to catalyze a color change reaction in a solution added to the test surface. Because the amount of enzyme present is inversely proportional to the concentration of native PCB contaminant, the amount of color development is inversely proportional to the concentration of PCB contaminant.

The D TECH PCB test kit uses latex particles as the solid support component of the assay. With this kit, antibodies are immobilized on thousands of latex particles that are free to interact and react with the analyte and the enzyme conjugate sample extract. These particles are then separated on a filtration device, the color development is induced, and the results are read visually or with a small reflectance meter. In this kit, sample solution (i.e., soil extract) is added to a dry mixture of antibody-coated latex particles and enzyme conjugate. The immunological reaction then occurs in this reconstituted suspension. When the reaction is complete, the particles are collected on the membrane surface of a collection cup and washed briefly. The test is completed by adding a color-developing solution to the surface of the collection cup. As with the other immunoassay formats, the enzyme linked to the antibody catalyzes a color change reaction. Measurement of test results can be completed instrumentally with a reflectance meter or visually with the included color card.

The D TECH PCB test kit is designed to provide quick, semi-quantitative, and reliable results for making environmental decisions. The kit can screen for highly contaminated samples that require pre-dilution prior to instrumental analysis. The D TECH PCB test kit has a working range of 0.5 to 25 ppm for soil samples and a 10 to 250 µg/100 cm^2 for wipe samples. This test specifically detects Aroclors 1254, 1260,

and 1262 equally; reacts well with Aroclors 1242, 1248, and 1268; reacts moderately with Aroclors 1232 and 1016; and shows little reactivity to Aroclor 1221.

Test Kit Description
The D TECH PCB test kit contains sufficient materials to perform four soil tests. All the materials needed to extract PCB from soils for semi-quantitation are included. This kit has a working temperature range from 7° to 38°C (45° to 100°F). It should not be frozen or stored in direct sunlight.

Procedure
Sampling and Extraction
The D TECH soil sampling tube is used to collect field soil samples. To achieve a more homogeneous distribution and to ensure reproducible test results, the soil sample should be mixed thoroughly. All debris, such as sticks, stones, and leaves, should be removed from the soil sample before using the D TECH soil sampling tube. (Note: Because the soil demonstration samples were sandy and dry, 5 g of soil was weighed out and poured into the soil sampling tube.)

In the D TECH procedure the soil is dispensed into **bottle 1,** which already contains 9 mL of methanol liquid, by positioning the barrel into the neck of the bottle and firmly pushing the plunger (Figure 2-1). If soil lodges in the neck of the bottle, the sampling tube is used to push it into the bottle. The threads of the bottle neck and cap should be wiped clean of any soil that has adhered to them before the cap is placed on the bottle. The bottle is then capped tightly, and the soil and liquid methanol are thoroughly mixed by continuous shaking for 3 min. The soil is then allowed to settle for approximately 1 min (Figure 2-2). Some soils will settle more slowly than others.
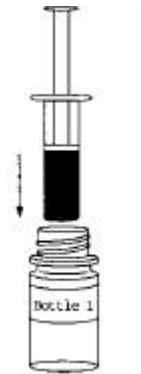
Figure 2-1. Dispensing soil into bottle 1.

The next sequence of steps, outlined in Figure 2-3, begins the extract analysis. The cap is removed from **bottle 2** (containing diatomaceous earth)**.** With the 2-mL **calibrated pipet**, 2 mL of the liquid layer is removed from **bottle 1** and dispensed into **bottle 2**; this solution is then mixed well. After the filter tip is snapped onto the neck of **bottle 2,** that bottle is squeezed to deliver the filtered solution into the **PCB test vial** to a level between the two lines (approximately 13-14 drops). At this point, the extract is prepared for assay.

Assay
Once the assay portion of the test is initiated, all steps must be executed sequentially and without stopping. The contents of **reagent C** (white cap) are squeezed to fill the **PCB reference vial** (red stopper) to a level between the 2 lines. The contents are
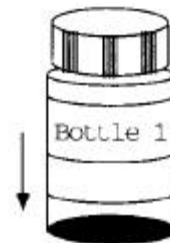
Figure 2-2. Allowing the soil to settle.

gently mixed by shaking the vial in a back and forth motion. After 5 min (±30 sec), the contents of the **PCB test vial** are poured into the **T** (test) side of the cup assembly (Figure 2-4). Then the contents of the **reference vial** are immediately poured into the **R** side of the cup assembly. The liquid is allowed to drain completely on both sides. The reference vial concentration is equivalent to the detection limit of

6
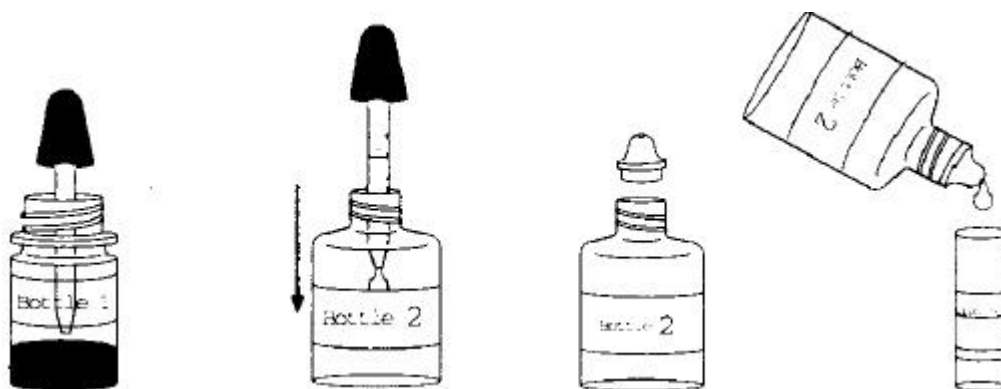
Figure 2-3. Diluting the extract.

the kit (0.5 ppm). For every sample analyzed, this vial is filled using a new bottle.

The next steps use dropper-tipped bottles. When dispensing these reagents, the analyst should not allow any dropper tip to contact any solution(s) or surface in the device. To assure uniform color development across the device, the drop should be dispensed onto the sloped side of the well to lessen its impact. The analyst should not allow the drop to fall into the middle of the well.

Figure 2-4. Filling the cup assembly.

Ten drops (±2 drops) of **reagent D** solution (yellow cap) are now added into each side of the cup assembly, and the liquid is allowed to drain completely. Then 5 drops (±1 drop) of **reagent E** solution (blue cap) are added into each side of the cup assembly. This solution must be added to the second well immediately after addition to the first well. Allow the wells to drain completely. Color development time is temperature-dependent and takes approximately 10 min at 75°F. More time is required at lower temperatures and less time is required at higher temperatures. The color in both wells is stable for approximately 4 h.

Determining PCB Concentration

The results from the D TECH PCB test kit are interpreted using the DTECHTOR and Table 2-1. First, the percent relative reflectance is determined by use of the DTECHTOR instrument. The DTECHTOR instrument is a hand-held reflectometer for interpreting the results of D TECH test samples. Completely portable and powered with a 9-V plug-in battery, the DTECHTOR uses a simple push-button procedure to provide results in 1 min. Given the reading of this instrument, the analyst then uses the data shown in Table 2-1 to determine the concentration range of total PCB in the sample. If the user does not know which Aroclor(s) are present in the sample, he or she should use the values for Aroclor 1254.

Table 2-1. Converting DTECHTOR readings to PCB concentration in soils

DTECHTOR Reading (% relative reflectance)	Aroclor Concentration Range (ppm)		
	1254	**1242**	**1248**
<10	<0.5	<1.5	<0.8
10–20	0.5–1.0	1.5–3.5	0.8–2.3
21–40	1.1–4.0	3.6–20	2.4–11
41–60	4.1–15	21–54	12–28
61–70	16–25	55–100	29–53
>70	>25	>100	>53

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 environmental soil) were analyzed. The ability to use

10

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

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 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 SDI team analyzed samples using the D TECH test kit outdoors July 26, and in the chamber on July 23.

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

12

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.

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.

13

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. SDI analyzed extracts prepared in methanol. 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).

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., SDI 1001 through SDI 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. SDI received information pertaining to which Aroclors were in the samples. This was provided at the request of SDI to simulate the type of information that would be available during actual field testing. 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 test kit's results for the predemonstration samples. Results indicated that SDI's D TECH PCB test kit was ready for field evaluation.

Table 3-2. Summary of D TECH predemonstration results

Sample Description	Matrix	Source	D TECH [a]		Reference Laboratory	
			Result (ppm)	Duplicate result (ppm)	Result (ppm)	Duplicate result (ppm)
2 ppm of Aroclor 1260	Soil	ORNL	[1, 4) [b]	[1, 4)	2.2	2.3
100 ppm (total) of Aroclors 1254 and 1260	Soil	ORNL	(25, ∞)	(25, ∞)	78.0	89.0
11 ppm of Aroclor 1260	Soil	EPA	[15, 25]	[15, 25]	11.0	9.5
50 ppm of Aroclor 1254	Soil	ERA	(25, ∞)	c	37.0	c
5 ppm of Aroclor 1242	Extract	ORNL	(3.5, 6.6)	(3.5, 6.6)	4.7	4.9

[a] Results were Aroclor-adjusted (see Section 2 for more details).
[b] The notation [1, 4) indicates sample concentration ≥1 and < 4. See Sections 2 and 5 for more information on interval reporting.
[c] Replicate was not analyzed because of lack of adequate sample for second analyses.

