N-0018

United States
Environmental Protection
Agency

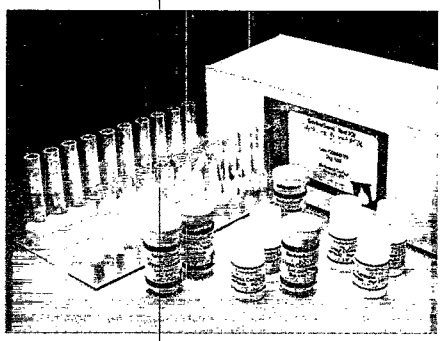
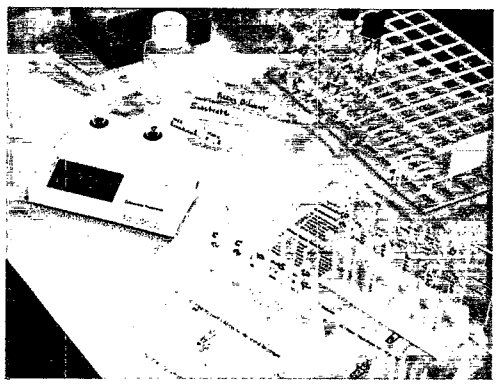
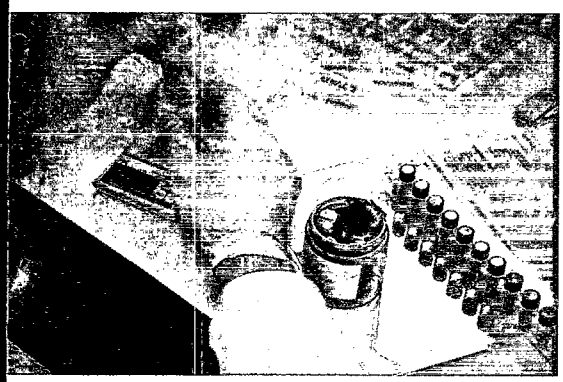
Office of Research and
Development
Washington DC 20460

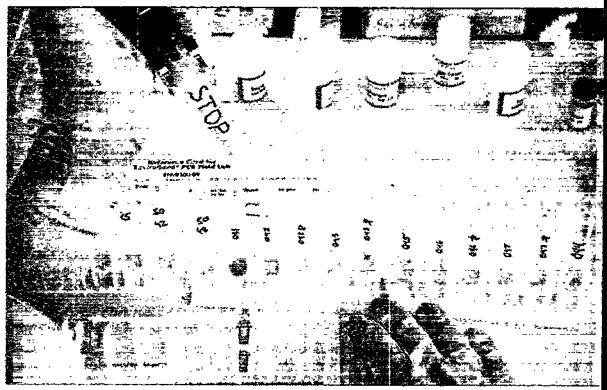
EPA/540/R-95/517
August 1995

EPA

EnviroGard PCB Test Kit, Millipore, Inc.

Innovative Technology Evaluation Report



SITE
SUPERFUND INNOVATIVE TECHNOLOGY EVALUATION

CONTACT

Jeanette Van Emon and Steve Rock are the EPA contacts for this report. Jeanette Van Emon is presently with the new Characterization Research Division (formerly the Environmental Monitoring Systems Laboratory) in Las Vegas, NV, which is under the direction of the National Exposure Research Laboratory with headquarters in Research Triangle Park, NC.

Steve Rock is presently with the new Land Remediation and Pollution Control Division (formerly the Risk Reduction Engineering Laboratory) in the newly organized National Risk Management Research Laboratory in Cincinnati, OH.

EPA/540/R-95/517
August 1995

ENVIROGARD PCB TEST KIT, MILLIPORE, INC.

INNOVATIVE TECHNOLOGY EVALUATION REPORT

NATIONAL RISK MANAGEMENT RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

NATIONAL EXPOSURE RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
LAS VEGAS, NEVADA 89193

Notice

The information in this document has been funded wholly or in part by the U.S. Environmental Protection Agency (EPA) in partial fulfillment of Contract No. 68-C0-0047, Work Assignment No. 0-40, to PRC Environmental Management, Inc. It has been subject to the Agency's peer and administrative review, and it has been approved for publication as an EPA document. The opinions, findings, and conclusions expressed herein are those of the contractor and not necessarily those of the EPA or other cooperating agencies. Mention of company or product names is not to be construed as an endorsement by the agency.

Foreword

The U.S. Environmental Protection Agency is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory is the Agency's center for investigation of technological and management approaches for reducing risks from threats to human health and the environment. The focus of the Laboratory's research program is on methods for the prevention and control of pollution to air, land, water and subsurface resources; protection of water quality in public water systems ; remediation of contaminated sites and ground water; and prevention and control of indoor air pollution. The goal of this research effort is to catalyze development and implementation of innovative, cost-effective environmental technologies; develop scientific and engineering information needed by EPA to support regulatory and policy decisions; and provide technical support and information transfer to ensure effective implementation of environmental regulations and strategies.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

E. Timothy Oppelt, Director
National Risk Management Research Laboratory

Abstract

This report describes the demonstration and evaluation of an immunoassay field screening technology designed to determine polychlorinated biphenyl (PCB) contamination in soil. The immunoassay technology was the EnviroGard PCB Test developed by Millipore, Inc. The technology was demonstrated in Kansas City, Missouri, in August 1992, by PRC Environmental Management, Inc. (PRC), under contract to the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV).

The principal objective of the demonstration was to evaluate the technology for accuracy and precision at detecting high and low levels of PCB in soil samples by comparing their results to those attained by a confirmatory laboratory using standard EPA analytical methods. The technology also was qualitatively evaluated for the length of time required for analysis, ease of use, portability, and operating cost. Accuracy was also assessed through analysis of performance evaluation (PE) samples, and precision was further assessed by comparing the results obtained on duplicate samples. A secondary objective of the demonstration was to evaluate the specificity of the technology. The evaluation of specificity was performed by examining any problems due to naturally occurring matrix effects, site-specific matrix effects, Aroclor sensitivity, and chemical cross reactivity. Information on specificity was gathered from the developer, from the analysis of demonstration samples, and from a specificity study performed during the demonstration.

This report was submitted in fulfillment of contract No. 68-CO-0047 by PRC, under sponsorship of the EPA. This report covers a period from February 10, 1992, to August 31, 1992, and work was completed as of February 28, 1993.

Table of Contents

Table of Contents (Continued)

List of Figures

List of Tables

List of Exhibits

List of Abbreviations and Acronyms

AICO	Abandoned Indian Creek Outfall
CCAL	continuing calibration
CLP	Contract Laboratory Program
CMS	corrective measures study
CRQL	contract required quantitation limit
DOE	Department of Energy
DQO	data quality objective
ECD	electron capture detector
EMSL-LV	Environmental Monitoring Systems Laboratory-Las Vegas
EPA	Environmental Protection Agency
ERA	Environmental Research Associates
GC	gas chromatograph
ICAL	initial calibration
IDW	investigation-derived waste
ITER	Innovative Technology Evaluation Report
KCP	Kansas City Plant
µL	microliter
µg/kg	micrograms per kilogram
mg/kg	milligrams per kilogram
Millipore	Millipore, Inc.
mL	milliliters
MMTP	Monitoring and Measurement Technologies Program
MS	mass spectrometer
NRMRL	National Risk Management Research Laboratory
ORD	Office of Research and Development
OSWER	Office of Solid Waste and Emergency Response
PCB	polychlorinated biphenyl
PE	performance evaluation
PRC	PRC Environmental Management, Inc.
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
r^2	correlation coefficient
RCRA	Resource Conservation and Recovery Act
RFI	RCRA facility investigation
RPD	relative percent difference
SARA	Superfund Amendments and Reauthorization Act of 1986
SITE	Superfund Innovative Technology Evaluation
SOP	standard operating procedures
SOW	statement of work
TCL	Target Compound List
TPM	technical project manager
UV	ultraviolet

Acknowledgments

This demonstration and the subsequent preparation of this report required the services of numerous personnel from the Environmental Protection Agency, Environmental Monitoring Systems Laboratory (Las Vegas, Nevada); Environmental Protection Agency, Region 7 (Kansas City, Kansas); Millipore, Inc. (Bedford, Massachusetts); the U.S. Department of Energy Kansas City Plant (Kansas City, Missouri); Allied-Signal, Inc. (Kansas City, Missouri); and PRC Environmental Management, Inc. (Kansas City, Kansas; Cincinnati, Ohio; and Chicago, Illinois). The cooperation and efforts of these organizations and personnel are gratefully acknowledged.

Additional information concerning the demonstration described in this report can be obtained by contacting Mr. Lary Jack, the Environmental Protection Agency, Environmental Monitoring Systems Laboratory technical project manager, at (702) 798-2373, or Mr. Eric Hess, the PRC Environmental Management, Inc., project manager, at (913) 573-1822.

Additional information on the innovative technology described in this report can be obtained by writing the developer, Millipore, Inc., at Mail Stop E-5A, 80 Ashby Road, Bedford, Massachusetts 01730, or by telephoning 1-800-225-1380.

Section 1
Executive Summary

This innovative technology evaluation report (ITER) presents information on the demonstration and evaluation of the EnviroGard PCB Test produced by Millipore, Inc. (Millipore). The EnviroGard PCB Test is designed to detect polychlorinated biphenyl (PCB) contamination in soil. The demonstration was conducted by PRC Environmental Management, Inc. (PRC), under contract to the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory—Las Vegas (EMSL-LV). The demonstration was developed under the Monitoring and Measurement Technologies Program (MMTP) of the Superfund Innovative Technology Evaluation (SITE) Program.

The EnviroGard PCB Test was demonstrated and evaluated in August 1992 at a site in Kansas City, Missouri. The demonstration of the test was done in conjunction with the demonstration of three other innovative field screening technologies: the Clor-N-Soil PCB Test Kit and the L2000 PCB/Chloride Analyzer, both manufactured by the Dexsil Corporation, and the Field Analytical Screening Program PCB Method developed under the Field Investigative Team Contract with the EPA Superfund Program. The demonstration results for these other technologies are presented in separate ITERs.

The EnviroGard PCB Test is designed to quickly provide semiquantitative results for PCB concentrations in soil samples. The technology can be customized to report specific results over a particular range of concentrations. As part of the SITE demonstration, the technology also was evaluated for its ability to produce quantitative results.

The EnviroGard PCB Test is an immunoassay system that uses polyclonal antibodies to produce compound-specific reactions to PCBs allowing their detection and quantitation. An anti-PCB antibody is fixed to the inside wall of a test tube to bind PCB compounds. An enzyme conjugate containing a PCB derivative labeled with horseradish peroxidase is added to the test tube where it competes with PCBs for anti-PCB antibody binding sites. Reagents are then added to the test tube where they react with the enzyme conjugate, causing a color change. Results can be estimated by observing the degree of color change. For a more precise quantitative measurement of the PCB concentration in the sample, the color of the solution can be compared to Aroclor standards using a differential photometer.

The EnviroGard PCB Test is portable, easy to operate, and useful under a variety of site conditions. The differential photometer requires electricity but can be operated using a rechargeable battery. Reagents must be kept refrigerated.

Calibrating the technology was initially difficult because of the small volume of Aroclor standards required. As the operator gained experience in using the EnviroGard PCB Test, the calibrations became easier.

The EnviroGard PCB Test costs $1,495, which includes the differential photometer and other equipment needed to run the test. Additional disposable equipment and reagents needed to perform 12 analyses cost $253. The differential photometer required to obtain quantitative results costs $799.

The developer reports that the detection limit is 3.3 milligrams per kilogram (mg/kg) for Aroclor 1248. This was the detection limit used during the demonstration. The detection limit differs depending on the Aroclor. The highest number of samples analyzed in an 8-hour day was 52; the average number analyzed per 8-hour day was 25.

Results produced with the EnviroGard PCB Test may be affected by the cross-reactivity of compounds other than PCBs to the anti-PCB antibody binding sites. The developer has evaluated a number of compounds to determine their levels of cross-reactivity, although this evaluation was not independently assessed during this demonstration.

PRC evaluated field and laboratory duplicate samples to determine the technology's precision in the semiquantitative mode. Thirty-seven duplicate sample pairs were used in this semiquantitative evaluation. Of these 37 duplicate sample pairs, the EnviroGard PCB Test produced the same results 35 times. One laboratory duplicate and one field duplicate did not agree with their respective soil sample results. Based on this data, the precision of the EnviroGard PCB Test was found to be 95 percent. This meets the demonstration's criteria for acceptable precision.

PRC evaluated the accuracy of the test in its semiquantitative mode by comparing its data to that of the confirmatory laboratory. This comparison showed that 71 percent of the time the technology was correct. The other 29 percent of the time, the technology gave false positive results. It never gave a false negative result. The technology is conservative when used in the semiquantitative mode. Using an absolute definition of accuracy, it was accurate only 71 percent of the time. The production of false positive results, though, may not affect its use in environmental assessments. False positive results will incorrectly label soil as being contaminated above a test's threshold level. At worst, this would lead to the overestimation of contaminated area or volume.

To assess this technology's precision in a quantitative mode, PRC evaluated the results produced from the analysis of laboratory and field duplicate sample pairs. The EnviroGard PCB Test had 27 duplicate sample pairs in which both a sample and its duplicate had positive results. PRC used the data from the duplicate sample analyses to establish precision control limits. The control limits were set at 0 and 91 percent. All but two of the 27 relative percent differences (RPD) fell within the control limits. This equates to a precision of 93 percent, which is below the 95 percent precision deemed acceptable for this demonstration. However, the technology's precision would have exceeded this threshold if only one more duplicate sample pair had fallen within the control limits. When PRC used the Dunnett's Test to compare the RPDs between the EnviroGard PCB Test's data and the confirmatory laboratory's data, a probability of 97.5 percent resulted. This indicates that the technology is as precise as the confirmatory laboratory.

PRC used linear regression analysis to assess the accuracy of the technology in its quantitative mode when compared to the confirmatory laboratory's data. The regression of 83 matched pairs of positive sample results defined a correlation coefficient (r^2) of 0.87, indicating that a relationship did exist between the two data sets. The regression line calculated had a y-intercept of 17.8 mg/kg and a slope of 0.76. These results indicate that the results from this technology are not accurate. Although the technology was found to be inaccurate in its quantitative mode, the results produced by the technology were linear, indicating that the results can be corrected mathematically. If 10 to 20 percent of the soil samples are sent to a confirmatory laboratory, then the results from the other 80 to 90 percent can be corrected. This could result in a significant savings in analytical costs. The Wilcoxon Signed Ranks Test was used to verify these results. It indicated, at a 95 percent confidence level, that the EnviroGard PCB Test's data was significantly different from that of the confirmatory laboratory. This confirmed the linear regression analysis and indicated that the EnviroGard PCB Test's data was not accurate.

Section 2
Introduction

This ITER summarizes the procedures used to demonstrate the EnviroGard PCB Test, discusses the results of the demonstration, and evaluates the effectiveness and possible uses of the test at various hazardous waste sites. The primary goal of the demonstration was to evaluate the technology and to provide Superfund decision makers with information on its performance and cost effectiveness.

EPA's SITE Program and MMTP: An Overview

At the time of the Superfund Amendments and Reauthorization Act of 1986 (SARA), it was well recognized that the environmental cleanup problem needed to be attacked with new and better methods. The Superfund Innovative Technology Evaluation Program (SITE) , therefore, was created to fulfill a requirement of SARA that the EPA address the potential of alternative or innovative technologies. The EPA made this program a joint effort between the Office of Solid Waste and Emergency Response (OSWER) and the Office of Research and Development (ORD). The SITE Program includes four component programs:

- The Demonstration Program (for remediation technologies)
- The Emerging Technology Program
- The Measuring and Monitoring Program (MMTP)
- The Technology Transfer Program

The largest part of the SITE Program, the Demonstration Program, is concerned with treatment technologies and is administered by ORD's National Risk Management Research Laboratory (NRMRL) in Cincinnati, Ohio. The MMTP is administered by EMSL-LV. The MMTP is concerned with monitoring and measurement technologies that identify, quantify, or monitor changes in contaminants occurring at hazardous waste sites or that are used to characterize a site.

The MMTP seeks to identify and demonstrate innovative technologies that may provide a less expensive, better, faster, or safer means of completing this monitoring or characterization. The managers of hazardous waste sites are often reluctant to use any method, other than conventional ones, to generate critical data on the nature and extent of contamination. It is generally understood that the courts recognize data generated with conventional laboratory methods; still, there is a tremendous need to generate data more cost effectively. Therefore, the EPA must identify innovative approaches, and through verifiable testing of the technologies under the SITE Program, to ensure that the innovative technologies are equivalent or better than conventional technologies.

The Role of Monitoring and Measurement Technologies

Effective measurement and monitoring technologies are needed to accurately assess the degree of contamination; to provide data and information to determine the effects of those contaminants on public health and the environment; to supply data for selection of the most appropriate remedial action; and to monitor the success or failure of a selected remedy. Thus, the MMTP is broadly concerned with evaluating screening (including remote sensing), monitoring, and analytical technologies for all media.

Candidate technologies may come from within the federal government or from the private sector. Through the program, developers are provided with the opportunity to rigorously evaluate the performance of their

technologies. Finally, by distributing the results and recommendations of those evaluations, the market for the technologies is enhanced.

Defining the Process

The innovative technology demonstration process begins by canvassing the EPA's 10 regional offices (with input by OSWER and ORD) to determine their needs. Concurrently, classes of technologies are identified. An ideal match is made when there is a single clear need by EPA's regions and a reasonable number of innovative technologies that can address that need. The demonstrations are designed to judge each technology against existing standards and not one against the other.

