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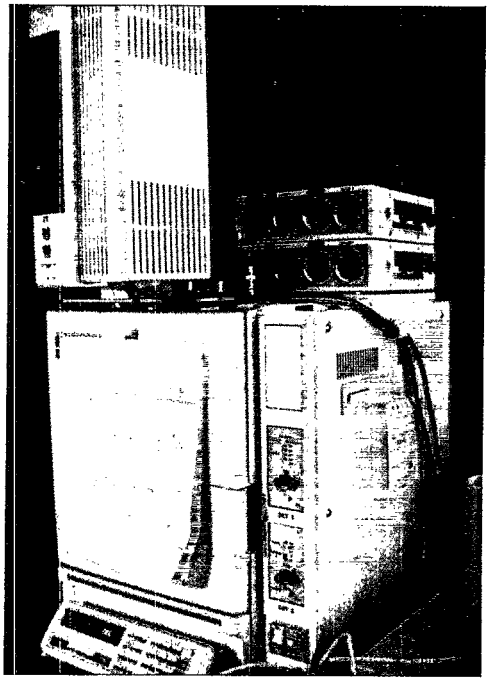
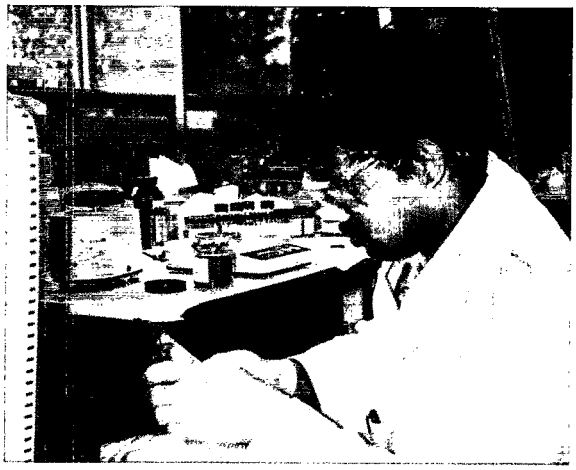
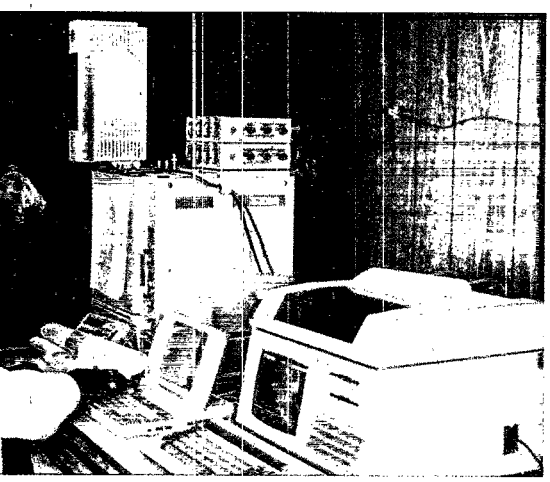
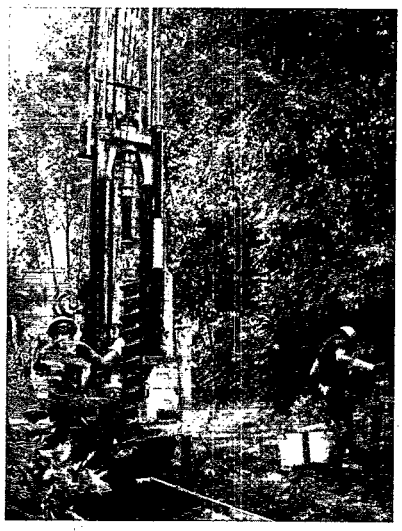
Field Analytical Screening Program: PCB Method

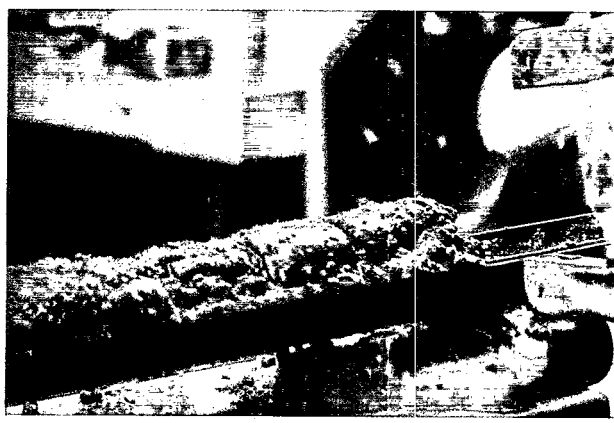
Innovative Technology Evaluation Report











SITE
SUPERFUND INNOVATIVE
TECHNOLOGY EVALUATION

EPA/540/R-95/521
August 1995

FIELD ANALYTICAL SCREENING PROGRAM: PCB METHOD

INNOVATIVE TECHNOLOGY EVALUATION REPORT

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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 innovative technology evaluation report (ITER) presents information on the demonstration of the U.S. Environmental Protection Agency (EPA) Region 7 Superfund Field Analytical Screening Program (FASP) method for determining polychlorinated biphenyl (PCB) contamination in soil. This method was demonstrated in Kansas City, Kansas, in August 1992.

The FASP PCB Method was developed by the EPA Superfund Branch for use at Superfund sites. The method uses a gas chromatograph (GC) equipped with a megabore capillary column and an electron capture detector (ECD). Gas chromatography is an EPA-approved method for determining PCB concentrations in soil, water, and waste samples. The FASP PCB Method is an abbreviated, modified version of approved methods. Soil samples require extraction before GC analysis. To remove matrix interferences, a sulfuric acid cleanup step is used during the FASP PCB Method.

The FASP PCB Method was found to be field-portable only in a mobile laboratory, must be done in a temperature-controlled environment, and requires a skilled chemist for operation. The detection limit reported by this method for is 0.4 part per million for soil samples. PRC used linear regression and inferential statistics to compare the method's data to that from the confirmatory laboratory. When the data sets were evaluated, the FASP PCB Method's results were statistically the same as the confirmatory laboratory. This method can produce Level 3 data. This method can also identify individual PCB isomers.

This report was submitted in partial fulfilment of contract 68-CO-0047 by PRC Environmental Management, Inc., under the sponsorship of the U.S. Environmental Protection Agency. This report covers a period from January 1992, to August 31, 1992, and work was completed as of February 1, 1993.

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List of Abbreviations and Acronyms

AICO	Abandoned Indian Creek Outfall
Allied-Signal	Allied-Signal, Inc.
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
EPA	U.S. Environmental Protection Agency
ERA	Environmental Research Associates
FASP	Field Analytical Screening Program
GC	gas chromatograph
HSP	health and safety plan
ICAL	initial calibration
ID	inside diameter
IDW	investigation-derived waste
ITER	Innovative Technology Evaluation Report
KCP	Kansas City Plant
L	liter
µg/L	micrograms per liter
µL	microliter
µg/kg	micrograms per kilogram
mg/kg	milligrams per kilogram
mL	milliliter
mm	millimeter
MMTP	Monitoring and Measurement Technologies Program
MS	mass spectrometer
NERL-LV	National Exposure Research Laboratory-Las Vegas
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	coefficient of determination
RCRA	Resource Conservation and Recovery Act
RFI	RCRA facility investigation
RPD	relative percent difference
SARA	Superfund Amendments and Reauthorization Act of 1986

List of Abbreviations and Acronyms (Continued)

SITE	Superfund Innovative Technology Evaluation
SOP	standard operating procedures
S O W	statement of work
TCL	target compound list
TIC	tentatively identified compounds
TPM	technical project manager
U V	ultraviolet

Acknowledgments

This demonstration and the subsequent preparation of this report required the services of numerous personnel from the U.S. Environmental Protection Agency, National Exposure Research Laboratory (Las Vegas, Nevada); U.S. Environmental Protection Agency, Region 7 (Kansas City, Kansas); 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 U.S. Environmental Protection Agency, National Exposure Research Laboratory **,** technical project manager, at (702) 798-2373 or Mr. Eric Hess, the PRC Environmental Management project manager, at (913) 573-1822.

Section 1
Executive Summary

This innovative technology evaluation report (ITER) presents information on the demonstration and evaluation of a field screening method for determining polychlorinated biphenyl (PCB) contamination in soil. PRC Environmental Management, Inc. (PRC), conducted the demonstration under contract with the U.S. Environmental Protection Agency (EPA) Superfund Innovative Technology Evaluation (SITE) Program. Specifically, this demonstration was conducted under the Monitoring and Measurement Technologies Program (MMTP) of the SITE Program, which is administered by the EPA National Exposure Research Laboratory, Las Vegas (NERL-LV).

