



# **Environmental Technology Verification Report**

## **Immunoassay Kit**

### **Strategic Diagnostics Inc. RaPID Assay System for PCB Analysis**

**ETV ✓ ETV ✓ ETV ✓**

# Environmental Technology Verification Report

## Immunoassay Kit

### Strategic Diagnostics Inc. RaPID Assay System for PCB Analysis

By

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Superfund Innovative Technology  
Evaluation Program



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## **Notice**

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**UNITED STATES ENVIRONMENTAL PROTECTION AGENCY**

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



**ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM  
VERIFICATION STATEMENT**

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

**PROGRAM OPERATION**

The EPA, in partnership with recognized testing organizations, objectively and systematically evaluates the performance of innovative technologies. Together, with the full participation of the technology developer, they develop plans, conduct tests, collect and analyze data, and report findings. The evaluations are conducted according to a rigorous demonstration plan and established protocols for quality assurance. EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, with the support of the U.S. Department of Energy's Environmental Management program, selected Oak Ridge National Laboratory (ORNL) as the testing organization for the performance verification of 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 ORNL in Oak Ridge, Tennessee, from July 22 through July 29. The study was conducted under two climatic 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., RaPID Assay System for PCB Analysis*, EPA/600/R-98/111.

## TECHNOLOGY DESCRIPTION

The RaPID Assay System applies the principles of enzyme-linked immunosorbent assay to the determination of PCBs. The sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles coated with PCB-specific antibodies. Both the analyte PCB (which may be in the sample) and the labeled PCB (the enzyme conjugate) compete for the antibody binding sites on the paramagnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (which contain the analyte PCB and labeled PCB bound to the antibodies in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with washing solution. The presence of PCBs is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5'-tetramethylbenzidine). The enzyme-labeled PCB conjugate bound to the PCB-specific antibody catalyzes the conversion of the enzyme substrate/chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Because the labeled PCB (enzyme conjugate) is in competition with the analyte PCBs (in the sample) for the antibody sites, the color development is inversely proportional to the concentration of PCB in the sample (e.g. the darker the color, the less analyte PCB is present in the sample).

## VERIFICATION OF PERFORMANCE

The following performance characteristics of the RaPID Assay System were observed:

**Detection limits:** EPA defines the method detection limit (MDL) as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL was calculated to be 1.5 ppm based on the performance evaluation sample analyses. This was slightly higher than SDI's specified MDL of 0.5 ppm.

**Throughput:** Throughput was 10 to 11 samples/hour. 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 trained operator. Minimal training (2 to 4 hours) is required to operate the RaPID Assay System, provided the user has a fundamental understanding of basic chemical and field analytical techniques.

**Completeness:** The RaPID Assay System generated results for all 232 PCB samples for a completeness of 100%.

**Blank results:** No PCBs were detected in either the soil or extract blanks above the RaPID Assay's MDLs; therefore, the percentage of false positive results was 0%. Two false negative results (1%) were reported for the nonblank soil samples.

**Precision:** The RaPID Assay System exhibited a significant "site effect" in terms of precision. The overall precision, based on average relative standard deviations (RSDs), was 25% (under outdoor conditions) and 12% (under chamber conditions) for soil samples. The outdoor precision was comparable to the reference laboratory's precision (21% RSD), while the chamber precision was better. The RaPID Assay's precision was comparable to the reference laboratory's (12% and 14%, respectively) for extract samples.

**Accuracy:** Accuracy was assessed using PE soil and extract samples. The data showed that the RaPID Assay System exhibited both positive and negative bias depending on the Aroclor type present in the sample. Because the bias was evenly distributed (positive and negative), this was not reflected in the overall accuracy (which was based on average percent recoveries) of 103% for the PE soil samples. Extract measurements were relatively unbiased, with an overall average

percent recovery of 101%. Evaluation of the data generated at each site indicated that there were no significant differences between the two data sets based on environmental conditions.

**Comparability:** This demonstration showed that the RaPID Assay System generated data that exhibited a linear correlation to the reference laboratory data. The coefficient of determination ( $R^2$ ), which is a measure of the degree of correlation between the reference laboratory and the RaPID Assay data, was 0.754 when all soil samples (0 to 700 ppm) were considered. For the concentration range from 0 to 125 ppm, the  $R^2$  value was 0.716. Approximately 36% of the soil sample results had percent difference values within the range of  $\pm 25\%$ . For extract samples, the data were highly correlated with the reference laboratory,  $R^2$  of 0.977.

**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  $\mu\text{g}/100\text{cm}^2$  for surface wipes. For PE and environmental soil samples in the range of 40 to 60 ppm, the precision was 21% RSD with a mean accuracy of 126% recovery. For extract samples representing surface wipe sample concentrations of 100  $\mu\text{g}/100\text{cm}^2$  and 1000  $\mu\text{g}/100\text{cm}^2$  (assuming a 100  $\text{cm}^2$  wipe sample), measurements were precise (12% RSD) and accurate (101% recovery).

**Data quality levels:** The overall performance of the RaPID Assay System was characterized as slightly biased and precise, under a given set of environmental conditions. Although there was a significant "site effect" in terms of the precision, it should be noted that the RaPID's worst-case precision (25% RSD) was comparable to the best case precision (21% RSD, excluding suspect values) for the reference laboratory.

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

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

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



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## Foreword

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

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

The U.S. Department of Energy's (DOE) 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 hinges on the deployment of innovative environmental characterization and monitoring technologies. To this end, DOE EM shares the goals and objectives of ETV.

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

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





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## Abstract

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

The RaPID Assay System is a field portable instrument that applies the principles of enzyme-linked immunosorbent assay to the determination of PCBs. The RaPID Assay System uses a PCB-labeled enzyme conjugate and paramagnetic particles coated with PCB-specific antibodies, where the analyte PCB (which may be in the sample) and the labeled PCB compete for the antibody binding sites and bind in proportion to their original concentration. The presence of PCBs is detected by a colored reaction, where the color development is inversely proportional to the concentration of PCBs in the sample (e.g., the darker the color,

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the less PCBs present in the sample). The RaPID Assay System provides no information on Aroclor identification.

The RaPID Assay System's quantitative results were based on initial calibrations. The method detection limit (MDL) is often defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The calculated field-based MDL (1.5 ppm) was slightly higher than SDI's specified MDL of 0.5 ppm. In general, the RaPID Assay System's results were slightly biased. Because the bias was evenly distributed (both positive and negative bias depending on the Aroclor type present in the sample), this was not reflected in the overall accuracy of 103% recovery for the performance evaluation soil samples. Extract measurements were relatively unbiased, with an overall average percent recovery of 101%. The overall precision exhibited a significant "site effect," where the average relative standard deviation (RSD) was 25% (under outdoor conditions) and 12% (under chamber conditions) for soil samples. The outdoor precision was comparable to the reference laboratory's precision (21% RSD), while the chamber precision was better. The precision for extract samples was comparable with the reference laboratory. Comparability, based on coefficients of determination ( $R^2$ ), was 0.754 for all soil samples (0 to 700 ppm), where an  $R^2$  of 1.0 denotes perfect correlation. Approximately 36% of the soil sample results had percent difference values within the range of  $\pm 25\%$ . The data for the extract samples were highly correlated with the reference laboratory.

The demonstration found that the RaPID Assay System was light, easily transportable, and rugged, requiring about one hour for initial setup and preparation for sample analysis. Once operational, the sample throughput of the RaPID Assay System was 10 to 11 samples/h. Three operators analyzed samples during the demonstration, but the technology can be run by a single operator. Minimal training (2 to 4 h) is required to operate the RaPID Assay System, provided the user has a fundamental understanding of basic chemical and field analytical techniques. No site effects (i.e., differences in performance due to environmental conditions) were observed in terms of the accuracy of the measurements; however, the significant (but comparable to the best case precision of the reference laboratory) site effect for precision should be considered when using this technology. Overall, the performance of the RaPID Assay System was characterized as slightly biased and precise, under a given set of environmental conditions.

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## List of Abbreviations and Acronyms

|       |   |
|-------|---|
| AL    | action level                                    |
| ANOVA | analysis of variance                            |
| ASTM  | American Society for Testing and Materials      |
| CCV   | continuing calibration verification standard    |
| CI    | confidence interval                             |
| CSCT  | Consortium for Site Characterization Technology |
| DCB   | decachlorobiphenyl                              |
| DOE   | U.S. Department of Energy                       |
| DQO   | data quality objective                          |
| EM    | Environmental Management (DOE)                  |
| EPA   | U.S. Environmental Protection Agency            |
| ERA   | Environmental Resource Associates               |
| ETTP  | East Tennessee Technology Park                  |
| ETV   | Environmental Technology Verification (Program) |
| ETVR  | Environmental Technology Verification Report    |
| EvTEC | Environmental Technology Evaluation Center      |
| fn    | false negative result                           |
| FN    | false negative decision error rate              |
| fp    | false positive result                           |
| FP    | false positive decision error rate              |
| ID    | identifier                                      |
| LCS   | laboratory control sample                       |
| LMES  | Lockheed Martin Energy Systems                  |
| MDL   | method detection limit                          |
| MS    | matrix spike                                    |
| MSD   | matrix spike duplicate                          |
| n     | number of samples                               |

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|------------------|---|
| NERL             | National Exposure Research Laboratory (EPA)                                 |
| NCEPI            | National Center for Environmental Publications and Information              |
| NRC              | U.S. Nuclear Regulatory Commission  |
| ORNL             | Oak Ridge National Laboratory   |
| ORO              | Oak Ridge Operations (DOE)  |
| PARCC            | precision, accuracy, representativeness, completeness, comparability        |
| PCB              | polychlorinated biphenyl  |
| PDF              | portable document format  |
| 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 <sup>2</sup>   | 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  |
| 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 Substances Control Act  |
| Z <sub>1-p</sub> | the (1 - p)th percentile for the standard normal distribution               |
| %D               | percent difference  |

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## Acknowledgments

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## **Section 1**

### **Introduction**

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

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

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

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



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(Hach Company), the 4100 Vapor Detector (Electronic Sensor Technology), and three immunoassay kits from Strategic Diagnostics Inc. (SDI): 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 RaPID Assay System. 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>).

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### **Demonstration Purpose**

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

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## Section 2 Technology Description

### Objective

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

### Principle

The RaPID Assay System for PCB analysis (formerly the Ohmicron RaPID Assay System) applies the principles of enzyme-linked immunosorbent assay (ELISA) to the determination of PCB [1]. 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 PCBs has been affixed. The native PCB and PCB-enzyme conjugate compete for a limited number of antibody sites. After a period of time, the solution is washed away, and what remains is either PCB-antibody complexes or enzyme-PCB-antibody complexes attached to the test surface. The proportion of the two complexes on the test surface is determined by the amount of native PCB in the original sample. The enzyme present on the test surface is used to catalyze a color change reaction in a solution added to the test surface. Because the amount of enzyme present is inversely proportional to the concentration of native PCB contaminant, the amount of color development is inversely proportional to the concentration of PCB contaminant.

In the case of the RaPID Assay System, the sample to be tested is added, along with an enzyme conjugate, to a disposable test tube, followed by paramagnetic particles with antibodies specific to PCBs attached. Both the PCBs (which may be in the sample) and the enzyme labeled PCB analog (the enzyme conjugate) compete for antibody binding sites on the magnetic particles. At the end of an incubation period, a magnetic field is applied to hold the paramagnetic particles (with PCBs and labeled PCB analog bound to the antibodies on the particles in proportion to their original concentration) in the tube and allow the unbound reagents to be decanted. After decanting, the particles are washed with washing solution.

The presence of PCBs is detected by adding the enzyme substrate (hydrogen peroxide) and the chromogen (3,3',5,5'-tetramethylbenzidine). The enzyme-labeled PCB analog that is bound to the PCB antibody catalyzes the conversion of the substrate/chromogen mixture to a colored product. After an incubation period, the reaction is stopped and stabilized by the addition of acid. Since the labeled PCBs (conjugate) were in competition with the unlabeled PCBs (sample) for the antibody sites, the color developed is inversely proportional to the concentration of PCBs in the sample. The color developed is quantified with a small, handheld photometer. Thus, the RaPID Assay System consists of three primary components, the kit used for

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sample preparation (the RaPID Prep Soil Collection Kit), the assay kit itself, and the RPA-I RaPID Analyzer, which is the small photometer.

### **Sample Preparation**

The RaPID Prep Soil Collection Kit is designed for collection, extraction, and subsequent filtration of soil samples before analysis of environmental contaminants. The reagents contained in the RaPID Prep PCB Sample Extraction Kit have been optimized for fast, efficient removal of PCB from soil and convenient preparation of the sample for immunoassay at levels of interest to the investigator. The system allows for reliable, convenient, and cost-effective determinations at the field testing or remediation site. This procedure was validated using 10 g soil samples. Soil sampling should be conducted in a uniform and consistent manner according to a plan appropriate for the site and the objectives of the study.

### ***Description of Kit Contents***

The following items are contained in the RaPID Assay Sample Preparation Kit:

- soil collection device with detachable plunger and screw cap
- filter caps
- extract collection vials
- chain-of-custody container labels
- portable Styrofoam tube holder
- PCB extraction solution (methanol with dispersion agent)
- PCB extract diluent (buffered saline solution containing preservatives and stabilizers without detectable PCBs)
- 25- $\mu$ L precision pipet
- Pipet tips

The components of the kit should be stored at 2 to 30°C, and reagents should not be used after the expiration date.

### ***Materials Not Provided in Kit***

In addition to materials provided, the following items will be necessary for the preparation of a soil sample:

- stopwatch
- permanent marking pen

- protective gloves
- digital balance

### **Procedure**

#### **Sample Weighing**

The soil collector (shown in Figure 2-1), with its plunger fully depressed (pushed to the top), will reproducibly collect a *volume* of soil. The weight of this volume will vary depending on soil type. Sand, clay, and loam soils collected with the device in this way will weigh approximately 12 g. The same volume of organic soil weighs much less but can be reproducibly collected. If a site is undergoing a preliminary screen for high levels of PCBs, the volume collection method could be used with an estimated average sample weight appropriate for that site.

Weighing the soil before extraction is recommended for soils with high organic content and for combinations of soil types. The following method was used during the demonstration: Remove the screw cap and plunger rod from an empty collection tube. Position the plunger at the bottom of the collection tube. Attach the red base piece provided, and place the tube in an upright position on the balance and tare weight. Weigh  $10 \pm 0.1$  g of soil into the tube. Record the soil weight.

#### **Extraction**

Position the soil collection tube containing a soil sample upright in the Styrofoam rack, and add one vial (20 mL) of the PCB extraction solution. Screw the cap (without filter) on tightly, and make sure that the luer cap is secured. Shake vigorously and continuously for at least 60 s. Additional shaking may be required to break up large or dry soil aggregates. Position the collection tube upright in the rack, and allow the mixture

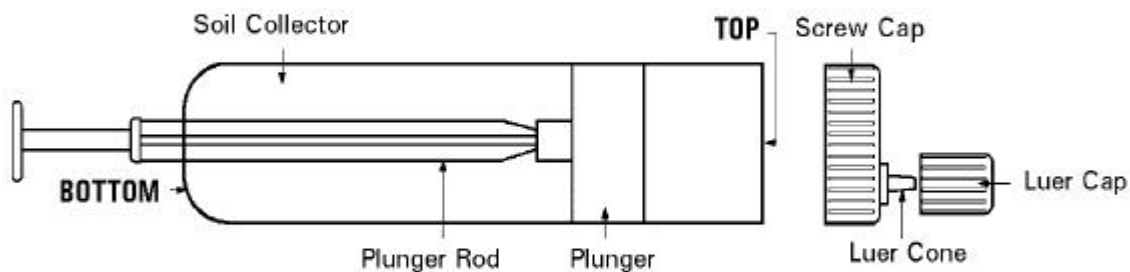


Figure 2-1. Soil sample collection device.

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to sit at least 5 min. Longer extraction times may be desirable for some situations. If batch processing is desired, up to 21 soil samples with added extraction solution can be loaded into the rack inside the kit box base; the box lid is put in place, and the box is shaken vigorously for at least 60 s.

#### **Filtration**

Remove the screw cap and attach the filter cap. Hand tighten until resistance is felt. Attach the plunger rod to the plunger of the soil collector. Invert the soil collector so that the luer cone is positioned over a collection vial. Keep the collector inverted for a few seconds to wet the filter and to allow the filtrate to drip through the filter into the luer cone. Apply slight pressure to the plunger handle. The filtrate will begin to flow more quickly as gentle pressure is applied continuously. Fill the vial with approximately 10 to 20 drops (0.5 to 1 mL). Cap the vial. This amount of filtrate is sufficient to perform multiple replicate analyses with the RaPID Assay Kit. The vial will hold up to 5 mL of filtrate if additional extract volume is desired. The filtrate is stable when stored in the extract collection vial for one week at room temperature (15 to 30°C).

#### **Dilution**

Using the pipet provided, transfer 25 µL of the extract directly into a vial of PCB extract diluent (25 mL). Mix by inverting the vial several times.

***NOTE: This is the step where the solvent extract analysis begins.***

#### ***Assay Procedure***

##### **Reagents**

The following are the reagents provided with the RaPID Assay System for PCB analysis:

- PCB antibody coupled paramagnetic particles—PCB antibody (rabbit anti-PCB) covalently bound to paramagnetic particles, which are suspended in buffered saline containing preservative and stabilizers.
- PCB enzyme conjugate—horseradish peroxidase-labeled PCB analog diluted in buffered saline containing preservative and stabilizers.
- PCB standards—three PCB standard solutions standards in buffered saline containing preservative and stabilizers. The concentrations of the standards are 0.25, 1.0, and 5.0 ppb as Aroclor 1254. Each vial contains 2.0 mL.
- Control—a PCB solution in buffered saline containing preservative and stabilizers. The concentration is approximately 3 ppb as Aroclor 1254. A 2.0-mL volume is supplied in one vial.
- Diluent/zero standard—buffered saline containing preservative and stabilizers without any detectable PCBs.
- Color solution—solution of hydrogen peroxide and 3, 3', 5, 5'-tetramethylbenzidine in an organic base.

- 
- Stopping solution—solution of sulfuric acid (0.5%).
  - Washing solution—preserved deionized water.
  - Test tubes—polystyrene tubes (36).