The demonstration is designed to provide for detailed quality assurance and quality control (QA/QC). This is done to ensure that a potential user can evaluate the accuracy, precision, representativeness, completeness, and comparability of data derived from the innovative technology. In addition, a description of the necessary steps and activities associated with operating the innovative technology is prepared. Cost data, critical to any environmental activity, is generated during the demonstration and allows a potential user to make economic comparisons. Finally, information on practical matters such as operator training requirements, detection levels, and ease of operation are reported. Thus, the demonstration report and other informational materials produced by the MMTP provide a real-world comparison of that technology to traditional technologies. With cost and performance data, as well as "how to" information, users can more comfortably determine whether a new technology better meets their needs.

Components of a Demonstration

Once a decision has been made to demonstrate technologies to meet a particular EPA need, the MMTP performs a number of activities. First, the MMTP identifies potential participants and determines whether they are interested in participating. Each developer is advised of the general nature of the particular demonstration and is provided with information common to all MMTP demonstrations. Information is sought from each developer about its technology to ensure that the technology meets the parameters of the demonstration. Then, after evaluation of the information, all respondents are told whether they have been accepted into the demonstration or not. While participants are being identified, potential sites also are identified, and basic site information is obtained. These activities complete the initial component of an MMTP demonstration.

The next component, probably the most important, is the development of plans that describe how various aspects of the demonstration will be conducted. A major part of the EPA's responsibility is the development of a demonstration plan, a quality assurance project plan (QAPP), and a health and safety plan. While the EPA pays for and has the primary responsibility for these plans, each is developed with input from all of the demonstration's participants. The plans define how activities will be conducted and how the technologies will be evaluated. MMTP also provides each developer with site information and often predemonstration samples so the developer can maximize the field performance of its innovative technology. Typically, the developers train demonstration personnel to ensure each operator is trained appropriately. This also ensures that potential users have valid information on training requirements and the types of operators who typically use a technology successfully.

The field demonstration itself is the shortest part of the process. During the field demonstration, data is obtained on cost, technical effectiveness (compared to standard methods), and limiting factors. In addition, standardized field methods are developed and daily logs of activities and observations (including photos or videotape) are produced. The EPA is also responsible for the comparative, conventional method analytical costs and the disposal of any wastes generated by the field demonstration.

The final component of an MMTP demonstration consists of reporting the results and ensuring distribution of demonstration information. The primary product of the demonstration is an ITER, like this one, which is peer-reviewed and distributed as part of the technology transfer responsibility of the MMTP. The ITER fully documents the procedures used during the field demonstration, QA/QC results, the field demonstration's results, and its conclusions. A separate QA/QC data package also is made available for those interested in evaluating the demonstration in greater depth. Also, short technical summaries are prepared to summarize the demonstration results and to ensure rapid and wide distribution of the information.

Each developer is responsible for providing the equipment or technology product to be demonstrated, funding its own mobilization, and training EPA-designated operators. The MMTP does not provide any funds to developers for costs associated with preparation of equipment for demonstration or for development, and it does not cover the costs developers incur to demonstrate their technologies.

Demonstration Purpose, Goals, and Objectives

During this demonstration, the EnviroGard PCB Test was evaluated for its accuracy and precision in detecting high and low levels of PCBs in soil samples, and the effects, if any, of matrix interferences on the test. The accuracy and precision of the test was statistically compared to the accuracy and precision attained in a conventional, fixed laboratory using standard EPA analytical methods. The EnviroGard PCB Test also was qualitatively evaluated for the length of time required for analysis, ease of use, portability, and operating cost.

Section 3
Predemonstration Activities

Several predemonstration activities were conducted by EMSL-LV, PRC, and the other demonstration participants. These activities included identifying developers, selecting the demonstration site, selecting the confirmatory laboratory and analytical method, conducting operator training, and conducting predemonstration sampling and analysis. This section summarizes these activities and presents the results of the predemonstration sampling and analysis.

Identification of Developers

EMSL-LV identified the EnviroGard PCB Test as one of four technologies showing promise for use in PCB field screening. After a review of available data on these four technologies, EMSL-LV concluded that they warranted evaluation under the MMTP.

Site Selection

The following criteria were used to select a hazardous waste site suitable for the demonstration:

- The four technologies had to be tested at a site with a wide range of PCB contamination.
- Contaminant concentrations had to be well characterized and documented. Thorough site background information was needed so that a demonstration sampling plan could be designed with a high degree of confidence that the desired range of PCB concentrations would be present in samples.
- The site had to be accessible so that demonstration activities could be conducted without interfering with other planned site activities.

Based on these criteria, the Abandoned Indian Creek Outfall (AICO) site at the Department of Energy (DOE) Kansas City Plant (KCP) was selected as the location for this demonstration. The soil at the AICO site is contaminated with a wide range of PCB concentrations. PCB levels range from not detected at a concentration of 0.16 mg/kg to 9,680 mg/kg. DOE has conducted numerous investigations at the site, including a Resource Conservation and Recovery Act (RCRA) facility investigation (RFI) and corrective measures study (CMS) in 1989 (DOE 1989). PCB concentrations at the AICO site are well documented, which made possible collecting samples with a wide range of PCB concentrations.

The DOE KCP is located about 20 miles south of downtown Kansas City, Missouri, at the northeast corner of Troost Avenue and 95th Street. The facility is owned by the government and operated by Allied-Signal, Inc., for DOE. The plant has been used since 1949 to manufacture nonnuclear components for nuclear weapons systems. The facility occupies more than 300 acres and includes three main buildings and numerous outbuildings with over 3 million square feet under roof. Land around the plant is primarily occupied by suburban residential and commercial developments (DOE 1989).

The AICO site is located immediately south of the DOE KCP between 95th Street and Bannister Road. The site is located in a former channel of Indian Creek and is the former location of a storm water outfall (Outfall 002), which discharged from KCP into the creek. In the early 1970s, Indian Creek was rerouted as part of a flood protection project and the construction of Bannister Road. When the creek was rerouted, the storm water outfall also was rerouted by extending a box culvert from the former outfall to the new creek channel. The outfall now discharges into Indian Creek about 500 feet south of the AICO site. The former creek channel in the AICO area was covered with about 10 feet of fill (DOE 1989).

PCBs are the only significant contaminants at the site. During the RFI and CMS, samples from 12 borings were analyzed for priority pollutants other than PCBs. Only one of these borings contained non-PCB priority pollutants. This boring was found to

contain several base neutral organics, including anthracene, fluoranthene, pyrene, and chrysene. It is believed that this sample included a piece of asphalt from the material used to fill the old creek channel and that the presence of these compounds was not the result of DOE KCP discharges through Outfall 002 (DOE 1989).

According to logbooks kept by Allied-Signal, Inc., when boreholes were drilled during the investigations of the AICO site, the former Indian Creek channel is overlain by 7 to 15 feet of fill material composed primarily of mottled clays. Shale and limestone fragments, wood, asphalt, and concrete slag up to 4 feet wide are found in the fill material. Near the surface, up to 20 percent of the fill is composed of organic matter, such as roots, peat, and wood (DOE 1989).

Sediments overlying bedrock consist of soft, dark brown to gray, homogenous, medium to high plasticity, moist clayey silt with traces of fine sand. This material varies in depth from 7 to 15 feet and appears to have low permeability (DOE 1989). The aquifer of concern beneath the AICO site is the shallow groundwater lying just above bedrock.

Selection of Confirmatory Laboratory and Method

EPA Region 7 Laboratory personnel selected one laboratory participating in the Contract Laboratory Program (CLP) to perform the confirmatory analysis of samples for this demonstration. All samples were analyzed using the method described in the CLP 1990 Statement of Work (SOW) for analyzing PCBs and pesticides. The EPA Region 7 Laboratory conducted a Level II data review of the confirmatory laboratory's data.

Operator Training

The EnviroGard PCB Test was demonstrated by PRC field personnel. Prior to the demonstration, the PRC operator was trained in the use of the technology. This training included a review of operating procedures and instructions provided by Millipore and informal field training conducted by Millipore at the start of the demonstration. Training was equivalent to that recommended by Millipore for actual site characterization projects.

Sampling and Analysis

In May 1992, PRC prepared a predemonstration sampling plan (PRC 1992a), and on July 14, 1992, PRC collected predemonstration soil samples from areas at the AICO site previously identified as containing high, medium, low, and not detected concentrations of PCBs. These samples were split into replicates. One replicate of each sample was submitted to Millipore, and the confirmatory laboratory analyzed one replicate.

The predemonstration sampling was conducted so that Millipore could refine its technology and revise its operating instructions, if necessary, before the demonstration. The sampling also allowed potential matrix effects or interferences to be evaluated prior to the demonstration. The principal finding from the predemonstration sampling was that the soil at the AICO site was more clayey than expected. The high clay content of the soil made homogenizing the samples difficult (see Section 4).

Section 4
Demonstration Design and Description

This section describes the sample collection procedures and the experimental design used to evaluate the EnviroGard PCB Test. This innovative technology was evaluated in conjunction with three other field screening technologies that also analyze PCBs in soil. The demonstration design and description, and the experimental design described in this section, are common to all four evaluations. The four evaluations also shared a single demonstration plan and QAPP. Key elements of the QAPP (PRC 1992b), field analysis operations, and data management activities are summarized in this section.

Sample Collection

For the demonstration, 112 soil samples and 32 field duplicate samples were collected from the AICO site. Each sample was thoroughly homogenized and then split into six replicate samples. One replicate from each sample was submitted to the confirmatory laboratory for analysis using the CLP 1990 SOW method. A second replicate was submitted to EMSL-LV for separate analysis at the request of the EPA technical project manager (TPM), although the data generated by EMSL-LV was not used in this demonstration. A third replicate was analyzed in the field using the EnviroGard PCB Test. The remaining replicates were analyzed in the field using the three other technologies described in separate ITERs.

Samples were collected using a drill rig to reach areas of the AICO site that, based on data from past investigations, exhibited a wide range of PCB concentrations. All samples were collected by PRC using the sample collection and homogenization procedures specified in the sampling plan (PRC 1992b). All PRC field activities also conformed with requirements in the health and safety plan prepared for this demonstration (PRC 1992b).

Samples were collected from areas known to exhibit PCB concentrations ranging from not detected (at a concentration of 0.16 mg/kg) to 9,680 mg/kg. Most of the samples were collected from areas previously identified as containing PCBs in the not detected to 100 mg/kg range, for two reasons. First, this range encompasses typical regulatory thresholds for PCBs, such as the 10 mg/kg level for cleanups in unrestricted access areas and the 50 mg/kg level for cleanups in industrial areas. Second, most of the four field screening technologies demonstrated, including the EnviroGard PCB Test, were designed primarily for operation in this range.

Samples were also collected from areas previously identified as containing PCBs at concentrations ranging from 100 to 1,000 mg/kg, and from areas previously identified as containing PCBs at concentrations between 1,000 and 10,000 mg/kg. These samples were analyzed to evaluate the abilities of the field screening technologies to monitor PCBs in higher concentrations as well as in the average range. After collection, soil samples were placed in plastic bags and thoroughly homogenized. Samples were then split and placed in sample containers. Samples to be submitted for confirmatory laboratory analysis were placed in 8-ounce, wide-mouth glass jars with Teflon-lined lids. Samples for submittal to EMSL-LV and for analysis by the field screening technologies were placed in 4-ounce, wide-mouth glass jars with Teflon-lined lids.

Homogenization of the samples was monitored by adding a small amount of powdered uranine, the sodium salt of fluorescein dye (fluorescein), to each soil sample. Homogenization was then performed. PRC then examined each sample under an ultraviolet (UV) lamp in a portable darkroom. Because fluorescein fluoresces under UV light, PRC was able to ensure that homogenization was complete. While under the UV light, PRC sliced each sample in a minimum of five different places and examined each slice for fluorescence. If any of the slices did not contain signs of fluorescence, then homogenization of the sample continued and the examination process was repeated.

The use of small amounts of fluorescein was found not to interfere with sample analysis for any of the field screening technologies, nor for the confirmatory laboratory.

After confirmatory laboratory results were received, PRC used the results from samples and their respective field duplicate samples to statistically determine whether the homogenization efforts were successful. Because the duplicate samples were collected as splits, the expected difference between a sample and its duplicate was zero. This assumes that there was perfect homogenization and that there was no difference introduced by analytical error. Using a matched pair Student's t-test, it was possible to determine if the mean of the differences between the samples and their duplicates was significantly different from zero at a 95 percent confidence level. The matched pair Student's t-test showed that this mean was not significantly different. Therefore, though the results of a few pairs of samples and duplicates seem to indicate that homogenization could have been better, overall the homogenization technique used was highly effective.

To apply the matched pair Student's t-test, it was necessary to have a normally distributed data population. The differences between confirmatory laboratory samples and their respective duplicates were statistically evaluated and found to be normally distributed. Two data point outliers were noted in the frequency plot. The matched pair Student's t-test, however, was found acceptable even when the outliers were included in the data set.

The statistical analysis indicates that the homogenization was acceptable, but even at a 95 percent confidence level, a few anomalous duplicate results can exist in a data set without the analysis being greatly affected. For example, a single pair of samples with high RPDs relative to the population's mean RPD is masked and does not affect the overall assessment. Therefore, even with a statistical assessment that indicates overall effective sample homogenization, a limited number of poorly homogenized samples may have been included in the demonstration. The analysis of such data could produce limited cases of inaccurate data. For this reason, a large number of samples were collected and analyzed to prevent any anomalous samples from affecting the overall results.

Quality Assurance Project Plan

To ensure that all activities associated with this demonstration met demonstration objectives, a QAPP was prepared (PRC 1992b). The QAPP, which was incorporated into the demonstration plan, defined project objectives, how those objectives would be achieved, data quality objectives (DQO), and the steps taken to ensure that these objectives were achieved. All demonstration participants were given the opportunity to contribute to the development of the QAPP, and ultimately, all participants agreed to its content.

The primary purpose of the QAPP was to outline steps to be taken to ensure that data resulting from the demonstration was of known quality and that a sufficient number of critical measurements were taken. Based on the EMSL-LV SOW, this demonstration is considered a Category II project. The QAPP addressed the key elements required for Category II projects prepared according to guidelines in "Preparing Perfect Project Plans" (EPA 1989) and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (Stanley and Verner 1983).

For sound conclusions to be drawn on the four field screening technologies, the data obtained during the demonstration had to be of known quality. For all monitoring and measurement activities conducted for EPA, the agency requires that DQOs be established based on how the data will be used. DQOs must include at least five indicators of data quality: representativeness, completeness, comparability, accuracy, and precision. Each of these indicators is discussed in more detail below. The success of the demonstration required that DQOs be met by the confirmatory laboratory. Some DQOs for the confirmatory laboratory were indicated in the CLP 1990 SOW, and others were derived from data generated while using the method. It was critical that the confirmatory laboratory analyses be sound and within CLP 1990 SOW method specifications so that the data it generated could be compared to that obtained by the technologies. High quality, well-documented confirmatory results were essential for making this comparison.

Representativeness refers to the degree to which the data accurately and precisely represents the condition or characteristic of the parameter represented by the data (Stanley and Verner 1983). In this demonstration, representativeness was ensured by executing a consistent sample collection, homogenization, and handling program. Representativeness also was ensured by using each technology at its optimum capability to provide results that represented the most accurate and precise measurements it was capable of achieving.

Completeness refers to the amount of data collected from a measurement process compared to the amount that was expected to be obtained (Stanley and Verner 1983). For this demonstration, completeness refers to

the proportion of valid, acceptable data generated using each of the technologies and the confirmatory laboratory. The completeness objective for each technology during this demonstration was 90 percent, which was achieved.

Comparability refers to the confidence with which one data set can be compared to another (Stanley and Verner 1983). The main focus of this demonstration was to compare data generated from the EnviroGard PCB Test and the other technologies with confirmatory laboratory results using the experimental design and statistical methods discussed in Section 4. Additional QC for comparability was achieved by analyzing QC samples, blanks, and Aroclor standards, and by adhering to standard EPA analytical methods and standard operating procedures (SOP) for preparing samples and operating instruments.

Accuracy refers to the difference between the sample result and the reference or true value for the sample. Bias, a measure of the departure from complete accuracy, can be caused by instrument calibration, loss of analyte in the sample extraction process, interferences, and systematic contamination or carryover of analyte from one sample to the next. During this demonstration, accuracy was measured by making the statistical comparisons discussed under Experimental Design in this section.

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Precision for this demonstration was measured by comparing the RPDs of samples and their duplicates to control limits established through the statistical methods detailed under Experimental Design in this section.

Experimental Design

The primary objective of the demonstration was to evaluate the efficiency of the EnviroGard PCB Test and three other technologies at determining PCB contamination in soil. This evaluation included defining the precision, accuracy, cost, and range of usefulness for each of these technologies. This objective also included determining the DQOs that each technology was capable of achieving. An additional objective was to evaluate the specificity of each technology to different Aroclors.

Accuracy and precision were the most important quantitative factors evaluated, particularly for PCB concentrations near 10 mg/kg, a common cleanup goal. A significant part of PRC's statistical evaluation was to evaluate these factors.

The cost of using each field screening technology was another important factor. Costs include expendable supplies, nonexpendable equipment, labor, and investigation-derived waste (IDW) disposal. These costs were tracked during the demonstration. Although batch analysis of samples can have major effects on per sample costs, the number of samples collected for this demonstration were within the range of a normal site investigation. Similar-sized sample batches were analyzed by each of the field screening technologies.