The method selected for this demonstration and evaluation was a modified version of the Field Analytical Screening Program (FASP) method developed for the Field Investigation Team contract, which is part of the Superfund program. The method uses a field gas chromatograph (GC) and an extraction process that is similar to that of a conventional fixed laboratory. In August 1992, the method was demonstrated and evaluated at a site in Kansas City, Missouri. It was demonstrated in conjunction with the demonstrations and evaluations of three other field screening technologies for PCBs in soil: the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer, both of which are manufactured by the Dexsil Corporation; and the EnviroGard PCB Test, which is manufactured by Millipore, Inc. Separate ITERs have been prepared on the evaluations of these technologies and are available. These ITERs are entitled "Innovative Technology Evaluation Report on the Dexsil Corporation's Demonstration of the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer" and "Innovative Technology Evaluation Report on the Demonstration of the Millipore, Inc., EnviroGard PCB Test. "

The FASP PCB Method is designed to quickly provide quantitative results for PCB concentrations in soil samples. It uses gas chromatography, which is an EPA-approved method for determining PCB concentrations in soil samples. In fact, the FASP PCB Method is a modified version of EPA SW-846 Manual Method 8000. The method determines results for PCB concentrations, in the micrograms per kilogram (μg/kg) range, by using a GC equipped with an electron capture detector (ECD). Chromatograrns produced by the GC and ECD for each sample are compared to the chromatograms from Aroclor standards.

The instrumentation and equipment required for the FASP PCB Method are not highly portable. However, when they are mounted in a mobile laboratory trailer, the method can be operated on or near most sites relatively easily. Use of this method requires electricity, and Aroclor standards require refrigeration. An exhaust hood and carrier gases are also needed. For the method to produce reliable results, it must be operated by a trained and experienced operator.

The initial purchase cost of the instrumentation and equipment is relatively high. The three major pieces of equipment used in this demonstration cost $23,214. Similar equipment can be rented for $1,500 to $2,500 per month. Additional accessory equipment, reagents, and glassware needed to extract, prepare, and analyze soil samples during the demonstration cost an additional $5,000. This cost includes nondisposable glassware and laboratory equipment, in addition to disposable items. During this demonstration, 400 Sam-ple extractions and injections were conducted.

The detection limit for the method is reported to be 400 μg/kg. During the demonstration, however, PRC found that the method can often achieve a detection limit as low as 100 μg/kg. The highest number of samples analyzed in an 8-hour day was 21; the average number analyzed per 8-hour day was 15.

The method is susceptible to interferences from some compounds, other than PCBs, that are sometimes found in soil samples. Common interferants include phthalates, sulfur, halogenated solvents, and halogenated pesticides. In some cases in which these interferants are present, higher detection limits must be used for PCBs. It is also difficult, at times, for the operator to correctly identify and quantify mixtures of more than one Aroclor. Correct

identification of Aroclors depends largely on the judgment and experience of the operator.

To assess this method's precision, PRC evaluated its performance on the analysis of field and laboratory duplicate samples. PRC used the data from the duplicate analyses to establish precision control limits. The FASP PCB Method analysis showed 34 sample pairs in which both a sample and its duplicate had positive results. The data from these 34 pairs had a mean relative percent difference (RPD) of 34 percent and a standard deviation of 29. During this evaluation, precision control limits were established by adding two times the standard deviation to the mean for the upper control limit and using zero for the lower control limit. Therefore, the control limits were set at 0 and 92 percent. All of the RPDs for the duplicate sample pairs fell within the control limits. Therefore, the precision of the method (100 percent) was considered acceptable.

PRC used a regression analysis approach to evaluate the accuracy of the FASP PCB Method. The regression analysis was based on 76 matched pairs of positive sample results, and it defined a coefficient of determination (r^2) factor of 0.86, indicating that there was a strong relationship between the two sets of data. It defined a regression line with a y-intercept of 3.57 milligrams per kilogram (mg/kg) and a slope of 1.09. This indicates that the method is accurate. No correction is needed for the FASP PCB Method data, as it is not statistically different from the confirmatory data. The Wilcoxon Signed Ranks Test was used to verify these results. It indicated, at a 95 percent confidence level, that the data from the FASP PCB Method was not significantly different from those of the conlirmatory laboratory. The accuracy of the FASP PCB Method data was verified.

PRC also used the Dunnett's Test to evaluate the precision of the FASP PCB Method. This test indicated that the FASP PCB Method and the confirmatory laboratory may not have achieved identical precision.

Section 2
Introduction

Document Purpose

This ITER summarizes the procedures used to demonstrate the FASP PCB Method, discusses the results of the demonstration, and evaluates the effectiveness and possible uses of the FASP PCB method at various hazardous waste sites. The main goal of the demonstration was to evaluate the technology and to provide Superfund decisionmakers with information on its performance and cost effectiveness for possible use in future site characterization or cleanup projects.

EPA SITE Program and MMTP: An Overview

When the Superfund Amendments and Reauthorization Act of 1986 (SARA) took effect, it was widely recognized that new and better methods were needed to attack the environmental cleanup problem. Therefore, EPA developed the SITE Program to fulfill a requirement of SARA that EPA address the potential of alternative or innovative technologies. EPA gave joint responsibiity for this program to its Office of Solid Waste and Emergency Response (OSWER) and the Office of Research and Development (ORD). The SITE Program includes four component programs:

* Demonstration Program (for remediation technologies)
* Emerging Technology Program
* Meawsuring and Monitoring Technologies Program (MMTP)
* Technology Transfer Program

The largest part of the SITE Program involves the treatment technologies and is administered by ORD National Risk Management Research Laboratory (NRMRL) in Cincinnati, Ohio. However, the MMTP component is administered by NERL-LV. MMTP involves monitoring and measurement technologies that (1) identify, quantity, or monitor changes in contaminants occurring at hazardous waste sites, or (2) are used to characterize a site.

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. 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. Courts generally recognize data generated by conventional laboratory methods; still, there is a need to generate data more cost-effectively. Therefore, EPA must identify innovative approaches and, through verifiable testing of the technologies under the SITE Program, ensure that the innovative technologies are equivalent to, or better than, traditional 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. Therefore, 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, distributing the results and recommending those evaluations enhances the market for the technologies.

Defining the Process

EPA begins the innovative technology demonstration process by canvassing its 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 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 against each other. "

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

Components of a Demonstration

After a decision has been made to demonstrate technologies to meet a particular EPA need, the MMTP performs several activities. First, 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. After evaluation of the information, all respondents are informed of whether they have been accepted for the demonstration. While participants are being identified, potential sites are also identified, and basic site information is obtained. These activities complete the initial component of an MMTP demonstration.

The next component, and probably the most important, is the development of plans that describe how various aspects of the demonstration will be conducted. A major part of EPA's responsibility is the development of a demonstration plan, a quality assurance project plan (QAPP), and a health and safety plan (HSP). Although 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 that the developer can maximize the field performance of its innovative technology. Typically, the developers train demonstration personnel to ensure that those operating a technology have been adequately trained. 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 is the shortest part of the process. During the field demonstration, data are 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. 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 main product of the demonstration is an lTER, which is reviewed by peers and distributed as part of the technology transfer responsibility of MMTP. The ITER fully documents the procedures used during the field demonstration, QA/QC results, the field demonstration results, and conclusions. A separate QA/QC data package is also made available for those interested in evaluating the demonstration in greater depth. Two-page Technical Briefs 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, its own mobilization costs, and the training of EPA-designated operators. 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 that developers incur to demonstrate their products.

Demonstration Purpose, Goals, and Objectives

For this demonstration, the FASP PCB Method 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 technology. The accuracy and precision of the method were statistically compared to the accuracy and precision attained in a

conventional, fixed laboratory by using standard EPA analytical methods. The FASP PCB Method was also evaluated qualitatively 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 NERL-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 findings and results of the predemonstration sampling and analysis.

Identification of Developers

NERL-LV identified the FASP PCB Method as a technology showing promise for use in PCB field screening. After a review of available data on this technology, NERL-LV concluded that it warranted evaluation under MMTP.

Site Selection

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

* Wide range of PCB contamination

* Thorough characterization and documentation of contaminant concentrations (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).