Store all reagents at 2 to 8°C. Do not freeze. Reagents may be used until the expiration date on the box. The test tubes require no special storage conditions and may be stored separately from the reagents. All reagents must be allowed to come to room temperature, and the antibody-coupled paramagnetic particles should be mixed thoroughly before use.

#### **Materials Required but Not Provided in Kit**

In addition to the reagents provided, the following items are essential for performance of the test:

- Pipets—precision pipets capable of delivering 200, 250, and 500 µL and a 1.0-mL repeating pipet.
- Vortex mixer—Thermolyne Maxi Mix, Scientific Industries Vortex Genie, or equivalent.
- Magnetic separation rack.
- RPA-I RaPID analyzer—or equivalent photometer capable of readings at 450 nm.

#### **Procedural Notes and Precautions**

- As with all immunoassay methods, a consistent technique is the key to optimal performance. To obtain the greatest precision, be sure to treat each tube in an identical manner.
- Add reagents directly to the bottom of the tube while avoiding contact between the reagents and the pipet tip. This will help ensure consistent quantities of reagent in the test mixture.
- Avoid cross-contaminations and carryover of reagents by using clean pipets for each sample addition and by avoiding contact between reagent droplets on the tubes and pipet tips.
- Avoid foam formation during vortexing.
- The magnetic separation rack consists of two parts: an upper rack that will securely hold the test tubes and a lower separator that contains the magnets used to attract the antibody-coupled paramagnetic particles. During incubations the upper rack is removed from the lower separator so that the paramagnetic particles remain suspended during the incubation. For separation steps, the rack and the separator are combined to pull the paramagnetic particles to the sides of the tubes.



- To obtain optimum assay precision, it is important to perform the separation steps carefully and consistently. Decant the rack by slowly inverting it away from the operator using a smooth turning action so that the liquid flows consistently along only one side of the test tube. While the rack is still inverted, place it on an absorbent pad and allow it to drain. Lifting the rack and replacing it gently onto the pad several times will ensure complete removal of the liquid from the rim of the tube (this technique is demonstrated on a training video available from SDI).
- Mix the antibody-coupled paramagnetic particles immediately before pipetting.
- Standard and control vials should remain capped when not in use to prevent evaporation.
- Do not use any reagents beyond their stated shelf life.
- Avoid contact of the stopping solution (sulfuric acid) with skin and mucous membranes. If this reagent comes in contact with skin, wash with water.
- A control solution with approximately 3 ppb of PCBs (as Aroclor 1254) is provided with the RaPID Assay PCB Kit. It is recommended that this solution be included in every run and be treated in the same manner as unknown samples. Acceptable limits should be established by each laboratory.

**Method**

Label test tubes for standards, control, and samples according to Table 2-1. Add 200 uL of the appropriate standard, control, or sample to each tube. Add 250 uL of PCB enzyme conjugate to each tube. Mix the PCB antibody-coupled paramagnetic particles thoroughly, and add 500 uL to each tube. Vortex for 1 to 2 s, minimizing foaming. Incubate for 15 min at room temperature. Separate in the magnetic separation rack for 2 min. Decant and gently blot all tubes briefly in a consistent manner. Add 1 mL of washing solution to each tube, and vortex tubes for 1 to 2 s. Return tubes and allow to remain in the magnetic separation unit for 2 min. Decant and gently blot all tubes briefly in a consistent manner. Repeat steps 10 and 11 one additional time. Remove the rack from the separator, and add 500 uL of color solution to each tube. Vortex for 1 to 2 s, minimizing foaming. Incubate for 20 min at room temperature. Add 500 uL of stopping solution to each tube. Add 1 mL of washing solution to a clean test tube to use as a blank. Within 15 min after adding the stopping solution, read the results using the RPA-I RaPID analyzer set at 450 nm.

**Table 2-1. Tube labels**

| Tube Label | Contents of Tube             |
|------------|------------------------------|
| 1,2        | Diluent/Zero Standard, 0 ppb |

|     |                      |
|-----|----------------------|
| 3,4 | Standard 1, 0.25 ppb |
| 5,6 | Standard 2, 1.0 ppb  |
| 7,8 | Standard 3, 5.0 ppb  |
| 9   | Control              |
| 10  | Sample 1             |
| 11  | Sample 2             |
| 12  | Sample 3             |

## **Results**

### **Manual Calculations**

1. Calculate the mean absorbance value for each of the standards.
2. Calculate the %B/Bo for each standard by dividing the mean absorbance value for the standard by the mean absorbance value for the diluent/zero standard.
3. Construct a standard curve by plotting the %B/Bo for each standard on vertical logit (Y) axis vs the corresponding PCB concentration on horizontal logarithmic (X) axis on the graph paper provided.
4. %B/Bo for controls and samples will then yield results in parts per billion (ppb) of PCBs by interpolation using the standard curve.

### **RPA-I RaPID Analyzer**

Using the RPA-I RaPID analyzer, calibration curves can be automatically calculated and stored. Refer to the RPA-I operating manual for detailed instructions. To obtain results from the RaPID Assay on the RPA-I, the recommended parameter settings are shown in Table 2-2. The following is a summary of the performance characteristics of the RPA-I RaPID:

*Recovery:* PCB recoveries will vary depending on soil type, retention mechanism, solvent and extraction apparatus used, length of extraction period, amount of agitation, and levels of potentially interfering substances in the soil.

**Table 2-2. Recommended parameter settings for the RPA-I RaPID analyzer**

| <b>Parameter</b>           | <b>Recommended Setting</b>                   |
|----------------------------|--|
| Data Reduct                | Linear Regression                            |
| Xformation                 | Ln/LogitB                                    |
| Read Mode                  | Absorbance                                   |
| Wavelength                 | 450 nm                                       |
| Units                      | ppb  |
| # Reagent Blk              | 0  |
| # of Cals                  | 4  |
| # of Reps                  | 2  |
| Calibration Concentrations | 0.00 ppb<br>0.25 ppb<br>1.00 ppb<br>5.00 ppb |
| Range                      | 0.10 - 5.00 ppb                              |
| Correlation                | > 0.990                                      |
| Replicate %CV              | < 10%  |

*Sensitivity:* The RaPID Assay System for PCB analysis has an estimated minimum detectable concentration, based on a 90% B/Bo of 100 ppt. Refer to appropriate application notes or procedures for sensitivity in specific sample matrices.

*Specificity:* The cross-reactivity of the RaPID Assay System for PCB analysis for various Aroclors, as shown in Table 2-3, can be expressed as the least detectable dose, which is estimated at 90% B/Bo, or as the dose required to produce a 50% B/Bo response. The following compounds demonstrated no reactivity in the RaPID Assay System for PCB analysis at concentrations up to 10,000 ppb: biphenyl, 2,5-dichlorophenol, 2,3,5-trichlorophenol, and di-n-octyl-phthalate.

***Limitations***

The RaPID Assay System for PCB analysis will detect PCBs to different degrees. Refer to specificity table for data on various Aroclors and congeners. The PCB RaPID Assay Kit provides screening results. As with any analytical technique (gas chromatography, high-pressure liquid chromatography, etc.), positive results requiring some action should be confirmed by an alternate method.

**Table 2-3. Cross-reactivity of the RaPID Assay System for PCB analysis**

| <b>Compound</b> | <b>Least Detectable Dose (ppb) <sup>a</sup><br/>[90% B/Bo]</b> | <b>Dose Required To Produce 50%<br/>B/Bo Response (ppb)</b> |
|-----------------|--|---|
| Aroclor 1254    | 0.10   | 1.80  |
| Aroclor 1260    | 0.10   | 1.15  |
| Aroclor 1248    | 0.11   | 2.11  |
| Aroclor 1242    | 0.17   | 4.40  |
| Aroclor 1262    | 0.18   | 2.37  |
| Aroclor 1232    | 0.42   | 9.38  |
| Aroclor 1268    | 0.46   | 10.9  |
| Aroclor 1016    | 0.47   | 12.8  |
| Aroclor 1221    | 6.77   | 81.3  |

<sup>a</sup> Concentration in the extract or calibration standard, not in the soil.

The total time required for pipetting the magnetic particles should be kept to 2 min or less; therefore, the total number of tubes that can be assayed in a run should be adjusted accordingly.

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## Section 3

### Site Description and Demonstration Design

#### **Objective**

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

#### **Demonstration Site Description**

##### *Site Name and Location*

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

##### *Site History*

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

In Oak Ridge, excavation activities occurred between 1991 and 1995. The Oak Ridge samples 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

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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 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 [2]. 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



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

| Concentration Range        | Sample ID <sup>a</sup>             |                         | Total # Samples Analyzed |
|----------------------------|------------------------------------|-------------------------|--------------------------|
|                            | Outdoor Site                       | Chamber Site            |                          |
| <i>PE Materials</i>        |                                    |                         |                          |
| 0                          | 126                                | 226                     | 8                        |
| 2.0 ppm                    | 118                                | 218                     | 8                        |
| 2.0 ppm                    | 124                                | 224                     | 8                        |
| 5.0 ppm                    | 120                                | 220                     | 8                        |
| 10.9 ppm                   | 122                                | 222                     | 8                        |
| 20.0 ppm                   | 119                                | 219                     | 8                        |
| 49.8 ppm                   | 125                                | 225                     | 8                        |
| 50.0 ppm                   | 121                                | 221                     | 8                        |
| 50.0 ppm                   | 123                                | 223                     | 8                        |
| <i>Environmental Soils</i> |                                    |                         |                          |
| 0.1–2.0 ppm                | 101, 107, 108, 109, 113, 114       | 201, 202, 206           | 36                       |
| 2.1–20.0 ppm               | 102, 103, 104, 115                 | 203, 207, 212, 213      | 32                       |
| 20.1–50.0 ppm              | 111, 116                           | 204, 208, 209, 214, 215 | 28                       |
| 50.1–700.0 ppm             | 105, 106, 110, 112, 117            | 205, 210, 211, 216, 217 | 40                       |
| <i>Extracts</i>            |                                    |                         |                          |
| 0                          | 129 <sup>b</sup> /132 <sup>c</sup> | 229/232                 | 8                        |
| 10 µg/mL                   | 127/130                            | 227/230                 | 8                        |
| 100 µg/mL                  | 128/131                            | 228/231                 | 8                        |
| <b>Grand Total</b>         | <b>116</b>                         | <b>116</b>              | <b>232</b> <sup>d</sup>  |

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

<sup>b</sup> Extract prepared in iso-octane for Dexsil and the reference laboratory.

<sup>c</sup> Extract prepared in methanol for Electronic Sensor Technology, SDI, and the reference laboratory.

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



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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 performed analyses with the RaPID Assay Kit outdoors on July 25 and in the chamber on July 22.

### **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 [2]. 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 [4] 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 semivolatiles blank soil matrix. This matrix was a topsoil that had been dried, sieved, and homogenized. Particle size was approximately 60 mesh. The soil was approximately 40% clay. The samples acquired from EPA's Analytical Operations and Data Quality Center had been prepared using contaminated soils from various sites around the country in the following manner: The original soils had been homogenized and diluted with a synthetic soil matrix (SSM). The SSM had a known matrix of 6% gravel, 31% sand, and 43% silt/clay; the remaining 20% was topsoil. The dilution of the original soils was performed by mixing known amounts of contaminated soil with the SSM in a blender for no less than 12 h. The samples were also spiked with target pesticides ( $\alpha$ ,  $\beta$ ,  $\Delta$ , and  $\delta$ -BHC, methoxychlor, and endrin ketone) to introduce some compounds that were likely to be present in an actual environmental soil. The hydrocarbon background from the original sample and the spiked pesticides

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produced a challenging matrix. The PE soils required no additional preparation by ORNL and were split for the developer and reference laboratory analyses as received.

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

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

### ***Extract Samples***

Traditionally, the amount of PCBs on a contaminated surface is determined by wiping the surface with a cotton pad saturated with hexane. The pad is then taken to the laboratory, extracted with additional hexane, and analyzed by gas chromatography. Unlike soil samples, which can be more readily homogenized and divided, equivalent wipe samples (i.e., contaminated surfaces or post-wipe pads) were not easily obtainable. Therefore, interference-free solutions of PCBs were analyzed to simulate an extracted surface wipe pad. Extract sample analyses provided evaluation data that relied primarily on the technology's performance rather than on elements critical to the entire method (i.e., sample collection and preparation). Because different developers required the extract samples prepared in different solvents (e.g., methanol and iso-octane), the reference laboratory analyzed sets of extracts in both solvents. 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

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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 was 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 [5].

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 (ID) 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 ID number was in the format of “xxx<sup>y</sup>zz,” 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 ID 101101 corresponded to sample ID “101” [an Oak Ridge soil from request for disposal (RFD) 40022, drum 02], “1” corresponded to the first replicate from that sample, and “01” corresponded to the first jar filled in that series.

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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 and at what ratio if multiple Aroclors were present. 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 [4]. 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 RaPID Assay’s results for the predemonstration samples. Results indicated that SDI’s RaPID Assay System was ready for field evaluation.

**Table 3-2. Summary of the RaPID Assay’s predemonstration results**

| Sample Description                        | Matrix  | Source | RaPID Assay <sup>a</sup> |                        | Reference Laboratory |                        |
|---|---------|--------|--------------------------|------------------------|----------------------|------------------------|
|   |         |        | Result (ppm)             | Duplicate result (ppm) | Result (ppm)         | Duplicate result (ppm) |
| 2 ppm of Aroclor 1260                     | Soil    | ORNL   | 1.2                      | 1.3                    | 2.2                  | 2.3                    |
| 100 ppm (total) of Aroclors 1254 and 1260 | Soil    | ORNL   | 66.3                     | 61.9                   | 78.0                 | 89.0                   |
| 11 ppm of Aroclor 1260                    | Soil    | EPA    | 4.2                      | 4.8                    | 11.0                 | 9.5                    |
| 50 ppm of Aroclor 1254                    | Soil    | ERA    | 66.0                     | <b>b</b>               | 37.0                 | <b>b</b>               |
| 5 ppm of Aroclor 1242                     | Extract | ORNL   | 4.4                      | 4.8                    | 4.7                  | 4.9                    |

<sup>a</sup> Results were Aroclor-adjusted (see Section 2 for more details).

<sup>b</sup> 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 [6], 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 [6] 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,

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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 did not note any deviations from the procedure described in the technology demonstration plan [6] for the RaPID Assay System.

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## **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 [7]. 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 [6] 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, 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.

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### **Reference Laboratory Method**

The reference laboratory's analytical method, also presented in the technology demonstration plan [6], followed the guidelines established in EPA SW-846 Method 8081 [5]. 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., " $\leq$  reporting detection limit").



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

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

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

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

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

### **Quality Control Results**

#### ***Objective***

The purpose of this section is to provide an assessment of the data generated by the reference laboratory's QC procedures. The QC samples included continuing calibration verification standards (CCVs), instrument blanks, method blanks, surrogate spikes, [laboratory control samples (LCSs)], and matrix spike/matrix spike duplicate (MS/MSD) samples. Each control type is described in more detail in the following text and in the technology demonstration plan [6]. 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.

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The requirement for acceptance was a percentage difference of less than 15% for the CCV relative to the initial calibration. This QC requirement was met for all Aroclors and surrogates, except for one standard that had a 16% difference for DCB. These results indicated that the reference laboratory maintained instrument calibrations during the course of sample analysis.

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

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

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

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

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

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

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

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

A laboratory control sample (LCS) is an aliquot of a clean soil that is spiked with known quantities of target analytes. The LCS is spiked with the same analytes and at the same concentrations as the MS. (MSs are described in the next section.) If the results of the MS analyses are questionable (i.e., indicating a potential

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

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

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

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 |  |

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

## **Data Assessment**

### ***Objective***

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

### ***Precision***

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

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

## **Performance Evaluation Samples**

The PE samples were homogenous soils containing certified concentrations of PCBs. Results for these samples represent the best estimate of precision for soil samples analyzed in the demonstration study. Table 4-2 summarizes the precision of the reference laboratory for the analysis of PE samples. One suspect

measurement (sample ID 225, 146.0 ppm) was reported for the PE soil samples. The RSDs for the combined data ranged from 9 to 33% when the suspect measurement was excluded, and from 9 to

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

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

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

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

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

79%, including the suspect measurement. The overall precision, determined by the mean RSD for all PE samples, was 21% for the worst case (including the suspect result) and 18% for the best case (excluding the suspect result).

### Environmental Soil Samples

The precision of the reference laboratory for the analysis of environmental soil samples is reported in Table 4-3. In this table, results including suspect measurements are presented in parentheses. Average concentrations were reported by the reference laboratory as ranging from 0.5 to 1,196 ppm with RSDs that ranged from 7 to 118% when the suspect results were included. Excluding the suspect results, the highest average concentration decreased to 660 ppm, and the largest RSD decreased to 71%. Because the majority of the samples fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low concentrations (< 125 ppm) and high concentrations (> 125 ppm). For the low concentrations, the average RSD was 23% excluding the suspect value and 26% including the suspect value. These average RSDs were only slightly larger than the RSDs for the PE soils samples of comparable concentration (18% for best case and 21% for worst case). Five soil sample sets (sample IDs: 106, 117, 205, 211 and 217) were in the high-concentration category. The average precision for high concentrations was 56% for the worst case and 19% for the best case. The precision estimates for the low and high concentration ranges were comparable when the suspect values were excluded. This indicated that the reference laboratory's precision

for the environmental soils was consistent (approximately 21% RSD), and comparable to the PE soil samples when the suspect values were excluded.