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 SDI team made one modification to the procedure described in the technology demonstration plan [5]: the demonstration samples were weighed instead of collected volumetrically using the D TECH soil sampling tube. This was necessary because of the dry, sandy characteristics of the soils.

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, 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 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 to the samples during preparation.

Surrogate Spike Results

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

$$percent\ recovery\ =\ \frac{measured\ amount}{actual\ amount}\ \times\ 100\% \tag{4-1}$$

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

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), a MS sample is an actual environmental soil sample into which target analytes are spiked at known concentrations. MS samples are used to assess the efficiency of the extraction and analytical methods for real samples. This is accomplished by determining the amount of spiked analyte that is quantitatively recovered from the environmental soil. A duplicate matrix spike (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:

$$RPD = \frac{\lfloor MS\ recovery - MSD\ recovery \rfloor}{average\ recovery} \times 100\% \tag{4-2}$$

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:

$$RSD = \frac{Standard\ Deviation}{Average\ Concentration} \times 100\% \tag{4-3}$$

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

25

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%).

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	b	b	b	215	26.7	3.2	12
111	38.7	4.3	11	216	55.1 (79.2)	8.5 (48.7)	15 (62)
112	b	b	b	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 parentheses include suspect values.
[b] 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 ≤ 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
This section presents the evaluation of the data generated by the D TECH PCB test 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. Evaluations of other aspects of the technology (such as cost, sample throughput, hazardous waste generation, and logistical operation) are also presented in this section.

Interval Reporting
The D TECH results were reported as concentration ranges that were designated as intervals incorporating parentheses/bracket notation. The parentheses indicate that the end points of the concentration range were excluded, while brackets indicate that the end points were included. For example, in Table 5-1 the interval (1, 4] indicates that the PCB concentration range is >1 and ≤4.

As discussed briefly in Section 2 of this report, this technology cannot distinguish between different Aroclors. The test kit has been calibrated to respond in a one-to-one ratio with Aroclor 1254. If site history or information indicates that Aroclor 1242 or 1248 is present in the sample, a conversion can be applied (see Section 2 for more information). The Aroclor-specific reporting intervals for the D TECH results are listed in Table 5-1. For the purposes of the demonstration, SDI was provided information about the type of Aroclor present in the samples. Dilution of samples during analysis to optimize method performance altered some of the standard intervals shown in Table 5-1 for select samples.

Table 5-1. D TECH PCB test kit reporting intervals

Default Mode		Conversion to Secific Aroclor			
Interval	Aroclor 1254 Concentration Range	Interval	Aroclor 1242 Concentration Range	Interval	Aroclor 1248 Concentration Range
[0, 0.5)	0≤ PCB ppm < 0.5	[0, 1.5)	0≤ PCB ppm < 1.5	[0, 0.8)	0≤ PCB ppm < 0.8
[0.5, 1]	0.5≤ PCB ppm ≤ 1	[1.5, 3.5]	1.5 ≤ PCB ppm ≤ 3.5	[0.8, 2.3]	0.8 ≤ PCB ppm ≤ 2.3
(1, 4]	1 < PCB ppm ≤ 4	(3.5, 20]	3.5 < PCB ppm ≤ 20	(2.3, 11]	2.3 < PCB ppm ≤ 11
(4, 15]	4 < PCB ppm ≤ 15	(20, 54]	20 < PCB ppm ≤ 54	(11, 28]	11 < PCB ppm ≤ 28
(15, 25]	15 < PCB ppm ≤ 25	(54, 100]	54 < PCB ppm ≤ 100	(28, 53]	28 < PCB ppm ≤ 53
(25, ∞)	PCB ppm > 25	(100, ∞)	PCB ppm > 100	(53, ∞)	PCB ppm > 53

Data Assessment

Objective

The purpose of the data assessment section is to present the evaluation of the performance of SDI's D TECH PCB test 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, 136 environmental soil samples, and 24 extract 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 D TECH 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.

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, 1.5), **[1.5, 3.5]**, (3.5, 20], (20, 54]	0	None
[0, 1.5), **[1.5, 3.5]**, **[1.5, 3.5]**, (3.5, 20]	2	Low
[0, 1.5), **[1.5, 3.5]**, **[1.5, 3.5]**, **[1.5, 3.5]**	3	Medium
[1.5, 3.5], **[1.5, 3.5]**, **[1.5, 3.5]**, **[1.5, 3.5]**	4	High

Performance Evaluation Samples

Table 5-3 summarizes the precision information for the D TECH test kit's analysis of the PE samples. The D TECH test kit reported all four replicates as the same interval (i.e., high precision) for two PE sample sets under the outdoor conditions but achieved the highest precision category for six of eight sample sets under the chamber conditions. Therefore, the test kit performed more precisely under the chamber conditions. Operating under the outdoor conditions, five of eight replicate sets were classified as low precision (i.e., two of four replicates were reported in the same interval). None of the replicate sets

34

Table 5-3. Precision of the D TECH PCB test kit for PE soil samples

Certified PE Conc. (ppm)	Outdoor Site					Chamber Site				
	Sample ID	Precision none ---------------- high				Sample ID	Precision none ---------------- high			
		# of Replicates Reported in Identical Intervals					# of Replicates Reported in Identical Intervals			
		0 [a]	2	3	4		0 [a]	2	3	4
0	126 [b]		x			226 [b]			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		0	6	1	2		0	2	2	5

[a] Indicates that all four replicates were reported as different intervals.
[b] Blank data were not included in the determination of the overall precision.

were reported with the lowest precision. A more detailed analysis of the data showed that the replicates having medium to low precision classifications were more than one interval away from the most frequently reported interval for sample IDs 120, 121, 122, and 124.

Environmental Soil Samples
The D TECH results for the replicate environmental soil sample measurements are presented in Table 5-4. The improved precision under the chamber conditions as described above for the PE samples was not observed for the environmental soils. Under the outdoor conditions, 3 of 17 replicate sets achieved the highest precision classification (i.e., the same interval was reported for all four replicates). Under the chamber conditions, 4 of 17 sample sets were classified as high precision. Under each set of conditions, 9 of 17 samples were classified as low precision. The D TECH test kit reported four different intervals for sample ID 112. A more detailed analysis of the data showed that, of the sample sets where precision was classified as medium to low, nine replicate results (i.e., sample IDs 103, 105, 106, 111, 112, 204, 208, 209, and 211) differed by more than one interval range.

Because most of the measurements fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low concentration (reference laboratory values <125 ppm) and high concentration (reference

laboratory values >125 ppm). See Section 4 for the delineation of which sample IDs were in the low and high categories. For the low concentrations, 20% of the sample sets were reported with all four replicates in the same interval (i.e., highest possible precision). For the high concentration category, 40% of the sample sets (two of five) were reported with the highest possible precision.

Table 5-4. Precision of the D TECH PCB test kit for environmental soil samples

	Outdoor Site					Chamber Site			
Sample ID	**Precision** none ---------------- high **# of Replicates Reported in Identical Intervals**				**Sample ID**	**Precision** none ---------------- high **# of Replicates Reported in Identical Intervals**			
	0 [a]	**2**	**3**	**4**		**0 [a]**	**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 [b]		x			**201 [b]**		x		
114				x	**202**		x		
115		x			**203**		x		
116			x		**204**		x		
117		x			**205**				x
# in each precision classification	**1**	**9**	**4**	**3**		**0**	**9**	**4**	**4**

[a] Indicates that all four replicates were reported as different intervals.
[b] Bold sample IDs were matching Paducah sample pairs (i.e., 113/201, 114/202, 115/203, 116/204, 117/205).

36

The Paducah soils (indicated by bold sample IDs in Table 5-4) were analyzed at both sites to provide an assessment of the D TECH'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, in which the 100 series were samples analyzed under the outdoor conditions and the 200 series were samples analyzed inside the chamber. Additional statistical analysis was used to compare the effect of the two environmental conditions on the measurements. Results from this analysis showed that there were no significant differences in the data generated at each site. This indicated that these different environmental conditions did not have an impact on the performance of the test kit.

Extract Samples
The D TECH results for the replicate extract measurements are presented in Table 5-5. Under the outdoor conditions, the 10- and 100-µg/mL sample sets were reported with medium and high precision, respectively, and medium and low precision under the chamber conditions. Of the sample sets reported with medium and low precision, all differed by more than one interval range. Under both testing conditions, the blank samples were reported with medium precision.

Table 5-5. Precision of the D TECH PCB test kit for extract samples

Outdoor Site					Chamber Site				
Sample ID	**Precision** none ------------------ high				**Sample ID**	**Precision** none ------------------ high			
	# of Replicates Reported in Identical Intervals					**# of Replicates Reported in Identical Intervals**			
	0 [a]	**2**	**3**	**4**		**0 [a]**	**2**	**3**	**4**
130 [b]			x		230 [b]			x	
131			x		231			x	
132				x	232		x		
# in each precision classification	**0**	**0**	**2**	**1**		**0**	**1**	**2**	**0**

[a] Indicates that all four replicates were reported as different intervals.
[b] Blank data were not included in the determination of the overall precision.