Many analytical techniques can have significant operator effects in which individual differences in technique have a significant effect on the numerical results. To reduce the potential impact of measurement variation, PRC used a single operator for each field screening technology and accepted that the error introduced by operator effect would not be distinguishable from error inherent in the various field screening technologies. This policy was selected because it approximates ordinary field conditions in which only one screening method is typically used.

All analytical methods have a specific usable range with lower and upper limits. The usable range for each field screening technology was determined by comparing results from each technology to those from the confirmatory laboratory. Statistical analysis of these results were then used to identify the contaminant range in which results from each technology were comparable to the confirmatory laboratory result.

The Aroclor expected to be found at the AICO site was Aroclor 1242, which is a common PCB. However, there are other common Aroclors as well. In the planning stages of this demonstration, interest was shown in the cross-reactivity between Aroclors for each technology. To assess this factor, cross-reactivity for each technology was evaluated through the use of matrix spikes for each of the seven Aroclors (1016, 1221, 1232, 1242, 1248, 1254, and 1260) typically analyzed using standard EPA analytical methods. This information was then used to determine the sensitivities of the technologies to each Aroclor.

Statistical Analysis of Results

This demonstration required comparisons of various groups of data. Sample results from each technology were statistically compared to duplicate sample results and other QA/QC sample results. These are called intramethod comparisons. The sample results, also, were statistically compared to the results from the confirmatory laboratory, which were considered as accurate and precise as possible. Finally, in some cases, the precision obtained by a technology was statistically

compared to the precision obtained by the confirmatory laboratory.

All of the statistical tests used for this demonstration were stipulated in the demonstration plan, which was approved in advance of data collection by all demonstration participants (PRC 1992b). Also stipulated in the demonstration plan was that all sample pairs that included a not detected result would be removed from data sets. PRC felt that the variance introduced by eliminating these data pairs would be less than, or no more than equal to, the variance introduced by giving not detected results an arbitrary value.

In cases where field duplicate samples were collected, the demonstration plan stated that the results of the two duplicates would be averaged and this average used in subsequent statistical analysis. PRC followed this guideline, as well. In this way, samples were not unduly weighted in the statistical analyses.

The intramethod comparisons involved a statistical analysis of RPDs. First, the RPDs of the results for each sample pair, in which both the sample and its duplicate were found to contain PCBs, were determined. The equation below was used to calculate the RPDs:

(4-1)

$$RPD = \frac{R_i - R_d}{(R_i + R_d)/2} \times 100$$

where

RPD = relative percent difference.
R_i = initial result.
R_d = duplicate result.

Acceptable RPD values for field duplicate data are difficult to assess. These ranges are rarely published for organic parameters and are mainly dependent on the heterogeneity of contaminant distribution, sample collection activities, and analytical precision. For this demonstration, acceptable RPD values for field duplicate data were established by determining an upper control limit using guidance stated in SW-846 Method 8000. Because the technologies being demonstrated were themselves being assessed, the control limits used were calculated from data provided during this investigation. To determine the upper control limit, the standard deviation of the RPDs was calculated for each technology. This standard deviation was then multiplied by two and added to its respective mean RPDs. This established the upper control limit for the technology. Because an RPD of zero would mean that the duplicate samples matched their respective samples perfectly, zero was used as the lower control limit. This resulted in a large range of acceptable values. Because duplicate analyses seldom match perfectly, even for established technologies, all samples that fell within the control limits were considered acceptable. PRC determined that if at least 95 percent of the duplicate samples fell within these control limits, the technology had acceptable precision.

Each field screening technology's data was compared to the confirmatory laboratory data to determine its accuracy. This comparison involved three statistical methods: linear regression analysis, the Wilcoxon Signed Ranks Test, and the Fisher's Test.

Linear regression was calculated for the technologies that were capable of determining quantitative results. One of those was the EnviroGard PCB Test. PRC calculated this data by the method of least squares. Calculating linear regression in this way makes it possible to determine whether two sets of data are reasonably related, and if so, how closely. Calculating linear regression results in an equation that can be visually expressed as a line. Three factors are determined during calculations of linear regression. These three factors are the y-intercept, the slope of the line, and the coefficient of determination, also called r^2. All three of these factors had to have acceptable values before a technology's accuracy was considered acceptable.

The r^2 expresses the mathematical relationship between two data sets. If r^2 is 1, then the two data sets are perfectly correlated. Lower r^2 values indicate less of a relationship. Because of the nature of environmental samples, r^2 values between 0.80 and 1 were considered acceptable for this demonstration.

If an r^2 below 0.80 was found, the data was reviewed to determine whether any particular results were skewing the r^2. This skewing may sometimes occurs because technologies are often more accurate when analyzing samples in one range than when analyzing samples in another range. In particular, samples with either very high or very low levels of contamination often skew the results. For this demonstration, the technique used to identify outliers that might have skewed the results was residual examination (Draper and Smith 1981). The computer program used for calculating the linear regression, in fact, identified most of the outliers. When outliers were identified, they were removed and linear regression was calculated again.

If the corrected data set resulted in an r^2 between 0.80 and 1, then the regression line's y-intercept and slope were examined to determine how closely the two

data sets matched. A slope of 1 and a y-intercept of zero would mean that the results of the technology matched those of the confirmatory laboratory perfectly. Theoretically, the farther the slope and y-intercept differ from these expected values, the less accurate the technology. Still, a slope or y-intercept can differ slightly from its expected value without that difference being statistically significant. To determine whether such differences were statistically significant, PRC used the normal deviate test statistic. This test statistic calculates a value that is compared to a table. The value at the 95 percent confidence level was used for the comparison.

If an r^2 between 0.80 and 1 was not found, then the technology's data was considered inaccurate. The technology's data was also considered inaccurate if an r^2 between 0.80 and 1 was found, but the normal deviate test statistic indicated that either the y-intercept or the slope differed significantly from its expected result. However, in this case, the data could be mathematically corrected if 10 to 20 percent of the samples were sent to a confirmatory laboratory. Analysis of a percentage of the samples by a confirmatory laboratory would provide a basis for determining a correction factor. Only in cases where the r^2, the y-intercept, and the slope were all found to be acceptable did PRC determine that the technology's data was accurate.

A second statistical method used to assess the accuracy of the data from each technology was the Wilcoxon Signed Ranks Test. This test is a nonparametric method for comparing matched pairs of data. It can be used to evaluate whether two sets of data are significantly different. The test requires no assumption regarding the population distribution of the two sets of data being evaluated other than that the distributions will occur identically. In other words, when one data point deviates, its respective point in the other set of data will deviate similarly. Because the only deviation expected during the demonstration was a difference in the concentrations reported by each technology, the two sets of data were expected to deviate in the same way.

The calculation performed in the Wilcoxon Signed Ranks Test uses the number of samples analyzed and a ranking of the number that results when a sample's result obtained by using one analytical method is subtracted from the corresponding result obtained by using another method. The rankings can be compared to predetermined values on a standard Wilcoxon distribution table, which indicates whether, overall, the two methods have produced similar results.

Although the Wilcoxon Signed Ranks Test and the linear regression analysis perform similar types of comparisons, the assumptions on which each is based are different. By running both tests on the data, PRC was able to determine whether either test's assumptions were violated, and if so, whether the statistical results were affected.

The EnviroGard PCB Test produced semiquantitative results. Linear regression analysis and the Wilcoxon Signed Ranks Test cannot be used to compare semiquantitative results. Instead, PRC used a 2 by 2 contingency table and a Fisher's Test. The Fisher's Test determines whether both data sets are correlated. When used in a two-tailed manner, as it was in this case, its formula is usually conservative. Therefore, use of a modified Chi-square formula is recommended (Pearson and Hartley 1976). This formula, as used in this demonstration, is:

(4-2)

$$X^2 = \frac{\Sigma[(observed\ value - expected\ value) - .5]^2}{expected\ value}$$

The Fisher's Test statistics were compared to the 95 percent confidence level obtained from a standard Chi-square distribution table. This comparison indicated whether, overall, there was a correlation between the results of the two methods. If a correlation existed, the technology's data was considered accurate.

Finally, the precision obtained by each technology was statistically compared to the precision obtained by the confirmatory laboratory using Dunnett's Test. This test was used to assess whether the precision of the technology and that of the confirmatory laboratory were statistically equivalent. First, the mean RPD for all samples and their respective duplicates analyzed by the confirmatory laboratory was determined. The RPDs of each duplicate pair analyzed by each of the technologies was then statistically compared to this mean. The Dunnett's Test results in a single statistical value which indicates the degree of certainty that the precision of the two methods is the same. In other words, a 90 percent value indicates that one can be 90 percent sure the precision is the same. During this demonstration, values of 95 percent or better indicated that the precisions were statistically the same.

It should be noted that results below 95 percent do not mean that the precision of the technology was not acceptable, only that it may be different from the precision of the confirmatory laboratory. In particular, Dunnett's Test has no way of determining whether or not

any difference between the two data sets actually resulted because a technology's data was more precise than the confirmatory laboratory's.

Field Analysis Operations

The field analysis portion of the demonstration was performed in a rented 28-foot trailer. Electricity was supplied for the equipment, refrigerators, and air conditioners. Space within the trailer was divided to provide an area for each technology, sample storage, and the storage of sample collection equipment. All of the equipment, supplies, reagents, and office supplies needed for the demonstration were moved into the trailer during the weekend before the start of the demonstration. All analytical equipment was powered up and checked to ensure that it was operable. All problems found were corrected.

Section 5
Confirmatory Analysis Results

All samples collected during this demonstration were submitted to the EPA Region 7 Laboratory for analysis under its CLP. The data supplied by the confirmatory laboratory is discussed in more detail in the following sections.

Confirmatory Laboratory Procedures

The samples collected during the demonstration were sent to the EPA Region 7 Laboratory where they were assigned EPA activity number DSX06. The samples were then shipped to the confirmatory laboratory for CLP 1990 SOW method analysis. This method requires that organochlorine pesticides and PCBs be analyzed using a gas chromatograph (GC) equipped with an electron capture detector (ECD).

EPA Region 7 Laboratory personnel conducted a Level II data review on the results provided by the confirmatory laboratory. This data review involved evaluating reported values and specific QC criteria. A Level II data review does not include an evaluation of the raw data or a check of calculated sample values. A review of the raw data and a check of the calculations was performed by the confirmatory laboratory before submitting the data package to EPA. PRC was not able to review the raw data generated from the analysis of samples. However, PRC did review the EPA's comments generated by the Level II data review.

The following sections discuss specific procedures used to identify and quantitate PCBs using the CLP 1990 SOW method. Most of these procedures involved requirements that were mandatory to guarantee the quality of the data generated.

In addition to being generally discussed in this Section, all of the confirmatory laboratory results used to assess the Millipore EnviroGard PCB Test are presented in tables in Section 6.

Soil Sample Holding Times

The CLP 1990 SOW method requires that all soil sample extractions be completed within 7 days from the laboratory's validated sample receipt. The analysis of soil samples must be completed within 40 days of validated sample receipt. The holding time requirements for the samples collected during this demonstration were met.

Soil Sample Extraction

Soil samples were extracted according to the procedures outlined in the CLP 1990 SOW method for organochlorine pesticides and PCBs. This procedure involves placing 30 grams of soil into a beaker and then adding 60 grams of purified sodium sulfate. This mixture is thoroughly mixed to a grainy texture. One hundred milliliters (mL) of a 50:50 ratio mixture of acetone and methylene chloride then is added to the beaker containing the soil and sodium sulfate. Pesticides and PCBs are extracted into the organic solvent with the aid of a sonic disrupter. This sonic disrupter bombards the soil with sonic waves, which facilitates the transfer of pesticides and PCBs into the organic solvent. The organic solvent is vacuum-filtered through filter paper to separate it from the soil particles. Sonication is repeated two more times with 100 mL of the acetone and methylene chloride mixture. The organic solvent is filtered and combined in a vacuum flask.

After filtration, the solvent is transferred to a Kuderna-Danish apparatus. The Kuderna-Danish apparatus is placed in a hot water bath, and the organic solvent is concentrated. Once concentrated, the solvent is transferred from the acetone and methylene chloride mixture into hexane by using a nitrogen evaporation system. The soil sample extract, now in hexane, is concentrated to a known volume using this system. The soil sample extract is taken through a florisil solid-phase

extraction column to remove any polar compounds from the extract. The soil sample extract is diluted to 10 mL with hexane and is transferred to a test tube to await sample analysis.

Initial and Continuing Calibrations

The CLP 1990 SOW method for analyzing PCBs involves an initial calibration (ICAL) for PCBs, which consists of analyzing one concentration of each of the seven Aroclors listed in the Target Compound List (TCL). The ICAL is used to determine peaks to identify Aroclors and to determine factors to quantitate PCBs in samples. The ICAL is performed before sample analysis begins. PCBs cause multipeak patterns when analyzed using gas chromatography. For each Aroclor, three to five peaks are chosen to monitor retention time shift and to determine factors used for quantitation.

Continuing calibrations (CCAL) are performed by analyzing instrument blanks and performance evaluation (PE) mixture standards. The retention times and calibration factors determined during the ICAL are monitored through CCALs. The CCAL standard is typically a mid-level pesticide standard; however, because PCBs were the compounds of interest, an Aroclor was used as the CCAL standard for analyzing these samples.

Retention times were monitored through evaluating the amount of retention time shift from the PCB CCAL standard as compared to the PCB ICAL standard. The retention time window was defined as $\pm$ 0.07 minute for each peak identified in the ICAL. According to the CLP 1990 SOW method, any time a peak of an Aroclor falls outside of its window, a new ICAL must be conducted. During the analysis of samples for this demonstration, the retention times of the peaks chosen for monitoring during the CCAL never exceeded the windows established for them in the ICAL.

Calibration factors were monitored in accordance with the CLP 1990 SOW method and were acceptable, as the CCAL calibration factor never exceeded 25 percent.

Once an ICAL has been performed, sample analysis begins. Usually, sample analysis begins by analyzing a method blank to verify that it meets the CLP 1990 SOW method requirements. After this, sample analysis may continue for 12 hours. After every 12-hour period, a CCAL standard must be analyzed. Sample analysis may continue as long as CCAL standards meet the CLP 1990 SOW method requirements.

Sample Analysis

PCBs are identified in samples by matching peak patterns found after analyzing the sample with those found in Aroclor standards. Peak patterns may not match exactly because of the way the PCBs were manufactured or because of the effects of weathering. When the patterns do not match, the analyst must choose the Aroclor that most closely matches the peak pattern present in the sample. For this reason, peak pattern identification is highly dependent on the experience and interpretation of the analyst.

Quantitation of PCBs is performed by measuring the response of the peaks in the sample to those same peaks identified in the ICAL standard. The reported results of this calculation are based on dry weights, as required by the CLP 1990 SOW method. Because the field screening technologies all reported wet weight results, PRC converted the results reported by the confirmatory laboratory from dry to wet weights to account for any loss of sample weight caused by drying.

The calculation to convert dry weight results to wet weight results is shown below:

(4-1)

$$WW = DW/1-MC$$

where

WW = Wet weight results.
DW = Dry weight results.
MC = Moisture content ratio provided by confirmatory laboratory.

Sample extracts frequently exceed the calibration range determined during the ICAL. When they do so, they must be diluted to obtain peaks that fall within the linear range of the instrument. For PCBs, this linear range is defined as 16 times the response of the Aroclor standards analyzed during the ICAL. Once a sample is diluted to within the linear range, it is analyzed again. Dilutions were performed when appropriate on the samples for this demonstration.

Detection Limits

One concentration of each Aroclor was analyzed during the ICAL. The concentration of each Aroclor standard should correspond to the contract required quantitation limit (CRQL) when corrected for the sample extraction concentration factors. The concentration used for Aroclor 1221 was 200 micrograms per kilogram (μg/kg); the level used for the other six Aroclors was

100 μg/kg. This corresponds to soil sample detection limits of 67 μg/kg for Aroclor 1221 and 33 μg/kg for the other Aroclors.

Because of CLP 1990 SOW method requirements, these detection limits are based on samples that have no moisture content. Because almost all soil samples contain moisture, the detection limits stated above are raised to correct for the percent moisture present in the soil sample. However, PRC did not correct the detection limits to account for the percent moisture present in the samples because the CRQLs were listed in μg/kg and the detection limits of the EnviroGard PCB Test and the other technologies were listed in mg/kg. Even when corrected to account for percent moisture, the CRQLs would be significantly below the detection limits for each technology.

Quality Control Procedures

A number of QC measures were used by the confirmatory laboratory as required in the CLP 1990 SOW method, including analysis of resolution standard mixes, method blanks, and instrument blanks, all requirements of which were met for this demonstration.

Also, surrogate standards were added to all standards, method blanks, matrix spikes, and soil samples analyzed using the CLP 1990 SOW method. The percent recovery of each surrogate was calculated and compared to the advisory control limits of 60 to 150 percent found in the CLP 1990 SOW. No corrective action is needed when surrogate recoveries fall outside of the advisory control limits. The surrogate recoveries, though, are reported with the other QC data. During this demonstration, 12 soil samples and field duplicate samples from the confirmatory laboratory analysis were outside the advisory control limits for surrogate recoveries.

During the demonstration, 46 samples and their respective duplicate samples required dilution to obtain peaks that were within the linear range required by the CLP 1990 SOW; however, the dilutions decreased the amount of the surrogate standards that were injected onto the GC. The result was that the surrogates were not detected in the samples. PRC was not able to obtain information regarding actual surrogate standard recovery for each of the samples analyzed by the confirmatory laboratory. Comments from the EPA Level II data review, though, indicated that 88 of the samples and their respective duplicate samples resulted in acceptable surrogate recovery data.