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

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

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

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

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

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

### 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 [8, 9] 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 <sup>a</sup> | 0                    | n/a        | n/a     | 229          | 0                    | n/a        | n/a     | 0                    | n/a        | n/a     |
| 132 <sup>a</sup> | 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       |                      |            |         |

<sup>a</sup> 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 [10] 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 <sup>a</sup> (n/a)                                  | 126          | 0                  | n/a          | 226          | 0                  | n/a             | 0                  | n/a             |
| 2.0 (0.7-2.2)   | 118          | 1.6                | 79           | 218          | 2.6                | 130             | 2.1                | 105             |
| 2.0 (0.9-2.5)   | 124          | 1.7                | 85           | 224          | 1.7                | 85              | 1.7                | 85              |
| 5.0 (2.1-6.2)   | 120          | 5.0                | 99           | 220          | 5.8                | 117             | 5.4                | 108             |
| 10.9 (4.0-12.8)                                       | 122          | 11.1               | 102          | 222          | 12.8               | 117             | 11.9               | 109             |
| 20.0 (11.4-32.4)                                      | 119          | 20.1               | 100          | 219          | 23.3               | 116             | 21.7               | 109             |
| 49.8 (23.0-60.8)                                      | 125          | 37.9               | 76           | 225          | 41.7 <sup>b</sup>  | 84 <sup>b</sup> | 39.5 <sup>c</sup>  | 79 <sup>c</sup> |
| 50.0 (19.7-63.0)                                      | 121          | 54.6               | 109          | 221          | 44.9               | 90              | 49.8               | 100             |
| 50.0 (11.9-75.9)                                      | 123          | 60.1               | 120          | 223          | 55.8               | 112             | 58.0               | 116             |

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

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

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

### 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- $\mu\text{g}/\text{mL}$  level (for both solvents) was 104%, compared with 64% at the 100- $\mu\text{g}/\text{mL}$  level. The reference laboratory classified all 16 samples spiked at 10  $\mu\text{g}/\text{mL}$  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  $\mu\text{g}/\text{mL}$  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- $\mu\text{g}/\text{mL}$  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 ( $\mu\text{g}/\text{mL}$ ) | Outdoor Site |  |              | Chamber Site |  |              | Combined Sites                           |              |
|---|--------------|--|--------------|--------------|--|--------------|--|--------------|
|   | Sample ID    | Average Conc ( $\mu\text{g}/\text{mL}$ ) | Recovery (%) | Sample ID    | Average Conc ( $\mu\text{g}/\text{mL}$ ) | Recovery (%) | Average Conc ( $\mu\text{g}/\text{mL}$ ) | Recovery (%) |
| 0 <sup>a</sup>                                  | 129          | 0  | n/a          | 229          | 0  | n/a          | 0  | n/a          |
| 0 <sup>a</sup>                                  | 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           |  |              |

<sup>a</sup> 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

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100 to express as a percentage. The completeness of the reference laboratory was 97%, where a completeness of 95% or better is typically considered acceptable.

### ***Comparability***

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

### **Summary of Observations**

Table 4-7 provides a summary of the performance of the reference laboratory for the analysis of all sample types used in the technology demonstration study. As shown in Table 4-7, the precision of the PE soils was comparable to the environmental soils. A weighted average, based on the number of samples, gave a best case precision of 21% and a worst-case precision of 28% for all the soil data (PE and environmental). The extract samples had a smaller overall RSD of 14%. Evaluation of overall accuracy was based on samples with certified or known spiked concentrations (i.e., PE and extract samples). The overall accuracy, based on percent recovery, for the PE samples was 105% for the worst case (which included the suspect value) and 101% for the best case (which excluded the suspect value). These results indicated that the reference laboratory measured values were unbiased estimates of the certified PE concentrations (for samples that contained  $\leq 50$  ppm of PCBs). Accuracy for the extract samples at 10 ppm was also unbiased, with an average percent recovery of 104%. However, the accuracy for the extract samples at 100 ppm was biased low, with an average recovery of 64%. Overall, the average percent recovery for all extract samples was 84%. The reference laboratory correctly reported all blank samples as non-detects, but had difficulty with two soil sample IDs (110 and 112) that contained chemical interferences. In general, the reference laboratory's completeness would be reduced, at the expense of an improvement in precision and accuracy, if the suspect measurements were excluded from the data analysis. Based on this analysis, it was concluded that the reference laboratory results were acceptable for comparison with the developer's technology.

**Table 4-7. Summary of the reference laboratory performance**

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

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

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

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## **Section 5**

### **Technology Performance and Evaluation**

#### **Objective and Approach**

The purpose of this section is to present the evaluation of data generated by SDI's RaPID Assay System. The technology's precision and accuracy performance are presented for the data generated in the demonstration study. In addition, an evaluation of comparability, through a one-to-one comparison with the reference laboratory data, is presented. An evaluation of other aspects of the technology (such as detection limits, cost, sample throughput, hazardous waste generation, and logistical operation) is also presented in this section.

#### **Data Assessment**

The purpose of the data assessment section is to present the evaluation of the performance of SDI's RaPID Assay System through a statistical analysis of the data. PARCC parameters were used to evaluate RaPID Assay'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 performance; see Section 3 for further details on the different sites. Evaluation of the measurements at each site indicated that there were no significant differences in the accuracy of the measurements made at each site. There was a significant "site effect" (i.e., significant differences in the data generated under the outdoor and chamber conditions), however, in the precision of the measured concentrations. In cases where the environmental conditions did not affect the results significantly (i.e., for accuracy), data from both sites were combined to determine 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 RaPID Assay System.

#### ***Precision***

Precision, as defined in Section 4, is the reproducibility of measurements under a given set of conditions. The standard deviation and relative standard deviation of four replicate measurements were used to quantify the technology's precision. The average PCB concentration for a replicate set was used to calculate the percent RSD for each Sample ID (see Equation 4-3). For comparative information on the reference laboratory's precision, refer to the data presented in Section 4 under the heading "Precision."

#### **Performance Evaluation Samples**

Table 5-1 summarizes the precision of the RaPID Assay System for the analysis of PE samples. Operating under the outdoor conditions, the RSDs ranged from 11 to 42%. RSDs ranged from 2 to 33% while

operating inside the chamber. Because there was a significant site effect for the samples that were analyzed under outdoor conditions, the precision data for both sites were not combined.

**Table 5-1. Precision of the RaPID Assay System for PE soil samples**

| Outdoor Site |                             |                  |         | Chamber Site |                             |          |         |
|--------------|-----------------------------|------------------|---------|--------------|-----------------------------|----------|---------|
| Sample ID    | Average Concentration (ppm) | SD (ppm)         | RSD (%) | Sample ID    | Average Concentration (ppm) | SD (ppm) | RSD (%) |
| 126          | < 0.5                       | n/a <sup>a</sup> | n/a     | 226          | < 0.5                       | n/a      | n/a     |
| 118          | 1.5                         | 0.2              | 11      | 218          | 1.3                         | 0.4      | 33      |
| 124          | 2.9                         | 0.7              | 25      | 224          | 2.3                         | 0.4      | 17      |
| 120          | 6.3                         | 1.5              | 24      | 220          | 5.4                         | 1.3      | 23      |
| 122          | 6.7                         | 1.2              | 18      | 222          | 6.2                         | 0.1      | 2       |
| 119          | 15.1                        | 6.2              | 41      | 219          | 10.8                        | 0.9      | 9       |
| 125          | 74.5                        | 9.4              | 13      | 225          | 77.3                        | 10.0     | 13      |
| 121          | 70.8                        | 24.0             | 34      | 221          | 63.7                        | 7.9      | 13      |
| 123          | 54.8                        | 22.8             | 42      | 223          | 44.5                        | 5.7      | 13      |

<sup>a</sup> SD and RSD cannot be calculated because all results were reported as < 0.5 ppm.

### Environmental Soil Samples

The precision of the RaPID Assay for the analysis of environmental soil samples is reported in Table 5-2. Operating under the outdoor conditions, the RSDs ranged from 1 to 51%. RSDs ranged from 7 to 22% while operating inside the chamber. Because the majority of measurements fell below 125 ppm, precision was also assessed by partitioning the results into two ranges: low concentrations (reference laboratory values <125 ppm) and high concentrations (reference laboratory values >125 ppm). See Section 4 for delineation of Sample IDs in each concentration range. For the low-concentration range, the average RSD was 24% at the outdoor site compared with 11% at the chamber site. The same trend was observed at the high-concentration range where the average RSD was 25% at the outdoor site compared with 6% at the chamber site. The site effect for the precision data is illustrated in Figure 5-1, which shows the standard deviations for the RaPID Assay's measurements vs the measured average concentrations for the environmental soil samples less than 100 ppm. The figure distinctly depicts how the variability of the measurements was significantly higher under the outdoor conditions compared with the chamber.

Table 5-2. Precision of the RaPID Assay System 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 <sup>a</sup>            | n/a                      | n/a     | 206          | 1.5                         | 0.3                      | 17      |
| 107                     | 1.9                         | 0.3                      | 16      | 212          | 4.0                         | 0.7                      | 18      |
| 108                     | 3.2                         | 0.9                      | 27      | 213          | 5.7                         | 0.4                      | 6       |
| 109                     | 3.4                         | 0.8                      | 23      | 207          | 19.3                        | 4.2                      | 22      |
| 102                     | 3.4                         | 0.3                      | 8       | 208          | 29.1                        | 3.1                      | 11      |
| 103                     | 5.1                         | 1.2                      | 24      | 215          | 29.8                        | 2.1                      | 7       |
| 104                     | 15.3                        | 7.8                      | 51      | 214          | 31.1                        | 2.0                      | 7       |
| 110                     | 31.0                        | 9.3                      | 30      | 209          | 48.4                        | 6.9                      | 14      |
| 111                     | 49.1                        | 12.6                     | 26      | 216          | 57.1                        | 8.3                      | 14      |
| 105                     | 91.4                        | 19.9                     | 22      | 210          | 80.8                        | 10.5                     | 13      |
| 112                     | 123.8                       | 19.4                     | 16      | 211          | >200                        | 0                        | 0       |
| 106                     | >200                        | 0                        | 0       | 217          | >200                        | 0                        | 0       |
| <b>113</b> <sup>b</sup> | 1.4                         | 0.2                      | 13      | <b>201</b>   | 0.8                         | 0.1                      | 18      |
| <b>114</b>              | 1.1                         | 0.4                      | 36      | <b>202</b>   | 0.9                         | 0.2                      | 22      |
| <b>115</b>              | 15.1                        | 3.2                      | 21      | <b>203</b>   | 17.7                        | 2.7                      | 15      |
| <b>116</b>              | 75.9                        | 29.3                     | 39      | <b>204</b>   | 88.5                        | 8.3                      | 9       |
| <b>117</b>              | 223.9                       | 56                       | 25      | <b>205</b>   | 198.0                       | 12.1                     | 6       |

<sup>a</sup> Three results reported as < 0.5 ppm; one result reported as 0.6 ppm.

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

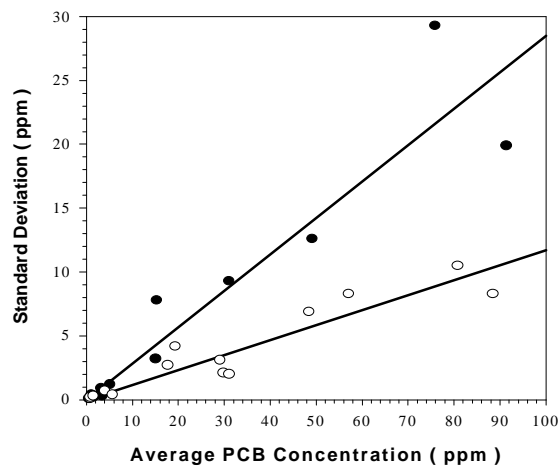


Figure 5-1. Standard deviation vs average PCB concentration for the RaPID Assay measurements with average values less than 100 ppm. Upper line and closed circles are for outdoor conditions and lower line and open circles are for chamber conditions.

The Paducah soils (indicated by bold Sample IDs in Table 5-2) were analyzed at both sites to provide an assessment of the RaPID Assay’s performance under different environmental conditions. Sample IDs 113 and 201, 114 and 202, 115 and 203, 116 and 204, and 117 and 205 represented replicate Paducah soil sample sets, where the 100 series were samples analyzed under the outdoor conditions and the 200 series were samples analyzed inside the chamber. An analysis of variance test (ANOVA) was used to compare the effect of the two environmental conditions on the average measurements. Results from this analysis showed that there were significant differences in the data generated at each site, further confirming that environmental conditions had an effect on the precision of the RaPID Assay. Because there was a significant site effect, the Paducah sample results were not combined to determine the overall precision for the Paducah samples.

**Extract Samples**

Table 5-3 summarizes the RaPID Assay’s results for the extract samples that were used to simulate surface wipe samples. Refer to Section 3, “Extract Samples,” for further clarification of this sample type. Operating under the outdoor conditions, the RSDs ranged from 5 to 8%. RSDs were both 6% while operating inside the chamber. In terms of concentration level, the average RSD at the 10 µg/mL level was 16%, while the average RSD at the 100 µg/mL level was 8%.

**Table 5-3. Precision of the RaPID Assay System for extract samples**

| Outdoor Site |                               |                  |         | Chamber Site |                               |            |         | Combined Sites                |            |         |
|--------------|-------------------------------|------------------|---------|--------------|-------------------------------|------------|---------|-------------------------------|------------|---------|
| Sample ID    | Average Concentration (µg/mL) | SD (µg/mL)       | RSD (%) | Sample ID    | Average Concentration (µg/mL) | SD (µg/mL) | RSD (%) | Average Concentration (µg/mL) | SD (µg/mL) | RSD (%) |
| 132          | <0.5                          | n/a <sup>a</sup> | n/a     | 229          | <0.3                          | n/a        | n/a     | <0.4                          | n/a        | n/a     |
| 130          | 12.5                          | 0.9              | 8       | 227          | 9.5                           | 0.6        | 6       | 11.0                          | 1.8        | 16      |
| 131          | 96.9                          | 5.2              | 5       | 228          | 86.3                          | 5.3        | 6       | 91.6                          | 7.5        | 8       |

<sup>a</sup> SD and RSD cannot be calculated because results were reported either as < 0.3 ppm or < 0.5 ppm.

**Precision Summary**

The overall precision was characterized by three summary values for the RSD: mean (i.e., average), median (i.e., 50<sup>th</sup> percentile value at which 50% of all individual RSD values are below and 50% are above), and 95<sup>th</sup> percentile (i.e., the value at which 95% of all individual RSD values are below and 5% are above). These values are summarized in Table 5-4 for each of the sample types. Because there was a significant site effect for the soil samples (i.e., PE and environmental) that were analyzed under the outdoor conditions, the precision data for both sites were not combined. Under the outdoor conditions, the mean RSD for the PE sample results was 26% and lower (at 15%) under the chamber conditions. Similarly, the mean RSD for the environmental soil samples was 24% under the outdoor conditions and 13% under the chamber conditions.



**Table 5-4. Overall precision of the RaPID Assay System for all sample types**

| Statistic                   | PE Samples |         | Environmental Soil Samples |         | Extract Samples  |         |          |
|-----------------------------|------------|---------|----------------------------|---------|------------------|---------|----------|
|                             | %RSD       |         | %RSD                       |         | %RSD             |         |          |
|                             | Outdoor    | Chamber | Outdoor                    | Chamber | Outdoor          | Chamber | Combined |
| Mean                        | 26         | 15      | 24                         | 13      | 7                | 13      | 12       |
| Median                      | 24         | 13      | 23                         | 14      | n/a <sup>a</sup> | n/a     | n/a      |
| 95 <sup>th</sup> percentile | 41         | 29      | 42                         | 22      | n/a              | n/a     | n/a      |

<sup>a</sup> Median and 95<sup>th</sup> percentile statistics were not applicable to extract samples.

The extract results did not indicate a significant site effect. This was most likely because the replicate extract measurements were performed over a shorter period of time outdoors (i.e., less temperature variability from the time the first and last extract sample were analyzed). All extract sample results were combined to determine an overall precision of 12% RSD.

**Accuracy**

Accuracy, as defined in Section 4, represents the closeness of the technology’s measured PCB concentrations to the accepted values. Accuracy was examined in terms of percent recovery (see Equation 4-1), and average percent recoveries were calculated by averaging the four replicates within a Sample ID. For comparative information on the performance of the reference laboratory, refer to Section 4 under the heading of “Accuracy.”

**Performance Evaluation Soil Samples**

The RaPID Assay’s performance for the PE samples is summarized in Table 5-5. Included in this table are the performance acceptance ranges and the certified PCB concentration values. Average percent recoveries ranged from 61 to 150% while operating under the outdoor conditions. Under chamber conditions, average percent recoveries ranged from 54 to 155%. Regression analyses indicated that there was not a significant site effect in terms of the accuracy of the measurements. However, three of the combined average concentrations (Sample IDs 121/221, 124/224, and 125/225) were outside of the acceptance ranges, and all were biased high. When the data for the two sites were combined, the average recoveries ranged from 60 to 152%, indicating both negative and positive bias. While the results were biased, the RaPID Assay data did correlate with the certified PE values. Although the correlation between the certified values and the RaPID Assay’s results in this study was most accurately described by a quadratic equation (which draws a curved line through the data points), a simpler linear equation (which draws a straight line through the data points) can be used with minimal loss of predictive capability. Further discussion on how the RaPID Assay data might be used in a decision-making process is presented in Appendix E.

The RaPID Assay’s bias appeared to be influenced by the type of Aroclor identified. An ANOVA test was used to determine which experimental factors (concentration level, Aroclor type, environmental conditions) affected the bias of the results. This analysis indicated that environmental conditions did not affect the accuracy, which confirmed the regression analysis results. Concentration level was a

**Table 5-5. Accuracy of the RaPID Assay System for PE soil samples**

| Certified Concentration (ppm)<br>(Acceptance Range, ppm) | Outdoor Site |               |              | Chamber Site |               |              | Combined Sites |              |
|--|--------------|---------------|--------------|--------------|---------------|--------------|----------------|--------------|
|  | Sample ID    | Average (ppm) | Recovery (%) | Sample ID    | Average (ppm) | Recovery (%) | Average (ppm)  | Recovery (%) |
| 0<br>(n/a)   | 126          | <0.5          | n/a          | 226          | <0.5          | n/a          | <0.5           | n/a          |
| 2.0<br>(0.7-2.2)   | 118          | 1.5           | 75           | 218          | 1.3           | 65           | 1.4            | 70           |
| 2.0<br>(0.9-2.5)   | 124          | 2.9           | 145          | 224          | 2.3           | 115          | 2.6            | 130          |
| 5.0<br>(2.1-6.2)   | 120          | 6.3           | 126          | 220          | 5.4           | 108          | 5.9            | 118          |
| 10.9<br>(4.0-12.8)                                       | 122          | 6.7           | 61           | 222          | 6.2           | 57           | 6.5            | 60           |
| 20.0<br>(11.4-32.4)                                      | 119          | 15.1          | 76           | 219          | 10.8          | 54           | 12.9           | 65           |
| 49.8<br>(23.0-60.8)                                      | 125          | 74.5          | 150          | 225          | 77.3          | 155          | 75.9           | 152          |
| 50.0<br>(19.7-63.0)                                      | 121          | 70.8          | 142          | 221          | 63.7          | 127          | 67.3           | 135          |
| 50.0<br>(11.9-75.9)                                      | 123          | 54.8          | 110          | 223          | 44.5          | 89           | 49.6           | 99           |

significant factor that was shown to affect the bias, as evidenced by the positive and negative bias described previously. More significantly, Aroclor type appeared to have a strong influence on the bias. Those samples quantified as Aroclor 1254 were biased particularly high at the higher (i.e., 50 ppm) PE concentrations.