* Accessible to the degree 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 U.S. Department of Energy (DOE) Kansas City Plant (KCP) was selected as the location of this demonstration. The soil at the AICO site is contaminated with a wide range of PCB concen-trations. PCB levels range from not detected, at a detection limit 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 (U.S. DOE 1989). PCB concentrations at the AICO site are well documented, which enabled PRC to collect samples having 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 federal government and operated by Allied-Signal, Inc. (Allied-Signal), for DOE. Since 1949, the plant has been used 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 occupied mainly by suburban residential and commercial developments (U.S. 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 was also 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 (U.S. DOE 1989).

PCBs comprise the only significant contaminant at the site. Before this demonstration, 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. PRC believes 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 did not result from DOE KCP discharges through Outfall 002 (U.S. DOE 1989).

According to logbooks recorded by Allied-Signal when boreholes were drilled during investigation of the AICO site, the former Indian Creek channel is overlain by 7 to 15 feet of till material composed mainly 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 (U.S. DOE 1989).

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

Selection of Confirmatory Laboratory and Method

EPA Region 7 Laboratory personnel selected one laboratory participating in the Contract Laboratory Program to perform the confirmatory analysis of samples for this demonstration. All samples were analyzed by using the method described in the 1990 CLP 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

Before the demonstration, the operator of the FASP PCB Method was trained in the use of the technology. This training included a review of operating procedures and instructions provided by the lead chemist for this demonstration.

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 that had previously been identified as containing high, medium, low, and nondetected concentrations of PCBs. These samples were split into replicates. One replicate of each sample was analyzed by using the FASP PCB Method, and the confirmatory laboratory analyzed one replicate of each sample by using standard EPA analytical methods. This sampling allowed potential matrix effects or interferences to be evaluated before the demonstration. The main finding from predemonstration sampling was that the soil at the AICO site was more clayey than expected, thereby making it difficult to homogenize the samples (see Section 4).

Section 4
Demonstration Design and Description

This section describes the sample collection procedures and the experimental design used to evaluate the FASP PCB Method. This section summarizes key elements of the QAPP (PRC 1992b), field analysis operations, and data management activities.

Sample Collection

For the demonstration, PRC collected 112 soil samples and 32 field duplicate samples 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 by using the 1990 CLP SOW method. At the request of the EPA technical project manager (TPM), a second replicate was submitted to NERL-LV for separate analysis, although the data generated by NERL-LV was not used in this demonstration. A third replicate was analyzed in the field by the FASP PCB Method. The remaining replicates were analyzed in the field by using the three other technologies, described in separate ITERs.

PRC collected samples by 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. PRC collected all samples by using the sample collection and homogenization procedures specified in the sampling plan (PRC 1992b). All PRC field activities conformed with requirements in the HSP prepared for this demonstration (PRC 1992b).

Samples were collected from areas known to exhibit PCB concentrations ranging from not detected (at a detection limit of 0.16 mg/kg) to 9,680 mg/kg. Most of the samples were collected from areas that had been identified as containing PCBs at concentrations ranging from nondetected to 100 mg/kg, 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 field screening technologies are designed for operation in this range.

PRC collected twenty samples from areas that had been identified as containing PCBs at concentrations ranging from 100 to 1,000 mg/kg, and from 1,000 to 10,000 mg/kg. These samples were analyzed to evaluate the abilities of the field screening technologies to monitor PCBs in higher concentrations. After col-lection, 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 NERL-LV and for analysis by the field-screening technologies were placed in 4-ounce wide-mouth glass jars with Teflon-lined lids.

PRC monitored homogenization of the samples by adding a small amount of powdered uranine, which is 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 that sample was 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 evenly distributed signs of fluorescence, homogenization of the sample continued, and the examination process was repeated. PRC found that of small amounts of fluorescein did not interfere with sample analysis for any of the field-screening technologies or for the confirmatory laboratory.

After PRC received confirmatory laboratory results, it 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 is based on the assumption that there was perfect homogenization and that there was no difference introduced by analytical error. Using a matched pair Student's t-test enabled PRC to determine whether 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 appear to indicate that their homogenization could have been better, the homo-genization technique used was highly effective overall.

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. Samples 91 and 102, respectively, were the low and high outliers. However, the Student's paired t-test 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 significantly affecting the analysis. For example, one 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, it is possible that some of the samples in the demonstration were poorly homogenized. The analysis of such data could produce limited cases of inaccurate data. Therefore, PRC collected and analyzed a large number of samples 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, PRC prepared a QAPP (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; all participants ultimately agreed to its content.

The main 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 NERL-LV SOW, this demonstration is considered a Category II project. The QAPP addressed the key elements required for Category II projects prepared in accordance with guidelines in the EPA booklet ***Preparing Perfect Project Plans*** (1989) and the ***Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans*** (U.S. EPA 1983).

For sound conclusions to be drawn concerning the field-screening technology, the data obtained during the demonstration needed to be of known quality. For all monitoring and measurement activities conducted for EPA, the agency requires that DQOs establish the basis of the way in which the data will be used. DQOs must include at least five indicators of data quality: represen-tativeness, completeness, comparability, accuracy, and precision. The following paragraphs discuss these indicators in greater detail. The success of the demonstration required that DQOs be met by the confirmatory laboratory. Some DQOs for the con-firmatory laboratory were indicated in the CLP 1990 SOW, and others were derived from data generated during use of the method. It was critical that the confirmatory laboratory analyses be sound and within CLP 1990 SOW method specifications so that the data that the method generated could be compared to that obtained by the technologies. High-quality, well-documented confirmatory results were essential to making this comparison.

Representativeness refers to the degree to which the data accurately and precisely represent the condition or characteristic of the parameter that is represented by the data (U.S. EPA 1983). In this demonstration, PRC ensured representativeness by (1) executing a consistent sample collection, homogenization, and handling program, and (2) using each technology at its optimum capability to provide results that represented the most accurate and precise measurements that it was capable of achieving.

Completeness refers to the amount of data collected from a measurement process compared to the amount that was expected (U.S. EPA 1983). For this demonstration, completeness refers to the proportion of valid, acceptable data generated by using each of the tecochnologies and the confirmatory laboratory. During the demonstration, the completeness objective for each technology was 90 percent, which was achieved.

Comparability refers to the confidence with which one data set can be compared to another (U.S. EPA 1983). The main focus of this demonstration was to compare data generated by the FASP PCB Method and the other technologies with confirmatory laboratory results by using the experimental design and statistical methods discussed in the QAPP. Additional QC for comparability was achieved by (1) analyzing QC samples, blanks, and Aroclor standards, and (2) adhering to standard EPA analytical methods and standard operating procedures (SOP) for.preparing samples and operating instruments.

Accuracy refers to the difference between the sam-ple 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, PRC assessed accuracy by using the statistical comparisons detailed in the QAPP.

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. For this demonstration, PRC measured precision by comparing the RPDs of samples and their duplicates with control limits established through the statistical methods detailed in the QAPP.

The main objective of the demonstration was to evaluate the efficiencies of the FASP PCB Method in determining PCB contamination in soil. This evaluation included defining the precision, accuracy, cost, and range of usefulness for the technology. This objective also included determining the DQOs that the technology was capable of achieving. A secondary objective was to evaluate the specificity of the 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 the field-screening technology was another important quantitative factor. Cost items include expendable supplies, nonexpendable equipment, labor, and disposal of investigation-derived waste (IDW). 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.

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 one operator for the field- screening technology, and accepted that the error introduced by operator effect would not be distinguishable from error inherent in the field- screening technology. 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 the field-screening technology was determined by comparing results from the technology with those from the confirmatory laboratory. PRC then used statistical analysis of these results to identify the contaminant range in which results from the 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. In the planning stages of this demonstration, interest was shown in the cross-reactivity between Aroclors for the technology. To assess this factor, PRC evaluated cross-reactivity for the technology by using matrix spikes for each of the seven Aroclors (1016, 1221, 1232, 1242, 1248, 1254, and 1260) typically analyzed by using standard EPA analytical methods. This information was then used to determine the sensitivities of the technology to each Aroclor.

Statistical Analysis of Results

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

All of the statistical tests used for this demonstration were stipulated in the demonstration plan, which all demonstration participants approved in advance of data collection. The demonstration plan also stipulated that all sample pairs that included a nondetect result would be removed from data sets. PRC believed that the variance introduced by eliminating these data pairs would be less than or equal to the variance introduced by giving an arbitrary value to nondetect results.