Precision Summary
A summary of the overall precision of the D TECH test kit is presented by sample type (PE, environmental soil, and extract samples) in Table 5-6. For PE and environmental soil samples, 44% and 21% of the samples, respectively, achieved the highest possible precision (i.e., all four samples replicates were reported as the same interval). For the extract samples, 25% of the samples achieved the highest precision.

Table 5-6. Overall precision of the D TECH PCB test kit for all sample types

Environmental Site	Percentage of Samples Classified in Each Precision Category											
	PE Samples				Environmental Soil Samples				Extract Samples			
	None	Low	Med	High	None	Low	Med	High	None	Low	Med	High
Outdoor Site	0	63	13	25	6	53	24	18	0	0	50	50
Chamber Site	0	25	13	63	0	53	24	24	0	50	50	0
Combined Sites	0	44	13	44	3	53	24	21	0	25	50	25

Accuracy

Accuracy represents the closeness of the D TECH test kit's measured PCB concentrations to the certified values. Because the D TECH 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 5-7 compares the D TECH interval results with the corresponding certified PE values. The listing of an interval in a particular column indicates how many of the four replicates were reported as that interval. For example, for sample ID 220, two replicates were reported as (4, 15], and two were reported as (15, 25]. For sample ID 124, three are reported as (1, 4], and one is reported as [0, 0.5]. Table 5-7 also presents performance acceptance ranges for the PE results, which are the guidelines established by the provider of the PE materials to gauge acceptable analytical results. These ranges were not used to evaluate the D TECH results because the acceptance ranges overlap several D TECH reporting intervals.

The data in Table 5-7 were used to derive the accuracy results presented in Table 5-8. Accuracy was based on a comparison of the certified PE value with the interval reported by the D TECH test kit. If the interval encompassed the certified PE value, the D TECH result "agreed" with the certified value. If the D TECH result was above the certified value, the result was classified as "biased high." If the D TECH result was below the certified value, the result was classified as "biased low." For example, for sample ID 219, the certified value was 20 ppm (for Aroclor 1248). The comparison would be classified as "agreed" for the D TECH interval result (11, 28], as "biased high" for the interval result (28, 53], and as "biased low" for the interval result (2.3, 11]. Separate comparisons were made for the two environmental conditions to determine if ambient temperature and humidity had an effect on the performance of the technology. Statistical analysis showed that there was no significant difference between the results obtained by the test kit under the two different environmental conditions evaluated in this demonstration. Therefore, all PE sample results were combined to determine the overall percentage of agreement between the D TECH results and the certified PE value. The overall percentage of agreement was 56%. Approximately 17% of the D TECH sample results were biased low. A total of 28% of the results from the test kit were biased high.

Table 5-7. D TECH test kit accuracy data for PE soil samples

Certified Conc. (ppm) (Acceptance Range, ppm)	Outdoor Site					Chamber Site				
	Sample ID	# of Replicates Reported at Each Interval				Sample ID	# of Replicates Reported at Each Interval			
		1	2	3	4		1	2	3	4
0 (n/a)	126		[0, 0.5) [0.5, 1]			226	[0, 0.5)		[0.5, 1]	
2.0 (0.7-2.2)	118				(2.3, 11]	218	[0.8, 2.3]		(2.3, 11]	
2.0 (0.9-2.5)	124	[0, 0.5)		(1, 4]		224				(1, 4]
5.0 (2.1-6.2)	120	(1, 4] (4, 15]	[0.5, 1]			220		(4, 15] (15, 25]		
10.9 (4.0-12.8)	122	[0.5, 1] (15, 25]	[4, 15]			222				(25, ∞)
20.0 (11.4-32.4)	119		(2.3, 11] (11, 28]			219	(2.3, 11] (28, 53]	(11, 28]		
49.8 (23.0-60.8)	125		(15, 25] (25, ∞)			225				(25, ∞)
50.0 (19.7-63.0)	121	(4, 15] (15, 25]	(25, ∞)			221				(25, ∞)
50.0 (11.9-75.9)	123				(25, ∞)	223				(25, ∞)

Table 5-8. Evaluation of agreement between D TECH'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	31%	50%	19%	36
Chamber Site	3%	61%	36%	36
Combined Sites	17%	56%	28%	72

Extract Samples

Table 5-9 compares the D TECH interval results with the corresponding spike concentrations for the extract samples. The D TECH test kit's percentage of agreement with the spike concentration of the extract samples is summarized in Table 5-10. Statistical analysis showed that environmental conditions had no significant effect upon the performance of the D TECH test kit. Therefore, the data sets generated under the outdoor and chamber conditions were combined. Overall, 10 of 24 extract samples (42%) agreed with the spike concentration. Approximately 29% were biased high, and 29% were biased low. All biased-high results were from samples analyzed under the chamber conditions, and all biased-low results were from samples analyzed under the outdoor conditions.

Table 5-9. Accuracy of the D TECH test kit for extract samples

Spike Conc. (µg/mL)	Outdoor Site					Chamber Site				
	Sample ID	# of Replicates Reported at Each Interval				Sample ID	# of Replicates Reported at Each Interval			
		1	2	3	4		1	2	3	4
0	132				[0, 0.5]	232	[0.5, 1] (4, 15]	[0, 0.5]		
10	130	(3.5, 20]		[1.5, 3.5]		230	(54, 100]		(20, 54]	
100	131	[0.5, 1]		(15, 25]		231	(100, ∞)		(25,∞)	

Table 5-10. Evaluation of agreement between D TECH's extract results and the spike concentration as a measure of accuracy

Environmental Site	Relative to Spike Concentration for Extract Samples			Number of Samples
	Biased Low	Agree	Biased High	
Outdoor Site	58%	42%	0%	12
Chamber Site	0%	42%	58%	12
Combined Sites	29%	42%	29%	24

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, five were reported as [0.5, 1], so the fp result was 62%. Of the 192 non-blank soil samples analyzed, D TECH reported one in the lowest reporting interval (e.g., 0 to 0.5 ppm), when the corresponding reference laboratory result was 1.8 ppm. Therefore, the fn result for the soil

samples was 0.5%. For the eight blank extract samples, the D TECH test kit reported one blank as [0.5, 1] and one as (4, 15]. Therefore, the fp result was 25%. All other extract samples were reported as non-blanks, so the fn result was 0%.

Representativeness

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

Completeness

Completeness is defined as the percentage of measurements that are judged to be useable (i.e., the result was not rejected). 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 D TECH results and the reference laboratory results was performed for all soil samples. Accuracy was evaluated in terms of the percentage of samples that agreed with, were above (i.e., biased high), and were below (i.e., biased low) the certified value. For comparability, the D TECH results were compared with the results generated by the reference laboratory, including both environmental soils and PE samples. Sample IDs 110 and 112 were excluded because the reference laboratory did not generate quantitative results for these samples. The results are summarized in Table 5-11. The percentage of D TECH results that agreed with the reference laboratory results was 53%. Approximately 28% were biased high, while approximately 20% were biased low relative to the results reported by the reference laboratory.

For the extract samples, the comparison of the D TECH test kit's result with the reference laboratory result was the same as the comparison with the spike concentrations (previously discussed in Table 5-10). There was 42% agreement between the laboratory method and the field technology, 29% of the D TECH results were biased high, and 29% were biased low.

The soil data not included in previous comparability evaluations (because the replicate data for the reference laboratory was considered suspect) are shown in Table 5-12. (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 D TECH's matching results. For sample IDs 110 and 112, the reference laboratory obtained qualitative results only. The D TECH test kit also had some difficulty with sample IDs 110 and 112, producing replicate results in multiple intervals. For the other five suspect values for the reference laboratory data, three of the D TECH test kit results agreed with the replicate means of the reference laboratory; two of the D TECH results were biased low relative to the reference laboratory replicate means. These comparisons demonstrated that the D TECH test kit had difficulty with most of the samples that were troublesome for the reference laboratory.

41

Table 5-11. Evaluation of agreement between D TECH'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	27%	46%	27%	96
Chamber Site	13%	59%	29%	104
Combined Sites	20%	53%	28%	200

Table 5-12. Comparison of the D TECH results with the reference laboratory's suspect measurements

Sample ID	Reference Laboratory		D TECH	
	Suspect Measurement (ppm)	Replicate Mean[a] (ppm)	Suspect Matching Result (ppm)	Number of Replicates Reported as This Interval
110	≤RDL [b]	≤RDL [b]	(4, 15] (15, 25)	1 3
112	≤RDL [b]	≤RDL [b]	(1, 4] (4, 15] (15, 25] (25, ∞)	1 1 1 1
106	649.6	281.0	(4, 15]	1
205	3,305.0	493.0	(53, ∞)	4
216	151.6	55.1	(15, 25]	2
217	1,913.3	659.8	(25, ∞)	4
225	146.0	41.7	(25, ∞)	4

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

Summary of PARCC Parameters

Table 5-13 summarizes the D TECH test kit's performance for precision, accuracy, and comparability. The percentage of replicate samples with the highest precision (i.e., in which all four replicates were reported as the same interval) was 44% for the PE, 21% for the environmental soil, and 25% for the extract samples. The agreement and disagreement of the D TECH test kit with certified values were based on the certified PE values (i.e., accuracy) and the reference laboratory results (i.e., comparability). Overall, the D TECH 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

Table 5-13. D TECH PCB test kit performance for precision, accuracy, and comparability

Sample Type	Precision [a]	Accuracy [b]			Comparability [c]		
	% High Precision	% Biased Low	% in Agreement	% Biased High	% Biased Low	% in Agreement	% Biased High
PE	44	17	56	28	19	57	24
Environmental Soil	25	n/a [b]	n/a	n/a	20	50	31
Extract	21	29	42	29	29	42	29

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

percentage in agreement ranged from 42 to 57%, the percentage biased high was 24 to 29%, and the percentage biased low was 17 to 29%.