**Extract Samples**

Percent recovery results for the extract samples are summarized in Table 5-6 for the RaPID Assay System. The average percent recoveries for extract samples ranged from 97 to 125% when the RaPID Assay was used under the outdoor conditions and from 86 to 95% inside the chamber. In terms of concentration levels (i.e., for the combined site data), the average recovery at the 10 µg/mL level was 110%, compared with 92% at the 100 µg/mL level.

**Accuracy Summary**

The overall accuracy was characterized by three summary values for percent recovery: mean, median, and 95<sup>th</sup> percentile. These values are summarized in Table 5-7 for the PE and extract samples. For the PE samples, despite a mean percent recovery of 103%, the overall accuracy of the RaPID Assay can be

**Table 5-6. Accuracy of the RaPID Assay System 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                           | 132          | <0.5              | n/a          | 232          | <0.3              | n/a          | <0.4              | n/a          |
| 10                          | 130          | 12.5              | 125          | 230          | 9.5               | 95           | 11.0              | 110          |
| 100                         | 131          | 96.9              | 97           | 231          | 86.3              | 86           | 91.6              | 92           |

**Table 5-7. Overall accuracy of the RaPID Assay System for all sample types**

| Statistic                   | PE Samples |         |          | Extract Samples  |         |          |
|-----------------------------|------------|---------|----------|------------------|---------|----------|
|                             | %Recovery  |         |          | %Recovery        |         |          |
|                             | Outdoor    | Chamber | Combined | Outdoor          | Chamber | Combined |
| Mean                        | 111        | 96      | 103      | 111              | 90      | 101      |
| Median                      | 115        | 88      | 94       | n/a <sup>a</sup> | n/a     | n/a      |
| 95 <sup>th</sup> percentile | 185        | 157     | 173      | n/a              | n/a     | n/a      |

<sup>a</sup> Median and 95<sup>th</sup> percentile statistics were not applicable to extract samples.

characterized as biased. As discussed previously in the “Accuracy” section, this is because of a statistically significant effect on recovery caused by Aroclor type. The result is that the recoveries ranged from 60% (biased low) to 152% (biased high), depending on the specific Aroclor(s) in the sample. Averaging across all samples produces an overall recovery close to 100%.

The overall accuracy for all extract samples was a mean percent recovery of 101% (which did not involve significant bias in either direction); the 95<sup>th</sup> percentile and median data were not presented because of the limited number of data points.

**False Positive/False Negative Results**

A false positive result (fp) [11] is one in which the technology detects PCBs in the sample when there actually are none. A false negative result (fn) [11] 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 (MDL) of the technology. Of the eight blank soil samples analyzed, none were reported as having detectable levels of PCBs (i.e., fp = 0%). Of the 192 non-blank soil samples analyzed, two were reported as non-detects. Therefore, the percentage of fn results was 1%. For the extract samples, the percentage of fp and fn results were both 0%.

### ***Representativeness***

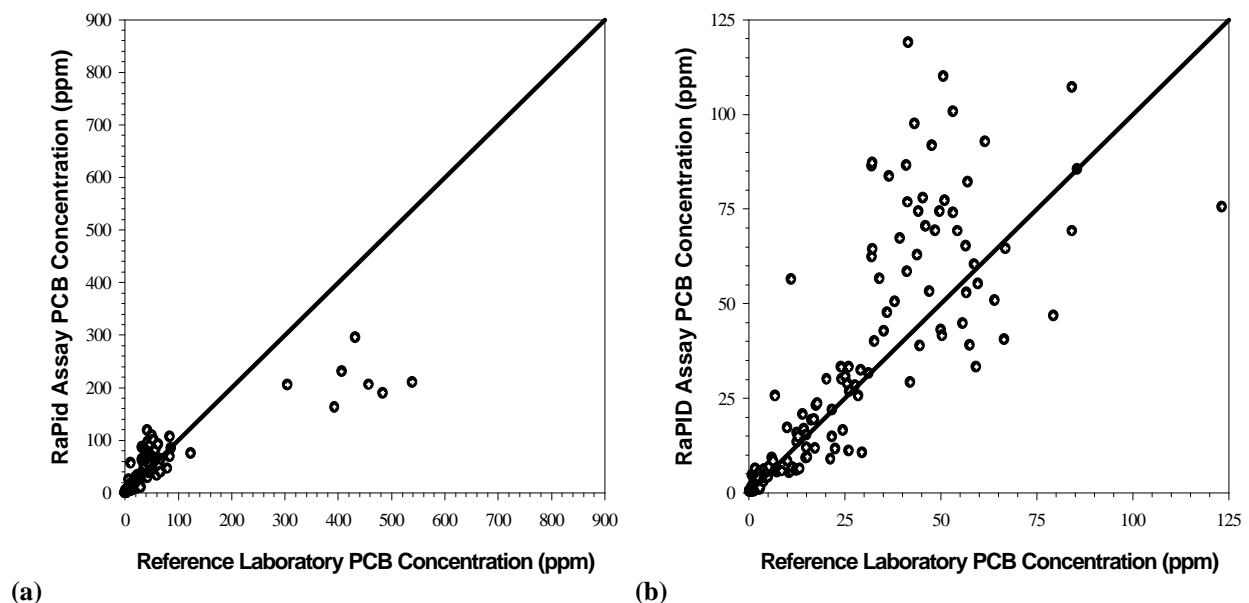
Representativeness expresses the degree to which sample data accurately and precisely represent the capability of the technology. The performance data were accepted as being representative of the technology because the RaPID Assay was capable of analyzing diverse sample types (PE, simulated surface wipe extract, and actual field environmental soil samples) under multiple environmental conditions. When using this technology, QC samples should be analyzed to assess the performance of RaPID Assay System 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, the completeness of the RaPID Assay System was 100%.

### ***Comparability***

Comparability refers to the confidence with which one data set can be compared with another. A one-to-one sample comparison was performed to assess the comparability of the PCB concentrations found in all soil samples (PE and environmental) for the RaPID Assay measured values vs the reference laboratory results. Additional statistical analysis of the PCB soil concentrations for paired samples showed that the RaPID Assay's results were significantly different (higher) than the reference laboratory results for low PCB concentrations (i.e., < 125 ppm). At higher concentrations (i.e., > 125 ppm), 12 measurements were reported semiquantitatively (i.e., > 200 ppm) by the RaPID Assay. This is illustrated in Figure 5-2, which is a plot of the RaPID Assay's measured PCB soil concentrations vs the corresponding reference laboratory



**Figure 5-2. Paired PCB measurements for RaPID Assay and reference laboratory for (a) all soil samples (RaPID Assay results reported as > 200 ppm not included) and (b) soil samples where the reference laboratory results were less than or equal to 125 ppm. Lines denote perfect correlation.**

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measured concentrations (excluding the suspect values listed in Table 4-1). Figure 5-2 (a) is a plot of all of the soil data, and (b) is a plot of the concentration region from 0 to 125 ppm, where most of the variation can be viewed. Note that the diagonal lines drawn in Figure 5-2 represent the line of theoretically perfect correlation ( $R^2 = 1.0$ ) between the reference laboratory data set (plotted along the x axis) and the RaPID Assay data set (plotted along the y axis). A value above the diagonal line indicates that the RaPID Assay's measurement was higher than the reference laboratory's measurement, while those below the diagonal line indicated a lower result.

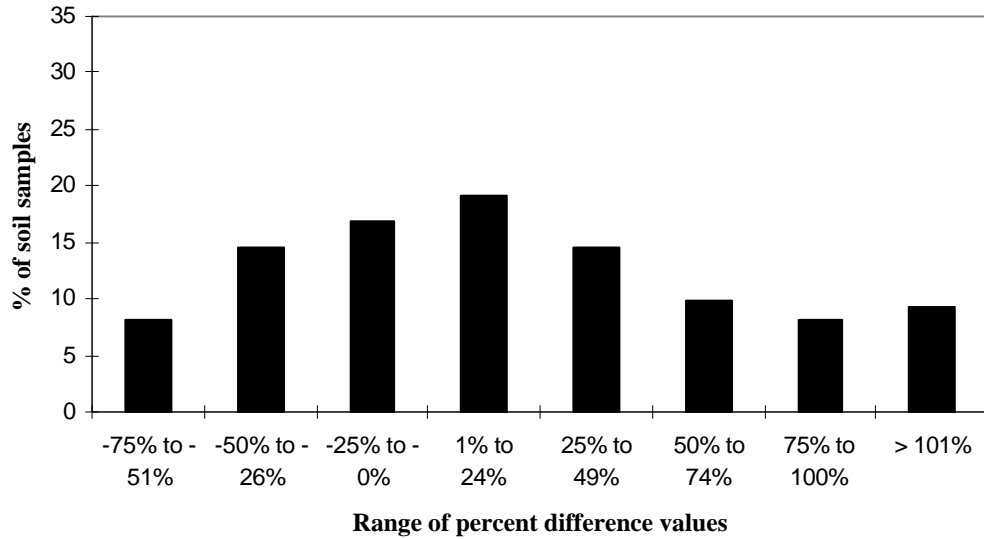
Coefficients of determination ( $R^2$ ) [10] were computed using a linear model fitted to the plot of the RaPID Assay PCB concentrations vs the reference laboratory PCB concentrations. Excluding the reference laboratory's suspect measurements, the coefficient of determination ( $R^2$ ) was 0.754 when all soil samples (0 to 900 ppm) were considered. As shown in Figure 5-2 (b), the majority of the soil samples were in the concentration range of 0 to 125 ppm. The  $R^2$  value for this concentration range was 0.716.

A direct comparison between the RaPID Assay and reference laboratory data was performed by evaluating the percent difference (%D) between the measured concentrations, defined as:

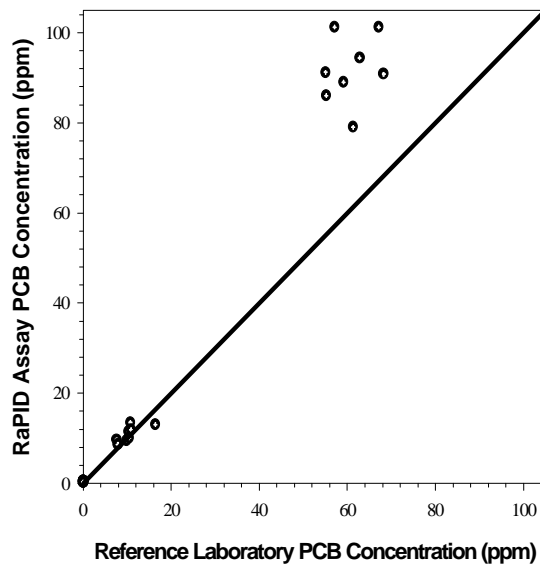
$$\%D = \frac{[RaPID] - [Ref Lab]}{[Ref Lab]} \times 100\% \quad (5-1)$$

Figure 5-3 provides a summary of the range of %D values for the soil samples, as calculated using Equation 5-1. The graph represents the percentage of samples that fall within each range of %D values but does not reflect any grouping according to the actual concentrations of the replicate sets. Results for Sample IDs 110, 112, 126, and 226 were not included because the reference laboratory did not report quantitative results for these samples. Results for samples that the RaPID Assay Kit reported as >200 ppm were also not included. As shown in Figure 5-3, the %D values were evenly distributed between -75% to 100%. Approximately 40% of the samples were biased low (%D < -1%) relative to the reference laboratory results. Approximately 36% of the soil sample results had %D values within the range of  $\pm 25\%$ .

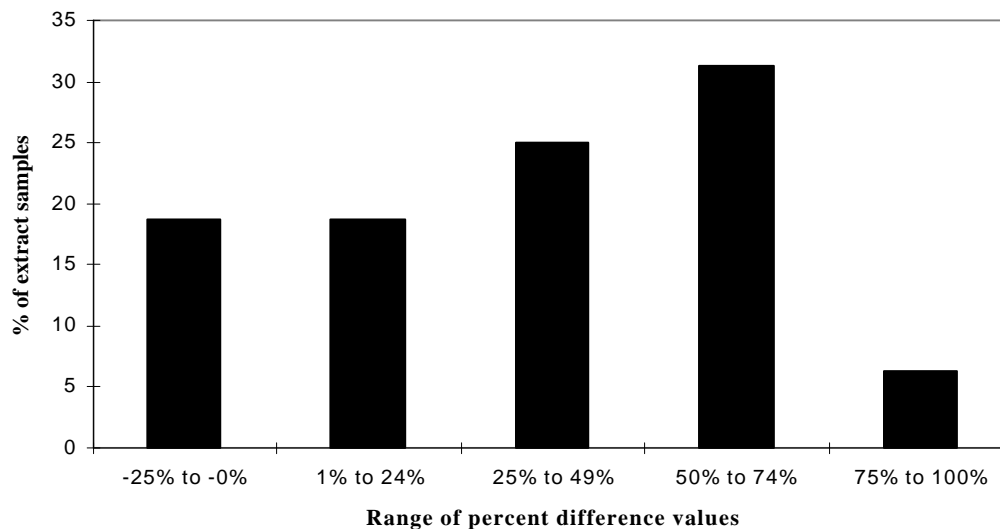
Comparability was also assessed for the extract samples. Figure 5-4 is a plot of the RaPID Assay measured PCB extract concentrations vs the corresponding reference laboratory measured concentrations. These data indicated that the RaPID Assay was biased high for the 100-ppm extract sample results relative to the reference laboratory. However, compared with the actual spike concentrations, the RaPID Assay's results for these extract samples were accurate because the reference laboratory results were biased low. The coefficient of determination ( $R^2$ ) for a line fitted to this data was 0.977, indicating near perfect correlation. The %D values for the extract samples were also assessed and are shown in Figure 5-5. Because the reference laboratory was biased low on the 100-ppm extract measurements relative to the actual spiked concentrations, when compared with the RaPID Assay measurements, approximately half of the %D values were between 50 and 75%. Approximately 56% of the extract sample results had %D values within  $\pm 25\%$ .



**Figure 5-3. Range of percent difference values for the comparison of the RaPID Assay's soil sample results with the reference laboratory results.**



**Figure 5-4. Paired extract PCB measurements for the RaPID Assay and reference laboratory. Measurements above the diagonal line indicate that the RaPID Assay's measurements are higher than the reference laboratory's measurements.**



**Figure 5-5. Range of percent difference values for the comparison of the RaPID Assay's extract sample results with the reference laboratory results.**

The soil data not included in previous comparability evaluations (because the replicate data for the reference laboratory were considered suspect) are shown in Table 5-8. 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 RaPID Assay's matching results. For sample IDs 110 and 112, the reference laboratory obtained qualitative results only, while SDI reported quantitative PCB concentrations for the four replicates that were precise. For two of the other five suspect measurements (sample IDs 106 and 217), SDI reported qualitative results only. For the other three suspect measurements, the RaPID Assay generated results for all four replicates that were consistent and that were comparable with the reference laboratory's replicate means that excluded the suspect value. These comparisons demonstrate the RaPID Assay's ability to successfully analyze some of the samples that were troublesome for the reference laboratory.

### **Summary of PARCC Observations**

Table 5-9 provides a summary of the performance of SDI's RaPID Assay System for the analysis of all sample types used in this demonstration. The reference laboratory's performance (excluding suspect data) is also presented in this table for comparison. In terms of precision, the overall average RSD for the RaPID Assay, weighted for the number of samples, was 12% for the soil data generated under the chamber conditions, compared with 25% under outdoor conditions. This data indicated that the RaPID Assay System exhibited a significant site effect in terms of the precision of the measurements. While the variability was lower under the controlled chamber conditions, the outdoor precision was still comparable to the overall precision for the reference laboratory (21% RSD). For the extract samples, the overall RSD of the RaPID Assay was comparable to the reference laboratory (12% and 14%, respectively).