In cases in which field duplicate samples were collected, the demonstration plan stated that the results of the two duplicates would be averaged and that this average would be used in subsequent statistical analysis. PRC followed this guideline. 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 following equation was used:

(4-I)

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

where

***RPD** = relative percent difference.*
***R,** = initial result.*
R_d = *duplicate result.*

The RPDs were then compared with upper and lower control limits. Because the technology being demonstrated was also being assessed, the control limits used were calculated from data provided during this investigation. To determine these control limits, PRC calculated the standard deviation of the RPDs for the 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 indicate 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 precision of the technology was acceptable.

Data from the field screening technology was compared with the confirmatory laboratory data to determine the accuracy of the technology. For the FASP PCB Method, two statistical methods were used: linear regression analysis and the Wilcoxon Signed Ranks Test.

PRC calculated the linear regression by using 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. Calculation of linear regression is expressed as an equation that can be visually expressed as a line. Three factors are determined during calculations of linear regression: the y-intercept, the slope of the line, and the correlation coefficient. Before a technology's accuracy could be considered acceptable, all three of these factors must have had acceptable values.

The r^2 expresses the mathematical relationship between two data sets. If the r^2 is 1, the two data sets are closely related. 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. Skewing of the r^2 can be caused by the greater accuracy of technologies in analyzing samples in one range than in 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, an examination of regression residuals (Draper and Smith 1981) technique was used to identify outliers that might have skewed the results. In fact, the computer program used to calculate the linear regression identified most of the outliers. After outliers were identified, they were removed, and linear regression recalculated.

If the corrected data set resulted in an r^2 between 0.80 and 1, 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 indicate that the results of the technology perfectly matched those of the confirmatory laboratory. 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 their expected values 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, the method's data was 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-the technology was found to be inaccurate; however, in this case, results from the technology 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 in which the r^2, the y-intercept, and the slope were all found to be acceptable did PRC determine that the technology was accurate.

The Wilcoxon Signed Ranks Test is a nonparametric method of 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. That is, 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 linear regression analysis and the Wilcoxon Signed Ranks Test perform similar types of comparisons, they are based on different assumptions. 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.

Finally, PRC used Dunnett's Test to statistically compare the intermethod precision of the method with the the precision of the confirmatory laboratory. This test was used to assess whether the precision of the technology and that of the confirmatory laboratory were statistically equivalent. PRC first determined the mean RPD for all samples and their respective duplicates analyzed by the confirmatory laboratory. It then statistically compared this mean RPD with the RPDs of each duplicate pair analyzed by each of the technologies. The Dunnett's Test results in a single statistical value that indicates the degree of certainty that the precision of the two methods is the same. In other words, a 90 per-cent 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.

Results below 95 percent do not indicate 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 any difference between the two data sets was caused by the precision of a technology's data being greater than that of the confirmatory laboratory.

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, storage of samples, and storage of sample collection equipment. All equipment, field 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 CLP laboratory is discussed in more detail in the following subsections.

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 analysis by the 1990 CLP SOW method. This method requires that organochlorine pesticides and PCBs be analyzed by using a GC equipped with an ECD.

EPA Region 7 Laboratory personnel conducted a Level II data review of 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. The confirmatory laboratory reviewed the raw data and checked the calculations before submitting the data package to EPA. PRC was not able to review the raw data generated from the analysis of samples. However, PRC reviewed EPA's comments generated by the Level II data review.

The following subsections discuss specific procedures used to identify and quantity PCBs by using the CLP 1990 SOW method. Most of these procedures involved requirements established to guarantee the quality of the data generated.

All of the confirmatory laboratory results used to assess the FASP PCB Method are presented in tables in Section 6.

Soil Sample Holding Times

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

Soil Sample Extraction

PRC extracted soil samples in accordance with the procedures outlined in the 1990 CLP SOW method for organochlorine pesticides and PCBs. This procedure involves placing 30 grams of soil into a beaker and 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 are then added to the beaker. 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 twice 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 Kudema-Danish apparatus. The Kuderna-Danish apparatus is placed in a hot water bath, and the organic solvent is concentrated. After it has been 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 by 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 1990 CLP 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 quantify PCBs in samples. The ICAL is performed before sample analysis begins. PCBs cause multipeak patterns when analyzed by 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.

The retention times were monitored by 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 ± 0.07 minutes for each peak identified in the ICAL. According to the 1990 CLP SOW method, when 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 1990 CLP SOW method and were acceptable, as the CCAL calibration factor never exceeded 25 per-cent.

After an ICAL has been performed, sample analysis begins. Sample analysis usually begins by analyzing a method blank to verify that it meets the 1990 CLP 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 1990 CLP 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. Because of the way in which the PCBs were manufactured or because of the effects of weathering, peak patterns may not match exactly. 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, identification of peak pattern depends largely on the experience and interpretation of the analyst.

PCBs are quantified 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 1990 CLP SOW method. Because the 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.

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 detined as 16 times the response of the Aroclor standards analyzed during the ICAL. After a sample has been diluted to within the linear range, it is analyzed again. When appropriate, dilutions were performed on the samples for this demonstration.

Detection Limits

During the ICAL, one concentration of each Aroclor was analyzed. 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 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 1990 CLP 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 in the preceding paragraph 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 FASP PCB Method was listed in mg/kg. Even when corrected to account for percent moisture, the CRQLs would be significantly below the detection limits for the technology.

Quality Control Procedures

As required in the 1990 CLP SOW method, the confirmatory laboratory used numerous QC measures including analysis of resolution standard mixes, method blanks, and instrument blanks; all requirements were met for this demonstration.

Also, surrogate standards were added to all standards, method blanks, matrix spikes, and soil samples analyzed by using the 1990 CLP SOW method. The percent

recovery of each surrogate was calculated and compared with the advisory control limits of 60 to 150 percent in the 1990 CLP SOW. No corrective action is needed when surrogate recoveries fall outside of the advisory control limits. However, the surrogate recoveries 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 witbin the linear range required by the 1990 CLP SOW; however, the dilutions decreased the amount of the surrogate standards that were injected onto the GC, resulting in nondetection of the surrogates 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. However, comments from the EPA Level II data review indicated that 88 of the samples and their respective duplicate samples resulted in acceptable surrogate recovery data.

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

Second Column Confirmation

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

The 1990 CLP 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 the results are estimated. For the analysis of the samples from this demonstration, 17 sample results were above the 25 per-cent requirement of the 1990 CLP SOW. These results were J-coded to indicate that the results were estimated but were not validated by approved QC procedures. Finally, following the 1990 CLP SOW method required when values obtained from the analysis of a sample on two columns were different, the reported value was the lower of the two values. This requirement was followed for the samples from this demonstration.

Gas Chromatographic Mass Spectrometer Confirmation

The 1990 CLP SOW requires that, when pesticides or PCBs are present in samples at sufficient quantities, they 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 was 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 to identifying individual congeners . Because all 20 sam-ples 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 1990 CLP SOW. PRC obtained data, from the confirmatory laboratory, on the percentage of solids in the sample and used these data to convert the results to wet weight. This conversion was required, because the data were to be compared with data obtained by the FASP PCB Method which reported concentrations on the basis of 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 were coded with a "K," indicating either (1) that the actual PCB concentration in the sample was less than the reported value, or (2) that PCBs were not found in the sample. The third code used was a "J" code, 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 RF1 and CMS results from April

1989, Aroclor 1242 was the only Aroclor believed to be present at the AICO site. Most of the samples analyzed by the confirmatory laboratory were found to contain either Aroclor 1242 or Aroclor 1248, however, the confirmatory laboratory found three additional Aroclors in the samples collected during the demonstration. Seventy-three samples were found to contain only Aroclor 1242, and 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 consisted of 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 at concentrations above the CRQLs.

Data Quality Assessment of Confirmatory Laboratory Data

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

Accuracy

Accuracy for the confirmatory laboratory was assessed through the use of PE samples containing a known quantity of Aroclor 1242, and purchased from Environmental Research Associates (ERA). For each PE sample, ERA supplied data sheets which included the true concentration and an acceptance range for the sample. The acceptance range was based on the 95 per-cent 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 were the other soil samples. The confirmatory laboratory knew that the samples were PE samples but did not know the true concentrations and acceptance ranges of the samples. The true concentration of sample 047-4024-l14 (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 per-cent. The true value concentration of sample 047-4024-l13 (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 1990 CLP SOW. Two other types of data commonly used to measure precision-matrix spike and matrix spike duplicate RPDs-were also unavailable, because matrix spike compounds required by the 1990 CLP SOW method are pesticide compounds, not PCBs.