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 and 100 $\mu g/100cm^2$ for surface wipes. To assess this ability, the D TECH 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 61% of the time. Results were biased high 7% of the time and biased low 32% of the time.. No false negatives were observed for this concentration range. Of the 24 PE samples that had a nominal concentration of 50 ppm, over 80% of the samples were reported correctly as (25, ∞); the other results were biased low.

Assuming a 10 mL extract volume, extract samples (at 10 and 100 $\mu g/mL$) represented surface wipe sample concentrations of 100 $\mu g/100cm^2$ and 1000 $\mu g/100cm^2$. For simulated wipe extract samples, the percentage of the D TECH's measurements that agreed with the reference laboratory results was 42%. Approximately 29% of the results were biased high and 29% were biased low. No false negative results were observed for the extract samples.

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 SDI team's sample throughput rate was 15 samples/hour. For work in the chamber, the rate was lower, around 11 samples/hour. The higher sample throughput under the outdoor conditions may be attributed to the analysis order; because the SDI staff analyzed samples under the chamber conditions first, they may have gained valuable experience that was applied during the analysis of the outdoor samples. Alternatively, SDI analysts may have had more difficulty with the sample matrices that were analyzed only under the outdoor conditions.

Cost Assessment

The purpose of this economic analysis is to provide an estimation of the range of costs for an analysis of PCB-contaminated soil samples using the D TECH PCB test kit and a conventional analytical reference laboratory method. The analysis was based on the results and experience gained from this demonstration, costs provided by SDI, 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 D TECH 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 5-14. This analysis assumed that the individuals performing the analyses were fully-trained to operate the technology. SDI does not offer a specific training course on the use of the D TECH kit, but does provide free assistance, on an as-needed basis, through its technical service department. Sample acquisition and pre-analytical sample preparation, which are tasks common to both methods, were costs that were not included here.

D TECH Costs

- **Sample shipment costs.** Because the samples were analyzed on-site, no sample shipment charges were associated with the cost of operating the D TECH test kit.

- **Labor costs.** Labor costs included mobilization/demobilization, travel, per diem, and on-site labor.
  — Labor mobilization/demobilization: This cost element included the time for one person to prepare for and travel to each site. The estimate ranged from 5 to 8 hours, at a rate of $50 per hour.
  — Travel: This element was the cost for the analyst(s) to travel to the site. If the analyst is located near the site, the cost of commuting to the site (estimated to be 50 miles at $0.30 per mile) would be minimal ($15). The estimated cost of an analyst traveling to the site for this demonstration ($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 zero (for a local site) to $150 per day per analyst.

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

D TECH PCB test kit Strategic Diagnostics, Inc.		EPA SW-846 Method 8080/8081/8082 Reference Laboratory	
Sample throughput rate: 15 samples per hour (outdoors) 11 samples per hour (chamber)		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	50–400	Mobilization/demobilization	Included[a]
Travel	15–1000 per analyst	Travel	Included
Per diem	0–150 per day per analyst	Per diem	Included
Rate	30–75 per hour per analyst	Rate	44–239 per sample
Equipment		**Equipment**	
Mobilization/demobilization	0–150	Mobilization/demobilization	Included
Photometer purchase price	299	Rental/purchase of system	Included
Reagents/supplies	36 per sample	Reagents/supplies	Included
		Waste Disposal	Included
Waste Disposal	75–1060		

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

— 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.** 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 photometer was $299.
— 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 D TECH PCB test kit was $36 per sample. This cost included the sample preparation supplies, assay supplies, and consumable reagents.

• **Waste disposal costs.** Waste disposal costs are estimated based on the 1997 regulations for disposal of PCB-contaminated waste. Using the D TECH test kit, SDI generated approximately 20 lb of vials containing soils and liquid solvents (classified as solid PCB waste suitable for disposal by incineration) and approximately 20 lb of other solid PCB waste (used and unused soil, gloves, paper towels,

ampules, etc.). The cost of disposing of PCB solid waste by incineration at a commercial facility was estimated at $1.50/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.** Sample shipment costs to the reference laboratory included overnight shipping charges, as well as labor charges associated with the various organizations involved in the shipping process.
    — Overnight shipping: The overnight express shipping service cost was estimated to be $50 for one 50-lb cooler of samples.
    — Labor: This cost element included all of the tasks associated with the shipment of the samples to the reference laboratory. Tasks included packing the shipping coolers, completing the chain-of-custody documentation, and completing the shipping forms. Because the samples contained PCBs, the coolers were inspected by qualified personnel to ensure acceptance with the U.S. Department of Transportation's shipping regulations for PCBs. The estimate to complete this task ranged from 2 to 4 h at $50 per hour.

- **Labor, equipment, and waste disposal costs.** The labor bids from commercial analytical reference laboratories who offered to perform the PCB analysis for this demonstration ranged from $44 per sample to $239 per sample. The bid was dependent on many factors, including the perceived difficulty of the sample matrix, the current workload of the laboratory, and the competitiveness of the market. In this case, the wide variation in bids may also be related to the cost of PCB waste disposal in a particular laboratory's state. LAS Laboratories was awarded the contract to complete the analysis as the lowest qualified bidder ($44 per sample). This rate was a fully loaded analytical cost that included labor, equipment, waste disposal, and report preparation.

Cost Assessment Summary
An overall cost estimate for the D TECH test kit vs the reference laboratory was not made due to the extent of variation in the difference cost factors, as outlined in Table 5-14. The overall costs for the application of each technology will be based on the number of samples requiring analysis, the sample type, and the site location and characteristics. Decision-making factors, such as turnaround time for results, must also be weighed against the cost estimate to determine the value of the field technology vs the reference laboratory.

General Observations

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

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

- Three operators were used for the demonstration because of the number of samples and the working conditions, but the technology can be operated by a single person. With three SDI technologies (D TECH, EnviroGard, and RaPID Assay) being demonstrated, SDI elected to work as a team to complete the analyses for each technology (as opposed to three SDI people working with three different technologies).

- Operators generally require 2 to 4 h of training and should have a basic knowledge of field analytical techniques.

- Data processing and interpretation was minimal. The results were quantified relative to the four calibration standards and reported in terms of intervals using the sample information provided (Aroclor type and ratio).

- New start and stop solutions were used with every four samples. All reagents were allowed to come to room temperature before use. Although it is recommended that all of the reagents in the test kit be stored under refrigerated conditions, the SDI team noted that the reagents can be stored at ambient conditions for several hours.

- The development times for the assay were altered as outlined in the D TECH protocol. The times, which were usually shorter as temperature increased, were determined on the basis of the assay of the reference standard. When the color development in the reference standard read the correct range, the assay was stopped. Temperature measurements and stop times were not recorded.

- The D TECH test 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 (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 SDI's D TECH PCB test kit, presented previously in this chapter, is shown in Table 5-15. The performance of D TECH test kit was characterized as biased, because nearly half (44%) of the D TECH results disagreed with the certified PE values, and imprecise, because over half (56%) of the PE replicate results were not reported as the same interval. For the soil samples, the test kit

reported 62% of the blank sample results as false positives. One non-blank result was classified as a false negative. For extract samples, the test kit had two false positive results (25%) and no false negative results.

Table 5-15. Performance summary for the D TECH PCB test kit

Feature/Parameter	Performance Summary
Blank Results	**Soils:** 3 samples reported correctly as [0, 0.5); 5 samples reported as [0.5, 1] **Extracts**: 6 samples reported correctly as [0, 0.5); 1 sample reported as [0.5, 1]; 1 sample reported as (4, 15]
Precision	**Percentage of combined sample sets in which all four replicates were reported as the same interval** PE soils: 44% Environmental soils: 21% Extracts: 25%
Accuracy	**PE soils** **Extracts** agreed = 56% agreed = 42% biased high =28 % biased high = 29% biased low = 17% biased low = 29%
False Positive Results	**Blank soils:** 62% (5 of 8 samples) **Blank extracts:** 25% (2 of 8 samples)
False Negative Results	**PE and environmental soils:** 0.5% (1 of 192 samples) **Spiked extracts:** 0% (0 of 16 samples)
Comparison with Reference Laboratory Results	**PE and environmental soils** **Extracts** agreed = 53% agreed = 42% biased high = 28% biased high = 29% biased low = 20% biased low = 29%
Regulatory Decision-Making Applicability	**PE and environmental soils** **Extracts** **(40–60 ppm)** **(100 μg/100cm^2, 1000 μg/100cm^2)** agreed = 61% agreed = 42% biased high = 7% biased high = 29% biased low = 32% biased low = 29%
Sample Throughput	11 samples/hour (chamber) 15 samples/hour (outdoors)
Power Requirements	Battery-operated photometer
Operator Requirements	Basic knowledge of chemical techniques; 2–4 h technology-specific training
Cost	Incremental: $36 per sample Instrumental: $299 (purchase photometer)
Hazardous Waste Generation	~20 lb of solid/liquid waste (classified as incinerable solid) ~20 lb of solid waste (used gloves, pipettes, paper towels, etc.) ~19 lb of liquid waste (aqueous with trace methanol)

Section 6
Technology Update and Representative Applications

Objective
The purpose of this section is to allow SDI to describe new technology developments that have occurred since the demonstration activities. In addition, the developer has provided a list of representative applications where the D TECH PCB test kit has been or is currently being utilized.