**Table 5-8. Comparison of the reference laboratory’s suspect data with the RaPID Assay System data**

| Sample ID | Reference Laboratory      |                                   | RaPID Assay System                 |                      |
|-----------|---------------------------|-----------------------------------|------------------------------------|----------------------|
|           | Suspect Measurement (ppm) | Replicate Mean <sup>a</sup> (ppm) | Suspect-matching Measurement (ppm) | Replicate Mean (ppm) |
| 110       | ≤RDL <sup>b</sup>         | ≤RDL <sup>b</sup>                 | n/a                                | 31.0                 |
| 112       | ≤RDL <sup>b</sup>         | ≤RDL <sup>b</sup>                 | n/a                                | 123.8                |
| 106       | 649.6                     | 281.0                             | > 200                              | > 200                |
| 205       | 3,305.0                   | 493.0                             | 185.4                              | 198.0                |
| 216       | 151.6                     | 55.1                              | 55.2                               | 57.1                 |
| 217       | 1,913.3                   | 659.8                             | > 200                              | > 200                |
| 225       | 146.0                     | 41.7                              | 73.2                               | 77.3                 |

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

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

**Table 5-9. Summary of PARCC observations for the RaPID Assay System**

| Sample Matrix | Sample Type                         | RaPID Assay Number of Samples | Precision (Average % RSD)        |                      | Accuracy (Average % Recovery)       |                              |
|---------------|-------------------------------------|-------------------------------|----------------------------------|----------------------|-------------------------------------|------------------------------|
|               |                                     |                               | RaPID Assay                      | Reference Laboratory | RaPID Assay                         | Reference Laboratory         |
| Blank         | Soil Extract                        | 8<br>8                        | n/a                              | n/a                  | All reported as < detection limits. | All reported as non-detects. |
| Soil          | PE                                  | 32                            | 26 (outdoor)                     | 18 <sup>a</sup>      | 103                                 | 101 <sup>a</sup>             |
|               |                                     | 32                            | 15 (chamber)                     |                      |                                     |                              |
|               | Environmental <125 ppm <sup>b</sup> | 52                            | 24 (outdoor)                     | 23 <sup>a</sup>      |                                     |                              |
|               |                                     | 56                            | 11 (chamber)                     |                      |                                     |                              |
|               | >125 ppm <sup>c</sup>               | 4                             | 25 (outdoor)                     | 19 <sup>a</sup>      |                                     |                              |
|               |                                     | 4                             | 6 (chamber)                      |                      |                                     |                              |
| Sample ID 110 | 4                                   | 30 (outdoor)                  | not quantified<br>not quantified |                      |                                     |                              |
| Sample ID 112 | 4                                   | 16 (outdoor)                  |                                  |                      |                                     |                              |
| Overall       |                                     | 96                            | 25 (outdoor)                     | 21 <sup>a</sup>      | 103                                 | 101 <sup>a</sup>             |
|               |                                     | 92                            | 12 (chamber)                     |                      |                                     |                              |
| Extract       | 10 ppm                              | 8                             | 16                               | 19                   | 110                                 | 104                          |
|               | 100 ppm                             | 8                             | 8                                | 8                    | 92                                  | 64                           |
|               | Overall                             | 16                            | 12                               | 14                   | 101                                 | 84                           |

<sup>a</sup> Average result excluding the suspect measurements.

<sup>b</sup> Samples where the reference laboratory values were <125 ppm.

<sup>c</sup> Samples where the reference laboratory values were >125 ppm, excluding measurements where SDI reported semiquantitative results (i.e., >200 ppm).



In terms of accuracy, the RaPID Assay’s PE soil measurements were biased both high (152% recovery) and low (60% recovery), such that the overall average percent recovery was 103%. However, the results are considered biased because the direction of bias was dependent on the Aroclor type present in the sample, as discussed in the “Accuracy” section. In comparison, the reference laboratory reported unbiased PCB concentrations (101% recovery) for the PE soil samples. Extract measurements by the RaPID Assay were unbiased at both 10 ppm (110% recovery) and 100 ppm (92% recovery) concentration levels. The reference laboratory results were unbiased at 10 ppm (104% recovery) but were biased low at 100 ppm (64% recovery) for the extract samples.

SDI correctly reported all blanks analyses as “less than the detection limit,” as did the reference laboratory (i.e., 0% fp). Two fn were reported by the RaPID Assay. Overall, the performance of the RaPID Assay System for the PCB demonstration samples was characterized as biased and precise.

**Regulatory Decision-Making Applicability**

One of the objectives 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<sup>2</sup> for surface wipes. To assess this, the RaPID Assay’s performance for soil samples (both PE and environmental soil samples) ranging in concentration from 40 to 60 ppm can be used, and the data are provided in Table 5-10. The performance of the RaPID Assay System for this concentration range showed comparable precision (21% RSD) and slightly higher percent recovery (129%) compared with the entire PE and environmental soil sample data set. The mean %D value was 39% when compared with the corresponding reference laboratory result. The RaPID Assay System’s performance on extract samples is provided in Tables 5-4 and 5-7. Assuming a 10-mL extract volume, extract samples (at 10 and 100 µg/mL) represented surface wipe sample concentrations of 100 and 1000 µg/100 cm<sup>2</sup>. For the simulated wipe extract samples, the RaPID Assay was precise (12% RSD) and accurate (101% recovery).

**Table 5-10. Performance of the RaPID Assay System for soil samples between 40 and 60 ppm**

| Overall Performance         | Precision (% RSD) | Accuracy (% Recovery) | Comparability (% Difference) |
|-----------------------------|-------------------|-----------------------|------------------------------|
| Mean                        | 21                | 129                   | 39                           |
| Median                      | 14                | 129                   | 37                           |
| 95 <sup>th</sup> percentile | 40                | 175                   | 77                           |

**Additional Performance Factors**

***Detection Limits***

The MDL is often defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is higher than zero. An MDL is determined from repeated analyses of a sample in a given matrix containing the analyte [12]. The reported MDL for the RaPID Assay Kit was 0.5 ppm. An MDL, calculated from the data for the PE samples, was 1.5 ppm.

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### ***Sample Throughput***

Sample throughput is representative of the average amount of time required to extract the PCBs, perform appropriate reactions, and to analyze the sample. SDI's sample throughput rate was relatively consistent under both environmental conditions at 10 to 11 samples/h.

### ***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 RaPID Assay System 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 RaPID Assay System 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 were considered in the estimate:

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

Each of these cost factors is defined and discussed in the following and serves as the basis for the estimated cost ranges presented in Table 5-11. This analysis assumed that the individuals performing the analyses were fully trained to operate the technology. SDI recommends that new users attend a training session that they offer on the use of the RaPID Assay System. Sample acquisition and preanalytical sample preparation costs, tasks common to both methods, are not included here.

### **RaPID Assay System Costs**

Because the samples were analyzed on-site, no sample shipment charges were associated with the cost of operating the RaPID Assay System. 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 h, at a rate of \$50/h.
- 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/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/day per analyst.
- Rate: The cost of the on-site labor was estimated at a rate of \$30 to \$75/h, 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.

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

| <b>RaPID Assay System<br/>Strategic Diagnostics Inc.</b> |                              | <b>EPA SW-846 Method 8080/8081/8082<br/>Reference Laboratory</b> |                       |
|--|------------------------------|--|-----------------------|
| <b>Sample throughput rate: 10 - 11 samples per hour</b>  |                              | <b>Typical turn-around time: 14 - 30 days</b>                    |                       |
| Cost Category  | Cost (\$)                    | Cost Category  | Cost (\$)             |
| <b>Sample Shipment</b>                                   | 0                            | <b>Sample Shipment</b>   |                       |
|  |                              | Labor  | 100 - 200             |
|  |                              | Overnight shipping charges                                       | 50 - 150              |
| <b>Labor</b>   |                              | <b>Labor</b>   |                       |
| Mobilization/demobilization                              | 250 - 400                    | Mobilization/demobilization                                      | Included <sup>a</sup> |
| Travel   | 15 - 1,000 per analyst       | Travel   | Included              |
| Per diem   | 0 - 150 per day per analyst  | Per diem   | Included              |
| Rate   | 30 - 75 per hour per analyst | Rate   | 44 - 239 per sample   |
| <b>Equipment</b>   |                              | <b>Equipment</b>   |                       |
| Mobilization/demobilization                              | 0 - 150                      | Mobilization/demobilization                                      | included              |
| Kit rental fee   | 450 per week                 | Rental/purchase of system  | Included              |
| Kit purchase price                                       | 1,665                        | Reagents/supplies  | Included              |
| Photometer purchase price                                | 595 - 3,985                  |  |                       |
| Training   | < 935                        |  |                       |
| Reagents/supplies  | 21 per sample                |  |                       |
| <b>Waste Disposal</b>                                    | 75 - 1,060                   | <b>Waste Disposal</b>  | Included              |

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

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.
- Rental/Purchase: The fee to rent the RaPID Assay System at the time of the demonstration study was \$450 per week. At the time of the demonstration, the cost of purchasing the equipment accessory kit was \$1,665. The price of the photometer was \$595 or \$3,985 (depending on the model).

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- **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 RaPID Assay was \$21 per sample. This cost included the sample preparation supplies, assay supplies, and consumable reagents.

Waste disposal costs were estimated based on the 1997 regulations for disposal of PCB-contaminated waste. Using the RaPID Assay, 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 (i.e., used and unused soil, gloves, paper towels, ampules). The disposal costs for the PCB solid waste by incineration at a commercial facility was estimated at \$1.50/lb. For comparison, the cost for PCB waste disposal at ETTP was estimated at \$18/lb for solids. The RaPID Assay 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; the cost at ETTA was estimated at \$11/lb.

#### **Reference Laboratory 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.

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

The labor bids from commercial analytical reference laboratories who offered to perform the PCB analysis for this demonstration ranged from \$44/sample to \$239/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 range 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/sample). This rate was a fully loaded analytical cost, including equipment, labor, waste disposal, and report preparation.

#### **Cost Assessment Summary**

An overall cost estimate for the RaPID Assay System vs the reference laboratory was not made because of the extent of variation in the different cost factors, as outlined in Table 5-11. The overall costs for the application of each technology will also 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.

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### **General Observations**

The following are general observations regarding the field operation of the RaPID Assay System:

- The system was light, easily transportable, and rugged. It took about 1 h 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 and brought everything inside at the close of each day. It took the SDI team less than 1 hour each morning to prepare for sample analysis.
- Three operators were used for the technology demonstration because of the number of samples and working conditions, but the technology can be run 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 techniques.
- The measurement system (RPA-I RaPID Analyzer) required 120-V ac power. Alternatively, it can be operated using a car battery.
- Using the RaPID Assay, 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 (i.e., used and unused soil, gloves, paper towels, ampules). The RaPID Assay also generated approximately 19 lb of liquid waste.

### **Performance Summary**

A summary of the performance characteristics of SDI's RaPID Assay System, presented previously in this section, is shown in Table 5-12. The overall performance of the RaPID Assay System was characterized as biased (with the direction of the bias dependent on Aroclor type) and precise for a given set of environmental conditions.

**Table 5-12. Performance Summary for the RaPID Assay System**

| Feature/Parameter                            | Performance Summary   |
|--|---|
| Blank samples                                | <b>Soils:</b> No PCBs detected<br><b>Extracts:</b> No PCBs detected   |
| Method detection limit                       | SDI specified: 0.5 ppm<br>Calculated: 1.5 ppm   |
| Precision                                    | <b>Average RSD</b><br>PE soils: 26% (outdoor); 15% (chamber)<br>Environmental soils: 24% (outdoor); 13% (chamber)<br>Extracts: 14%  |
| Accuracy                                     | <b>Average Percent Recovery</b><br>PE soils: 103% (both positive and negative Aroclor-dependent biases)<br>Extracts: 101%   |
| False positive results                       | <b>Blank Soils:</b> 0% (0 of 8 samples)<br><b>Blank Extracts:</b> 0% (0 of 8 samples)   |
| False negative results                       | <b>PE and Environmental Soils:</b> 1% (2 of 192 samples)<br><b>Spiked Extracts:</b> 0% (0 of 16 samples)  |
| Comparison with reference laboratory results | <b>PE and Environmental Soil Samples</b><br>Percent difference: 40% of samples were $\pm 25\%$ D<br>Coefficients of determination ( $R^2$ ): 0.754 (all data)<br>0.716 (<125 ppm)<br><b>Extract Samples</b><br>Percent difference: 56% of samples were $\pm 25\%$ D<br>Coefficient of determination ( $R^2$ ): 0.977  |
| Regulatory decision-making applicability     | <b>40 to 60 ppm PE and Environmental Soil Samples</b><br>precision: 21% average RSD<br>accuracy: 129% average recovery<br>comparability: 39% average percent difference<br><b>100 <math>\mu\text{g}/100\text{cm}^2</math> and 100 <math>\mu\text{g}/100\text{cm}^2</math> Extract Samples</b><br>precision: 12% average RSD<br>accuracy: 101% average recovery<br>comparability: 46% average difference |
| Sample throughput                            | 10 - 11 samples/h   |
| Operator requirements                        | Basic knowledge of field chemical techniques; 2-4 h technology-specific training  |
| Power requirements                           | 120V AC or car battery (for RPA-I Analyzer)   |
| Cost   | Consumables: \$21 per sample<br>Instrument: \$450 weekly rental; \$1,665 purchase accessories; \$595 - 3,985 purchase photometer  |
| Hazardous waste generation                   | Approximately 20 lb of solid/liquid<br>(classified as solids suitable for disposal by incineration)<br>Approximately 19 lb of liquid<br>Approximately 20 lb of solid (used gloves, pipettes, paper towels, etc.)<br>Approximately 19 lb of liquid waste (aqueous with trace methanol)   |

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## **Section 6**

### **Technology Update and Representative Applications**

#### **Objective**

In this section, SDI describes new technology developments that have occurred since the demonstration activities. In addition, the developer has provided a list of representative applications where the SDI RaPID Assay System 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 SDI's four remediation immunoassay test kit product lines. The affected product lines include the EnviroGard, EnSys (not demonstrated), 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 in 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.

##### ***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.

Please note that the previously listed product improvements are ongoing projects; therefore, the information presented is subject to change.

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### **Representative Applications**

In a 1997 report entitled, "Field Analytical and Site Characterization Technologies: Summary of Applications" [13], the use of SDI immunoassay kits is documented at more than 30 remediation sites under state or federal oversight. Contact information is provided 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 at <http://www.epa.gov/ncepihom/>. The summary report is available for viewing or downloading as a Portable Document Format (PDF) file from the CLU-IN Internet Web site: <http://clu-in.com/pubichar.htm>.

### **Data Quality Objective Example**

This application of SDI's RaPID Assay System is based on data quality objective (DQO) methods for project planning advocated by the American Society for Testing and Materials (ASTM) [14, 15] and EPA [16]. ORNL derived a DQO example from the performance results in Section 5. This example, which is presented in Appendix E, illustrates the use of the RaPID Assay's performance data from the ETV demonstration in the DQO process to select the number of samples and to quantify the action level for the decision rule.



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## Section 7

### References

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[12] “Definition and Procedure for the Determination of the Method Detection Limit,” *Code of Federal Regulations*, CFR 40, Pt. 136, Appendix B, rev. 1.11.

[13] *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.

[14] 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.

[15] 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.

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

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**Appendix A**  
**Description of Environmental Soil Samples**



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

| <b>Location</b>          | <b>Request for Disposal (RFD) #</b> | <b>Drum #</b>   | <b>Description</b>   |
|--------------------------|-------------------------------------|---|--|
| 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<br>02<br>03<br>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<br>02<br>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<br>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<br>02<br>03<br>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<br>1069<br>1096<br>1898<br>2143<br>2528<br>3281<br>538<br>940<br>4096 | Soil from the DOE Portsmouth Gaseous Diffusion Plant in Ohio. This soil is PCB-contaminated soil from a probable PCB oil spill into the East Drainage Ditch that was excavated in 1986.  |
| Tennessee Reference Soil | n/a                                 | n/a   | Captina silt loam from Roane County, Tennessee; used as a blank in this study (i.e., not contaminated with PCBs)   |



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





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

| Location   | Sample ID | RFD Drum # <sup>a</sup> | Composition |        |               | Total Organic Carbon (mg/kg) | pH   |
|------------|-----------|-------------------------|-------------|--------|---------------|------------------------------|------|
|            |           |                         | % gravel    | % sand | % silt + clay |                              |      |
| Oak Ridge  | 101       | 40022-02                | 0           | 91.8   | 8.2           | 5384                         | 7.12 |
|            | 102       | 40267-03                | 0.5         | 99.3   | 0.2           | 13170                        | 7.30 |
|            | 103       | 40267-01                | 0.2         | 96.7   | 3.1           | 13503                        | 7.21 |
|            | 104       | 40267-04                | 0.6         | 98.2   | 1.2           | 15723                        | 7.07 |
|            | 105       | 40267-01S <sup>b</sup>  | 0.5         | 94.8   | 4.7           | 14533                        | 7.28 |
|            | 106       | 24375-03                | 0.5         | 87.8   | 11.7          | 19643                        | 7.36 |
|            | 107       | 24375-01                | 2.5         | 92.5   | 5.0           | 1196                         | 7.26 |
|            | 108       | 40267-02                | 0.4         | 94.2   | 5.4           | 9007                         | 7.30 |
|            | 109       | 24375-02                | 0.3         | 93.1   | 6.6           | 1116                         | 7.48 |
|            | 110       | 43275-01                | 0           | 89.2   | 10.8          | 14250                        | 7.57 |
|            | 111       | 134555-03S <sup>b</sup> | 0.5         | 88.1   | 11.4          | 10422                        | 7.41 |
|            | 112       | 43275-02                | 0.1         | 91.4   | 8.5           | 38907                        | 7.66 |
|            | 126, 226  | non-PCB soil            | 0           | 85.6   | 14.4          | 9249                         | 7.33 |
| Paducah    | 113, 201  | 97002-04                | 0           | 92.4   | 7.6           | 1296                         | 7.71 |
|            | 114, 202  | 97002-01                | 0.2         | 87.6   | 12.2          | 6097                         | 7.64 |
|            | 115, 203  | 97002-03                | 0.1         | 83.6   | 16.3          | 3649                         | 7.59 |
|            | 116, 204  | 97002-02                | 0.4         | 93.7   | 5.8           | 4075                         | 7.43 |
|            | 117, 205  | 97002-02S <sup>b</sup>  | 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       | 7515-0538               | 0.5         | 73.3   | 26.3          | 1328                         | 7.78 |
|            | 211       | 7515-0538S <sup>b</sup> | 0.5         | 73.3   | 26.3          | 1328                         | 7.78 |
|            | 217       | 7515-0538S <sup>b</sup> | 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 |

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

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



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

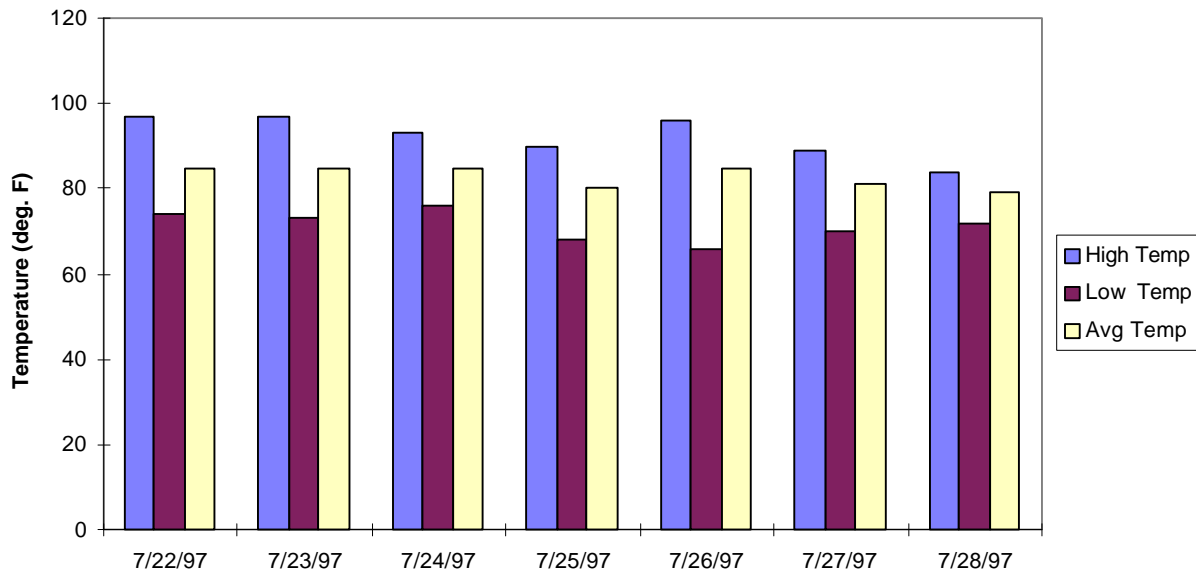


**Table C-1. Average temperature and relative humidity conditions during testing periods**

| Date    | Outdoor Site             |                               | Chamber Site             |                               |
|---------|--------------------------|-------------------------------|--------------------------|-------------------------------|
|         | Average Temperature (°F) | Average Relative Humidity (%) | Average Temperature (°F) | Average Relative Humidity (%) |
| 7/22/97 | 85                       | 62                            | 70 <sup>a</sup>          | 38 <sup>a</sup>               |
| 7/23/97 | 85                       | 70                            | 60 <sup>a</sup>          | 58 <sup>a</sup>               |
| 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</b>                 | <b>b</b>                      | 55                       | 50                            |

<sup>a</sup> The chamber was not operating properly on this day. See discussion in Section 3.