PRC used the evaluation of field duplicate sample results 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, whereas 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 97 had a result of 1.23 mg/kg, whereas its duplicate had a result of 0.285 mg/kg. The RPD for Samples 97 and 97D was 124.8 percent. However, the other RPDs had much lower percentages. Without these two samples, the mean RPD fell from 31.8 to 20 percent. Overall, these data show 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 EPA Region 7 Laboratory defines the J-code as data estimated but not validated by approved QC procedures. Based on the definition of completeness stated in the QAPP (PRC 1992b), 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, PRC and NERL-LV determined that the J-coded

data were acceptable. For this reason, the actual completeness of data used was 100 percent.

Use of Qualified Data for Statistical Analysis

As noted, 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 these J-coded data were not valid, because they had failed at least one of the two QA/QC criteria specified in the 1990 CLP 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 in 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 magnitude similar to that introduced by using the data. For these reasons, PRC, after consulting with NERL-LV, chose to use the J-coded data regardless of the determination by the EPA Region 7 Laboratory that the results were invalid under approved QC procedures.

Section 6
EPA Superfund: Field Analytical Screening Program PCB Method

This section discusses the FASP PCB Method, including background information, operational characteristics, performance factors, a data quality assessment, and a comparison of its results with those of the confnmatory laboratory. Observations made by the operator are presented throughout this section.

Theory of Operation and Background Information

The FASP PCB Method is designed to provide quick and accurate results for PCB concentrations in soil samples. PCB concentrations are reported in μg/kg. This method was developed as part of the Field Investigation Team contract administered by the EPA Region 7 Superfund program.

The FASP PCB Method uses a GC equipped with an ECD to identify and quantify PCBs. Gas chromatography is used in EPA SW-846 Manual Method 8000, an EPA-approved method for determining PCB concentrations in soil samples. The FASP PCB Method is an abbreviated version of this method. Soil samples must be extracted before analysis begins. First, 5 grams of a soil sample are placed into an extraction vial. For removal of the water from the soil sample, 10 grams of anhydrous sodium sulfate are added to the extraction vial and thoroughly mixed with the soil. Twenty mL of pesticide-grade hexane are added to the extraction vial, which is then mixed with a vortex mixer for 2 minutes. The extraction vial is allowed to sit so that the soil and sodium sulfate mixture separates from the hexane extract. A part of the hexane extract is transferred to a test tube. One to 2 mL of concentrated sulfuric acid is added to the test tube, where it is mixed with the hexane extract. The hexane and sulfuric acid mixture is allowed to sit for 10 minutes so that they can separate. This step is used to remove potential interferences from the resulting soil sample extract.

Next, the GC must undergo an ICAL. The ICAL involves analyzing standards containing three different concentrations of seven Aroclors. Peak patterns are observed, and retention time windows are determined for three to five peaks in each Aroclor. Calibration factors for each Aroclor are calculated and evaluated. When an acceptable ICAL has been completed, analysis of soil sample extracts may begin.

Analysis begins with the injection of microliter (μL) amounts of the soil sample extract into a GC equipped with a megabore capillary column. The GC megabore capillary column effluent is directed through an ECD. Sample chromatograms are compared to PCB standard chromatograms to identify and quantify Aroclors. Peak patterns and retention times from the chromatograms are used to identify PCBs in the soil sample extract. Peak patterns are used to identify and quantify PCBs in the soil sample extract.

Chromatograms for standards and samples can be plotted on a strip chart, an integrator, or a data system. Samples analyzed during the demonstration were plotted on a data system. The data system plotted chromatograms, measured retention times of peaks, calculated peak areas and peak heights, stored chromatographic data on floppy disks, and manipulated data for baseline correction. The data generated were transferred to a spreadsheet program for the evaluation of standards and samples.

Daily CCALs are performed for four of the seven Aroclors. CCALS are used to monitor the performance of the GC. .Acceptable CCALs must be performed before sample analysis can continue. When unacceptable CCALs occur, a new ICAL must be performed before sample analysis can continue.

Operational Characteristics

Because they are large, the instruments and equipment required for the FASP PCB Method are not very portable. The instruments used for this demonstration included (1) a Shimadzu GC-14A equipped with an ECD, (2) a Shimadzu AOC-14 auto-sampler, (3) a Shimadzu CR-4AX

equipped with an ECD, (2) a Shimadzu AOC-14 auto-sampler, (3) a Shimadzu CR-4AX data system, and (4) a COMPAQ SLT/286 laptop computer. The GC with the auto sampler was 18 by 24 by 42 inches and weighed about 100 pounds. The data system was 18 by 24 by 12 inches and weighed 25 pounds. Laboratory equipment needed for sample preparation and analysis included: (1) one cylinder of Nitrogen gas, (2) a GC Column: DB-608, 30 meter, 0.53 millimeter (mm) ID, or equivalent, (3) 10, 50, and 100 μL micropipettes, (4) 10, 25, 50, 100, and 500 PL micro syringes, (5) 1, 5, and 10 mL glass volumetric pipettes, (6) 2 to 10 mL repipettors with Teflon liners, (7) 10, 50, and 100 mL volumetric flasks made of glass and with ground-glass stoppers, (8) 2 mL glass vials with Teflon-lined caps for storing stock standards, (9) 2 mL autosampler vials with Teflon-lined cap, (10) a bubble flow meter used, used to check the GC column flows, (11) 4-fluid-ounce standard bottles with Teflon-lined screw cap for calibration standard storage, (12) Pasteur pipets: 5.75- and 9-inch size Pasteur pipettes made of disposable glass, (13) 4OmL extraction vials with Teflon-lined screw caps, (14) 13 mm by 100 mm test tubes with Teflon-lined screw caps, (15) stainless-steel spatulas, (16) a top loader balance with 0.01 gram accuracy, (17) a high speed vortex mixer, (18) a digital timer capable of timing up to 99 minutes, (19) both large and small pipet Bulbs to fit the volumetric and Pasteur pipets, (20) labels, (21) markers, (22) polypropylene or latex gloves, (23) safety glasses, (24) laboratory coats or protective clothing, (25) a refrigerator or ice chest for standards and samples, (26) a hood to remove solvent fumes from work area, and (27) floppy disks for data storage.

The standard reagents used for sample extraction and analysis are pesticide-grade hexane; granular, pesticide-grade sodium sulfate; concentrated sulfuric acid; powdered sodium bicarbonate (for acid spills); and Aroclor standards of 96 percent purity or greater.

The FASP PCB Method must be performed indoors to protect the analytical equipment from moisture and temperature extremes. Most of the other equipment and reagents also require this protection. During this demonstration, this method was performed in a trailer. Another logistical requirement was electricity. Electricity was provided to the trailer through a temporary power pole. An alternative source of electricity may be used, such as a gasoline-powered generator. A generator allows the analytical equipment to be operated at even the most remotely located PCB-contaminated sites. The FASP PCB Method requires using a hood to evacuate solvent and acid fumes from the work area. The hood may be vented directly outdoors or through a charcoal trap in which the harmful **fumes are** trapped. Regular maintenance of the charcoal trap is required to ensure that it is operating correctly. A flammable solvent storage cabinet is recommended for storing solvents safely. A refrigerator is needed for storing the Aroclor standards. The refrigerator used for this demonstration was 3.5 cubic feet in size. Carrier gases are required for gas chromatography. These gases may be purchased before arrival at the site, or agreements for delivering the carrier gases to the site may be made with local carrier gas suppliers. The work space required for the setup of analytical equipment is 12 square feet. Another 8 square feet of space is needed to perform the sample extraction and preparation. Storage space is also needed for the equipment, reagents, and glassware.

Soil samples should be refrigerated until sample analysis. However, they should not be stored with Aroclor standards, which should be stored in a second refrigerator. The refrigerator is recommended for storing samples overnight or until it is determined which of the samples will be transported to a formal laboratory for further analysis.

A minimum of 6 months of experience in using a GC and a minimum of 1 month of experience in analyzing PCBs is required to effectively use the FASP PCB Method. The operator noted that the method is relatively easy to run with some prior GC experience and that any person with a basic knowledge of analytical chemistry can be expected to quickly learn the method and techniques. PRC tested the FASP PCB Method by using the equipment from its mobile laboratories. The ruggedness of PRC's equipment has been proven. This equipment has been transported and used in Hawaii, California, Arizona, Montana, Kansas, Missouri, Iowa, Wisconsin, Illinois, Michigan, and Ohio. No problems have been observed in the instrumentation.