The CLP 1990 SOW requires that matrix spike and matrix spike duplicate samples be prepared with six organochlorine pesticides and analyzed with each batch of samples. Because the demonstration was only concerned with PCB results, the matrix spike results were not reported.

Confirmation of Analytical Results

The CLP 1990 SOW also requires that all positive sample results be confirmed. There are two methods of confirming sample results. The first, required in all cases, is to analyze the sample again using a second GC column. If concentrations identified this way are sufficiently high, the second method, analyzing the sample again using a GC and mass spectrometer (MS), must also be used.

Second Column Confirmation

As required, all samples that were found to contain PCBs during analysis on the first column were analyzed on the second column. In all cases, the presence of PCBs were confirmed. There were 122 samples that required second column confirmations.

The CLP 1990 SOW states that results from the two columns should be within 25 percent of each other. When this requirement is not met, the result for that sample must be coded to indicate that it is estimated. For the analysis of the samples from this demonstration, 17 sample results were above the 25 percent requirement of the CLP 1990 SOW. These results were J-coded to indicate that the results were estimated but were not validated by approved QC procedures. Finally, following the CLP 1990 SOW method, the lower of the two values was reported.

Gas Chromatographic and Mass Spectrometer Confirmation

The CLP 1990 SOW requires that when pesticides or PCBs are present in samples at sufficient quantities, they must be confirmed by GC and MS analysis. Twenty samples from this demonstration contained sufficient quantities of PCBs to require GC and MS confirmation. These samples were compared to Aroclor standards. None of the 20 samples were confirmed through GC and MS analysis. Lack of GC and MS confirmation is not uncommon for Aroclors because they are a mixture of congeners, and the GC and MS analysis is better suited for identifying individual congeners. Because all 20 samples were confirmed on the second GC column, the lack of GC and MS confirmation was determined to be insignificant during the EPA Level

II data review. Therefore, these samples were not coded.

Data Reporting

The data report PRC received from the EPA Region 7 Laboratory included a standard EPA Region 7 Analysis Request Report. PCBs were the only compounds reported. Results were reported on a dry weight basis, as required in the CLP 1990 SOW. PRC obtained data on the percentage of solids in the sample from the confirmatory laboratory and used this data to convert the results to wet weight. This conversion was required because the data was to be compared to data from the EnviroGard PCB Test and three other technologies, all of which reported concentrations based on wet soil weight. PRC also converted the confirmatory laboratory results from μg/kg to mg/kg.

The results reported by the confirmatory laboratory contained three different codes. Every result was coded with a "V," indicating that the data had been reviewed and reported correctly. Some data was coded with a "K," indicating that the actual PCB concentration in the sample was less than the reported value, or that PCBs were not found in the sample. The third code used was "J", which indicated that the data was estimated but not validated by approved QC procedures. Twenty-nine of the 146 total samples submitted for analysis were J-coded.

Aroclors Reported by the Confirmatory Laboratory

According to RFI and CMS results from April 1989, the only Aroclor believed to be present at the AICO site was Aroclor 1242. However, the confirmatory laboratory found three additional Aroclors in the samples collected during the demonstration. Most of the samples analyzed by the confirmatory laboratory were found to contain either Aroclor 1242 or Aroclor 1248. Seventy-three samples were found to contain only Aroclor 1242, while 33 samples were found to contain only Aroclor 1248. Sixteen samples were found to contain mixtures of two of the four Aroclors found. The predominant mixture was Aroclor 1242 and Aroclor 1248. Seven samples were found to contain this mixture. Four samples were found to contain a mixture of Aroclor 1242 and Aroclor 1260. Three samples were found to contain a mixture of Aroclor 1248 and Aroclor 1260. Two samples were found to contain a mixture of Aroclor 1242 and Aroclor 1254. In all, 122 soil samples submitted to the confirmatory laboratory for this demonstration were found to contain detectable levels of PCBs. Twenty-four samples were reported as not containing PCBs above the CRQLs.

Data Quality Assessment of Confirmatory Laboratory Data

This section discusses the accuracy, precision and completeness of the confirmatory laboratory data.

Accuracy

Accuracy for the confirmatory laboratory results was assessed through the use of PE samples, purchased from Environmental Research Associates (ERA), that contained a known quantity of Aroclor 1242. ERA supplied data sheets for each PE sample, which included the true concentration and an acceptance range for the sample. The acceptance range was based on the 95 percent confidence interval taken from data generated by ERA and EPA interlaboratory studies.

The two PE samples contained different concentrations, one low and one high. These samples were extracted and analyzed in exactly the same manner as the other soil samples. The confirmatory laboratory knew that the samples were PE samples, but the true concentrations and acceptance ranges of the samples were not known to the confirmatory laboratory. The true concentration of sample 047-4024-114 (the high-level sample) was 110 mg/kg, with an acceptance range of 41 to 150 mg/kg. The result reported for this sample by the confirmatory laboratory was 67 mg/kg of Aroclor 1242, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 61 percent. The true value concentration of sample 047-4024-113 (the low-level sample) was 32.7 mg/kg, with an acceptance range of 12 to 43 mg/kg. The result reported by the confirmatory laboratory for this sample was 15 mg/kg, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 46 percent. Based on the results of the PE samples, the accuracy of the confirmatory laboratory was acceptable.

Precision

Precision for the confirmatory laboratory results was determined by evaluating field duplicate sample results. Other types of data typically used to measure precision were not available. Laboratory duplicate samples were not required by the CLP 1990 SOW. Two other types of data commonly used to measure precision, matrix spike and matrix spike duplicate RPDs, also were not available because matrix spike compounds required by the CLP 1990 SOW method are pesticide compounds, not PCBs.

The evaluation of field duplicate sample results was used to assess the precision of the analytical method.

Precision can be evaluated by determining the RPDs for sample results and their respective field duplicate sample results. The RPDs for the 32 field duplicates and their respective samples averaged 31.8 percent, but this included two pairs of samples with extremely dissimilar results. Sample 102 had a result of 293 mg/kg while its duplicate, Sample 102D, had a result of 1.77 mg/kg. The RPD for the sample pair was calculated as 197.6 percent. Also, Sample 097 had a result of 1.23 mg/kg while its duplicate had a result of 0.285 mg/kg. The RPD for Sample 097 and 97D was 124.8 percent. The other RPDs, though, had much lower percentages. Without these two samples, the mean RPD fell to 20 percent. Overall, this data shows excellent agreement between the samples and their respective field duplicates, indicating a high degree of precision by the confirmatory laboratory. The mean RPD also indicated that the method used to homogenize the samples before splitting them for analysis was highly effective.

Completeness

This demonstration resulted in the collection of 112 samples, 32 field duplicate samples, and two PE samples. Results were obtained for all of these samples. Of the 146 total samples analyzed by the confirmatory laboratory, 29 were J-coded. The J-code is defined by EPA Region 7 Laboratory as data estimated but not validated by approved QC procedures. Based on the definition of completeness given above, these 29 samples cannot be considered complete. Because of this, completeness for the samples analyzed by the confirmatory laboratory was 80 percent, which is below the completeness objective of 90 percent. However, the J-coded data was determined to be acceptable by PRC and EMSL-LV. For this reason, the actual completeness of data used was 100 percent.

Use of Qualified Data for Statistical Analysis

As noted previously, 20 percent of the confirmatory laboratory results were reported as data not validated by approved QC procedures. The EPA Level II data review indicated that this J-coded data was not valid because it had failed at least one of the two QA/QC criteria specified in the CLP 1990 SOW.

Twelve samples were determined to be invalid because one of the two surrogate compound recoveries was outside of the advisory control limits. In all cases, the second surrogate recovery was within the advisory control limit. The remaining 17 samples were considered invalid because results from the two GC columns used for sample quantitation differed by more than 25 percent.

Neither of these QA/QC problems was considered serious enough to preclude the use of J-coded data for this demonstration. The surrogate recovery control limits are for advisory purposes only, and no corrective action was required for the surrogate recoveries that were outside of this range. High percent differences between the sample results analyzed on the two GC columns is a frequent problem when analyzing samples with very complex chromatograms. In all cases, the reported value was the lower of the two, reducing the effect of interferants on the results.

As discussed in the QAPP (PRC 1992b), a rejection of a large percentage of data would increase the apparent variation between the confirmatory laboratory data and the data from the technologies. This apparent variation would be of a similar magnitude to that introduced by using the data. For these reasons, PRC, after consulting with EMSL-LV, elected to use the J-coded data despite the fact that the EPA Region 7 Laboratory had determined the results to be invalid under approved QC procedures.

Section 6
Millipore EnviroGard PCB Test

This section provides information on the EnviroGard PCB Test, including background information, operational characteristics, performance factors, specificity, a data quality assessment, and a comparison of its results with those of the confirmatory laboratory. Observations on the technology made by the operator during the demonstration are presented throughout this section.

The EnviroGard PCB Test was designed by Millipore for use in a semiquantitative mode. Most of this section discusses the technology as a semiquantitative analytical method. However, after consulting with both Millipore and EMSL-LV, PRC also tested the technology's ability to produce quantitative results. Details of its quantitative abilities are included in this section.

Theory of Operation and Background Information

The EnviroGard PCB Test is designed to provide quick, semiquantitative results for PCB concentrations in soil samples using an immunoassay approach. The approach uses polyclonal antibodies to detect and quantify PCBs in a sample. These results indicate whether PCB concentrations are above or below a specific level within a 99 percent confidence level. The technology can be customized to report specific results over a particular range of concentrations.

The EnviroGard PCB Test is designed to report levels of PCBs that are above or below three known concentrations. These known concentrations are defined by the standards used to calibrate the technology. The EnviroGard PCB Test comes with standards of Aroclor 1248 in the following PCB concentrations: 5, 10, and 50 mg/kg.

Before sample analysis can take place, soil samples must be extracted. Soil samples are extracted using an organic solvent and steel ball bearings. An aliquot of the soil sample extract is then diluted and placed in a test tube coated with the anti-PCB antibodies. This solution is mixed and allowed to incubate for 5 minutes so that all of the PCBs in the sample extract can attach to the anti-PCB antibody binding sites. After the incubation period, the test tube is emptied and vigorously washed with four test-tube volumes of tap or distilled water. Four drops of enzyme conjugate are then added to the test tube and allowed to incubate for 5 minutes while periodically mixed. The enzyme conjugates compete with PCBs for the binding sites of anti-PCB antibodies on the test tube. After the 5-minute incubation time, the test tube is again emptied and vigorously washed with four test-tube volumes of tap or distilled water.

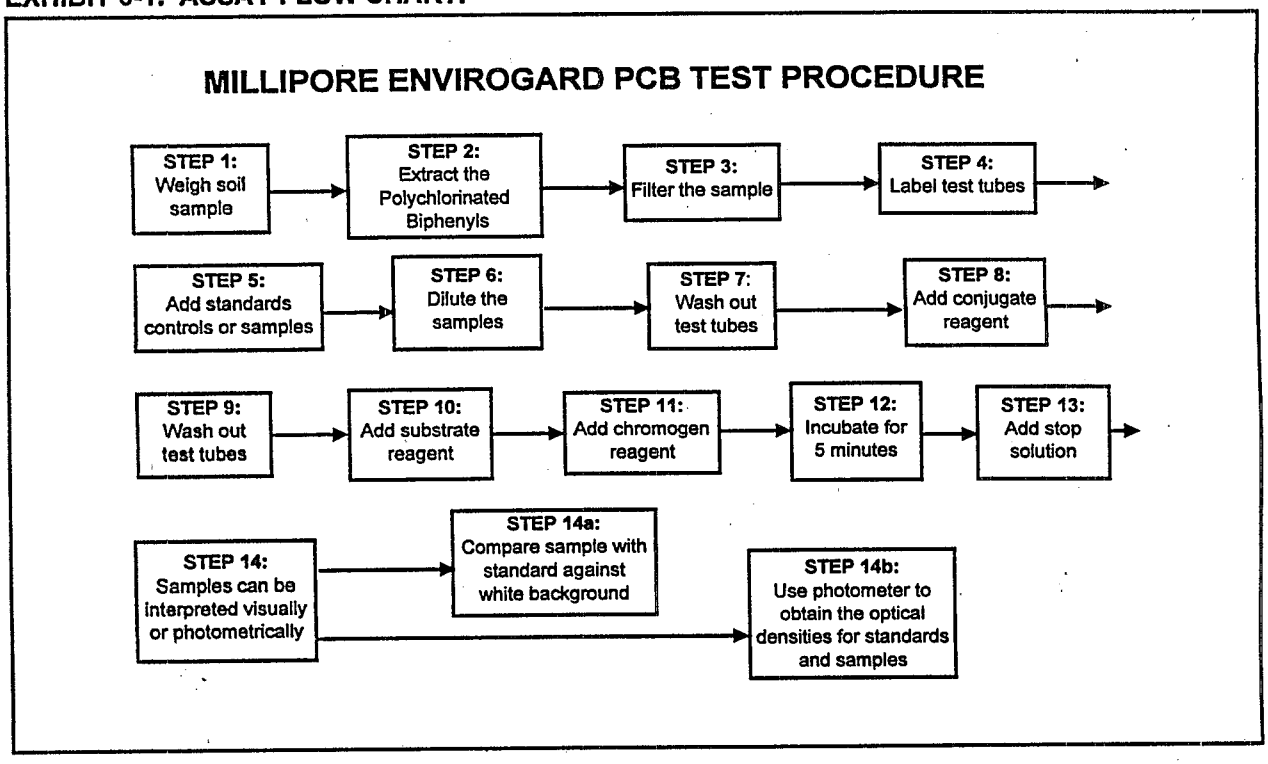
Next, color reagents are added to the test tube. Four drops of substrate are added to the test tube, followed by four drops of chromogen. This mixture is swirled in the test tube and allowed to incubate for 5 minutes. After 5 minutes, a stop solution is added to the test tube to stop color development. Test results are estimated visually by observing the color development. A blue color indicates the absence of PCBs, while a lighter blue or clear color indicates the presence of PCBs. To obtain semiquantitative results, the color of the solution is compared to the colors resulting from the analysis of the Aroclor standards and a negative control sample. For a more precise measurement of the PCB concentration, the color of the solution is compared to the color resulting from the analysis of the Aroclor standards and a negative control sample using a differential photometer (see Exhibit 6-1).

Operational Characteristics

Overall, the EnviroGard PCB Test was found to be portable. The test is made up of three kits: the EnviroGard PCB Field Lab, the EnviroGard Field Soil Extraction Kit II, and the EnviroGard PCB Test Kit.

The EnviroGard PCB Field Lab is a starter kit and consists of a portable carrying case about 18 inches long,

EXHIBIT 6-1. ASSAY FLOW CHART.



18 inches wide, and 6 inches high. The carrying case and all the equipment contained in it weighs nearly 10 pounds. That equipment includes (1) a differential spectrophotometer equipped with an electrical cord and rechargeable battery, (2) a positive displacement precision pipettor for dispensing volumes between 1 and 25 microliter (μL), (3) an Eppendorf Repeater pipettor, (4) an electronic timer, (5) a portable balance with a 50-gram calibrator weight, (6) a 500-mL wash bottle, (7) two six-position test tube racks, (8) a rack of pipette tips for the positive displacement precision pipettor, and (9) an instruction card. Several other items needed to complete this kit were provided by Millipore in a cardboard box. These items included (1) one 12- by 75-mm polystyrene test tube used for blanking the spectrophotometer, (2) eight 5-mL pipette tips for the Eppendorf Repeater pipettor, for dispensing volumes between 0.1 and 0.5 mL, (3) four 12.5-mL pipette tips for the Eppendorf Repeater pipettor for dispensing volumes between 0.25 and 0.625 mL, and (4) one 50-mL pipette tip for the Eppendorf Repeater pipettor, for dispensing volumes between 1.0 and 5.0 mL.

The EnviroGard Field Soil Extraction Kit II and its associated equipment are provided in a small cardboard box. The equipment includes (1) 12 polypropylene extraction bottles with screw caps containing five steel ball bearings, (2) 12 filter devices consisting of 12 upper and 12 lower units, (3) 12 polyethylene prefilters, (4) 15 wooden spatulas, (5) 12 4-mL, screw-top glass vials, and (6) 15 polyethylene weigh boats.

The EnviroGard PCB Test Kit comes in a small cardboard box and includes enough reagents and other equipment to analyze 16 samples. Most of its contents require refrigeration. This kit contains (1) 20 polystyrene tubes coated with anti-PCB antibody, (2) a 14-mL vial of assay diluent, (3) a 4.8-mL vial of PCB enzyme conjugate, (4) a 0.5-mL vial of negative control solution, (5) a 4.8-mL vial of peroxide solution (substrate), (6) a 4.8-mL vial of chromogen solution, (7) a 15-mL vial of 1.0-normal sulfuric acid (stop solution), (8) three 0.5-mL vials of 5, 10, and 50 mg/kg Aroclor 1248 standards, and (9) a plastic six-position test tube rack. Aroclor standards can be obtained in different concentrations by contacting Millipore.

Electricity is needed for the differential photometer. Millipore offers a photometer that can be operated using either a 115-volt or 230-volt electrical supply. The differential photometer also is equipped with a rechargeable battery. This battery requires 8 to 24 hours of charging, and when fully charged can perform up to 500 tests without being recharged. A battery also is needed to operate the portable balance. A refrigerator must be available to keep reagents cool to avoid a decrease in the activity of the enzyme conjugate. The recommended storage temperature of the reagents is

between 2 and 8 °C. The activity of the enzyme conjugate can be decreased by freezing the reagents, by exposing them to temperatures above 37 °C, or by repeated exposure to ambient temperatures.