<sup>b</sup> No developers were working outdoors on this day.



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

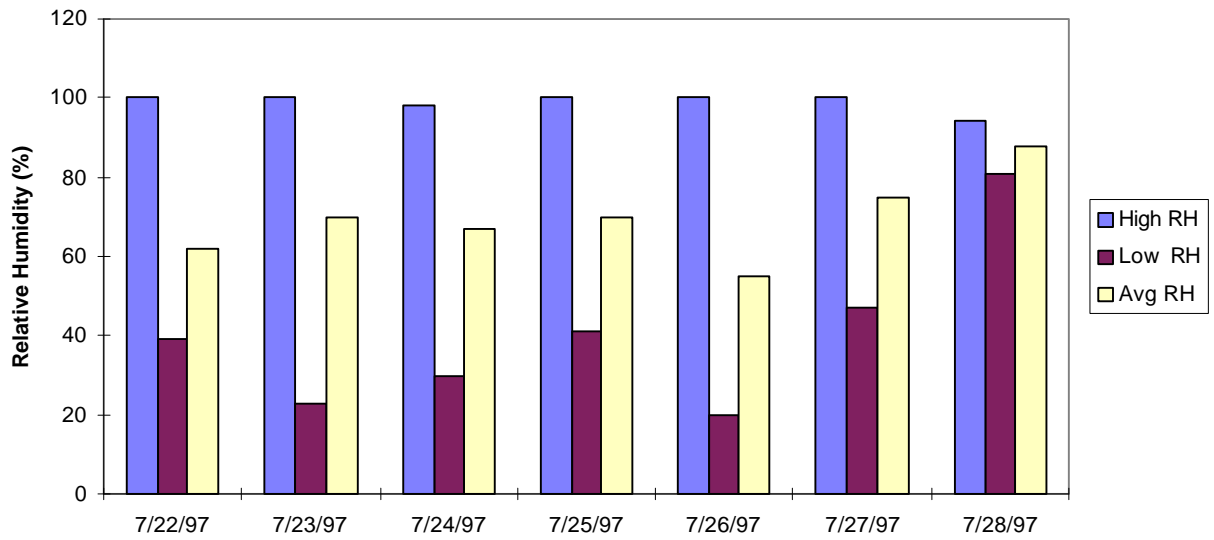


Figure C-2. Summary of relative humidity conditions for the outdoor site.

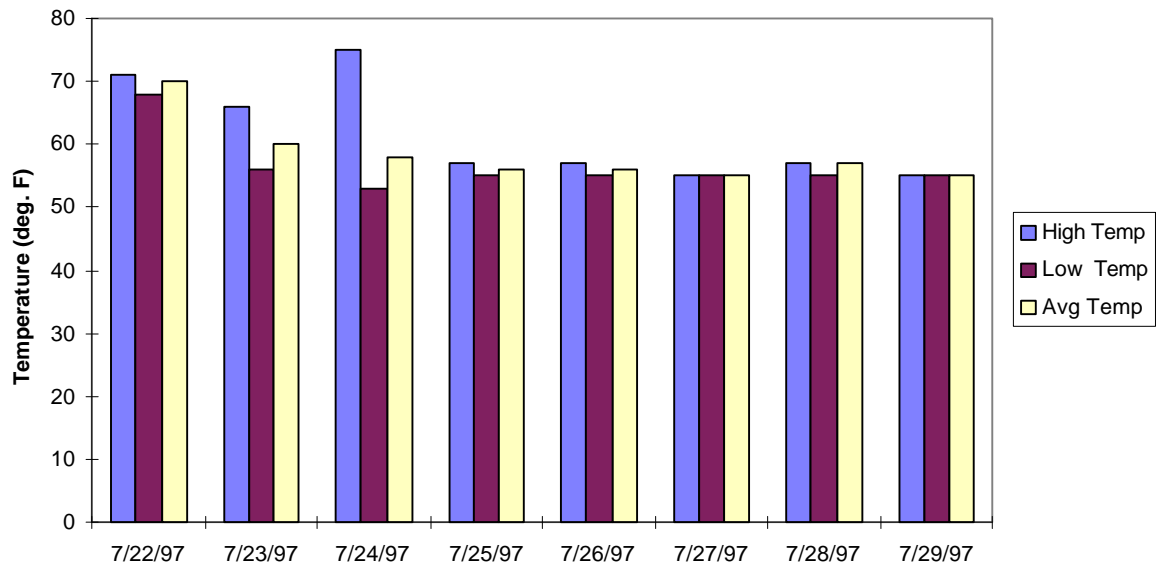


Figure C-3. Summary of temperature conditions for chamber site.

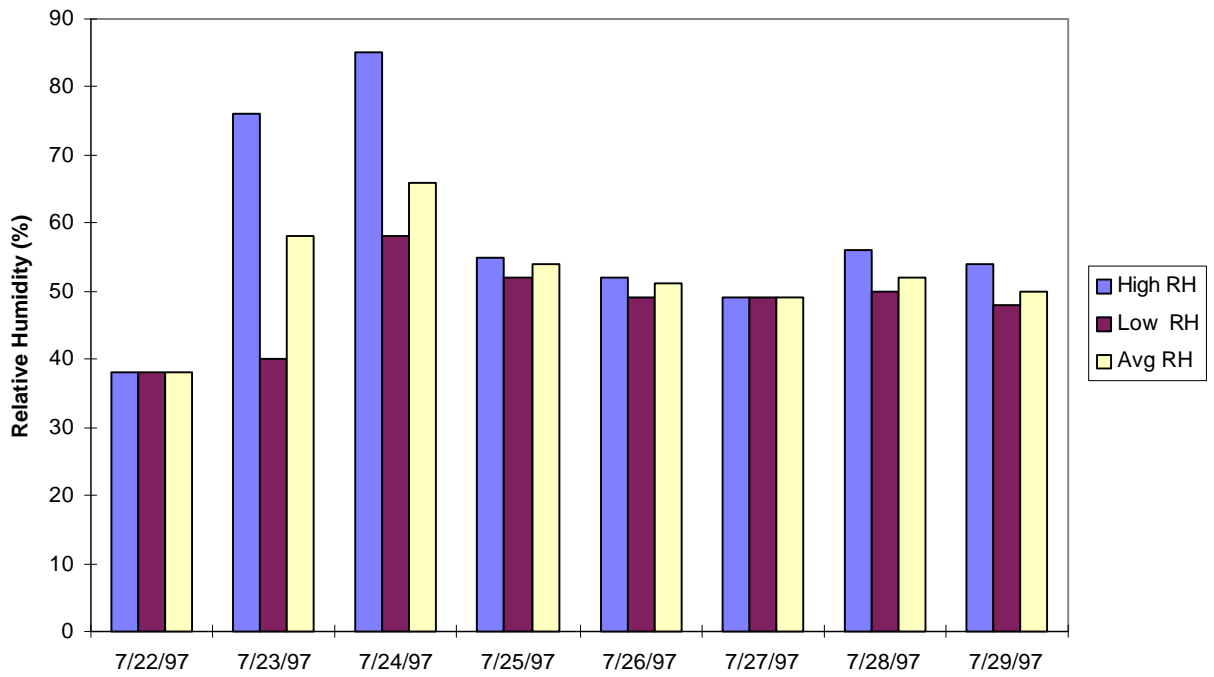


Figure C-4. Summary of relative humidity conditions for chamber site.





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**Appendix D**  
**RaPID Assay System for PCB Analysis**  
**PCB Technology Demonstration Sample Data**



**Legend for Appendix D Tables**

| <b>Table Heading</b> | <b>Definition</b>   |
|----------------------|---|
| Obs                  | Observation   |
| Sample ID            | Sample identification<br>101 to 126 = outdoor site soil samples<br>127 to 130 = outdoor site extract samples<br>201 to 226 = chamber site soil samples<br>227 to 230 = chamber site extract samples |
| Rep                  | Replicate of sample ID (1 through 4)  |
| RaPID Result         | RaPID Assay System measured PCB concentration (ppm)   |
| Ref Lab Result       | LAS reference laboratory measured PCB concentration (ppm)<br>Values with “≤” are samples that the reference laboratory reported as<br>“≤ reporting detection limit”                                 |
| Reference Aroclor    | Aroclor(s) identified by the reference laboratory   |
| Type                 | Sample = environmental soil<br>1242, 1248, 1254, 1260 = Aroclor in PE samples<br>Blank = non-PCB-contaminated sample  |
| Order                | Order of sample analysis (started with 2001–2116, then 1001–1116)   |

**Table D-1. RaPID Assay System PCB technology demonstration soil sample data**

| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type   | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|--------|-------|
| 1   | 101       | 1   | <0.5               | 0.6                  | 1254              | Sample | 1079  |
| 2   | 101       | 2   | <0.5               | 0.4                  | 1254              | Sample | 1069  |
| 3   | 101       | 3   | <0.5               | 0.5                  | 1254              | Sample | 1020  |
| 4   | 101       | 4   | 0.6                | 0.5                  | 1254              | Sample | 1025  |
| 5   | 102       | 1   | 3.2                | 2.2                  | 1254              | Sample | 1016  |
| 6   | 102       | 2   | 3.5                | 2.1                  | 1254              | Sample | 1055  |
| 7   | 102       | 3   | 3.2                | 1.7                  | 1260              | Sample | 1022  |
| 8   | 102       | 4   | 3.8                | 2.5                  | 1260              | Sample | 1024  |
| 9   | 103       | 1   | 5.8                | 3.0                  | 1254              | Sample | 1021  |
| 10  | 103       | 2   | 3.8                | 2.4                  | 1254              | Sample | 1059  |
| 11  | 103       | 3   | 4.3                | 2.0                  | 1260              | Sample | 1103  |
| 12  | 103       | 4   | 6.4                | 1.6                  | 1260              | Sample | 1049  |
| 13  | 104       | 1   | 25.6               | 6.8                  | 1260              | Sample | 1029  |
| 14  | 104       | 2   | 9.2                | 6.0                  | 1254              | Sample | 1094  |
| 15  | 104       | 3   | 9.3                | 14.8                 | 1254              | Sample | 1004  |
| 16  | 104       | 4   | 17.2               | 9.9                  | 1254              | Sample | 1028  |
| 17  | 105       | 1   | 74.4               | 49.7                 | 1260              | Sample | 1099  |
| 18  | 105       | 2   | 107.2              | 84.1                 | 1260              | Sample | 1031  |
| 19  | 105       | 3   | 110.0              | 50.6                 | 1260              | Sample | 1040  |
| 20  | 105       | 4   | 74.0               | 53.2                 | 1260              | Sample | 1005  |
| 21  | 106       | 1   | >200.0             | 269.6                | 1254              | Sample | 1001  |
| 22  | 106       | 2   | >200.0             | 255.9                | 1254              | Sample | 1102  |
| 23  | 106       | 3   | >200.0             | 317.6                | 1254              | Sample | 1088  |
| 24  | 106       | 4   | >200.0             | 649.6                | 1254              | Sample | 1014  |
| 25  | 107       | 1   | 1.8                | 1.0                  | 1254              | Sample | 1071  |
| 26  | 107       | 2   | 1.5                | 1.6                  | 1254              | Sample | 1043  |
| 27  | 107       | 3   | 2.2                | 1.2                  | 1254              | Sample | 1042  |
| 28  | 107       | 4   | 2.0                | 1.2                  | 1254              | Sample | 1050  |
| 29  | 108       | 1   | 2.5                | 1.7                  | 1254              | Sample | 1018  |
| 30  | 108       | 2   | 2.9                | 2.0                  | 1254              | Sample | 1077  |
| 31  | 108       | 3   | 4.5                | 1.7                  | 1254              | Sample | 1046  |
| 32  | 108       | 4   | 3.0                | 1.9                  | 1254              | Sample | 1101  |
| 33  | 109       | 1   | 4.1                | 1.5                  | 1254              | Sample | 1053  |
| 34  | 109       | 2   | 3.9                | 2.1                  | 1254              | Sample | 1058  |
| 35  | 109       | 3   | 2.8                | 1.8                  | 1254              | Sample | 1067  |
| 36  | 109       | 4   | 2.6                | 2.4                  | 1254              | Sample | 1082  |
| 37  | 110       | 1   | 26.4               | ≤490.0               | Non-Detect        | Sample | 1066  |
| 38  | 110       | 2   | 30.8               | ≤99.0                | Non-Detect        | Sample | 1091  |
| 39  | 110       | 3   | 44.0               | ≤66.0                | Non-Detect        | Sample | 1009  |
| 40  | 110       | 4   | 22.8               | ≤98.0                | Non-Detect        | Sample | 1097  |

| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type   | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|--------|-------|
| 41  | 111       | 1   | 38.8               | 44.5                 | 1254              | Sample | 1063  |
| 42  | 111       | 2   | 47.6               | 36.0                 | 1254              | Sample | 1052  |
| 43  | 111       | 3   | 67.2               | 39.3                 | 1254              | Sample | 1083  |
| 44  | 111       | 4   | 42.8               | 35.1                 | 1254              | Sample | 1070  |
| 45  | 112       | 1   | 97.2               | ≤66.0                | Non-Detect        | Sample | 1096  |
| 46  | 112       | 2   | 125.2              | ≤200.0               | Non-Detect        | Sample | 1081  |
| 47  | 112       | 3   | 143.6              | ≤130.0               | Non-Detect        | Sample | 1032  |
| 48  | 112       | 4   | 129.2              | ≤200.0               | Non-Detect        | Sample | 1078  |
| 49  | 113       | 1   | 1.3                | 0.7                  | 1260              | Sample | 1027  |
| 50  | 113       | 2   | 1.3                | 1.1                  | 1260              | Sample | 1017  |
| 51  | 113       | 3   | 1.4                | 0.6                  | 1260              | Sample | 1036  |
| 52  | 113       | 4   | 1.7                | 1.9                  | 1248/1260         | Sample | 1038  |
| 53  | 114       | 1   | 0.9                | 1.1                  | 1260              | Sample | 1045  |
| 54  | 114       | 2   | 1.2                | 1.2                  | 1260              | Sample | 1100  |
| 55  | 114       | 3   | 0.7                | 1.3                  | 1260              | Sample | 1011  |
| 56  | 114       | 4   | 1.6                | 1.7                  | 1260              | Sample | 1104  |
| 57  | 115       | 1   | 12.0               | 14.9                 | 1248              | Sample | 1089  |
| 58  | 115       | 2   | 13.4               | 12.4                 | 1016              | Sample | 1092  |
| 59  | 115       | 3   | 15.4               | 15.0                 | 1248              | Sample | 1057  |
| 60  | 115       | 4   | 19.4               | 16.9                 | 1248              | Sample | 1061  |
| 61  | 116       | 1   | 119.0              | 41.4                 | 1248              | Sample | 1035  |
| 62  | 116       | 2   | 58.5               | 41.2                 | 1016              | Sample | 1015  |
| 63  | 116       | 3   | 69.3               | 48.5                 | 1248              | Sample | 1047  |
| 64  | 116       | 4   | 56.6               | 34.0                 | 1016              | Sample | 1075  |
| 65  | 117       | 1   | 296.0              | 431.6                | 1016              | Sample | 1034  |
| 66  | 117       | 2   | 230.6              | 406.3                | 1016              | Sample | 1048  |
| 67  | 117       | 3   | 205.6              | 304.7                | 1016              | Sample | 1003  |
| 68  | 117       | 4   | 163.3              | 392.8                | 1016              | Sample | 1076  |
| 69  | 118       | 1   | 1.7                | 2.1                  | 1248              | 1248   | 1026  |
| 70  | 118       | 2   | 1.5                | 1.9                  | 1016              | 1248   | 1013  |
| 71  | 118       | 3   | 1.3                | 0.7                  | 1248              | 1248   | 1044  |
| 72  | 118       | 4   | 1.6                | 1.6                  | 1248              | 1248   | 1023  |
| 73  | 119       | 1   | 9.0                | 21.2                 | 1016              | 1248   | 1093  |
| 74  | 119       | 2   | 11.8               | 17.2                 | 1248              | 1248   | 1006  |
| 75  | 119       | 3   | 23.1               | 17.4                 | 1248              | 1248   | 1019  |
| 76  | 119       | 4   | 16.5               | 24.4                 | 1248              | 1248   | 1010  |
| 77  | 120       | 1   | 4.8                | 4.5                  | 1254              | 1254   | 1074  |
| 78  | 120       | 2   | 6.3                | 4.0                  | 1254              | 1254   | 1073  |
| 79  | 120       | 3   | 8.4                | 6.3                  | 1254              | 1254   | 1039  |
| 80  | 120       | 4   | 5.7                | 5.0                  | 1254              | 1254   | 1060  |

| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type      | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|-----------|-------|
| 81  | 121       | 1   | 60.4               | 58.7                 | 1254              | 1254      | 1065  |
| 82  | 121       | 2   | 44.8               | 55.7                 | 1254              | 1254      | 1054  |
| 83  | 121       | 3   | 100.8              | 53.2                 | 1254              | 1254      | 1012  |
| 84  | 121       | 4   | 77.2               | 50.9                 | 1254              | 1254      | 1002  |
| 85  | 122       | 1   | 6.0                | 12.2                 | 1260              | 1260      | 1090  |
| 86  | 122       | 2   | 5.8                | 10.9                 | 1260              | 1260      | 1072  |
| 87  | 122       | 3   | 6.7                | 11.3                 | 1260              | 1260      | 1084  |
| 88  | 122       | 4   | 8.4                | 10.0                 | 1260              | 1260      | 1087  |
| 89  | 123       | 1   | 33.3               | 59.2                 | 1260              | 1260      | 1095  |
| 90  | 123       | 2   | 82.1               | 56.9                 | 1260              | 1260      | 1030  |
| 91  | 123       | 3   | 64.6               | 66.8                 | 1260              | 1260      | 1041  |
| 92  | 123       | 4   | 39.0               | 57.5                 | 1260              | 1260      | 1062  |
| 93  | 124       | 1   | 2.5                | 1.8                  | 1254              | 1254/1260 | 1007  |
| 94  | 124       | 2   | 2.7                | 1.4                  | 1260              | 1254/1260 | 1086  |
| 95  | 124       | 3   | 4.0                | 1.9                  | 1254              | 1254/1260 | 1037  |
| 96  | 124       | 4   | 2.5                | 1.8                  | 1254              | 1254/1260 | 1085  |
| 97  | 125       | 1   | 86.4               | 32.0                 | 1254              | 1254/1260 | 1080  |
| 98  | 125       | 2   | 76.8               | 41.3                 | 1254              | 1254/1260 | 1098  |
| 99  | 125       | 3   | 70.4               | 46.0                 | 1254              | 1254/1260 | 1064  |
| 100 | 125       | 4   | 64.4               | 32.2                 | 1260              | 1254/1260 | 1051  |
| 101 | 126       | 1   | <0.5               | ≤0.1                 | Non-Detect        | Blank     | 1008  |
| 102 | 126       | 2   | <0.5               | ≤0.1                 | Non-Detect        | Blank     | 1033  |
| 103 | 126       | 3   | <0.5               | ≤0.2                 | Non-Detect        | Blank     | 1068  |
| 104 | 126       | 4   | <0.5               | ≤1.3                 | Non-Detect        | Blank     | 1056  |
| 105 | 201       | 1   | 0.8                | 1.0                  | 1016/1260         | Sample    | 2037  |
| 106 | 201       | 2   | 0.7                | 1.0                  | 1016/1260         | Sample    | 2056  |
| 107 | 201       | 3   | 1.0                | 1.1                  | 1016/1260         | Sample    | 2006  |
| 108 | 201       | 4   | 0.7                | 0.6                  | 1260              | Sample    | 2104  |
| 109 | 202       | 1   | 0.9                | 1.4                  | 1260              | Sample    | 2040  |
| 110 | 202       | 2   | 1.0                | 1.6                  | 1260              | Sample    | 2025  |
| 111 | 202       | 3   | 0.6                | 1.2                  | 1260              | Sample    | 2050  |
| 112 | 202       | 4   | 1.0                | 1.5                  | 1260              | Sample    | 2002  |
| 113 | 203       | 1   | 20.7               | 14.0                 | 1248              | Sample    | 2022  |
| 114 | 203       | 2   | 14.9               | 12.8                 | 1248              | Sample    | 2102  |
| 115 | 203       | 3   | 19.2               | 16.2                 | 1248              | Sample    | 2045  |
| 116 | 203       | 4   | 15.9               | 12.4                 | 1248              | Sample    | 2100  |
| 117 | 204       | 1   | 97.6               | 43.1                 | 1248              | Sample    | 2068  |
| 118 | 204       | 2   | 78.0               | 45.3                 | 1248              | Sample    | 2039  |
| 119 | 204       | 3   | 86.6               | 41.0                 | 1248              | Sample    | 2019  |
| 120 | 204       | 4   | 91.7               | 47.7                 | 1248              | Sample    | 2036  |

| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type   | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|--------|-------|
| 121 | 205       | 1   | 185.4              | 3305.0               | 1016/1260         | Sample | 2038  |
| 122 | 205       | 2   | 210.4              | 538.7                | 1016              | Sample | 2001  |
| 123 | 205       | 3   | 206.1              | 457.0                | 1016              | Sample | 2030  |
| 124 | 205       | 4   | 190.1              | 483.3                | 1016              | Sample | 2073  |
| 125 | 206       | 1   | 1.4                | 2.9                  | 1260              | Sample | 2083  |
| 126 | 206       | 2   | 1.3                | 1.1                  | 1260              | Sample | 2008  |
| 127 | 206       | 3   | 1.5                | 1.1                  | 1016/1260         | Sample | 2087  |
| 128 | 206       | 4   | 1.9                | 2.5                  | 1260              | Sample | 2027  |
| 129 | 207       | 1   | 23.6               | 17.8                 | 1260              | Sample | 2043  |
| 130 | 207       | 2   | 16.8               | 14.3                 | 1260              | Sample | 2029  |
| 131 | 207       | 3   | 22.0               | 21.6                 | 1260              | Sample | 2062  |
| 132 | 207       | 4   | 14.8               | 21.6                 | 1254              | Sample | 2009  |
| 133 | 208       | 1   | 29.2               | 42.0                 | 1260              | Sample | 2061  |
| 134 | 208       | 2   | 28.4               | 27.7                 | 1016/1260         | Sample | 2103  |
| 135 | 208       | 3   | 33.2               | 24.0                 | 1254              | Sample | 2096  |
| 136 | 208       | 4   | 25.6               | 28.4                 | 1260              | Sample | 2054  |
| 137 | 209       | 1   | 40.0               | 32.7                 | 1260              | Sample | 2059  |
| 138 | 209       | 2   | 46.8               | 79.3                 | 1260              | Sample | 2094  |
| 139 | 209       | 3   | 56.4               | 11.0                 | 1260              | Sample | 2041  |
| 140 | 209       | 4   | 50.5               | 37.9                 | 1260              | Sample | 2021  |
| 141 | 210       | 1   | 75.6               | 123.2                | 1260              | Sample | 2090  |
| 142 | 210       | 2   | 92.8               | 61.5                 | 1260              | Sample | 2047  |
| 143 | 210       | 3   | 69.2               | 84.1                 | 1260              | Sample | 2015  |
| 144 | 210       | 4   | 85.6               | 85.5                 | 1260              | Sample | 2065  |
| 145 | 211       | 1   | >200               | 387.8                | 1254              | Sample | 2075  |
| 156 | 211       | 2   | >200               | 581.4                | 1254              | Sample | 2055  |
| 157 | 211       | 3   | >200               | 330.0                | 1254              | Sample | 2084  |
| 158 | 211       | 4   | >200               | 318.7                | 1254              | Sample | 2042  |
| 149 | 212       | 1   | 3.0                | 3.8                  | 1260              | Sample | 2086  |
| 150 | 212       | 2   | 4.1                | 3.9                  | 1260              | Sample | 2032  |
| 151 | 212       | 3   | 4.0                | 4.3                  | 1260              | Sample | 2093  |
| 152 | 212       | 4   | 4.7                | 0.8                  | 1260              | Sample | 2095  |
| 153 | 213       | 1   | 6.2                | 6.9                  | 1260              | Sample | 2076  |
| 154 | 213       | 2   | 5.5                | 7.3                  | 1260              | Sample | 2057  |
| 155 | 213       | 3   | 5.8                | 7.8                  | 1260              | Sample | 2028  |
| 156 | 213       | 4   | 5.4                | 10.5                 | 1260              | Sample | 2089  |
| 157 | 214       | 1   | 33.2               | 26.0                 | 1260              | Sample | 2018  |
| 158 | 214       | 2   | 28.8               | 25.6                 | 1260              | Sample | 2070  |
| 159 | 214       | 3   | 32.4               | 29.1                 | 1260              | Sample | 2016  |
| 160 | 214       | 4   | 30.0               | 20.2                 | 1260              | Sample | 2007  |

| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type      | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|-----------|-------|
| 161 | 215       | 1   | 30.8               | 25.1                 | 1260              | Sample    | 2014  |
| 162 | 215       | 2   | 30.0               | 24.1                 | 1260              | Sample    | 2020  |
| 163 | 215       | 3   | 26.8               | 26.2                 | 1260              | Sample    | 2034  |
| 164 | 215       | 4   | 31.6               | 31.2                 | 1016/1260         | Sample    | 2097  |
| 165 | 216       | 1   | 55.2               | 151.6                | 1260              | Sample    | 2017  |
| 166 | 216       | 2   | 53.2               | 47.0                 | 1260              | Sample    | 2079  |
| 167 | 216       | 3   | 69.2               | 54.3                 | 1260              | Sample    | 2010  |
| 168 | 216       | 4   | 50.8               | 64.0                 | 1260              | Sample    | 2063  |
| 169 | 217       | 1   | >200.0             | 886.7                | 1254              | Sample    | 2099  |
| 170 | 217       | 2   | >200.0             | 549.8                | 1254              | Sample    | 2067  |
| 171 | 217       | 3   | >200.0             | 542.8                | 1254              | Sample    | 2024  |
| 172 | 217       | 4   | >200.0             | 1913.3               | 1016/1260         | Sample    | 2074  |
| 173 | 218       | 1   | 0.9                | 2.8                  | 1248              | 1248      | 2049  |
| 174 | 218       | 2   | 1.0                | 2.4                  | 1248              | 1248      | 2091  |
| 175 | 218       | 3   | 1.8                | 2.6                  | 1248              | 1248      | 2046  |
| 176 | 218       | 4   | 1.5                | 2.6                  | 1248              | 1248      | 2066  |
| 177 | 219       | 1   | 11.7               | 22.4                 | 1248              | 1248      | 2013  |
| 178 | 219       | 2   | 11.2               | 26.0                 | 1016              | 1248      | 2004  |
| 179 | 219       | 3   | 10.7               | 29.4                 | 1248              | 1248      | 2053  |
| 180 | 219       | 4   | 9.5                | 15.2                 | 1248              | 1248      | 2085  |
| 181 | 220       | 1   | 5.8                | 8.5                  | 1254              | 1254      | 2081  |
| 182 | 220       | 2   | 4.2                | 4.9                  | 1254              | 1254      | 2052  |
| 183 | 220       | 3   | 4.6                | 4.7                  | 1254              | 1254      | 2080  |
| 184 | 220       | 4   | 7.0                | 5.2                  | 1254              | 1254      | 2072  |
| 185 | 221       | 1   | 62.4               | 32.0                 | 1016/1260         | 1254      | 2064  |
| 186 | 221       | 2   | 74.4               | 44.1                 | 1016/1260         | 1254      | 2088  |
| 187 | 221       | 3   | 62.8               | 43.8                 | 1254              | 1254      | 2058  |
| 188 | 221       | 4   | 55.2               | 59.6                 | 1254              | 1254      | 2069  |
| 189 | 222       | 1   | 6.4                | 13.2                 | 1260              | 1260      | 2078  |
| 190 | 222       | 2   | 6.1                | 12.4                 | 1260              | 1260      | 2031  |
| 191 | 222       | 3   | 6.2                | 12.7                 | 1260              | 1260      | 2051  |
| 192 | 222       | 4   | 6.2                | 12.7                 | 1260              | 1260      | 2077  |
| 193 | 223       | 1   | 52.8               | 56.6                 | 1260              | 1260      | 2044  |
| 194 | 223       | 2   | 41.5               | 50.3                 | 1260              | 1260      | 2005  |
| 195 | 223       | 3   | 43.1               | 49.9                 | 1260              | 1260      | 2092  |
| 196 | 223       | 4   | 40.5               | 66.4                 | 1260              | 1260      | 2011  |
| 197 | 224       | 1   | 2.6                | 2.2                  | 1254              | 1254/1260 | 2035  |
| 198 | 224       | 2   | 2.4                | 1.2                  | 1260              | 1254/1260 | 2033  |
| 199 | 224       | 3   | 2.4                | 1.4                  | 1260              | 1254/1260 | 2012  |
| 200 | 224       | 4   | 1.7                | 2.1                  | 1254              | 1254/1260 | 2098  |



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| Obs | Sample ID | Rep | RaPID Result (ppm) | Ref Lab Result (ppm) | Reference Aroclor | Type      | Order |
|-----|-----------|-----|--------------------|----------------------|-------------------|-----------|-------|
| 201 | 225       | 1   | 65.2               | 56.4                 | 1260              | 1254/1260 | 2060  |
| 202 | 225       | 2   | 83.6               | 36.5                 | 1016/1260         | 1254/1260 | 2026  |
| 203 | 225       | 3   | 87.2               | 32.1                 | 1260              | 1254/1260 | 2048  |
| 204 | 225       | 4   | 73.2               | 146.0                | 1254              | 1254/1260 | 2003  |
| 205 | 226       | 1   | <0.5               | ≤0.1                 | Non-Detect        | Blank     | 2071  |
| 206 | 226       | 2   | <0.5               | ≤0.8                 | Non-Detect        | Blank     | 2082  |
| 207 | 226       | 3   | <0.5               | ≤0.1                 | Non-Detect        | Blank     | 2101  |
| 208 | 226       | 4   | <0.5               | ≤0.1                 | Non-Detect        | Blank     | 2023  |

**Table D-2. RaPID Assay System PCB technology demonstration extract sample data**

| OBS | Sample |     | RaPID           | Ref Lab         | Reference  |       | Spike <sup>a</sup><br>(ppm) | Order |
|-----|--------|-----|-----------------|-----------------|------------|-------|-----------------------------|-------|
|     | ID     | Rep | Result<br>(ppm) | Result<br>(ppm) | Aroclor    | Type  |                             |       |
| 1   | 130    | 1   | 13.0            | 16.4            | 1016       | 1242  | 10                          | 1111  |
| 2   | 130    | 2   | 11.9            | 10.9            | 1016       | 1242  | 10                          | 1114  |
| 3   | 130    | 3   | 11.5            | 10.3            | 1016       | 1242  | 10                          | 1108  |
| 4   | 130    | 4   | 13.5            | 10.7            | 1016       | 1242  | 10                          | 1109  |
| 5   | 131    | 1   | 101.2           | 67.1            | 1254       | 1254  | 100                         | 1105  |
| 6   | 131    | 2   | 101.2           | 57.1            | 1254       | 1254  | 100                         | 1112  |
| 7   | 131    | 3   | 94.4            | 62.8            | 1254       | 1254  | 100                         | 1113  |
| 8   | 131    | 4   | 90.8            | 68.2            | 1254       | 1254  | 100                         | 1115  |
| 9   | 132    | 1   | <0.5            | ≤0.1            | Non-Detect | blank | 0                           | 1107  |
| 10  | 132    | 2   | <0.5            | ≤0.1            | Non-Detect | blank | 0                           | 1106  |
| 11  | 132    | 3   | <0.5            | ≤0.1            | Non-Detect | blank | 0                           | 1110  |
| 12  | 132    | 4   | <0.5            | ≤0.1            | Non-Detect | blank | 0                           | 1116  |
| 13  | 230    | 1   | 9.5             | 9.8             | 1016       | 1242  | 10                          | 2114  |
| 14  | 230    | 2   | 10.0            | 10.4            | 1016       | 1242  | 10                          | 2112  |
| 15  | 230    | 3   | 9.7             | 7.6             | 1016       | 1242  | 10                          | 2109  |
| 16  | 230    | 4   | 8.6             | 7.9             | 1016       | 1242  | 10                          | 2115  |
| 17  | 231    | 1   | 86.0            | 55.2            | 1254       | 1254  | 100                         | 2111  |
| 18  | 231    | 2   | 91.2            | 55.0            | 1254       | 1254  | 100                         | 2116  |
| 19  | 231    | 3   | 79.0            | 61.3            | 1254       | 1254  | 100                         | 2110  |
| 20  | 231    | 4   | 89.0            | 59.1            | 1254       | 1254  | 100                         | 2105  |
| 21  | 232    | 1   | <0.3            | ≤0.1            | Non-Detect | blank | 0                           | 2113  |
| 22  | 232    | 2   | <0.3            | ≤0.1            | Non-Detect | blank | 0                           | 2108  |
| 23  | 232    | 3   | <0.3            | ≤0.1            | Non-Detect | blank | 0                           | 2107  |
| 24  | 232    | 4   | <0.3            | ≤0.1            | Non-Detect | blank | 0                           | 2106  |

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

**Table D-3. Corrected reference laboratory data**

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

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

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



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



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### Disclaimer

The following hypothetical example serves to demonstrate how the information provided in this report may be used in the 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 characterization determined that the PCB concentration in a single drum was homogenous, but PCB concentrations varied greatly from drum to drum. The company's DQO team was considering the use of SDI's RaPID to measure the PCB concentration in each drum. The DQO team decided that drums will be disposed of by incineration if the PCB concentration is greater than or equal to 50 ppm ("hot"). A concentration of 50 ppm is the Toxic Substances Control Act (TSCA) regulatory threshold (RT) for this environmental problem. Those drums with PCB concentrations less than 50 ppm will be put into a landfill because incineration of soil is very expensive. With regulator agreement, the DQO team determined that a decision rule for disposal would be based on the average concentration of PCBs in each drum.