In addition to the GC, the operator used an autosampler that automatically injected equal amounts of sample extracts into the GC's column. Using the autosampler analysis can occur even when the analyst is not present. In addition, the autosampler ensures that the correct amount of extract is used for each analysis. Maintenance of the GC and other equipment is essential to ensure quick and accurate PCB results in the field. Because of the rigors of working in the field, the equipment should be on site and operating correctly at least 1 to 2 days before sample collection begins. This lead time allows any needed maintenance to be performed before sample collection begins. Routine maintenance includes carrier gas changes, injector port septum changes, and column conditioning. Nonroutine maintenance may include column changes or electronic board replacement. Agreements with equipment

suppliers for overnight delivery of replacement parts, and in-the-field servicing by equipment service representatives may be required to provide all of the necessary maintenance.

PRC evaluated instrument reliability by monitoring the equipment's ability to maintain its calibration. An ICAL was performed at the start of the demonstration. The instrument calibration was monitored daily through CCALs by using the mid-level concentrations for four Aroclor standards. Three to five peaks were identified in the mid-level Aroclor standards to monitor CCALs. The height of these peaks in the ICAL were compared with the height of these peaks in subsequent CCALs. The heights of these peaks were compared by calculating their RPDs. An RPD value of less than or equal to 25 percent is required to continue sample analysis. CCAL RPD checks were performed on four Aroclors: 1016, 1242, 1254, and 1260.

During the 17 days of the demonstration, 16 CCALs were performed. The first 14 CCALs analyzed were determined acceptable. On the 15th day, the CCAL for Aroclor 1260 was above the 25-percent RPD criterion. Corrective action was taken to determine the cause of the calibration failure. It was determined that the septum needed replacement. The total number of sample and standard injections performed on the GC before the 15th day was greater than 400. This is an extremely high number of analyses without a septum change. The septum was changed, and another set of CCAL standards was analyzed. The CCAL RPD values for the reanalysis were less than 25 percent. Sample analysis was continued. On the 16th day, a CCAL was performed after sample analysis was completed. The RPD values for this final CCAL were less than 25 percent and considered acceptable.

The retention times of specific Aroclor peaks were also monitored through the CCALs. The retention times of these peaks shifted throughout the demonstration. The windows were adjusted to maintain accurate retention time windows for these peaks. Thii adjustment was made by using the CCAL PCB peaks as the mean of the window, and the absolute retention time windows stated in the FASP PCB Method were applied to this mean. Another reliability factor evaluated was the consistency of the data system to properly draw a baseline for standards and samples. Overall, the data system was consistent in its baseline determinations. However, because of numerous interference peaks in the samples, numerous chromatograms had to be reprocessed to correct the baseline. The operator of this method was able to redraw the baselines of these samples to more correctly simulate the baseline of the standards.

The FASP PCB Method uses three chemicals to remove potential interferants from samples and to extract the PCBs. Hexane, a flammable solvent that can be absorbed through the skin, is used to extract PCBs from samples and to dilute standard solutions. Sodium sulfate, which can irritate the skin and eyes, is used to bind to the water in soil samples. Sulfuric acid, which can burn the skin, was used to remove possible interferences from soil sample extracts. While using any of these chemicals, personnel should wear chemical-resistant clothing, gloves, and safety glasses. Fire extinguishers, safety equipment, and an adequate source of water should be available in case the acid comes into contact with the skin. Eyewash solutions should also be available.

The ECD contains nickel-63, which is a radioactive material. The amount of radioactive material in the ECD is minimal, and it is stored in a sealed container. The container must be checked for possible leakage twice each year. Leakage of the radioactive material of above federally-regulated limits requires immediate attention. The manufacturer of the material must be contacted to determine the appropriate action.

Mr. Ramarao Rayavarapu, an employee of PRC, was the operator chosen to analyze samples by using the FASP PCB Method. Mr. Rayavarapu earned a B.S. degree in civil engineering in 1983 from Andhra University in India. Mr. Rayavarapu also earned an M.S. in environmental engineering in 1990 from the University of Missouri. While at PRC, Mr. Rayavarapu has conducted RCRA facility inspections, compliance evaluation inspections, and field sampling and oversight work. Before joining PRC, he was involved in designing industrial and municipal waste treatment systems. Mr. Rayavarapu has more than 5 years of experience in designing these types of systems. His experience also includes using several types of wet chemical analytical methods. The lead chemist for the demonstration trained Mr. Rayavarapu in using the FASP PCB Method. This training included 1 week of hands-on work using and maintaining the GC. Training also included specific procedures required for the extraction, preparation, and analysis of samples.

Mr. Rayavarapu analyzed all soil samples collected during the predemonstration activities using the FASP PCB Method. Mr. Rayavarapu's training culminated with the analysis of two PE samples to determine whether he could produce acceptable results by using this method. Mr. Rayavarapu completed the PE samples with acceptable results. After analyzing these samples, Mr. Rayavarapu noted that he felt comfortable using the method.

The analytical instrumentation used to analyze soil samples during this demonstration was purchased through a Shimadzu sales representative. The total cost of the analytical equipment was $23,214. This cost includes the GC equipped with an ECD, the autosampler, the data system, all equipment required for set-up of the GC, and installation. Costs per component were $13,149 for the GC, $3,865 for the autosampler, and $6,200 for the data system. Similar analytical equipment can be purchased from other analytical equipment manufacturers. The costs are expected to be comparable to the Shimadzu prices. Analytical equipment can also be rented from numerous companies. The costs for renting comparable equipment range from $1,500 to $2,500 per month. Most rental companies require a minimum rental period, but this may be negotiable. Rental rates sometimes include delivery and set-up. Many companies also offer rent-to-own or lease-to-own options for analytical equipment.

The reagents and equipment needed to perform the extraction, preparation, and analysis of soil samples by using the FASP PCB Method are estimated to cost $5,000. This demonstration required over 400 sample extractions and injections. These 400 sample extractions include initial sample analysis, subsequent sample dilutions, and QA/QC samples. These supplies may be purchased from numerous analytical supply companies.

Waste disposal is another operating cost. During this demonstration the waste generated filled a 55-gallon barrel. The appropriate way to dispose of this waste is through an approved PCB incinerator facility. Disposing of one barrel of this waste is estimated to cost $1,000.

Performance Factors

The following subsections describe the FASP PCB Method's performance factors, including its detection limits and sensitivity, sample throughput, linear range, specificty, and drift.

Detection Limits and Sensitivity

The sensitivity of the FASP PCB Method depends on the detection limit of the ECD. For most methods that use this sort of instrument, the detection limit is defined as the minimum amount of a compound that will give a response that is greater than three times the noise level of the instrument. The ECD is so sensitive that it almost always detects something in its environment; this level of detection is termed "noise level." During the demonstration, the noise level of the instrument's detection limit was not evaluated. However, PRC noted that the responses of all of the low-concentration Aroclor standards were significantly above three times the noise level for the instrument.

The detection limit of the FASP PCB Method was stated to be 0.4 mg/kg for soil samples. This detection limit was based on the low-concentration Aroclor standards used to calibrate the GC, and the dilution factors used to extract and prepare samples. The low-concentration standard for all seven Aroclors was 100 μg/kg. No further dilution or concentration of the sample was performed during the extraction process. The detection limit is calculated by dividing the number of liters (L) of solvent (.02 L) by the number of grams of soil (5 grams), and multiplying this number by the concentration of the low Aroclor standard (100 μg/kg). This results in the detection limit of 0.4 mg/kg.

The ECD is used to detect organochlorine pesticides and PCBs in soil and water samples by using standard EPA-approved methods. The ECD is sensitive to halogenated compounds, especially chlorine. PCBs contain an appreciable amount of chlorine, making the ECD a sensitive detector for analyzing PCBs.

The range and attenuation of the GC were set to provide 100 percent, full-scale deflection for high-concentration Aroclor standards. This means that the heights of the peaks for these standards were set to reach the top of the chromatographs. The low-concentration Aroclor standards exhibited 10 percent of a full-scale deflection. This means that the peaks reached 10 percent of the distance to the top of the chromatogram. When the samples were analyzed, numerous samples exhibited responses that fell below 10 percent of full-scale deflections. That is, the responses of these samples were below the responses of the low-concentration Aroclor standards. This indicated that the ECD detected levels of PCBs below the low-concentration Aroclor standard of 100 μg/kg. This means that the GC was detecting levels of PCBs below the calculated detection limit.

Of the 146 total samples analyzed during this demonstration, 17 exhibited PCB patterns below the 10 percent deflection. The concentrations of these samples was reported as containing PCBs below 0.4 mg/kg. These values were below the stated detection limit of the FASP PCB Method and were J-coded to indicate that the reported results were estimated. From this information, it appears that the detection limit of the FASP PCB Method may actually be lower than 0.4 mg/kg.