Technology Update
Reconfiguration of Soil Extraction (Sample Preparation) Products
SDI is in the process of commercializing a common extraction kit for three of its four remediation immunoassay test kit product lines. The affected product lines include the EnviroGard, EnSys (not demonstrated here), and RaPID Assay test kit systems. The new "Universal Extraction Kit" will be used with assay kits of these three product lines, with extraction solvents or dilution reagents specifically formulated to match individual kits available as kit component options where required. The new test kit configuration will provide increased user convenience and simplify the product specification and ordering process without affecting test kit analytical performance. Commercialization of the new Universal Extraction Kit was initiated April 1998. The new kits are not for use with the D TECH product line, which will continue to use the existing SDI Soil Extraction Pac products.

D TECH Test Format Changes
Two modifications to SDI's D TECH product line to enhance analytical performance are presently under development. First, the intervals between the tests' existing semi-quantitative range values will be significantly increased by reducing the number of ranges. For example, the D TECH PCB semi-quantitative ranges will be reduced from the current six (0 to 0.5 ppm, 0.5 to 1 ppm, >1 to 4 ppm, >4 to 15 ppm, >15 to 25 ppm, >25 ppm) to about four (0 to 1 ppm, 1 to 10 ppm, 10 to 25 ppm, and >25 ppm). This will simplify the interpretation of results and broaden the range of results, which will make the results more accurate. Secondly, each assay kit will include results' interpretation data based on the actual lot-specific performance (calibration curve) of that production lot. The combination of these two modifications is expected to significantly increase the analytical performance of the D TECH PCB test kit, while maintaining the speed, simplicity, and convenience of this format.

Instrument Consolidation
Associated with the incorporation of several independently developed product lines into SDI's product offerings, some consolidation of equipment and instrumentation is anticipated in the near future. This will consist primarily of reducing the number of pipet types and photometers used to perform the assays. While pipet types and procedures for pipeting reagents and reading and interpreting assay results may change slightly, no effect of assay performance will result.

Representative Applications
A 1997 report entitled *Field Analytical and Site Characterization Technologies: Summary of Applications* [11] documents the use of SDI immunoassay kits at more than 30 remediation sites under state or federal oversight. The report provides contact information for many of the immunoassay kit users at these sites. The summary report can be obtained from the National Center for Environmental Publications and Information (NCEPI). Hard copies of the report can be ordered, free of charge, by telephone (513-891-6561), by fax (513-891-6685), or through the NCEPI home page on the Internet (http://www.epa.gov /ncepihom). The report is available for viewing or download as a .pdf file from the CLU-IN Internet web site (http://clu-in.com/pubichar.htm).

Data Quality Objective Example
This application of SDI's D TECH PCB immunoassay kit is based on data quality objective (DQO) methods for project planning advocated by ASTM [12, 13] and EPA [14]. ORNL derived a DQO example from the performance results in Section 5. The example, which is presented in Appendix E, illustrates the use of SDI's D TECH 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

[1]   Erickson, M. D. *Analytical Chemistry of PCBs,* 2nd ed., CRC Press/Lewis Publishers, Boca Raton, Fla., 1997.

[2]   "Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution in Commerce, and Use Prohibitions," *Code of Federal Regulations,* 40 CFR, pt. 761, rev. 7, December 1994.

[3]   Maskarinec, M.P., et al. *Stability of Volatile Organics in Environmental Soil Samples,* ORNL/TM-12128, Oak Ridge National Laboratory, Oak Ridge, Tenn., November 1992.

[4]   U.S. Environmental Protection Agency. "Method 8081: Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique," in *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods (SW-846),* 3d ed., Final Update II, Office of Solid Waste and Emergency Response, Washington, D.C., September 1994.

[5]   Oak Ridge National Laboratory. *Technology Demonstration Plan: Evaluation of Polychlorinated Biphenyl (PCB) Field Analytical Techniques,* Chemical and Analytical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tenn., July 1997.

[6]   U.S. Environmental Protection Agency. *Data Quality Objectives for Remedial Response Activities,* EPA 540/G-87/003, EPA, Washington, D.C., March 1987.

[7]   Sachs, Lothar. *Applied Statistics: A Handbook of Techniques,* 2nd ed., Springer-Verlag, New York, 1984.

[8]   Snedecor, G. W., and William G. Cochran. *Statistical Methods,* Iowa State University Press, Ames, Iowa, 1967.

[9]   Draper, N. R., and H. Smith. *Applied Regression Analysis,* 2nd ed., John Wiley & Sons, New York, 1981.

[10]   Berger, Walter, Harry McCarty, and Roy-Keith Smith. *Environmental Laboratory Data Evaluation,* Genium Publishing Corp., Schenectady, N.Y., 1996.

[11]   U.S. Environmental Protection Agency. *Field Analytical and Site Characterization Technologies: Summary of Applications,* EPA-542-R-97-011, Office of Solid Waste and Emergency Response, Washington, D.C., November 1997.

[12]   American Society for Testing and Materials (ASTM). *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation,* D5283-92, 1997.