Miscellaneous provisions needed to analyze samples include (1) methanol for sample extraction and dilution, (2) distilled or tap water for washing the test tubes coated with anti-PCB antibodies, (3) a table or work space of at least 8 square feet, (4) a permanent marker for writing sample numbers on test tubes and sample containers, (5) a logbook or a report form for recording sample results, and (6) an ink pen.

The EnviroGard PCB Test is designed for use in the field or laboratory. Some of its instrumentation and equipment require special handling, such as the differential photometer, portable balance, and two pipettors. This equipment must be handled carefully to avoid damage.

The reliability of the EnviroGard PCB Test was evaluated by monitoring instrument calibrations. The ICAL problems experienced by the operator were attributed to the small volumes used to prepare the Aroclor standards and to the operator's inexperience in using the pipette required to measure these small volumes. Preparing the Aroclor standards required measuring 5 μL each of the three concentrations. Using such a small volume may produce large errors, especially if a single drop of Aroclor standard is left outside the pipette tip or if the pipette tip is not placed completely into the vial containing the Aroclor standard, allowing air into the pipette tip rather than the Aroclor standard.

Thirty-one total calibrations were performed during this demonstration to produce both the semiquantitative and quantitative data on PCB concentrations in the samples. Initially, each calibration consisted of two sets of Aroclor standards, each containing three concentration levels: Aroclor 1248 (5, 10, and 50 mg/kg) provided by Millipore, and Aroclor 1242 (1, 5, and 25 mg/kg) prepared by PRC. PRC prepared the Aroclor 1242 standards and used them to perform the quantitative evaluation, discussed later in this section. The Aroclor 1248 standards were used for the semiquantitative evaluation of the technology. Semiquantitative and quantitative analyses were conducted concurrently, and therefore, the semiquantitative and quantitative batch calibrations were done at the same time. The following paragraphs discuss problems with the semiquantitative calibrations; a discussion of problems found during calibrations for the quantitative evaluation is found later in this section.

Five calibration failures occurred within the first 16 semiquantitative calibrations. The first calibration failure was attributed to operator error and errors attributed to measuring the small volumes associated with the analysis step. This problem appeared to be resolved after a reanalysis of these standards and further training of the operator. Because of the failures, the use of the three Aroclor 1248 standards was discontinued. All subsequent calibrations with the Aroclor 1248 standard involved only the 10 mg/kg concentration. This concentration was selected as the sole Aroclor 1248 calibration standard because it is a common action level for PCB cleanups.

Reanalysis of samples associated with the first 16 Aroclor 1248 standards was required for only two of the unacceptable calibrations. This is because acceptable Aroclor 1242 calibration was obtained for the other three, even though the Aroclor 1248 calibrations failed. It also should be noted that the predominant Aroclor expected at the site was Aroclor 1242.

The frequent problems with the Aroclor 1248 standards may have occurred because the actual concentrations of the standards were not the concentrations shown on the standards' containers. The actual concentrations were different because Millipore built a safety margin into the Aroclor standards. The stated concentrations of the Aroclor 1248 standards were 5, 10, and 50 mg/kg. The actual concentrations of these standards were 3, 5, and 22 mg/kg, respectively. Three of the five Aroclor 1248 calibrations that failed did so because the response for the 5 mg/kg standard was greater than the response of the 10 mg/kg standard. Because the concentrations of these two standards were actually 3 and 5 mg/kg, the color changes that resulted when they were analyzed were very similar. The small differences in the actual concentrations of the standards magnified the possibility of errors associated with analysis of the standards.

Between the 17th and 31st calibrations, there were two failures. These failures were not associated with either the Aroclor 1242 or 1248 standards. The first calibration failure was due to a positive response exhibited by the negative control sample. A negative control sample was analyzed with each standard and used as an assay blank. The samples analyzed during this unacceptable calibration were reanalyzed during another acceptable calibration. The second calibration failure was due to a lack of color formation in any of the samples, Aroclor standards, or negative control samples. The cause of this failure was unknown.

The EnviroGard PCB Test requires the use of chemicals to perform the analysis. The chemicals used

include methanol, a flammable solvent that can be absorbed through the skin, and sulfuric acid, which can cause chemical burns. Chemical resistant clothing, gloves, and safety glasses should be worn when using this solution to protect against injury while using these chemicals

The operator chosen to use the EnviroGard PCB Test was Mr. Keith Brown, an employee of PRC. He earned a B.G.S. degree in Environmental Science from the University of Kansas in 1990. While at PRC, Mr. Brown has conducted preliminary site assessments and investigations at hazardous waste sites throughout EPA Regions 5, 7, and 9. He also has performed hydrogeologic investigations at similar sites. Mr. Brown has more than 2 years experience performing these investigations with PRC and a previous employer.

Mr. Brown reviewed the information provided by Millipore concerning the analysis of soil samples using the EnviroGard PCB Test before the start of the demonstration. This information included a 20-minute videotape that explained the fundamentals of immunoassay analysis and introduced the sample extraction and analysis techniques used. Additional training was provided by Dr. Alan Weiss of Millipore at the start of the demonstration. This training included an in-depth explanation of the sample preparation and analysis steps. In addition, Mr. Brown analyzed five predemonstration samples using the technology under the supervision of Dr. Weiss. Mr. Brown noted that he felt comfortable using the EnviroGard PCB Test after analyzing the five predemonstration samples.

Millipore states that this product is intended for use by individuals with little or no training in PCB testing. The operator for this technology had no prior PCB testing experience and noted that the sample preparation and analysis steps were simple and straightforward. As described above, the operator did experience some problems with the technology's ICAL. The first attempt at calibration resulted in a higher PCB concentration for the 5 mg/kg Aroclor 1248 standard than for the 10 mg/kg Aroclor 1248 standard. These standards were reanalyzed, and an acceptable calibration was obtained. Analysis of samples then proceeded. All samples analyzed following the unacceptable calibrations were reanalyzed.

The EnviroGard PCB Field Lab Kit includes most of the equipment needed to analyze soil samples. The cost of this kit is $1,495. Most of this equipment can be reused for a number of projects, although a few of the items will wear out over time and eventually need to be replaced. The EnviroGard Field Soil Extraction Kit II includes the equipment required to extract 12 soil samples. The cost of this kit is $60. The kit must be purchased each time an analysis is conducted because the equipment is disposable. The EnviroGard PCB Test Kit includes the reagents needed to perform 16 analyses. The cost of this kit is $185. According to Millipore, these reagents have a 6-month shelf life. Reagents should not be used after the expiration date printed on the product label. Millipore also sells the methanol used to extract PCBs from soil. The cost for 100 mL of methanol is $7.90. Methanol also may also be purchased from a number of other chemical supply companies. A differential photometer can be used with the technology to obtain more accurate results. Millipore offers a differential photometer that uses either 115- or 230-volt sources of electricity. The cost of this photometer is $799. The photometer should be a one-time purchase.

Operator costs using the EnviroGard PCB Test will vary depending on the technical level of the operator. During this demonstration, about 200 samples were analyzed with this technology, including QA/QC samples. The waste generated from this analysis filled half a 55-gallon drum. The appropriate way to dispose of this waste would be through an approved PCB incinerator facility. The cost for disposal of one drum of this waste is estimated at $1,000.

Performance Factors

The following sections describe the EnviroGard PCB Test's performance factors, including detection limits and sensitivities, sample matrix effects, sample throughput, and drift. Specificity, due to its complexity, it is discussed separately in Section 6.

Detection Limits and Sensitivity

Millipore reports that the limit of quantitation for the EnviroGard PCB Test is 3.3 mg/kg for Aroclor 1248. This is the lowest concentration of Aroclor 1248 that can be accurately identified in a soil sample 99 percent of the time. The limit of quantitation differs for each Aroclor.

The limit of quantitation for the EnviroGard PCB Test depends on a number of factors. These factors include the number of active sites present on the wall of the anti-PCB antibody-coated test tube, the amount of PCBs present in the sample analyzed, the amount of enzyme conjugate added to the test tube, and the amount of coloring reagent added to the test tube. Millipore has designed the test so that the only factor that will change when analyzing a soil sample is the amount of PCBs present.

The technology is designed so that the number of active sites in the test tube will always be the same within a certain percentage of error. This will be especially true when the same test-tube lots are used. There may be some variance between different test-tube lots, so it is advisable to use the same lot throughout any particular project.

The kits are also designed so that the amount of enzyme conjugate and coloring reagent will be the same for each sample analyzed. Measured amounts of the enzyme conjugate solution and coloring reagents are added to each Aroclor standard, negative control sample, and sample analyzed.

Another factor that will affect the limit of quantitation is the particular Aroclor in the sample. The EnviroGard PCB Test cannot differentiate between Aroclors, but it will respond differently to each Aroclor. Millipore states that the response of the technology to Aroclors 1016, 1242, 1254, and 1260 should be within two times its response for Aroclor 1248. Aroclor specificity for this technology is discussed later in this section.

Sample Matrix Effects

Most of the soil samples collected during this demonstration consisted of clay, which caused some problems during extraction and analysis. The most common problem was that a colloidal suspension formed for some of the samples after the initial sample extraction. The colloidal suspension either (1) prevented good recovery of the organic extraction solvent from the filtering device or (2) clogged the filter, making it impossible to push the extraction solvent through the filter. Many of the samples resulted in organic extraction solvent recoveries of less than 2 mL. Millipore suggested that PRC extract another sample when this filter problem occurred. It was recommended that this extraction be performed using the standard 5 grams of soil sample and doubling the standard volume of extraction solvent from 5 mL to 10 mL. If this approach did not solve the problem, less than 5 grams of the soil sample was used in the extraction step. Nineteen samples required this approach: 033, 034, 035, 035D, 048, 056, 057, 065, 070, 071, 071D, 072, 077, 080, 083D, 086, 086D, 104, and 106. Results for these 19 samples were S-coded by the operator to indicate that due to sample matrix effects, the extraction and analysis process was modified as described above.

This extra dilution required that a correction factor be used when calculating the results for these samples. The correction factor caused results and detection limits to double those normally used. For semiquantitative results, when the response of the sample was less than the response of the 10 mg/kg Aroclor 1248 standard, the reported result of the sample was raised from "less than 10 mg/kg" to "less than 20 mg/kg."

Another sample matrix effect observed was differences between some samples and their corresponding field duplicate samples. This problem was noted during the predemonstration activities, and steps were taken to improve sample homogenization.

Sample Throughput

Millipore recommends that no more than 20 individual immunoassay analyses be performed at the same time. Every analysis performed requires that three concentrations of Aroclor standards and a negative control sample be prepared. This means a total of 16 actual sample analyses can be performed at a time. The operator found that a minimum of 30 minutes was required to extract 16 samples and that between 30 and 45 minutes were required to analyze the sample extracts. The operator also noted that the time required for the extraction and analysis steps did not include the time required for sample handling, data documentation, diluting samples when required, difficult extractions, or the preparation of QC samples. During this demonstration, the highest number of samples analyzed in a single 8-hour day was 52. The average number of samples analyzed during an 8-hour day was 25. This information indicates that the EnviroGard PCB Test can provide rapid analysis of PCBs in soil samples.

Drift

Drift normally is a measurement of an instrument's variability in quantitating a known amount of a standard. The EnviroGard PCB Test eliminates the variability associated with drift by requiring that a new calibration be performed with each set of samples analyzed.

During this demonstration, the optical density values obtained from the Aroclor standards placed into the differential photometer were evaluated. These values were found to drift during the 31 calibrations performed. The optical density values for the Aroclor 1242 standards decreased over time. The standards were stored in a refrigerator when not in use to reduce evaporation. However, the decrease may have been caused by the evaporation of solvent used to dilute the standards.

The optical density values for the Aroclor 1248 standards also decreased over time, although not as dramatically as values for the Aroclor 1242 standards. This is also believed to result from evaporation of solvent. Although Millipore supplied standards with

each EnviroGard PCB Test, new standards were not used for each new calibration. This was because the volume of standards provided with the technology was more than enough to perform a single or multiple calibration. This may have contributed to the decrease in optical density for the Aroclor 1248 standards.

Specificity

Immunoassay techniques typically use a "lock and key" type of chemical bonding to trap both the PCB and enzyme conjugate. This chemical must be specific to PCBs to prevent a large number of similar chemicals from binding to the active sites. The affinity of other compounds to these sites is called cross-reactivity. Millipore has designed the EnviroGard PCB Test to produce a specific binding site for PCBs that is not cross-reactive to most other compounds.

Millipore has performed studies of the cross-reactivity of other chemicals to the active sites of the anti-PCB antibody used. The following compounds were tested by Millipore and found to have a cross-reactivity of 0.5 percent or less when compared to Aroclor 1248 on a weight-to-weight basis: 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichloro-benzene, 1,2,4-trichloro-benzene, 2,4-dichlorophenol, 2,5-dichlorophenol, 2,4,5-trichlorophenol, 2,4,6-tri-chlorophenol, biphenyl, and pentachlorophenol. Millipore explains that for compounds with a cross-reactivity value of 0.5 percent, a concentration of 200 mg/kg would be required to produce the same response as a sample containing 1 mg/kg of Aroclor 1248.

Matrix effects also can influence an immunoassay. Millipore noted that soils containing greater than 5 percent oil may cause false negative results. However, careful field observations can reduce the impact of this effect. Soil samples containing greater than 5 percent oil should be visibly different from soil samples containing less than 1 percent oil, and the extract from soils containing 5 percent oil will appear cloudy during the first incubation step of the immunoassay process.

This technology is designed specifically to react to the chlorinated biphenyls present in Aroclor 1248. Many of these chlorinated biphenyls also are present in other Aroclors. Therefore, the technology will produce a positive response to other Aroclors, but in varying degrees. During this demonstration, an Aroclor specificity test was used to evaluate the response of the technology to each of the seven Aroclors. For this test, seven soil samples were each divided into four aliquots. The aliquots were then spiked with different Aroclors at approximately 10 mg/kg. This concentration was chosen because it is a common action level at contaminated sites. While the actual concentration of the spike was often slightly less than 10 mg/kg, the spike was always close enough that the technology result should have indicated a positive response. The results of the Aroclor specificity test for the technology in its semiquantitative mode are tabulated in Table 6-1.

The Aroclor-spiked samples were identified with 9-character digit, alphanumeric identification codes. The first three characters of this code referred to the soil sample number used for spiking. The next four characters, "ARSP," are an abbreviation of the words "Aroclor spike." The next character was a letter, A through G, identifying which Aroclor was used to spike the sample. The last character was a number, 1 through 4, referring to the aliquot of the sample. To ensure that results of the assessment were unbiased by operator effects, the operator did not know which Aroclor was used for spiking or the Aroclor concentration in the samples.

Sample 077 was spiked with approximately 10 mg/kg of Aroclor 1016. The four spiked samples indicated that the PCB concentrations of the samples were less than 10 mg/kg when compared to the Aroclor 1248 standard. It appears that the technology is less responsive to Aroclor 1016 than to Aroclor 1248.

Sample 058 was then spiked with 10 mg/kg of Aroclor 1248. Three of the four spiked sample results indicated PCB concentrations of greater than 10 mg/kg when compared to the Aroclor 1248 standard. Two of these aliquots had been spiked with exactly 10 mg/kg of Aroclor 1248; the third, with 9.90 mg/kg of Aroclor 1248; and the fourth, with 9.73 mg/kg of Aroclor 1248. The response of the technology to the fourth aliquot containing 9.73 mg/kg of Aroclor 1248 was less than its response to the 10 mg/kg standard. This indicates that the detection limit of the technology for Aroclor 1248 is 10 mg/kg, as expected.

Sample 021 was spiked with about 10 mg/kg of Aroclor 1232. The four spiked sample results all indicated that the PCB concentrations of the samples were less than 10 mg/kg when compared to the Aroclor 1248 standard. This indicates that the technology cannot identify a soil sample containing 10 mg/kg of Aroclor 1232 using the Aroclor 1248 standard.

Sample 012 was spiked with 10 mg/kg of Aroclor 1260. The four spiked sample results indicated that the PCB concentrations in the samples were greater than 10 mg/kg when compared to the Aroclor 1248 standard. This indicates that a soil sample containing 10 mg/kg of Aroclor 1260 can be identified using the Aroclor 1248 standards.

TABLE 6-1. SEMIQUANTITATIVE RESULTS FOR THE AROCLOR SPECIFICITY TEST.