Sample Matrix Effects

Sample matrices were less than ideal, because most of the samples consisted of clay, which caused problems with extraction and analysis. The first set of soil samples extracted was not prepared properly. During the extraction process, anhydrous sodium sulfate was added to these soil samples to remove water from them. The mixture of soil and anhydrous sodium sulfate must be thoroughly mixed to a fine granular texture. Because the operator failed to thoroughly mix the first set of soil samples extracted, there were many large lumps of soil. The PRC lead chemist instructed the operator on how to thoroughly mix the samples. The first set of extractions was then discarded and these samples were reextracted. The operator extracted a second set of soii samples by using the directions provided by the PRC lead chemist. The lead chemist evaluated the mixing process and found it to be acceptable.

Because of the nature of the soils, a fine granular texture could not be achieved for all samples extracted. The operator was instructed to perform the mixing as thoroughly as possible in a reasonable amount of time. Although some samples still exhibited small lumps of soil, this was not viewed as a major problem, because the vortex mixer was able to break these small lumps apart.

The GC analyses of some of the soil samples showed large interference peaks in their chromatograms. About 85 percent of the samples analyzed exhibited these interference peaks. The interference peaks consisted of three peaks. These peaks eluted at retention times of 4.9, 9.8, and 14.4 minutes. The 4.9~minute peak eluted before any of the Aroclors. The 9.8-minute peak eluted in the middle of the Aroclor 1016, 1232, 1242, and 1248 peak patterns, and at the beginning of the Aroclor 1254 peak pattern. The 14.4~minute peak eluted at the end of the Aroclor 1016, 1232, 1242, and 1248 peak patterns; in the middle of the Aroclor 1254 peak pattern; and at the beginning of the Aroclor 1260 peak pattern. The source of the interference peaks was determined to be from the samples and not from any type of laboratory-induced contamination. These interference peaks were not found in any of the reagent blanks analyzed with the GC.

The interference peaks in the chromatograms ranged in size from less than a 100 percent, full-scale deflection to many times above a 100 percent, full-scale deflection. The interference peak that eluted at 14.4 minutes was found in more chromatograms, and appeared to contain a higher concentration, than the other two interference peaks.

Three soil samples showing interference peaks were sent to the EPA Region 7 Laboratory for an EPA SW-846 Method 8270 analysis to determine the identity of the compounds causing the interference peaks. The three samples sent for Method 8270 analysis were samples 026, 052, and 054. No Method 8270 analytes were identified. The laboratory also performed a library search for tentatively identified compounds (TIC). The results of the TICS showed only low concentrations of chlorinated biphenyl congeners, polynuclear aromatic hydrocarbons, and hydrocarbons. These TIC compounds did not appear to match the‘ interference peaks for the three samples analyzed by the FASP PCB Method, because (1) the PCBs identified through the TIC search had already been detected by the FASP PCB Method, and (2) because polynuclear aromatic hydrocarbons and other hydrocarbons do not exhibit responses when analyzed with an ECD.

PRC originally theorized that the interference peaks in the samples analyzed by the FASP PCB Method were phthalates. During the predemonstration activities, Dexsil analyzed the predemonstration samples by using a GC and MS. Dexsil tentatively identified buty l-2-methyl propyl phthalate in some of the predemonstration samples. A subsequent library search indicated a 70 percent probability that this compound would also be present in the demonstration samples.

A review of the predemonstration chromatograms generated by the FASP PCB Method showed that the 14.4-minute interference peak found in the demonstration samples was also present in the predemonstration samples. An assumption was made that the phthalate found by Dexsil was causing this peak. The EPA Region 7 Laboratory's TIC search did not indicate the presence of any phthalates. Therefore, the compounds causing these interference peaks cannot be positively identified. The interference peaks did not prevent PCBs in the soil samples from being identified and quantified, because PCBs consist of many resolved peaks, which elute throughout a relatively wide area of the chromatogram. The interference peaks masked part of some of the Aroclor peak patterns. However, enough of the patterns were unaffected to allow PRC to identify and quantify PCBs.

Another sample matrix effect was the high levels of PCBs found in many of the samples. Any sample that was analyzed and found to contain levels of PCBs above a full-scale deflection of 100 percent required dilution. Of the 146 samples analyzed during the demonstration, 48 required dilution, which is 33 percent. To avoid contaminating the ECD by injecting large amounts of PCBs into the GC, PRC diluted any samples suspected of containing high levels of PCBs before sample

analysis. PRC used two methods that helped the operator determine samples that potentially contained high levels of PCBs First, those samples with highly-colored organic sample extract were diluted before sample analysis. Second, highly-colored sulfuric acid layers were also diluted before sample analysis. However, the operator noted that this visual observation did not always correctly indicate high PCB concentrations. Samples that were found to contain no PCBs in the diluted sample extract were reanalyzed at a more concentrated sample extract solution until either a positive result was obtained or the 20-mL sample extract solution was analyzed.

Sample Throughput

The FASP PCB Method uses an autosampler to increase the number of samples that can be analyzed in 1 day. The autosampler allows samples to be analyzed 24 hours a day. After it is loaded with samples, the autosampler can be operated without an operator present. The FASP PCB Method claimed that the analysis of up to 20 samples per day could be completed. During this demonstration, the most samples analyzed in 1 day by using the FASP PCB Method was 21. The average number of samples analyzed in 1 day was 15 samples.

Linear Range

The linear range of the ECD was not established during the demonstration. PRC established the linear range of the analysis for PCBs by analyzing the three concentrations for each Aroclor standard during the ICAL. The three concentrations used during the ICAL ranged from 100 to 1,000 **µg/kg**. These concentrations correspond with PCB concentrations ranging from 0.4 to 4 mg/kg in the soil samples.

Based on the ICAL Aroclor standard concentrations, any sample that exceeded a PCB concentration above 4 mg/kg required dilution. As discussed previously, 48 samples collected during this demonstration re-quired dilution to obtain Aroclor peaks within the linear range of the calibration. Twenty-three of the samples required a 1: 10 dilution; 12 required a 1: 100 dilution; 11 required a 1: 1,000 dilution; and 2 required a 1: 10,000 dilution. These dilutions were used to obtain the sample results.

The linear range for the FASP PCB Method is comparable to the linear ranges used in formal laboratory analysis. Although this range is very narrow, as indicated by the high number of dilutions, it may be extended upward if the response of the ECD to higher levels of PCBs meets the ICAL. This is a suggested improvement for the FASP PCB Method.

Drift

Drift is a measurement of an instrument's variability in quantifying a known amount of a standard. The drift associated with the FASP PCB Method was evaluated through daily CCALs. For sample analysis to continue, the CCALs were required to meet specific criteria. The responses exhibited by the CCALs were compared with those exhibited by the ICAL. As discussed previously, all but one of the CCALs were found to be acceptable. The reason for the unacceptable CCAL was that the GC injection port septa had worn out. The GC septa was replaced, and a new CCAL was performed. The new CCAL was found to be acceptable, and sample analysis continued. Replacement of GC injection port septa is a normal maintenance requirement for GC analysis. The performance during this demonstration, of more than 400 injections before the septa required replacement indicates that the instrument was not highly susceptible to drift. GC septa replacement is typically done after 100 injections.

Drift of Aroclor retention times was also monitored through CCALs. During the analysis of samples, PRC noticed that the retention times of the Aroclors were shifting. These shifts were small, but they exceeded the acceptable retention time ranges calculated during the ICAL. Shifting of retention times was also observed in the sample chromatograms. PRC compensated for the shifting of retention times by establishing new acceptable retention time ranges. The retention times of the Aroclor peaks for the CCALs were used.

Specificity

Most of the interferences associated with analyzing PCBs by using the FASP PCB Method are attributed to phthalates and sulfur. Phthalates and sulfur produce large, late-eluting peaks, which partially or totally mask PCB patterns. These interferants are found in many environmental samples. Phthalates are also common laboratory contaminants and can be found in many of the plastic materials used in some extraction processes. Using organic solvents to extract PCBs from samples will also extract the phthalates from these plastics. For this reason, plastic materials should not be used in any stage of sample storage, extraction, or analysis.