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

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

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 % gravel	% sand	% silt + clay	Total Organic Carbon (mg/kg)	pH
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.
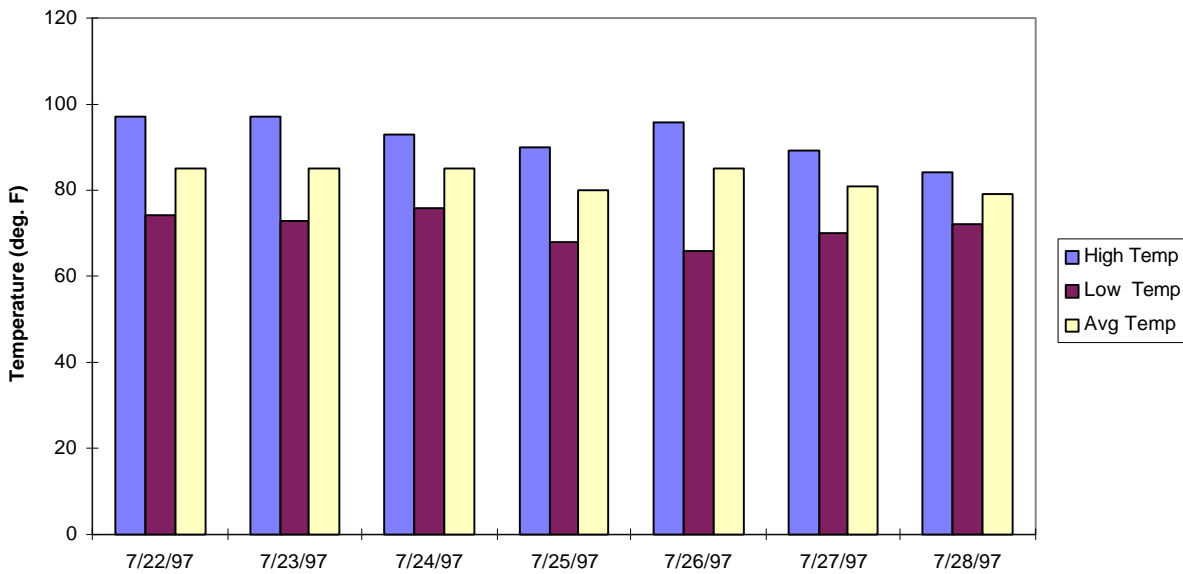
Figure C-1. Summary of temperature conditions for outdoor site.
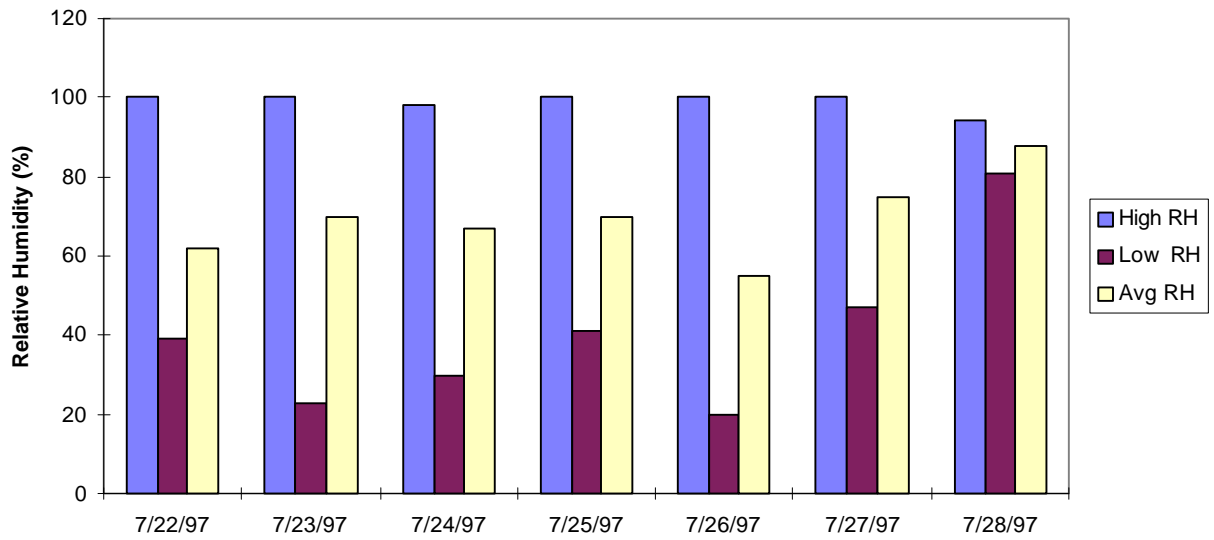
Figure C-2. Summary of relative humidity conditions for the outdoor site.
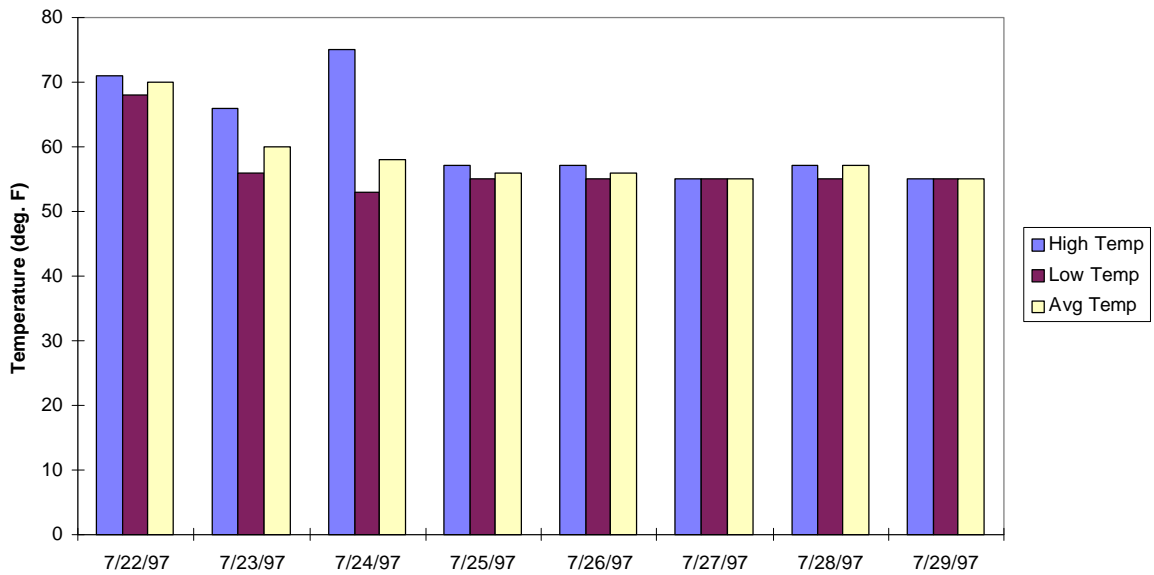
Figure C-3. Summary of temperature conditions for chamber site.
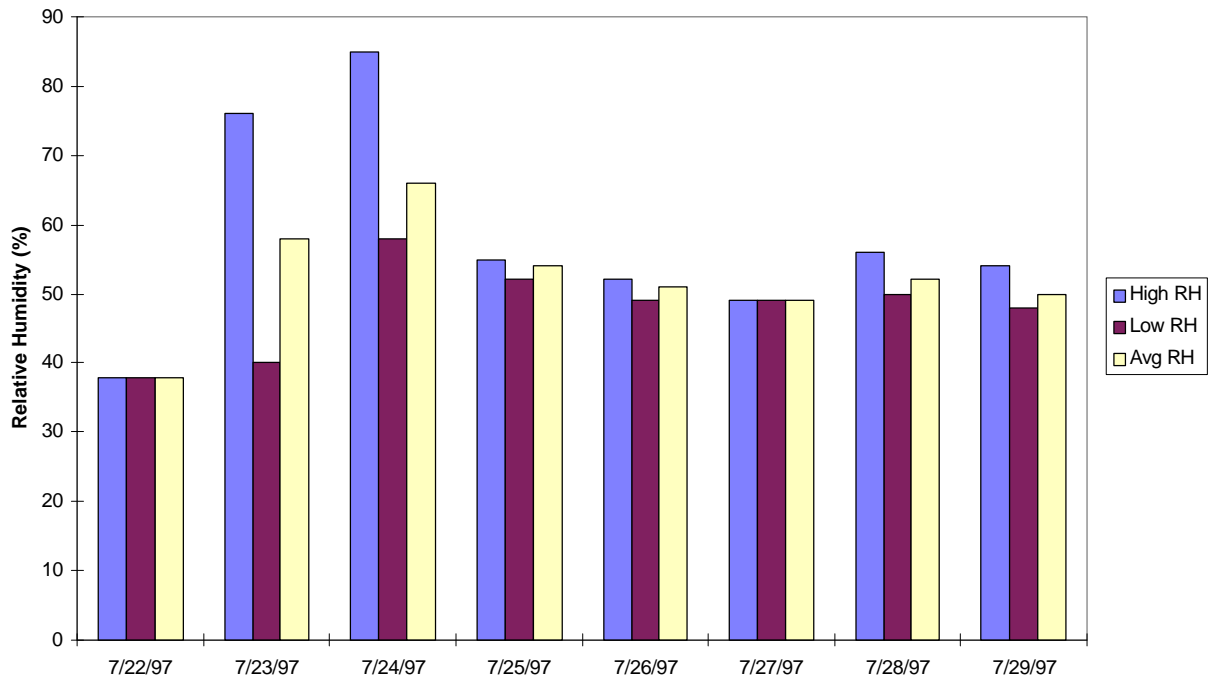
Figure C-4. Summary of relative humidity conditions for chamber site.

Appendix D
SDI's D TECH PCB Test Kit
PCB 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)
D TECH Result	Measured PCB concentration (ppm) for SDI's D TECH
Ref Lab Result	LAS reference laboratory measured PCB concentration (ppm) 　　　Values with "$\leq$" are samples that the reference 　　　laboratory reported as "$\leq$ 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 by SDI (started with 2001–2116, then 1001–1116)

Table D-1. SDI's D TECH PCB test kit technology demonstration soil sample data

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
1	101	1	(2, 8]	0.6	1254	Sample	1103
2	101	2	(1, 2]	0.4	1254	Sample	1097
3	101	3	(2, 8]	0.5	1254	Sample	1092
4	101	4	(2, 8]	0.5	1254	Sample	1075
5	102	1	(4, 15]	2.2	1254	Sample	1052
6	102	2	(4, 15]	2.1	1254	Sample	1084
7	102	3	(4, 15]	1.7	1260	Sample	1077
8	102	4	(4, 15]	2.5	1260	Sample	1018
9	103	1	(25, ∞)	3.0	1254	Sample	1007
10	103	2	(4, 15]	2.4	1254	Sample	1004
11	103	3	(1, 4]	2.0	1260	Sample	1096
12	103	4	(1, 4]	1.6	1260	Sample	1005
13	104	1	(4, 15]	6.8	1260	Sample	1071
14	104	2	(1, 4]	6.0	1254	Sample	1100
15	104	3	(4, 15]	14.8	1254	Sample	1043
16	104	4	(15, 25]	9.9	1254	Sample	1010
17	105	1	(1, 4]	49.7	1260	Sample	1060
18	105	2	(25, ∞)	84.1	1260	Sample	1062
19	105	3	(15, 25]	50.6	1260	Sample	1032
20	105	4	(25, ∞)	53.2	1260	Sample	1045
21	106	1	(25, ∞)	269.6	1254	Sample	1014
22	106	2	(25, ∞)	255.9	1254	Sample	1041
23	106	3	(25, ∞)	317.6	1254	Sample	1038
24	106	4	(4, 15]	649.6	1254	Sample	1087
25	107	1	(1, 4]	1.0	1254	Sample	1012
26	107	2	[0.5, 1]	1.6	1254	Sample	1030
27	107	3	[0.5, 1]	1.2	1254	Sample	1070
28	107	4	(1, 4]	1.2	1254	Sample	1061
29	108	1	(1, 4]	1.7	1254	Sample	1088
30	108	2	(4, 15]	2.0	1254	Sample	1020
31	108	3	(4, 15]	1.7	1254	Sample	1048
32	108	4	(1, 4]	1.9	1254	Sample	1046
33	109	1	(1, 4]	1.5	1254	Sample	1039
34	109	2	(1, 4]	2.1	1254	Sample	1050
35	109	3	(1, 4]	1.8	1254	Sample	1080
36	109	4	(1, 4]	2.4	1254	Sample	1033
37	110	1	(15, 25]	≤490.0	Non-Detect	Sample	1049
38	110	2	(4, 15]	≤99.0	Non-Detect	Sample	1028
39	110	3	(4, 15]	≤66.0	Non-Detect	Sample	1006
40	110	4	(4, 15]	≤98.0	Non-Detect	Sample	1089