Sample No.	Confirmatory Laboratory Result (mg/kg)	Soil Sample Result (mg/kg)	Aroclor Spike	Spike Amount (mg/kg)	Spiked Sample Result (mg/kg)
003ARSPA1	0.1	< 10	AR1221	9.94	< 10
003ARSPA2	0.1	< 10	AR1221	9.84	< 10
003ARSPA3	0.1	< 10	AR1221	9.92	< 10
003ARSPA4	0.1	< 10	AR1221	9.77	< 10
012ARSPB1	ND	< 10	AR1260	9.78	> 10
012ARSPB2	ND	< 10	AR1260	9.80	> 10
012ARSPB3	ND	< 10	AR1260	9.65	> 10
012ARSPB4	ND	< 10	AR1260	9.86	> 10
021ARSPC1	0.06	< 10	AR1232	9.98	< 10
021ARSPC2	0.06	< 10	AR1232	9.84	< 10
021ARSPC3	0.06	< 10	AR1232	9.88	< 10
021ARSPC4	0.06	< 10	AR1232	9.82	< 10
034ARSPD1	34.0	> 10	AR1254	9.86	> 10
034ARSPD2	34.0	> 10	AR1254	9.77	> 10
034ARSPD3	34.0	> 10	AR1254	9.77	> 10
034ARSPD4	34.0	> 10	AR1254	9.67	> 10
040ARSPE1	4.3	> 10	AR1242	9.88	> 10
040ARSPE2	4.3	> 10	AR1242	9.69	> 10
040ARSPE3	4.3	> 10	AR1242	9.86	> 10
040ARSPE4	4.3	> 10	AR1242	9.75	> 10
058ARSPF1	0.7	< 10	AR1248	9.73	< 10
058ARSPF2	0.7	< 10	AR1248	10.00	> 10
058ARSPF3	0.7	< 10	AR1248	10.00	> 10
058ARSPF4	0.7	< 10	AR1248	9.90	> 10
077ARSPG1	ND	< 10	AR1016	9.65	< 10
077ARSPG2	ND	< 10	AR1016	9.96	< 10
077ARSPG3	ND	< 10	AR1016	9.75	< 10
077ARSPG4	ND	< 10	AR1016	9.90	< 10

Notes:

ND PCBs not detected above the detection limit of 1.0 mg/kg.
mg/kg Milligrams per kilogram.

Sample 003 was spiked with 10 mg/kg of Aroclor 1221. Results for the four spiked samples indicated that the PCB concentrations in the samples were less than 10 mg/kg when compared to the Aroclor 1248 standard. This indicates that a soil sample containing 10 mg/kg of Aroclor 1221 cannot be identified using the Aroclor 1248 standard.

Sample 034 was then spiked with 10 mg/kg of Aroclor 1254, but because the confirmatory laboratory indicated that Sample 034 had greater than 10 mg/kg of PCBs prior to these samples being spiked, no conclusions can be drawn.

Sample 040 was spiked with 10 mg/kg of Aroclor 1242. The results from the four spiked samples indicated that the PCB concentrations of the samples were greater than 10 mg/kg when compared to the Aroclor 1248 standard.

Intramethod Assessment

Six reagent blanks were prepared and analyzed to evaluate laboratory-induced contamination. These samples were taken through all extraction, filtration, and immunoassay steps of the analysis. No PCBs were detected in any of the reagent blanks analyzed.

For this demonstration, completeness refers to the proportion of valid, acceptable data generated using the EnviroGard PCB Test. Semiquantitative results were obtained for all of the samples; therefore, completeness for the samples analyzed by the EnviroGard PCB Test during the demonstration was 100 percent.

Intramethod accuracy was assessed for the EnviroGard PCB Test by using PE samples and matrix spike and matrix spike duplicate samples. Accuracy also was determined by comparing the results of the technology to those of the confirmatory laboratory. A discussion of this intermethod accuracy is presented later in Section 6.

Each PE sample had a different concentration of PCBs: one was low and the other was high. These samples were extracted and analyzed in exactly the same manner as all other soil samples. The operator knew that the samples were PE samples but did not know their true values, nor their ranges. The true result for sample 047-4024-114 (the high-level sample) was 110 mg/kg of Aroclor 1242, with an acceptance range of 41 to 150 mg/kg. The semiquantitative result for this sample showed that the PCB concentration was greater than the 10 mg/kg Aroclor 1248 standard. The semiquantitative result, therefore, was acceptable. The true result for sample 047-4024-113 (the low-level sample) was 32.7 mg/kg of Aroclor 1242. The semiquantitative result for this sample showed that the PCB concentration was greater than the 10 mg/kg Aroclor 1248 standard. The semiquantitative result, therefore, also was acceptable.

Matrix spike samples, prepared by adding a known quantity of PCBs to a sample, were used to evaluate the extraction and analysis efficiency of the technology and to determine accuracy. Enough Aroclor 1242 was added to a 5-gram soil sample to produce a matrix spike concentration of 25 mg/kg. The spiked sample was duplicated to produce a matrix spike duplicate sample.

Six matrix spike samples and six matrix spike duplicate samples were extracted and analyzed using the EnviroGard PCB Test. Semiquantitative results were compared to the 10 mg/kg Aroclor 1248 standard. The results of the soil samples before being spiked showed that all six samples contained less than 10 mg/kg of PCBs as determined by the Aroclor 1248 standards. Of the 12 spiked sample results, 11 were found to contain greater than 10 mg/kg of PCBs as determined by the Aroclor 1248 standards. The EnviroGard PCB Test was able to determine that the matrix spike samples contained more than 10 mg/kg of PCBs, as determined from the Aroclor 1248 standards, in 92 percent of the samples.

For this demonstration, three types of precision data were generated: laboratory duplicate samples, field duplicate samples, and matrix spike duplicate samples. Semiquantitative results for laboratory and field duplicate samples are provided in Table 6-2.

Laboratory duplicate samples are single samples on which two analyses are performed. Laboratory duplicate samples were analyzed after each set of 20 samples submitted for analysis. Five pairs of laboratory duplicate samples and their respective soil samples were analyzed with the EnviroGard PCB Test. Field duplicate samples are two samples collected together but delivered to the laboratory under separate sample numbers. PRC collected 32 field duplicate samples during this demonstration. Each sample and its duplicate was analyzed by the technology and by the confirmatory laboratory.

Typically, field and laboratory duplicate samples are used to determine problems with collection and analysis, not problems with the technology itself. The laboratory duplicates are compared to a window of acceptable values and if one fell outside that window, corrective action is taken by the laboratory. Field duplicates are used to ensure that contamination of samples does not occur during sample collection and to set boundaries of variance due to the lack of homogenization inherent in soil contamination.

PRC was tasked, though, not with determining the precision of those collecting the samples or of the laboratory, but with determining the precision of the technology itself. To do this, PRC attempted to control any factor other than those inherent in the technology that might contribute to a difference between a sample

TABLE 6-2. SEMIQUANTITATIVE RESULTS FOR LABORATORY AND FIELD DUPLICATE SAMPLES.

Sample No.	Sample Result (mg/kg)	Duplicate Result (mg/kg)	Sample No.	Sample Result (mg/kg)	Duplicate Result (mg/kg)
047-4024-001LD	> 10	< 10	047-4024-082FD	< 10	< 10
047-4024-015FD	> 10	> 10	047-4024-083FD	< 10	< 10
047-4024-022FD	< 10	< 10	047-4024-084FD	> 10	> 10
047-4024-024FD	< 10	< 10	047-4024-085FD	> 10	> 10
047-4024-028FD	< 10	< 10	047-4024-086FD	< 10	< 10
047-4024-035FD	< 10	< 10	047-4024-087FD	< 10	< 10
047-4024-037FD	< 10	< 10	047-4024-088FD	> 10	> 10
047-4024-040LD	> 10	> 10	047-4024-090FD	< 10	< 10
047-4024-042FD	> 10	> 10	047-4024-091FD	> 10	> 10
047-4024-043FD	> 10	> 10	047-4024-092FD	< 10	< 10
047-4024-046FD	< 10	< 10	047-4024-095FD	> 10	> 10
047-4024-047FD	< 10	< 10	047-4024-097FD	< 10	< 10
047-4024-050FD	> 10	> 10	047-4024-098FD	> 10	> 10
047-4024-060FD	> 10	< 10	047-4024-098LD	< 10	< 10
047-4024-062LD	> 10	> 10	047-4024-100FD	> 10	> 10
047-4024-063FD	< 10	< 10	047-4024-102FD	> 10	> 10
047-4024-069FD	< 10	< 10	047-4024-102LD	> 10	> 10
047-4024-071FD	< 10	< 10	047-4024-109FD	< 10	< 10
047-4024-081FD	< 10	< 10			

Notes:

mg/kg Milligrams per kilogram.
LD Laboratory duplicate.
FD Field duplicate.

and its duplicate. To control the problems usually detected by laboratory duplicates, PRC used only one operator for each technology. It was assumed that any variance in that operator's laboratory techniques would be the same for each sample, and therefore, statistically insignificant. For the field duplicates, PRC put each sample through a homogenization process designed to ensure that there was little difference between the contamination in a sample and its duplicate. Confirmatory laboratory data on the field duplicates and their respective samples indicate that, overall, this technique worked (see Section 4). Only in a few samples did the homogenization appear not to have been complete.

PRC used both the laboratory and field duplicates to determine the technology's precision. Thirty-seven duplicate pairs were used in the semiquantitative evaluation. Of these 37 duplicate pairs, the EnviroGard PCB Test produced the same results 35 times. One laboratory duplicate, Sample 001, and one field duplicate, Sample 060D, did not agree with their respective corresponding sample results. Based on this data, the precision of the EnviroGard PCB Test was

found to be 95 percent, which meets the criteria for precision.

Matrix spike duplicate samples were used to further evaluate the precision of this technology. The matrix spike duplicate sample results were compared to the matrix spike sample results. The semiquantitative results for the matrix spike samples were compared to the Aroclor 1248 standards supplied by Millipore. Aroclor 1242 was added to each of the matrix spike samples at a concentration of 25 mg/kg. Five of the six matrix spike duplicate sample results matched those of the matrix spike samples. The matrix spike duplicate result for Sample 024 did not match the original matrix spike sample result. From this data, it was determined that the precision of the semiquantitative data for the EnviroGard PCB Test was 83 percent.

Comparison of Results to Confirmatory Laboratory Results

The following sections compare the accuracy and precision of the semiquantitative data from the EnviroGard PCB Test to that of the confirmatory laboratory. The results from the confirmatory laboratory are considered accurate, and its precision is considered acceptable. The results are summarized in Table 6-3 and on Figure 6-1, following the table.

Accuracy

To measure the accuracy of the EnviroGard PCB Test, PRC compared the data from the technology to the data from the confirmatory laboratory by using a 2 by 2 contingency table and a Fisher's Test, as approximated by a corrected Chi-square statistic.

To obtain semiquantitative results with the technology, its results must be evaluated relative to a set of points or ranges. PRC evaluated the technology's semiquantitative accuracy in two ways: using three concentrations of Aroclor 1242 standards and using one concentration of Aroclor 1248 standard.

Results were statistically evaluated with two 2 by 2 contingency tables, one for each range, to compare the number of times the technology's results indicated results were within each range to the number of times the confirmatory laboratory indicated results were within that range. A Fisher's Test, at a 95 percent confidence level, was then used to determine whether a relationship existed between the two sets of results.

Aroclor 1242 Standards (5, 10, and 50 mg/kg)

PRC used three Aroclor 1242 standards to determine the semiquantitative results of 52 samples. These results placed the concentration of each sample within one of four ranges: (1) less than 5 mg/kg; (2) between 5 and 10 mg/kg; (3) between 10 and 50 mg/kg; and (4) greater than 50 mg/kg. The confirmatory laboratory's results also were placed within one of these ranges.

For the first range, less than 5 mg/kg, the technology indicated that 25 sample results were within the range and 27 results were above this range. The confirmatory laboratory indicated that 39 were within the range and 13 were above it. The Fisher's Test showed that results from the two sets of data were statistically different. Since the confirmatory laboratory's data is assumed to be accurate, the lack of correlation indicates that within the first range the semiquantitative results were not accurate when these standards were used.

For the second range, between 5 and 10 mg/kg, the technology had no sample results within the range and 52 outside the range. The confirmatory laboratory had five sample results within the range and 47 outside the range. The Fisher's Test indicated that these two sets of data were statistically different and that within this range the semiquantitative results were not accurate.

The technology had 10 results within the third range, which was between 10 and 50 mg/kg, and 42 outside it. The confirmatory laboratory had five results within the range and 47 outside. The Fisher's Test indicated that the data was not statistically different. Therefore, within this range and using these standards, the semiquantitative results were accurate.

In the final range, greater than 50 mg/kg, the technology had 17 sample results above that level and 35 below it. The confirmatory laboratory had three results above that level and 49 below it. The Fisher's Test indicated that the data was statistically different. Therefore, within the fourth range, the results from the technology were not accurate.

A comparison of the technology's sample results to the confirmatory laboratory's results shows that 28 of 52 times the technology was correct, or 53.9 percent of the time. The other 24 times the technology gave false positive results. It never gave a false negative result when the three Aroclor 1242 standards were used.

TABLE 6-3. COMPARISON OF SEMIQUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy	Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy
001	> 10	0.593	FP	029	> 10	0.229J	Correct
002	> 10	1.50	FP	030	> 10	1.15	Correct
003	> 10	0.114	Correct	031	> 10	0.263	Correct
004	> 10	6.71J	FP	032	> 10	47.6	Correct
005	> 10	1.37	FP	033	> 1+0	6.00J	FP
006	> 10	0.679	FP	034	> 10	34.0	Correct
007	> 10	0.552	FP	035	> 10	ND	Correct
008	> 10	2.00	FP	035D	> 10	ND	Correct
009	> 10	1.30J	FP	036	> 10	816	Correct
010	> 10	0.172J	FP	037	> 10	0.055J	Correct
011	> 10	1.15J	FP	037D	> 10	0.040J	Correct
012	> 10	ND	Correct	038	> 10	1,030J	Correct
013	> 10	1.13	Correct	039	> 10	0.676	Correct
014	> 10	0.18	Correct	040	> 10	4.25	FP
015	> 10	9.13	FP	041	> 10	ND	Correct
015D	> 10	9.84	FP	042	> 10	0.517	FP
016	> 10	2,110	Correct	042D	> 10	0.462J	FP
017	> 10	2.55	FP	043	> 10	1.69J	FP
018	> 10	45.4	Correct	043D	> 10	1.74	FP
019	> 10	6.70	FP	044	> 10	0.592J	Correct
020	> 10	0.068J	Correct	045	> 10	ND	Correct
021	> 10	0.063	Correct	046	> 10	ND	Correct
022	> 10	0.535	Correct	046D	> 10	ND	Correct
022D	> 10	0.718	Correct	047	> 10	0.094J	Correct
023	> 10	20.8	Correct	047D	> 10	0.098J	Correct
024	> 10	0.055	Correct	048	> 10	ND	Correct
024D	> 10	0.049	Correct	049	> 10	ND	Correct
025	> 10	11.7	FP	050	> 10	3.60	FP
026	> 10	1.96	Correct	050D	> 10	4.41	FP
027	> 10	0.057	Correct	051	> 10	ND	Correct
028	> 10	0.216	Correct	052	> 10	4.21	FP
028D	> 10	0.224J	Correct	053	> 10	0.958	Correct

TABLE 6-3 (Continued). COMPARISON OF SEMIQUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy	Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy
054	> 10	0.516J	Correct	081	> 10	0.687	Correct
055	> 10	2.40	Correct	081D	> 10	0.450	Correct
056	> 10	0.505	Correct	082	> 10	ND	Correct
057	> 10	ND	Correct	082D	> 10	0.244	Correct
058	> 10	0.681	Correct	083	> 10	0.484	Correct
059	> 10	7.86	FP	083D	> 10	0.413	Correct
060	> 10	0.624J	FP	084	> 10	1.16	FP
060D	> 10	0.577	Correct	084D	> 10	1.08	FP
061	> 10	580	Correct	085	> 10	428	Correct
062	> 10	2.35	FP	085D	> 10	465	Correct
063	> 10	0.092J	Correct	086	> 10	1.42	Correct
063D	> 10	0.154J	Correct	086D	> 10	1.25	Correct
064	> 10	19.0	Correct	087	> 10	0.076	Correct
065	> 10	3.08	FP	087D	> 10	ND	Correct
066	> 10	1.98	Correct	088	> 10	2.70	FP
067	> 10	0.081	Correct	088D	> 10	1.77	FP
068	> 10	0.504J	Correct	089	> 10	45.0	Correct
069	> 10	ND	Correct	090	> 10	1.01	Correct
069D	> 10	ND	Correct	090D	> 10	1.40	Correct
070	> 10	ND	Correct	091	> 10	1,630	Correct
071	> 10	0.052J	Correct	091D	> 10	1,704	Correct
071D	> 10	ND	Correct	092	> 10	1.21	Correct
072	> 10	0.035J	Correct	092D	> 10	ND	Correct
073	> 10	15.8	Correct	093	> 10	0.295	Correct
074	> 10	13.3	Correct	094	> 10	0.362J	Correct
075	> 10	23.0	Correct	095	> 10	17.5	Correct
076	> 10	46.7	Correct	095D	> 10	31.2	Correct
077	> 10	ND	Correct	096	> 10	0.059J	Correct
078	> 10	2.27	FP	097	> 10	1.23	Correct
079	> 10	42.8	Correct	097D	> 10	0.285	Correct
080	> 10	3.77	Correct	098	> 10	1.17	FP

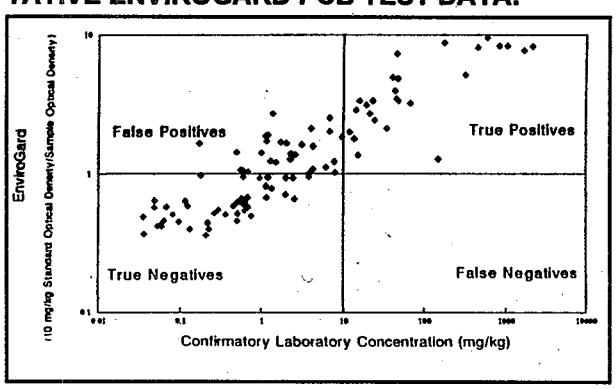
TABLE 6-3 (Continued). COMPARISON OF SEMIQUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy	Sample No.	EnviroGard PCB Test (10 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Technology Accuracy
098D	> 10	0.825	FP	106	> 10	2.50	Correct
099	> 10	ND	Correct	107	> 10	14.1J	Correct
100	> 10	177	Correct	108	> 10	3.84J	FP
100D	> 10	167	Correct	109	> 10	ND	Correct
101	> 10	1.21	FP	109D	> 10	ND	Correct
102	> 10	293	Correct	110	> 10	ND	Correct
102D	> 10	1.77	FP	111	> 10	ND	Correct
103	> 10	40.3	Correct	112	> 10	315	Correct
104	> 10	7.66	FP	113	> 10	14.9	Correct
105	> 10	0.210	Correct	114	> 10	66.3	Correct

Notes:

a Detection limit.
FP False positive.
J Reported amount is below detection limit or not valid by approved QC procedures.
ND PCBs not detected above the detection limit.
NA The technology, the confirmatory laboratory, or both did not detect PCBs.