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| <p style="text-align: center;"><b>General Decision Rule</b></p> <p>If average PCB concentration &lt; than action level, then send the soil drum to the landfill.</p> <p>If average PCB concentration ≥ action level, then send the soil drum to the incinerator.</p> |
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### Data Quality Objective Goals

EPA's Guidance for Data Quality Assessment [16] states in Section 1.2: "The true condition that occurs with the more severe decision error . . . should be defined as the null hypothesis." The 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 50 ppm. Therefore, the null hypothesis is constructed to assume that a drum's true PCB concentration is greater than 50 ppm; and as a "hot" drum, it would be sent to an 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 50 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 50 ppm PCBs (i.e., the null hypothesis is rejected), when actually the drum is "hot" (i.e., the null hypothesis is true). The team required that the error rate for 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

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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 50 ppm. This scenario represents a 5% chance of sending a drum containing 50 ppm or more of PCBs to the landfill.

The DQO team did not want to send an excessive number of drums to the incinerator if the average PCB concentration was less than 50 ppm because of the expense. In this situation, a false negative decision is made when it is concluded that a drum is “hot” (i.e., the null hypothesis is not rejected), when in actuality, the drum contains soil with less than 50 ppm PCBs (i.e., the null hypothesis is actually false). After considering the guidelines presented in Section 1.1 of EPA’s Guidance for Data Quality Assessment [16] for developing limits on decision errors, the team selected the false negative decision error rate (FN) to be 0.10 if the true drum concentration was 40 ppm. That is, there would be a 10% probability (Pr) of sending a drum to the incinerator (denoted as Pr[Take Drum to Incinerator]) if the true PCB concentration for a drum was 40 ppm.

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

FP: Pr[Take Drum to Landfill]  $\leq$  0.05 when true PCB concentration = 50 ppm

FN: Pr[Take Drum to Incinerator]  $\leq$  0.10 when true PCB concentration = 40 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 RaPID Assay Kit, the performance of this technology (as reported in this ETV report) was used to assess its applicability to this project. Two questions arise:

1. *How many samples are needed* from a single drum to permit a valid estimation of the true average concentration of PCBs in the drum to the specified certainty? Recall that the simplifying assumption was made that the PCB distribution throughout the soil within a single drum is homogeneous, and thus, matrix heterogeneity will not contribute to overall variability. The only variability, then, to be considered in this example is the variability in the RaPID Assay’s method, which is determined by precision studies.
2. *What is the appropriate action level (AL)* for using the RaPID Assay Kit to make decisions in the field? After the required number of samples have been collected from a drum and analyzed, the results are averaged together to get an estimate of the “true” PCB concentration of the drum. When using the RaPID Assay Kit, what is the value (here called “the action level for the decision rule”) to which that average is compared to decide if the drum is “hot” or not? This method-specific or site-specific action level is derived from evaluations of the method’s accuracy using an appropriate quality control regimen.



### RaPID Assay Accuracy

In the ETV study, the overall data generated by SDI's RaPID Assay Kit was slightly biased when compared with the certified PCB values for the PE samples, which had concentrations ranging from 0 to 50 ppm. As summarized in Table 5-5, environmental conditions (temperature and humidity) showed no effect on the accuracy achieved by the RaPID Assay Kit; therefore, the data from both sites can be plotted to create Figure E-1. Similarly, environmental soil data in the concentration range of 0 to 60 ppm, which was generated by the reference laboratory, can be plotted against the corresponding SDI results to create Figure E-2.

The lines on these graphs depict the lines of best fit (with 95% confidence intervals), and can be used to predict the results that the RaPID Assay would produce for a particular "true" PCB concentration. The arrows on the plots demonstrate how to quickly estimate this. The same task can be performed mathematically, if desired, by using equations which to define the lines in Figures E-1 and E-2.

Table E-1 presents the prediction performance of the linear model for the SDI RaPID Assay kit at the 40 and 50 ppm critical decision points. Table E-1 shows that the RaPID Assay results tend to be slightly biased high within this range of PCB values.

The 95% confidence intervals (CIs) for the environmental soils and PE data sets overlap for a single nominal value. At a nominal value of 40 ppm, for example, the 95% CI predicted for RaPID's results from comparison with the PE sample set is 43 to 55 ppm, which overlaps the 95% CI predicted from the environmental soils sample set (53 to 65 ppm). Since these ranges overlap, they can be considered to be statistically similar. Although this similar

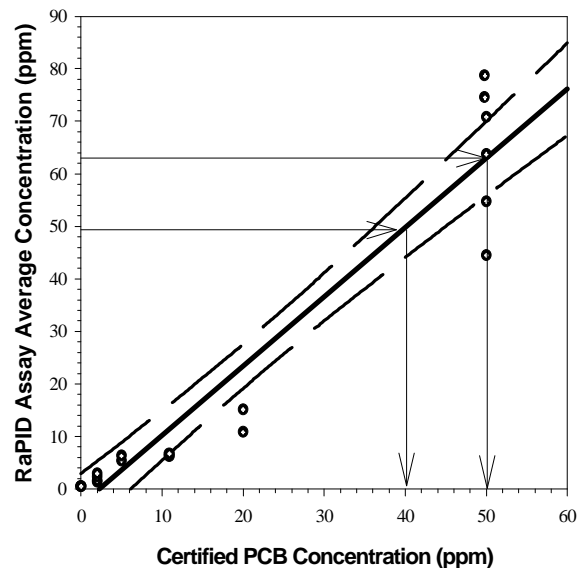


Figure E-1. A linear model for predicting RaPID Assay PCB concentrations from certified PCB concentrations with 95% confidence intervals (dashed lines).

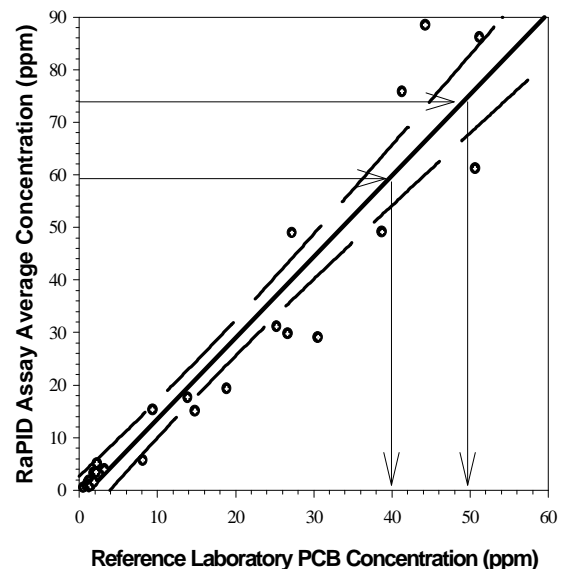


Figure E-2. A linear model for predicting RaPID Assay PCB concentrations from reference laboratory PCB concentrations for environmental soils up to 60 ppm with 95% confidence intervals (dashed lines).

**Table E-1. Predicting SDI’s RaPID Assay Kit results from graphs of performance data**

| Soil Matrix<br>(concentration range)                 | Critical Decision Point | Predicted SDI Results (95% CI) |
|--|-------------------------|--------------------------------|
| Certified PE Soil (0 to 50 ppm)<br>from Figure E-1   | 40 ppm                  | 49 (43 - 55)                   |
|  | 50 ppm                  | 63 (65 - 70)                   |
| Environmental Soils (0 to 60 ppm)<br>from Figure E-2 | 40 ppm                  | 59 (53 - 65)                   |
|  | 50 ppm                  | 74 (66 - 82)                   |

performance with a variety of soil types in the ETV study increases the confidence that the RaPID Assay Kit may perform equivalently on other soil types, site-specific quality control is always advisable to detect unforeseen matrix effects or interferences.

After considering the matrix characteristics and Aroclor types of the PE and environmental soils in this ETV study and comparing these to the soils that had been placed into the drums that the DQO team planned to test, the DQO team reasoned that there was a strong likelihood that the SDI Kit would perform in a similar manner for their testing. However, they decided to design an appropriate QC regimen (which included matrix spikes, split samples sent for confirmatory laboratory analysis, and duplicates) to ensure that the SDI Kit was performing as expected under their site-specific conditions.

Additionally, to address the possibility that these QC samples would reveal that the kit’s performance was different from that expected, the team created a backup plan (which became part of the Sampling and Analysis Plan) that would permit them to document and account for deviations in expected performance. The team reasoned that, even if the kit’s performance varied somewhat from their expectations, being able to account for any deviations would permit them to produce verifiable and defensible data that might still be able to support decision-making at the site, without the need for resampling.

The DQO team also decided not to compensate for the positive bias they expected to see based on the ETV study. Until the members of the team had more field experience with the kit’s performance under a variety of conditions, they believed that the bias would provide a margin of safety in their project that would be valuable for regulator and stakeholder acceptance. Therefore, the critical decision points of 40 and 50 ppm (which correspond to  $C_{FN}$  and  $RT$  in the following equations) were selected for use with the SDI RaPID Assay Kit when calculating the sample size and action level to meet the project DQO goals.

**Determining the Number of Samples**

With the critical decision points selected for the kit, the team could determine the number of samples needed from each drum to calculate its “true” average PCB concentration. For a homogeneous matrix, the number of samples required depends on the precision of the analytical method.

The ETV demonstration results indicated the standard deviation increased with concentration levels and that the RSD would be a more appropriate precision measurement than the standard deviation. For SDI’s RaPID

Assay measurements, the mean RSDs were 15% for controlled environmental conditions and 26% for outdoor conditions (see Table 5-4). The DQO team could set up a controlled environment at the remediation site with additional effort and cost. The DQO team decided to calculate the number of samples for both environmental conditions to judge whether the additional effort would be cost effective. A formula (Equation E-1) is provided in EPA's *Guidance for Data Quality Assessment* [16] (pp. 3.2-3, Box 3.2-1) that can be adapted to this example for calculating the number of samples required to meet the false positive and false negative error rate for the decision. This formula uses a constant SD for the analytical method's precision but can be modified to use RSD by dividing the numerator and denominator by  $(RT)^2$  and multiplying by  $(100\%)^2$ , as shown in Equation E-2. The final form of the formula appears as Equation E-3.

$$n = \frac{(SD)^2 (Z_{1-FP} + Z_{1-FN})^2}{(RT - C_{FN})^2} + (0.5)Z_{1-FP}^2 \quad (E-1)$$

$$n = \frac{(100\% \times SD/RT)^2 (Z_{1-FP} + Z_{1-FN})^2}{[100\% \times (RT - C_{FN})/RT]^2} + (0.5)Z_{1-FP}^2 \quad (E-2)$$

$$n = \frac{RSD^2 (Z_{1-FP} + Z_{1-FN})^2}{(\%D)^2} + (0.5)Z_{1-FP}^2 \quad (E-3)$$

where

- n = number of samples from a drum to be measured,
- RSD = RSD at the regulatory threshold [e.g.,  $RSD^2 = (15\%)^2$  or  $RSD^2 = (26\%)^2$  ],
- RT = regulatory threshold (e.g., RT = 50 ppm),
- $C_{FN}$  = concentration at which the FN is specified (e.g.,  $C_{FN} = 40$  ppm),
- %D = percent difference of  $C_{FN}$  relative to RT [e.g.,  $(\%D)^2 = (20\%)^2$  ],
- FP = false positive decision error rate (e.g., FP = 0.05),
- FN = false negative decision error rate (e.g., FN = 0.10),
- $Z_{1-p}$  = the (1- p)th percentile of the standard normal distribution (see ref. 16, Table A-1 of Appendix A). Example  $Z_{(1-FP)} = Z_{0.95} = 1.645$ .

Equation E-3 is then used to determine the number of samples to be analyzed from each drum. As seen in the following, the greater precision performance of the SDI RaPID Assay Kit under controlled environmental conditions has the effect of reducing by the number of samples per drum in half compared with the number needed under uncontrolled heat and humidity.

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$$n = \frac{(15\%)^2 (1.645 + 1.282)^2}{(20\%)^2} + (0.5)(1.645)^2 = 6.2 \quad 7 \quad \text{chamber conditions}$$

$$n = \frac{(26\%)^2 (1.645 + 1.282)^2}{(20\%)^2} + (0.5)(1.645)^2 = 15.8 \quad 16 \quad \text{outdoor conditions}$$

Note that, to be conservative, the sample size was rounded up to the next integer. The DQO team was able to compare the cost of an additional 9 samples per drum vs the cost of establishing a controlled environment (such as an air-conditioned camper). Because of the large number of drums to be tested during a hot summer, the DQO team opted for a controlled analytical environment to reduce the total number of samples. The action levels for the decision rule will then be calculated based on taking 7 samples from each drum.

### Determining the Action Level

Since the DQO team decided not to compensate for the kit's positive bias, seven sample results from each drum will be averaged (arithmetic mean) to produce an estimate of the drum's "true" PCB concentration. This average PCB concentration will be compared with the AL for the decision rule. The AL for the decision rule is calculated based on regulation-driven requirements (the TSCA regulatory threshold of 50 ppm) and on controlling the FP established in the DQO process. Recall that the team set the permissible FP error rate at 5%.

ASTM D5283-92 [13] shows the formula for the AL based on a constant SD over a relevant concentration range (Equation E-4). Since the RaPID Assay Kit did not produce data with a constant standard deviation, this formula must be adapted to this example by using the relationship between SD and RSD, which is  $SD = (\text{Concentration}) \times RSD/100\%$ . Thus Equation E-4 becomes Equation E-5, and the regulatory threshold ( $RT = 50$  ppm) is the concentration used in the formula.

$$AL = RT - Z_{1-FP} \times \frac{SD}{\sqrt{n}} \quad (\text{E-4})$$

$$AL = RT - Z_{1-FP} \times \frac{RT \times RSD}{100\% \times \sqrt{n}} \quad (\text{E-5})$$

The AL for the decision rule using SDI's RaPID Assay Kit to satisfy a 5% FP and a 10% FN for seven samples under chamber conditions is

$$AL = 50 \text{ ppm} - (1.645) \times \frac{50 \text{ ppm} \times 15\%}{100\% \times \sqrt{7}} = 45.3 \text{ ppm} \quad \text{chamber conditions.}$$

Note that if the analytical testing was done under uncontrolled environmental conditions with a sample size of 16, the AL for the decision rule would become

$$AL = 50 \text{ ppm} - (1.645) \times \frac{50 \text{ ppm} \times 26\%}{100\% \times \sqrt{16}} = 44.7 \text{ ppm} \quad \text{outdoor conditions.}$$

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

If the average PCB concentration of 7 (or 16) random soil samples on a drum is less than 45.3 ppm (or 44.7 ppm), then send the drum to the landfill.

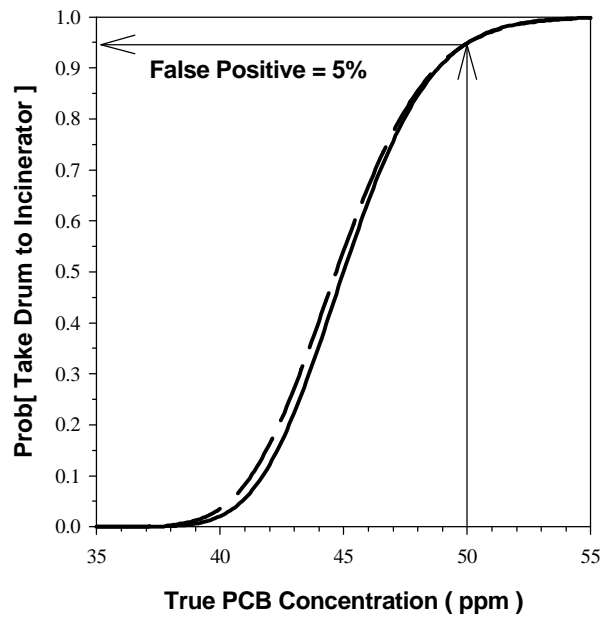
If the average PCB concentration of 7 (or 16) random soil samples on a drum is greater or equal to 45.3 ppm (or 44.7), then consider the drum “hot,” and send it to the incinerator.

The decision performance curve (see ref. 16) calculates the probability of sending a drum to the incinerator for different values of true PCB concentration in a drum. Figure E-3 illustrates that the decision performance curves for the controlled and uncontrolled environments both have the value of  $\text{Pr}[\text{Take Drum to Incinerator}] = 0.95$  for  $\text{True} = 50$  ppm. This indicates that the decision rule meets the FP requirement of 5% for both environmental conditions. The  $\text{Pr}[\text{Take Drum to Incinerator}] = 0.02$  and  $0.04$  at  $\text{True} = 40$  ppm for the controlled ( $\text{RSD} = 15\%$ ) and uncontrolled environment ( $\text{RSD} = 25\%$ ), respectively. These false negative probabilities are better than the  $\text{FN} = 10\%$  that the DQO team had specified. This improved performance is caused by rounding up the number of samples to the next integer in the calculation of the number of samples required.

#### **Alternative FP Error Rate**

Because of random sampling and analysis error, there is always some chance that analytical results will not accurately reflect the true nature of a decision unit (such as a drum, in this example). Often, 95% certainty (a 5% FP) is customary and sufficient to meet stakeholder comfort. But suppose that the DQO team wanted to be even more cautious about limiting the possibility that a drum might be sent to a landfill when its true value is 50 ppm. If the team wanted to be 99% certain that a drum was correctly sent to a landfill, the following discussion describes how changing the FP from 5% to 1% would affect the decision rule. Using  $\text{FP} = 0.01$ , the sample sizes are calculated to be 9 and 23, and the ALs for the decision rule are 44.2 and

43.7 ppm for the controlled and uncontrolled environments, respectively. The decision performance curves has the value of Pr [Take Drum to Incinerator] = 0.99 for True = 50 ppm. This indicates that the decision rule meets the FP of 1%. The Pr [Take Drum to Incinerator] = 0.02 and 0.04 at True = 40 ppm for the controlled and uncontrolled environment, respectively. These probabilities are better than the FN of 10% than the DQO team had specified. This improved performance is caused by rounding up the number of samples to the next integer in the calculation of number of samples required. The decision rule for the lower FP would be:



**Figure E-3. Decision performance curves for RSD = 15% (solid line) and RSD = 25% (dashed lined) for the PCB drum example.**

**Decision Rule for FP = 1% and FN = 10%**

If the average PCB concentration of 9 (or 23) random soil samples on a drum is less than 44.2 (or 43.7) ppm, then send the drum to the landfill.

If the average PCB concentration of 9 (or 23) random soil samples on a drum is greater or equal to 44.2 (or 43.7) ppm, then consider the drum to be “hot,” and send it to the incinerator.