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
41	111	1	(1, 4]	44.5	1254	Sample	1074
42	111	2	(15, 25]	36.0	1254	Sample	1040
43	111	3	(4, 15]	39.3	1254	Sample	1008
44	111	4	(1, 4]	35.1	1254	Sample	1065
45	112	1	(4, 15]	≤66.0	Non-Detect	Sample	1086
46	112	2	(25, ∞)	≤200.0	Non-Detect	Sample	1044
47	112	3	(1, 4]	≤130.0	Non-Detect	Sample	1058
48	112	4	(15, 25]	≤200.0	Non-Detect	Sample	1047
49	113	1	[0.8, 2.3]	0.7	1260	Sample	1013
50	113	2	(2.3, 11]	1.1	1260	Sample	1073
51	113	3	(2.3, 11]	0.6	1260	Sample	1017
52	113	4	[0.8, 2.3]	1.9	1248/1260	Sample	1076
53	114	1	(1, 4]	1.1	1260	Sample	1093
54	114	2	(1, 4]	1.2	1260	Sample	1034
55	114	3	(1, 4]	1.3	1260	Sample	1099
56	114	4	(1, 4]	1.7	1260	Sample	1001
57	115	1	(20, 54]	14.9	1248	Sample	1083
58	115	2	(3.5, 20]	12.4	1016	Sample	1063
59	115	3	(3.5, 20]	15.0	1248	Sample	1094
60	115	4	[1.5, 3.5]	16.9	1248	Sample	1057
61	116	1	(3.5, 20]	41.4	1248	Sample	1064
62	116	2	(20, 54]	41.2	1016	Sample	1095
63	116	3	(20, 54]	48.5	1248	Sample	1027
64	116	4	(20, 54]	34.0	1016	Sample	1091
65	117	1	(53, ∞)	431.6	1016	Sample	1021
66	117	2	(28, 53]	406.3	1016	Sample	1067
67	117	3	(28, 53]	304.7	1016	Sample	1069
68	117	4	(53, ∞)	392.8	1016	Sample	1090
69	118	1	(2.3, 11]	2.1	1248	1248	1042
70	118	2	(2.3, 11]	1.9	1016	1248	1056
71	118	3	(2.3, 11]	0.7	1248	1248	1035
72	118	4	(2.3, 11]	1.6	1248	1248	1016
73	119	1	(2.3, 11]	21.2	1016	1248	1026
74	119	2	(11, 28]	17.2	1248	1248	1003
75	119	3	(11, 28]	17.4	1248	1248	1079
76	119	4	(2.3, 11]	24.4	1248	1248	1051
77	120	1	(1, 4]	4.5	1254	1254	1098
78	120	2	(4, 15]	4.0	1254	1254	1101
79	120	3	[0.5, 1]	6.3	1254	1254	1085
80	120	4	[0.5, 1]	5.0	1254	1254	1068

70

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
81	121	1	(25, ∞)	58.7	1254	1254	1011
82	121	2	(25, ∞)	55.7	1254	1254	1029
83	121	3	(4, 15]	53.2	1254	1254	1053
84	121	4	(15, 25]	50.9	1254	1254	1031
85	122	1	(4, 15]	12.2	1260	1260	1024
86	122	2	[0.5, 1]	10.9	1260	1260	1066
87	122	3	(15, 25]	11.3	1260	1260	1078
88	122	4	(4, 15]	10.0	1260	1260	1015
89	123	1	(25, ∞)	59.2	1260	1260	1009
90	123	2	(25, ∞)	56.9	1260	1260	1036
91	123	3	(25, ∞)	66.8	1260	1260	1037
92	123	4	(25, ∞)	57.5	1260	1260	1072
93	124	1	(1, 4]	1.8	1254	1254/1260	1023
94	124	2	(1, 4]	1.4	1260	1254/1260	1102
95	124	3	(1, 4]	1.9	1254	1254/1260	1054
96	124	4	[0, 0.5)	1.8	1254	1254/1260	1059
97	125	1	(25, ∞)	32.0	1254	1254/1260	1081
98	125	2	(15, 25]	41.3	1254	1254/1260	1025
99	125	3	(25, ∞)	46.0	1254	1254/1260	1002
100	125	4	(15, 25]	32.2	1260	1254/1260	1055
101	126	1	[0, 0.5)	≤0.1	Non-Detect	Blank	1104
102	126	2	[0.5, 1]	≤0.1	Non-Detect	Blank	1082
103	126	3	[0.5, 1]	≤0.2	Non-Detect	Blank	1019
104	126	4	[0, 0.5)	≤1.3	Non-Detect	Blank	1022
105	201	1	(2.3, 11]	1.0	1016/1260	Sample	2092
106	201	2	[0.8, 2.3]	1.0	1016/1260	Sample	2044
107	201	3	[0.8, 2.3]	1.1	1016/1260	Sample	2023
108	201	4	(2.3, 11]	0.6	1260	Sample	2073
109	202	1	[0.5, 1]	1.4	1260	Sample	2002
110	202	2	(1, 4]	1.6	1260	Sample	2059
111	202	3	(4, 15]	1.2	1260	Sample	2058
112	202	4	(4, 15]	1.5	1260	Sample	2033
113	203	1	(54, 100]	14.0	1248	Sample	2070
114	203	2	(20, 54]	12.8	1248	Sample	2019
115	203	3	(53, ∞)	16.2	1248	Sample	2011
116	203	4	(20, 54]	12.4	1248	Sample	2094
117	204	1	(100, ∞)	43.1	1248	Sample	2042
118	204	2	(100, ∞)	45.3	1248	Sample	2055
119	204	3	(54, 100]	41.0	1248	Sample	2091
120	204	4	(3.5, 20]	47.7	1248	Sample	2051

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
121	205	1	(53, ∞)	3305.0	1016/1260	Sample	2068
122	205	2	(53, ∞)	538.7	1016	Sample	2045
123	205	3	(53, ∞)	457.0	1016	Sample	2066
124	205	4	(53, ∞)	483.3	1016	Sample	2021
125	206	1	(4, 15]	2.9	1260	Sample	2101
126	206	2	(4, 15]	1.1	1260	Sample	2078
127	206	3	(1, 4]	1.1	1016/1260	Sample	2077
128	206	4	(1, 4]	2.5	1260	Sample	2007
129	207	1	(15, 25]	17.8	1260	Sample	2076
130	207	2	(15, 25]	14.3	1260	Sample	2041
131	207	3	(15, 25]	21.6	1260	Sample	2084
132	207	4	(25, ∞)	21.6	1254	Sample	2017
133	208	1	(25, ∞)	42.0	1260	Sample	2064
134	208	2	(25, ∞)	27.7	1016/1260	Sample	2024
135	208	3	(25, ∞)	24.0	1254	Sample	2087
136	208	4	(4, 15]	28.4	1260	Sample	2082
137	209	1	(15, 25]	32.7	1260	Sample	2031
138	209	2	(4, 15]	79.3	1260	Sample	2029
139	209	3	(4, 15]	11.0	1260	Sample	2050
140	209	4	(25, ∞)	37.9	1260	Sample	2032
141	210	1	(25, ∞)	123.2	1260	Sample	2085
142	210	2	(25, ∞)	61.5	1260	Sample	2020
143	210	3	(25, ∞)	84.1	1260	Sample	2072
144	210	4	(25, ∞)	85.5	1260	Sample	2096
145	211	1	(25, ∞)	387.8	1254	Sample	2016
146	211	2	(4, 15]	581.4	1254	Sample	2065
147	211	3	(25, ∞)	330.0	1254	Sample	2053
148	211	4	(25, ∞)	318.7	1254	Sample	2009
149	212	1	(4, 15]	3.8	1260	Sample	2095
150	212	2	(4, 15]	3.9	1260	Sample	2039
151	212	3	(4, 15]	4.3	1260	Sample	2010
152	212	4	(4, 15]	0.8	1260	Sample	2102
153	213	1	(4, 15]	6.9	1260	Sample	2079
154	213	2	(4, 15]	7.3	1260	Sample	2100
155	213	3	(15, 25]	7.8	1260	Sample	2038
156	213	4	(1, 4]	10.5	1260	Sample	2030
157	214	1	(25, ∞)	26.0	1260	Sample	2104
158	214	2	(25, ∞)	25.6	1260	Sample	2014
159	214	3	(25, ∞)	29.1	1260	Sample	2093
160	214	4	(15, 25]	20.2	1260	Sample	2054