FIGURE 6-1. ASSESSMENT OF SEMIQUANTITATIVE ENVIROGARD PCB TEST DATA.



Aroclor 1248 (10 mg/kg standard)

A 10 mg/kg Aroclor 1248 standard was used to assess the results of 94 samples. Each sample result was reported as above 10 mg/kg of PCBs or below 10 mg/kg of PCBs. The confirmatory laboratory results also were placed in one of these two ranges.

The technology obtained 53 sample results below the 10 mg/kg level and 41 above it. The confirmatory laboratory obtained 72 results below that level and 22 above it. The results of the analysis indicated that the two data sets were statistically different. Since the confirmatory laboratory's data is considered accurate, the results from the technology, overall, are considered not accurate when this Aroclor 1248 standard was used.

Of the 94 results, the technology had 75 correct results and 19 false positive results. Again, no false negative results were found.

Summary

Overall, the EnviroGard PCB Test is a conservative kit when used semiquantitatively. It is not accurate when assessed in this mode, but in all cases where its results did not match those from the confirmatory laboratory, it produced false positive results.

False negatives are the principal concern with semiquantitative technologies. False negative results could lead to the belief that soil is not contaminated

above an action level when in fact it is. For this reason, the EnviroGard PCB Test is designed to produce numerous false positive results, but few false negative results.

It should be noted that depending on the situation, false positive results also can be of concern because they indicate soil is more contaminated than it actually is. This results in more soil being excavated and disposed of than necessary. This can be costly and does not comply with the waste minimization policies of many regulatory agencies.

Precision

When used to produce semiquantitative results, the precision of the EnviroGard PCB Test cannot be compared to that of the confirmatory laboratory.

Quantitative Evaluation

After consulting with both Millipore and EMSL-LV, PRC determined that with a minimal amount of additional effort the EnviroGard PCB Test's ability to produce quantitative results could be evaluated. This evaluation, therefore, was included in the demonstration. The following sections detail its results. Table 6-4 and Figure 6-2 summarize the quantitative results.

Theory of Operation and Background Information

The EnviroGard PCB Test used during the quantitative analysis was the same as that used during the semiquantitative analysis with one exception. Instead of using the three Aroclor 1248 standards supplied by Millipore to analyze samples, PRC used the Aroclor 1242 standards it had prepared. Originally, the three concentrations of Aroclor 1242 used were 1, 5, and 25 mg/kg. The 1 mg/kg concentration was used to define the detection limit of the technology. However, a large number of soil samples analyzed with these concentrations required dilution because the response of the samples was not within the response range of the standards. To decrease the number of samples requiring dilution, the three concentrations were changed to 5, 10, and 50 mg/kg.

Samples were prepared for analysis in the same manner used for the semiquantitative evaluation. The test tube was then placed into the differential photometer, and the optical density was recorded.

Quantitative results were obtained for the samples through the use of standard curves. These curves were produced for each set of Aroclor standards used. The curves were manually plotted on graph paper. The x-axis of the graph represented the PCB concentration in the sample; the y-axis represented the optical density, or absorbance, in the sample. The Aroclor standards and the negative control sample were plotted on the graph. A curve was generated by connecting these points. The optical density values obtained for each soil sample were then traced from the y-axis to the intersection of the standard curve line, then to the x-axis. The point where this line intercepted the x-axis indicated the concentration of PCBs in the sample. Before the results were reported, calculations were performed as needed to correct the concentration for any dilutions performed.

The standard curve produced for each concentration of the Aroclor 1242 standard resulted in a relatively linear curve. The curve had a negative slope. The curve resulted in average slope of -0.29. The slope of the line did vary somewhat from analysis to analysis. When the lowest standard was 1 mg/kg, the region of the standard curve connecting the lowest concentration standard and the negative control sample, which was always placed at the y-intercept, changed slope dramatically from sample to sample. When 5 mg/kg was used as the lowest standard, the slope of the line from the 5 mg/kg standard to the y-intercept was not as dramatic. This suggests that a 5 mg/kg detection limit for Aroclor 1242 may be more reliable than the 1 mg/kg detection limit used for this demonstration

Operational Characteristics

The portability, logistical requirements, ease of operation, and health and safety requirements for the EnviroGard PCB Test operated in the quantitative mode are the same as those detailed earlier in this section. The same operator operated the technology in both modes, and the costs of the analyses were the same. In addition to the calibration problems detailed earlier, the first calibration using the Aroclor 1242 standards was unacceptable. This was attributed to operator error and the small amount of standard required for analysis. The problem was corrected, and all other calibrations with these Aroclor standards were acceptable.

Performance Factors

Quantitative analysis of soil samples during this demonstration was performed using 1, 5, 25, and 50 mg/kg Aroclor 1242 standards. The 50 mg/kg Aroclor standard replaced the 25 mg/kg Aroclor standard to reduce the number of dilutions needed to obtain quantitative results. Throughout the demonstration, the 1 mg/kg Aroclor 1242 standard consistently caused a greater response than the negative control sample, which was expected. Not all samples analyzed

TABLE 6-4. COMPARISON OF QUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference	Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference
001	10	0.593	9.4	177.7	033	64S	6.00J	58	165.7
002	22	1.50	20.5	174.5	034	40S	34.0	6.0	16.2
003	2.5	0.114	2.4	182.6	035	ND,S	ND	NA	NA
004	61	6.71J	54.3	160.4	035D	ND,S	ND	NA	NA
005	30	1.37	28.6	182.5	036	2,300	816	1,484	95.3
006	13	0.679	12.3	180.1	037	3	0.055J	2.9	192.8
007	19	0.552	18.5	188.7	037D	4	0.040J	3.9	196.0
008	16	2.00	14.0	155.6	038	1,000	1,030J	-30	3.0
009	17	1.30J	15.7	171.6	039	5	0.676	4.3	152.4
010	40	0.172J	39.8	198.3	040	44	4.25	39.8	164.8
011	24	1.15J	22.9	181.7	041	ND	ND	NA	NA
012	17	ND	NA	NA	042	25	0.517	24.5	191.9
013	12	1.13	10.9	165.6	042D	9	0.462J	8.5	180.5
014	20	0.18	19.8	196.4	043	30	1.69J	28.3	178.7
015	50	9.13	40.9	138.2	043D	20	1.74	18.3	168.0
015D	50	9.84	40.2	134.2	044	2	0.592J	1.4	108.6
016	300	2,110	-1,810	150.2	045	ND	ND	NA	NA
017	12	2.55	9.5	129.9	046	ND	ND	NA	NA
018	370	45.4	324.6	156.3	046D	ND	ND	NA	NA
019	29	6.70	22.3	124.9	047	ND	0.094J	NA	NA
020	2.5	0.068J	2.4	189.4	047D	ND	0.098J	NA	NA
021	1	0.063	0.9	176.3	048	ND,S	ND	NA	NA
022	5	0.535	4.5	161.3	049	ND	ND	NA	NA
022D	4	0.718	3.3	139.1	050	20	3.60	16.4	139.0
023	50	20.8	29.2	82.5	050D	30	4.41	25.6	148.7
024	ND	0.055	NA	NA	051	2	ND	NA	NA
024D	ND	0.049	NA	NA	052	25	4.21	20.8	142.3
025	50	11.7	38.3	124.1	053	15	0.958	14.0	176.0
026	14	1.96	12.0	150.9	054	3	0.516J	2.5	141.3
027	ND	0.057	NA	NA	055	15	2.40	12.6	144.9
028	1	0.216	0.8	128.9	056	4S	0.505	3.5	155.2
028D	ND	0.224J	NA	NA	057	ND,S	ND	NA	NA
029	ND	0.229J	NA	NA	058	4	0.681	3.3	141.8
030	6	1.15	4.9	135.7	059	18	7.86	10.1	78.4
031	6	0.263	5.7	183.2	060	17	0.624J	16.4	185.8
032	50	47.6	2.4	4.9	060D	12	0.577	11.4	181.6

TABLE 6-4 (Continued). COMPARISON OF QUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference	Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference
061	470	580	-110	21.0	085D	400	465	-65	15.0
062	25	2.35	22.6	165.7	086	14S	1.42	12.6	163.2
063	3	0.092J	2.9	188.1	086D	15S	1.25	13.7	169.2
063D	3	0.154J	2.8	180.5	087	6	0.076	5.9	195.0
064	60	19.0	41.0	103.8	087D	3	ND	NA	NA
065	78S	3.08	74.9	184.8	088	20	2.70	17.3	152.4
066	14	1.98	12.0	150.4	088D	23	1.77	21.2	171.4
067	2	0.081	1.9	184.4	089	20	45.0	-25	76.9
068	4	0.504J	3.5	155.2	090	10	1.01	9.0	163.3
069	ND	ND	NA	NA	090D	8	1.40	6.6	140.4
069D	1	ND	NA	NA	091	1,300	1,630	-330	22.7
070	ND,S	ND	NA	NA	091D	1,600	1,704	-104	6.3
071	ND,S	0.052J	NA	NA	092	2	1.21	0.8	49.2
071D	ND,S	ND	NA	NA	092D	2	ND	NA	NA
072	2S	0.035J	1.9	193.1	093	2	0.295	1.7	148.6
073	40	15.8	24.2	86.7	094	2	0.362N	1.6	138.7
074	41	13.3	27.7	102.0	095	10	17.5	-7.5	54.5
075	35	23.0	12.0	41.4	095D	10	31.2	-21.2	102.9
076	40	46.7	-6.7	15.5	096	ND	0.059J	NA	NA
077	ND,S	ND	NA	NA	097	ND	1.23	NA	NA
078	23	2.27	20.7	164.1	097D	3	0.285	2.7	165.3
079	70	42.8	27.2	48.2	098	46	1.17	44.8	190.1
080	19S	3.77	15.2	133.8	098D	30	0.825	29.2	189.3
081	4	0.687	3.3	141.4	099	3	ND	NA	NA
081D	3	0.450	2.5	147.8	100	400	177	233	77.3
082	ND	ND	NA	NA	100D	470	167	303	95.1
082D	ND	0.244	NA	NA	101	--	1.21	NA	NA
083	1	0.484	0.5	69.5	102	30	293	-263	162.8
083D	5S	0.413	4.6	169.5	102D	30	1.77	28.2	177.7
084	50	1.16	48.8	190.9	103	190	40.3	149.7	130.0
084D	49	1.08	48.9	191.4	104	54S	7.66	56.3	150.3
085	370	428	-58	14.5	105	ND	0.210	NA	NA

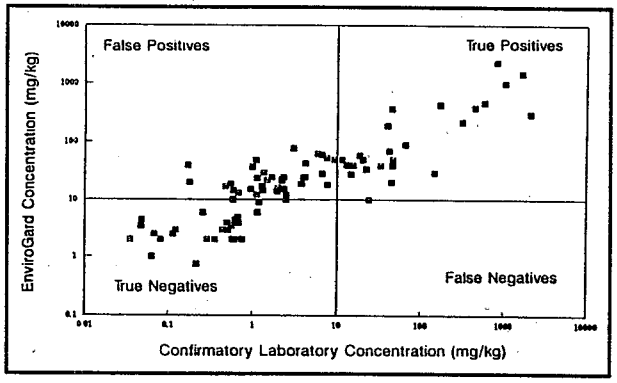
TABLE 6-4 (Continued). COMPARISON OF QUANTITATIVE DATA FOR ENVIROGARD PCB TEST AND CONFIRMATORY LABORATORY.

Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference	Sample No.	EnviroGard PCB Test (1.0 mg/kg[a])	Confirmatory Laboratory (0.033 mg/kg[a])	Difference	Relative Percent Difference
106	10S	2.50	7.5	120.0	110	2	ND	NA	NA
107	40	14.1J	25.9	95.7	111	ND	ND	NA	NA
108	19	3.84J	15.1	132.8	112	220	315	-95	35.5
109	2	ND	NA	NA	113	28	14.9	13.1	61.0
109D	ND	ND	NA	NA	114	90	66.3	23.7	30.3

Notes:

[a] Detection limit.
J Reported amount is below detection limit or not valid by approved QC procedures.
S Sample matrix effects raised detection limits.
ND PCBs not detected above detection limit.
NA The technology, the confirmatory laboratory, or both did not detect PCBs.
-- No sample result obtained.

FIGURE 6-2. ASSESSMENT OF QUANTITATIVE ENVIROGARD PCB TEST DATA



were calibrated using the 1 mg/kg Aroclor standard. However, because the 1 mg/kg Aroclor 1242 standard appeared correct each time it was used, all quantitative results reported for the EnviroGard PCB Test were based on a detection limit of 1 mg/kg.

The sample matrix effects noted in the semi-quantitative evaluation also were noticed during the quantitative evaluation. High concentrations of PCBs in many of the soil samples presented an additional sample matrix problem in the quantitative evaluation. While these high concentrations had no effect on the semiquantitative data, the quantitative data was affected. The samples that exhibited a higher response than the highest concentration of the Aroclor 1242 standard had to be diluted to obtain results. The dilution was performed by serially diluting the sample extract at a ratio of 1 to 10. If, after the first dilution, the response still exceeded the response of the highest Aroclor 1242 standard, the sample was diluted again at the same ratio. This procedure was continued until the response of the sample fell within the range of the Aroclor 1242 standards. Of the 146 total samples analyzed during this demonstration, 42 required dilution. Thirty-seven of these samples fell within the range of the Aroclor 1242 standards after only one 1 to 10 ratio dilution. The other five samples required a second 1 to 10 ratio dilution to provide a response within the range of the Aroclor 1242 standards. When dilutions were required, the detection limit was raised appropriately.

Specificity

The specificity of the EnviroGard PCB Test in the quantitative mode was assessed using the approach discussed earlier in this section. Table 6-5 summarizes the quantitative results of the Aroclor specificity test.

Sample 077 was spiked with about 10 mg/kg of Aroclor 1016. Results ranged from 8 to 12 mg/kg. The average percent recovery of the four spiked samples was 92 percent; the standard deviation of the percent recovery was 22 percent. The technology's response to Aroclor 1016 was close to its response to the Aroclor 1242 standards.

Sample 058 was spiked with 10 mg/kg of Aroclor 1248. Results ranged from 8 to 19 mg/kg. The average percent recovery of the samples was 118 percent; the standard deviation of the percent recovery was 48 per-

TABLE 6-5. QUANTITATIVE RESULTS FOR THE AROCLOR SPECIFICITY TEST.

Sample No.	Confirmatory Laboratory Result (mg/kg)	Soil Sample Result (mg/kg)	Aroclor Spike	Spike Amount (mg/kg)	Spiked Sample Result (mg/kg)	Percent Recovery (%)
003ARSPA1	0.1	2.5	AR1221	9.94	5	25
003ARSPA2	0.1	2.5	AR1221	9.84	3	5
003ARSPA3	0.1	2.5	AR1221	9.92	2	0
003ARSPA4	0.1	2.5	AR1221	9.77	4	15
012ARSPB1	ND	17	AR1260	9.78	45	286
012ARSPB2	ND	17	AR1260	9.80	36	194
012ARSPB3	ND	17	AR1260	9.65	36	197
012ARSPB4	ND	17	AR1260	9.86	34	172
021ARSPC1	0.06	1	AR1232	9.98	10	98
021ARSPC2	0.06	1	AR1232	9.84	12	111
021ARSPC3	0.06	1	AR1232	9.88	6	51
021ARSPC4	0.06	1	AR1232	9.82	8	71
034ARSPD1	34.0	40	AR1254	9.86	120	811
034ARSPD2	34.0	40	AR1254	9.77	120	819
034ARSPD3	34.0	40	AR1254	9.77	120	819
034ARSPD4	4.3	40	AR1254	9.67	120	827
040ARSPE1	4.3	44	AR1242	9.88	44	0
040ARSPE2	4.3	44	AR1242	9.69	28	0
040ARSPE3	4.3	44	AR1242	9.86	42	0
040ARSPE4	4.3	44	AR1242	9.75	33	0
058ARSPF1	0.7	4	AR1248	9.73	8	50
058ARSPF2	0.7	4	AR1248	10.00	16	120
058ARSPF3	0.7	4	AR1248	10.00	19	150
058ARSPF4	0.7	4	AR1248	9.90	19	152
077ARSPG1	ND	ND J	AR1016	9.65	12	124
077ARSPG2	ND	ND J	AR1016	9.96	8	80
077ARSPG3	ND	ND J	AR1016	9.75	8	82
077ARSPG4	ND	ND J	AR1016	9.90	8	81

Notes:

mg/kg	Milligrams per kilogram.
ND	Not detected above the 1 mg/kg detection limit.
J	Detection limit raised to 2 mg/kg due to dilution of sample.

cent. The response of the technology to the samples spiked with the Aroclor 1248 standard was greater than its response to the Aroclor 1242 standards. Three of the four sample results were higher than that of the 10 mg/kg Aroclor 1242 standard. This suggests that a 10 mg/kg sample containing Aroclor 1248 should produce a response greater than the technology's response to a 10 mg/kg Aroclor 1242 standard.

Sample 021 was then spiked with 10 mg/kg of Aroclor 1232. Results ranged from 6 to 12 mg/kg. The average percent recovery of the samples was 83 percent; the standard deviation of the percent recovery was 27 percent. Results indicated that a soil sample containing 10 mg/kg of Aroclor 1232 should produce a response within two times the technology's response to the Aroclor 1242 standard.

Sample 012 was spiked with 10 mg/kg of Aroclor 1260. Results ranged from 34 to 45 mg/kg. The average percent recovery of the samples was 212 percent; the standard deviation of the percent recovery was 50 percent. The response of the technology to samples containing Aroclor 1260 was two to three times its response to the Aroclor 1242 standards. A soil sample containing 10 mg/kg of Aroclor 1260 should produce a response within two to three times its response to a 10 mg/kg Aroclor 1242 standard.

Sample 003 was spiked with 10 mg/kg of Aroclor 1221. Results ranged from 2 to 5 mg/kg. The average percent recovery of the samples was 11 percent; the standard deviation of the percent recovery was 11 percent. The response of the technology to samples containing Aroclor 1221 was one-fourth, or less, than its response to the Aroclor 1242 standards. A soil sample containing 10 mg/kg of Aroclor 1221 should produce a response equal to or less than one-fourth the response of the technology to a 10 mg/kg Aroclor 1242 standard.

Sample 034 was found to contain 40 mg/kg of PCBs when compared to the Aroclor 1242 standards. For the specificity evaluation, this sample was spiked with 10 mg/kg of Aroclor 1254. The result for all four aliquots was 120 mg/kg. The average percent recovery of the samples was 819 percent; the standard deviation of the percent recovery was 1 percent. The confirmatory laboratory result for Sample 034 was 34 mg/kg. This indicates that the high results obtained for all four aliquots were caused by the high recoveries of Aroclor 1254 in these samples.

The response of the technology to Sample 034 may have been affected by the high concentrations of PCBs in the soil samples prior to spiking. Results were nearly eight times higher than expected, which suggests the technology is much more sensitive to Aroclor 1254 than to Aroclor 1242.

Sample 040 was found to contain 44 mg/kg of PCBs when compared to the Aroclor 1242 standards. This sample was spiked with 10 mg/kg of Aroclor 1242. Results ranged from 28 to 44 mg/kg. The average percent recovery of the samples was 0 percent; the standard deviation of the percent recovery was 0 percent. The confirmatory laboratory result for Sample 040 was 4.2 mg/kg. EnviroGard PCB Test result of 44 mg/kg does not compare well with the result from the confirmatory laboratory. It was suspected that the original EnviroGard PCB Test result for this soil sample was a false positive result. The results of the Aroclor specificity test seem to support this suspicion. The response of the technology was affected by the high concentration of PCBs in Sample 040. All four spiked samples were 0.0 percent, indicating that the 10 mg/kg spikes were masked by the high concentration of PCBs already in the four aliquots. Because the original EnviroGard PCB Test result for this sample was much greater than the result from the confirmatory laboratory, it is not possible to determine with certainty the technology's specificity to Aroclor 1242.

Intramethod Assessment

No PCBs were found in any of the reagent blanks analyzed during either evaluation for this technology. Quantitative results were obtained for all but Sample 101 because the soil sample required dilution. The dilution of this sample resulted in a response comparable to the negative control sample. This problem was not recognized until after the demonstration activities were completed. The measure of completeness, therefore, for the quantitative analysis was 99 percent.

Intramethod accuracy was assessed using PE samples and matrix spike and matrix spike duplicate samples. The true result for PE sample 047-4024-114 (the high-level sample) was 110 mg/kg of Aroclor 1242, with an acceptance range of 41 to 150 mg/kg. The quantitative result for the PE sample was determined using the Aroclor 1242 standards prepared by PRC. The actual reported result for this sample when analyzed by the EnviroGard PCB Test was 90 mg/kg. This value was within the acceptance range. The percent recovery for the high-level PE sample was 82 percent. The true result for PE sample 047-4024-113 (the low-level sample) was 32.7 mg/kg of Aroclor 1242. It had an acceptance range of 12 to 43 mg/kg. The quantitative result for the PE sample when analyzed was determined with the Aroclor 1242 standards prepared by PRC. The result for this sample when analyzed with the Enviro-

Gard PCB Test was 28 mg/kg. This value was within the acceptance range. The percent recovery for the low-level PE sample was 86 percent.

The quantitative recovery results of the matrix spike and matrix spike duplicate samples are listed in Table 6-6. They were evaluated by comparing them to Aroclor 1242 standard responses plotted on a standard curve. The quantitative results of the soil samples, before they were spiked, showed that four contained less than 1 mg/kg of PCBs as determined by the Aroclor 1242 standards. One soil sample was found to contain 1 mg/kg of PCBs. Another soil sample was found to contain 17 mg/kg PCBs. The average recovery of the matrix spike samples was 114 percent or 28.5 mg/kg. The standard deviation of the matrix spike samples was 40.1 percent or 10.0 mg/kg. These results are summarized in Table 6-6. Following guidelines outlined in EPA SW—846 Method 8000, control limits can be established as $\pm$ 2 standard deviations from the mean percent recovery. For the matrix spike samples analyzed during this demonstration, the calculated control limits ranged from 34 to 194 percent recovery. All matrix spike samples analyzed fell within these control limits. The quantitative accuracy, as measured by the matrix spike and matrix spike duplicate samples, therefore, is acceptable. Based on an evaluation of PE samples and matrix spike and matrix spike duplicate samples, the intramethod accuracy of the EnviroGard PCB Test is acceptable.

As with the semiquantitative evaluation, three types of precision data were generated for the quantitative evaluation: laboratory duplicate samples, field duplicate samples, and matrix spike duplicate samples. Again, laboratory and field duplicate samples were used together because the soil samples were homogenized and only one operator was used.

Quantitatively, the results of the soil samples ranged from 1 to 1,300 mg/kg. The results of the duplicates for those samples ranged from 2 to 1,600 mg/kg. Quantitative matrix spike duplicate sample results are given in Table 6-6. Quantitative laboratory and field duplicate sample results are presented in Table 6-7.

Even the best technology which determines results quantitatively can not reproduce its results every time. Therefore, PRC established control limits like those used to evaluate laboratory duplicates. These control limits were then used to determine whether the difference between a result from a duplicate and the result from its respective sample was reasonable. To establish the control limits, all sample pairs (sample and duplicate) that did not produce two positive results were removed from the data population. Then, the RPD for each sample pair was calculated and the mean RPD and population standard deviation were determined. The lower control limit was set at zero because this would mean that the results from a duplicate and its respective soil sample matched perfectly. The upper control limit was set by multiplying the standard deviation by two and adding it to the mean RPD. The RPD of each sample pair was then compared to these control limits. Each RPD was expected to fall within the control limits. If greater than 95 percent fell within this range, the technology's precision was considered adequate. If fewer than 95 percent fell within this range, the data was reviewed, and if no explanation could be found, the technology's precision was considered inadequate.

The EnviroGard PCB Test had 27 sample pairs in which both a sample and its duplicate had positive results. The data from these sample pairs had a mean RPD of 29 percent and a standard deviation of 31. The control limits were, therefore, set at 0 and 92 percent. All but two of the 27 RPDs fell within the control limits. The first of these two sample pairs was Sample 042 and Sample 042D, which had results of 25 and 9 mg/kg, respectively. This resulted in an RPD of 94 percent, 2 percent above the upper control limit. The second of the two sample pairs was Sample 083 and 083D, which had results of 1 and 5 mg/kg, respectively. This resulted in an RPD of 133 percent, 41 percent above the upper control limit. Still, 91.5 percent of the sample pairs had RPDs within the control limits. While 91.5 percent is not between the demonstration's acceptable range of 95 to 100 percent, the technology only had two sample pairs not within the control limits. If one more pair had been accurate, the percentage acceptable would have been 96.3. Because so few pairs were involved in the statistical evaluation, this percentage was deemed acceptable.

The quantitative results for the matrix spike samples were compared to the Aroclor 1242 standards prepared by PRC. Precision of the matrix spike duplicate samples was evaluated through the RPD of the matrix spike result compared to the matrix spike duplicate result. RPD is the difference between these two results divided by the mean of the two results, expressed as a percentage. RPD values for the six matrix spike samples ranged from 0 to 90 percent. The mean RPD value from these six samples was 29 percent, and the standard deviation was 34 percent. If an upper control limit of two times the standard deviation is used, the upper control limit was 97 percent. All RPD values for the matrix spike duplicate samples fell within this range (Table 6-6). Therefore, based on the three types of intramethod precision data generated, the precision of the EnviroGard PCB Test is acceptable when used quantitatively.

TABLE 6-6. QUANTITATIVE MATRIX SPIKE AND MATRIX SPIKE DUPLICATE RESULTS.

Sample No.	Soil Sample Result (mg/kg)	Matrix Spike Amount (mg/kg)	Matrix Spike Recovery (%)	Matrix Spike Duplicate Recovery (%)	RPD (%)
047-4024-012	17	25	112	100	11
047-4024-024	ND	25	136	76	57
047-4024-049	ND	25	96	84	13
047-4024-069D	1.0	25	84	84	0
047-4024-082	ND	25	64	168	90
047-4024-111	ND	25	184	176	4

Notes:

mg/kg Milligrams per kilogram.
ND Not detected above the detection limit of 1 mg/kg.

Comparison of Results to Confirmatory Laboratory Results

The quantitative results of the EnviroGard PCB Test were analyzed by using the linear regression techniques detailed in Section 4. The initial linear regression analysis was based on results from 89 samples. The other results indicated that no PCBs were detected above the detection limit 1.0 mg/kg. The r^2 for this regression was 0.45, indicating that little or no relationship exists between the data sets. Therefore, the technology is not accurate. A residual analysis of the data, though, identified that the r^2 was greatly influenced by the results for Samples 16, 18, 38, 91, and 100. All of these samples show high levels of contamination. PRC removed these six points as outliers and recalculated the linear regression.

When the regression was recalculated on the 83 remaining sample results, it defined an r^2 factor of 0.87, indicating that a relationship did exist between the two data sets. The regression line that was calculated had a y-intercept of 17.8 mg/kg and a slope of 0.76. The normal deviate test statistic, though, indicated that the slope of 0.76 is significantly different from 1, and the y-intercept of 17.8 mg/kg is significantly different from 0. This means that the results from this technology are not accurate. However, if 10 to 20 percent of the soil samples were sent to a confirmatory laboratory, then the results from the other 80 to 90 percent could be corrected. This could result in a significant savings in analytical costs.

The Wilcoxon Signed Ranks Test was used to verify these results. It indicated, at a 95 percent confidence level, that the EnviroGard PCB Test's data was significantly different from that of the confirmatory laboratory. This confirmed the linear regression analysis and indicated that the EnviroGard PCB Test's data was not accurate.

To compare the precision of the EnviroGard PCB Test's results to the precision of the confirmatory laboratory's results, a Dunnett's Test was performed on the RPDs determined from the field duplicate samples and their respective samples. The Dunnett's Test determines the probability that the data sets on which it is based are the same. If the RPDs from the confirmatory laboratory and those from the technology are the same, then it can be assumed that the precisions are also similar. Dunnett's Test results in a percentage. For this demonstration, probabilities above 95 percent indicate that the precision of the technology and that of the confirmatory laboratory are considered the same. When the Dunnett's Test compared the RPDs between the EnviroGard PCB Test's data and the confirmatory laboratory's data, a probability of 97.5 percent resulted. This indicates that the technology is as precise as the confirmatory laboratory.

TABLE 6-7. QUANTITATIVE LABORATORY AND FIELD DUPLICATE SAMPLE RESULTS.

Sample No.	Soil Sample Result (mg/kg)	Duplicate Sample Result (mg/kg)	Relative Percent Difference (%)	Sample No.	Soil Sample Result (mg/kg)	Duplicate Sample Result (mg/kg)	Relative Percent Difference (%)
047-4024-001LD	10	5	67	047-4024-082FD	ND	ND	NA
047-4024-015FD	50	50	0	047-4024-083FD	1	5	133
047-4024-022FD	5	4	22	047-4024-084FD	50	49	2
047-4024-024FD	ND	ND	NA	047-4024-085FD	370	400	8
047-4024-028FD	1	ND	NA	047-4024-086FD	14	15	7
047-4024-035FD	ND, J	ND	NA	047-4024-087FD	6	3	67
047-4024-037FD	3	4	29	047-4024-088FD	20	23	14
047-4024-040LD	40	44	10	047-4024-090FD	10	8	21
047-4024-042FD	25	9	94	047-4024-091FD	1,300	1,600	21
047-4024-043FD	30	20	40	047-4024-092FD	2	2	0
047-4024-046FD	ND	ND	NA	047-4024-095FD	10	10	0
047-4024-047FD	ND	ND	NA	047-4024-097FD	ND	3	NA
047-4024-050FD	20	30	40	047-4024-098FD	46	30	42
047-4024-060FD	17	12	34	047-4024-098LD	46	31	39
047-4024-062LD	25	21	17	047-4024-100FD	400	470	16
047-4024-063FD	3	3	0	047-4024-102FD	30	30	0
047-4024-069FD	ND	1	NA	047-4024-102LD	30	41	31
047-4024-071FD	ND, J	ND	NA	047-4024-109FD	2	ND	NA
047-4024-081FD	4	3	29				

Notes:

mg/kg	Milligrams per kilogram.
LD	Laboratory duplicate.
FD	Field duplicate.
ND	Not detected above the 1 mg/kg detection limit.
NA	Not analyzed; the sample, duplicate, or both did not contain PCBs.
J	Detection limit raised to 2 mg/kg due to dilution of sample.

Section 7
Applications Assessment

The EnviroGard PCB Test can be operated in either a semiquantitative or quantitative mode. In either mode, it is relatively inexpensive and easy to operate. The technology involves a number of different steps, which increases the chance of operator error. In particular, errors can occur during measurements because of the small quantities of Aroclor standards and samples used. For this reason, operators must be thoroughly trained. Experience in common laboratory practices is helpful, although the technology can still be operated by nontechnical personnel.

The technology is very portable. Electricity is required to operate it; however, electricity can be supplied by a rechargeable battery. Reagents used with the technology must be refrigerated. It has a high sample throughput and is capable of quickly providing results, particularly when used in the semiquantitative mode. The ability of the technology to provide quantitative results is an additional advantage. When it is used in the quantitative mode, however, samples must frequently be diluted to bring their PCB concentrations into the linear range of the technology.

The EnviroGard PCB Test has slight reactions to some contaminants other than PCBs, such as halogenated organic compounds. For this reason, the technology does not appear to be highly susceptible to false positive results due to interferants, unless the interferants are present in very high concentrations (generally above 200 mg/kg depending on the interferant). The technology also did not appear prone to produce false negative results.

The results of the Aroclor specificity test indicated that the EnviroGard PCB Test will react differently to different Aroclors. However, Millipore can provide Aroclor-specific standards if the Aroclor of concern is known. When used with the correct standard, the technology can provide semiquantitative or quantitative results for each Aroclor.

The EnviroGard PCB Test can provide semiquantitative or quantitative results at sites where the Aroclor of concern is positively known so that the appropriate Aroclor standard can be used. If the Aroclor is known and if no interferants are suspected to be present at high concentrations, this technology would be useful at sites where results are needed quickly. If quantitative results are required, the quantitative results reported by the technology must be corrected if there is a need for them to be accurate.

Section 8
References

Department of Energy (DOE). 1989. "RCRA Facility Investigation and Corrective Measures Study for the Abandoned Indian Creek Outfall." Albuquerque Operations Office, Environment and Health Division, Environmental Programs Branch, Kansas City Plant.

Draper, N. R., and H. Smith. 1981. *Applied Regression Analyses*. John Wiley & Sons, Inc. New York. 2nd ed.

Environmental Protection Agency (EPA). 1989. "Preparing Perfect Project Plans." U.S. Environmental Protection Agency. Cincinnati, OH. EPA/600/9-89/087.

Pearson, E. S. and H. O. Hartley. 1976. *Biometrika Tables for Statisticians*. Charles Griffin and Company, Ltd. Third edition with corrections.

PRC Environmental Management, Inc. (PRC). 1992a. "SITE Demonstration: EnSys, Inc., Immunosystems, and Dexsil Corporation, PCB Field Kits, Pre-Demonstration Sampling Plan." May.

—. 1992b. "Final Demonstration Plan and Quality Assurance Plan for Demonstration of PCB Immunoassay and Field Screening Technologies." July 24.

Stanley, T. W. and S. S. Verner. 1983. "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans." U.S. Environmental Protection Agency, Washington D.C. EPA/600/4-83/004.

☆U.S. GOVERNMENT PRINTING OFFICE: 1995- 653-291

United States
Environmental Protection Agency
National Risk Management
Research Laboratory (G-72)
Cincinnati, OH 45268

Official Business
Penalty for Private Use
$300

EPA/540/R-95/517

Please make all necessary changes on the below label, detach or copy, and return to the address in the upper left-hand corner.

If you do not wish to receive these reports CHECK HERE ☐; detach, or copy this cover, and return to the address in the upper left-hand corner.

BULK RATE
POSTAGE & FEES PAID
EPA
PERMIT No. G-35