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
161	215	1	(15, 25]	25.1	1260	Sample	2069
162	215	2	(25, ∞)	24.1	1260	Sample	2022
163	215	3	(25, ∞)	26.2	1260	Sample	2056
164	215	4	(15, 25]	31.2	1016/1260	Sample	2086
165	216	1	(15, 25]	151.6	1260	Sample	2083
166	216	2	(25, ∞)	47.0	1260	Sample	2027
167	216	3	(15, 25]	54.3	1260	Sample	2048
168	216	4	(25, ∞)	64.0	1260	Sample	2063
169	217	1	(25, ∞)	886.7	1254	Sample	2046
170	217	2	(25, ∞)	549.8	1254	Sample	2036
171	217	3	(25, ∞)	542.8	1254	Sample	2047
172	217	4	(25, ∞)	1913.3	1016/1260	Sample	2057
173	218	1	(2.3, 11]	2.8	1248	1248	2075
174	218	2	(2.3, 11]	2.4	1248	1248	2080
175	218	3	(2.3, 11]	2.6	1248	1248	2008
176	218	4	[0.8, 2.3]	2.6	1248	1248	2062
177	219	1	(28, 53]	22.4	1248	1248	2025
178	219	2	(11, 28]	26.0	1016	1248	2060
179	219	3	(2.3, 11]	29.4	1248	1248	2001
180	219	4	(11, 28]	15.2	1248	1248	2005
181	220	1	(15, 25]	8.5	1254	1254	2067
182	220	2	(4, 15]	4.9	1254	1254	2088
183	220	3	(4, 15]	4.7	1254	1254	2028
184	220	4	(15, 25]	5.2	1254	1254	2074
185	221	1	(25, ∞)	32.0	1016/1260	1254	2004
186	221	2	(25, ∞)	44.1	1016/1260	1254	2015
187	221	3	(25, ∞)	43.8	1254	1254	2103
188	221	4	(25, ∞)	59.6	1254	1254	2089
189	222	1	(25, ∞)	13.2	1260	1260	2012
190	222	2	(25, ∞)	12.4	1260	1260	2090
191	222	3	(25, ∞)	12.7	1260	1260	2006
192	222	4	(25, ∞)	12.7	1260	1260	2034
193	223	1	(25, ∞)	56.6	1260	1260	2026
194	223	2	(25, ∞)	50.3	1260	1260	2003
195	223	3	(25, ∞)	49.9	1260	1260	2035
196	223	4	(25, ∞)	66.4	1260	1260	2098
197	224	1	(1, 4]	2.2	1254	1254/1260	2049
198	224	2	(1, 4]	1.2	1260	1254/1260	2013
199	224	3	(1, 4]	1.4	1260	1254/1260	2037
200	224	4	(1, 4]	2.1	1254	1254/1260	2099

Obs	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Order
201	225	1	(25, ∞)	56.4	1260	1254/1260	2018
202	225	2	(25, ∞)	36.5	1016/1260	1254/1260	2052
203	225	3	(25, ∞)	32.1	1260	1254/1260	2071
204	225	4	(25, ∞)	146.0	1254	1254/1260	2040
205	226	1	[0, 0.5)	≤0.1	Non-Detect	Blank	2061
206	226	2	[0.5, 1]	≤0.8	Non-Detect	Blank	2081
207	226	3	[0.5, 1]	≤0.1	Non-Detect	Blank	2043
208	226	4	[0.5, 1]	≤0.1	Non-Detect	Blank	2097

Table D-2. SDI's D TECH PCB test kit's technology demonstration extract sample data

OBS	Sample ID	Rep	D TECH Result (ppm)	Ref Lab Result (ppm)	Reference Aroclor	Type	Spike [a] (ppm)	Order
1	130	1	(3.5, 20]	16.4	1016	1242	10	1107
2	130	2	[1.5, 3.5]	10.9	1016	1242	10	1116
3	130	3	[1.5, 3.5]	10.3	1016	1242	10	1112
4	130	4	[1.5, 3.5]	10.7	1016	1242	10	1114
5	131	1	(15, 25]	67.1	1254	1254	100	1105
6	131	2	(15, 25]	57.1	1254	1254	100	1111
7	131	3	(15, 25]	62.8	1254	1254	100	1110
8	131	4	[0.5, 1]	68.2	1254	1254	100	1113
9	132	1	[0, 0.5)	≤0.1	Non-Detect	blank	0	1108
10	132	2	[0, 0.5)	≤0.1	Non-Detect	blank	0	1115
11	132	3	[0, 0.5)	≤0.1	Non-Detect	blank	0	1109
12	132	4	[0, 0.5)	≤0.1	Non-Detect	blank	0	1106
13	230	1	(20, 54]	9.8	1016	1242	10	2113
14	230	2	(54, 100]	10.4	1016	1242	10	2109
15	230	3	(20, 54]	7.6	1016	1242	10	2114
16	230	4	(20, 54]	7.9	1016	1242	10	2107
17	231	1	(100, ∞)	55.2	1254	1254	100	2116
18	231	2	(25, ∞)	55.0	1254	1254	100	2110
19	231	3	(25, ∞)	61.3	1254	1254	100	2106
20	231	4	(25, ∞)	59.1	1254	1254	100	2108
21	232	1	[0, 0.5)	≤0.1	Non-Detect	blank	0	2115
22	232	2	[0.5, 1]	≤0.1	Non-Detect	blank	0	2111
23	232	3	[0, 0.5)	≤0.1	Non-Detect	blank	0	2112
24	232	4	(4, 15]	≤0.1	Non-Detect	blank	0	2105

[a] Nominal spike concentration of the extract sample prepared by ORNL.

Table D-3. Corrected reference laboratory data

Error	Sample ID	Reported Result (ppm)	Corrected Result (ppm)
Transcription	106	≤490	255.9
	130	5.6	10.3
	205	32,000	3,305.0
	207	180	17.8
	210	160	123.2
Calculation	118	3.6	2.1
	119	4.3	17.4
	209	2.3	37.9
	214	43.0	26.0
	219	29.0	22.4
Interpretation	101[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 SDI's D TECH PCB kit to measure the PCB concentration in each drum. Because the type of Aroclor was unknown, all measurements would be reported as Aroclor 1254. The plan was to randomly select soil samples collected from each drum and test with SDI's kit to determine if the concentration was in one of the three intervals: [0, 1], (1, 4], or (4, ∞). Recall that this notation describes the concentration ranges 0 ppm ≤ PCB ≤ 1 ppm, 1 ppm < PCB ≤ 4 ppm, and PCB > 4 ppm, as used in Section 5. The DQO team decided that a drum would be reprocessed by incineration if any of SDI's D TECH results indicated a concentration in the intervals (1, 4], or (4, ∞). In agreement with the regulator, 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, 4], or (4, ∞).

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* [14] states in Secion 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 drum's true PCB concentration exceeds the 2 ppm limit and that, 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 the drum is actually "hot" (i.e., the null hypothesis is true). The DQO 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 D TECH 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* [14], the 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 the D TECH PCB kit, the performance of this technology (as reported in this ETV report) was used to assess its applicability to this project. The question which then arises is, How many samples are needed from a single drum to permit a statistically valid decision at the specified certainty? Recall that a simplifying assumption was made that the PCB distribution throughout the soil within a single drum is homogeneous and that therefore matrix heterogeneity will not contribute to overall variability. The only variability to be considered in this example, then, is the variability in performance of the D TECH kit's analytical method, which is determined by precision and accuracy studies.

Determining the Number of Samples
The number of samples needed to satisfy the FP and FN requirements depends on the misclassification error rates of the D TECH 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 than 1 ppm, and
2.   overestimating the PCB concentration— classifying a sample concentration in (1, 4] or (4, ∞) when the PCB concentration is less than or equal to 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 as follows:

P_U = Pr[Underestimating the PCB concentration] = 0.066,
P_O = Pr[Overestimating the PCB concentration] = 0.412.

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

$$FP = Pr[\ All\ D\ TECH\ results\ <\ 1\ ppm\ for\ PCB\ \geq\ 1\ ppm\ ] = (P_U)^n \qquad \text{(E-1)}$$

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

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

where

n = number of samples from a drum to be measured,
FP = false positive decision error rate (e.g., $FP = 0.05$),
P_U = probability of underestimating the PCB concentration (e.g., $P_U = 0.066$).

Incorporating the appropriate values for the D TECH PCB test kit into Equation E-2 gives

$$n = \frac{Log(0.05)}{Log(0.066)} = \frac{-1.301}{-1.180} = 1.10 \quad 2 \quad .$$

To be conservative, the sample size was rounded up to the next integer, which will decrease the FP. The DQO team would have to take two samples to meet the decision rule's false positive requirement. The FN for the decision rule is related to P_O by

$$FN = Pr[\ Some\ of\ D\ TECH\ results\ \geq\ 1\ ppm\ for\ PCB\ <\ 1\ ppm\ ] = 1 - (1 - P_O)^n \qquad \text{(E-3)}$$

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., $FN = 0.10$),
   P_O =   probability of overestimating a PCB concentration.

$$n = \frac{Log(1 - 0.10)}{Log(1 - 0.412)} = \frac{-0.046}{-0.231} = 0.20 \quad .$$

The sample size must be rounded up to $n = 2$ because that is the number of samples required to meet the specified FP. When $n = 2$ 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 65%. The DQO team would be able to meet the DQO goals only for the FP requirement, not for the FN requirement. This situation occurs because of the 41% overestimation error rate of the kit. If the decision about sending a drum for reprocessing is based on having a single overestimate out of two samples, and each sample has a 41% chance of being overestimated, there is a 65% chance that the drum will unnecessarily be sent for reprocessing through the incinerator (which is 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 decided that the sampling procedure would be to randomly select two soil samples from each drum and test the samples with SDI's D TECH PCB kit. The DQO team would send the drum to the landfill if both of SDI's D TECH results were less than 1 ppm, and send the drum to be reprocessed by incineration if any of SDI's D TECH results were greater than 1 ppm. To meet the FP requirement of 5%, the DQO team would have to accept the FN of 65%.

<div style="border: 1px solid black; padding: 10px;">

Decision Rule for 5% FP and 65% FN

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

</div>

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 $n = 2.54$, so the sample size would be rounded up to $n = 3$. The actual FP would then be 0.03% and the actual FN would be 80%. The decision rule for the lower FP requirement would be as shown.

<div style="border: 1px solid black; padding: 10px;">

Decision Rule for FP = 0.1% and FN = 80%

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

If any of the three randomly selected soil samples have PCB test results different than [0, 1], then reprocess the soil drum by incineration.

</div>