Interferences may be introduced through other means, such as carrier-gas contamination, carryover from extremely contaminated samples, and sample matrix interferences. Potential for carrier-gas contamination is reduced by using ultra-high, purity-grade gases. The use of carrier gas filters also helps to eliminate these problems. The analysis of highly contaminated samples, followed by the analysis of less contaminated samples,

frequently results in sample carryover contamination. During this demonstration, PRC reduced sample carryover contamination by using disposable glassware. Glassware was replaced after the extraction and analysis of each sample. To reduce contamination of the GC and ECD, PRC analyzed those samples believed to be highly contaminated after the analysis of those believed to be less contaminated. The use of solvent washes added to the GC after the analysis of highly contaminated samples also reduced the amount of sample carryover contamination.

Sample matrix interferences are more difficult to eliminate. Extensive sample cleanup may be required to determine the amount of PCBs in these samples. Common sample matrix interferants present in environmental samples include phthalates, sulfur, halogenated solvents, halogenated pesticides, polar halogenated compounds, and chlorinated paraffins. Although this section discusses a simple cleanup, such may not eliminate all sources of matrix interferences that may be found in environmental samples. For these samples, alternative methods of identifying and quantifying PCBs may be needed. This may include using different peaks for identification and quantitation. In samples in which interferences totally mask the PCB pattern, higher detection limits may need to be reported.

Another source of matrix interference that may be found in samples consists of high PCB concentrations. High concentrations of one Aroclor may prevent the identification or quantitation of other Aroclors. PCBs are mixtures of chlorinated biphenyls. There are 209 congeners of chlorinated biphenyls, differentiated by the number and position of chlorine atoms on the biphenyl compound. PCBs were manufactured as mixtures of chlorinated biphenyls called Aroclors. These Aroclors differ in the percentage of chlorine present. The number nomenclature associated with the Aroclors refers to the percent chlorination. Each Aroclor contains five to 100 different congeners of the chlorinated biphenyl group. This large number of congeners results in many resolved peaks when the Aroclor is analyzed by using an ECD. Each Aroclor exhibits a characteristic peak pattern when analyzed by an ECD. Although the peak pattern of each Aroclor is characteristic, many of the Aroclors exhibit similar peak patterns. Aroclors 1016, 1221, 1232, 1242, and 1248 all exhibit similar peak patterns. These Aroclors contain many of the same peaks when analyzed by using the GC and ECD, but the major differences between these Aroclors is the peak response ratio. When a sample is found to contain one of these Aroclors, the peak responses are evaluated to determine which of the Aroclors that the sample peak pattern most resembles.

If any one of these five Aroclors is found in a sample, identifying and quantifying the other four Aroclors within this group is nearly impossible. When this occurs, the FASP PCB Method will report results for only the Aroclor that is found. The other four Aroclors will not be reported. Aroclors 1254 and 1260 do not present this problem. The peak patterns of these two Aroclors elute in a different region of the chromatogram and can be discerned from Aroclors 1016, 1221, 1232, 1242, and 1248. Aroclor 1254 and 1260 can be identified and quantified when present in a sample containing the other five Aroclois. Aroclor 1254 can also be identified and quantified in samples containing Aroclor 1260 and vice versa.

For many reasons, Aroclor peak patterns may not exactly match those of Aroclor standards. This may be because (1) different manufacturing batches of a specific Aroclor may not have exactly the same ratio of chlorinated biphenyls as the standards, (2) Aroclors can be physically weathered in the environment, and (3) sample interferences may affect the peak pattern.

During this SITE demonstration, PRC measured the specificity of the FASP PCB Method to each of the seven Aroclors. Seven soil samples were spiked with known amounts of each Aroclor. Each sample was spiked with a different Aroclor. First, each sample was divided into four aliquots. All four aliquots were then spiked with an Aroclor at a concentration of about 10 mg/kg. This concentration was chosen, because it is a common cleanup goal at PCB-contaminated sites. Table 6-l shows the results of the Aroclor specificity test.

The Aroclor-spiked samples were identified with nine-digit, alpha-numeric identification numbers. The first three digits of this code refer to the soil sample number. The next four digits, "ARSP," are an abbreviation of the words "Aroclor spike.' The next digit is a letter, A through G, referring to a code used to identity the Aroclor that was used to spike the sample. The last digit is a number, 1 through 4, referring to the aliquot of the sample. To ensure that the results of the assessment were unbiased by operator effects, the operator did not know which Aroclor was used for spiking or the concentration used for spiking.

At the time at which the Aroclor spikes were prepared, the concentrations of the PCBs in the soil samples were not known. Initial indications from the FASP PCB Method were available, but the results had not been finalized. All but two of the samples chosen for the Aroclor specificity test were found to

TABLE 6-1. AROCLOR SPECIFICITY TEST RESULTS.

Sample No.	Soil Sample Result (mg/kg)	Aroclor Spike	Spike Amount (mg/kg)	Spiked Sample Result (mg/kg)	Percent Recovery (%)
003ARSPA1	ND	AR1221	9.86	3.83	39
003ARSPA2	ND	AR1221	9.71	3.75	39
003ARSPA3	ND	AR1221	9.67	3.59	37
003ARSPA4	ND	AR1221	9.73	4.13	42
012ARSPB1	ND	AR1260	10.00	6.40	64
012ARSPB2	ND	AR1260	9.96	7.24	73
012ARSPB3	ND	AR1260	10.02	9.30	93
012ARSPB4	ND	AR1260	9.98	8.80	88
021ARSPC1	ND	AR1232	9.78	30.30	34
021ARSPC2	ND	AR1232	9.94	3.60	36
021ARSPC3	ND	AR1232	10.06	3.60	36
021ARSPC4	ND	AR1232	10.10	2.70	27
034ARSPD1	ND	AR1254	9.75	6.60	68
034ARSPD2	ND	AR1254	9.96	5.20	52
034ARSPD3	ND	AR1254	10.02	5.01	50
034ARSPD4	ND	AR1254	9.84	5.14	52
040ARSPE1	4.6	AR1242	9.92	18.4	139
040ARSPE2	4.6	AR1242	9.96	12.3	78
040ARSPE3	4.6	AR1242	9.92	14.6	101
040ARSPE4	4.6	AR1242	9.75	7.80	33
058ARSPF1	0.58	AR1248	9.77	4.43	39
058ARSPF2	0.58	AR1248	9.94	5.40	48
058ARSPF3	0.58	AR1248	9.71	4.50	46
058ARSPF4	0.58	AR1248	9.94	5.70	52
077ARSPG1	ND	AR1016	9.90	3.40	34
077ARSPG2	ND	AR1016	9.94	4.60	46
077ARSPG3	ND	AR1016	9.98	4.80	48
077ARSPG4	ND	AR1016	9.84	4.30	44

Note:

ND Not detected above the 0.4 mg/kg limit

contain less than 0.4 mg/kg of the Aroclor used for the spike, as determined by the FASP PCB Method.

Sample 077 was spiked with Aroclor 1016. The unspiked soil sample result (1016) reported by the FASP PCB Method was less than 0.4 mg/kg of any Aroclor. The amount of Aroclor 1016 found in the four spiked aliquots ranged from 3.4 to 4.8 mg/kg. Therefore, the percent recoveries obtained for the Aroclor 1016-spiked samples ranged from 34 to 48 percent. From the Aroclor specificity test results, PRC determined that a sample containing 10 mg/kg of Aroclor 1016 can be detected with the FASP PCB Method, but less than half of the amount of the Aroclor spiked into the sample can be detected.

Sample 058 was spiked with Aroclor 1248. The unspiked soil sample result (1248) reported by the FASP PCB Method was 0.58 mg/kg. The amount of Aroclor 1248 found in the four spiked aliquots ranged from 3.9 to 5.1 mg/kg. Therefore, the percent recoveries obtained for the Aroclor 1248-spiked samples ranged from 39 to 52 percent. From these Aroclor specificity 10 mg/kg of Aroclor 1248 can be detected with the FASP PCB Method, but less than 60 percent of the amount of the Aroclor spiked into the sample can be detected.

Sample 040 was spiked with Aroclor 1242. The unspiked soil sample result (1242) reported by the FASP PCB Method was 4.6 mg/kg. The amount of Aroclor 1242 found in the four spiked aliquots ranged from 7.8 to 18.4 mg/kg. The percent recoveries obtained for the Aroclor 1242-spiked samples ranged from 33 to 139 percent. From these specificity test results, PRC determined that a sample containing 10 mg/kg of Aroclor 1242 can be detected with the FASP PCB Method. However, the four reported results varied widely. It is believed that the relatively high concentration of Aroclor 1242 in the soil sample affected the outcome of the specificity test.

Sample 034 was spiked with Aroclor 1254. The unspiked soil sample result (1254) reported by the FASP PCB Method was less than 0.4 mg/kg The amount of Aroclor 1254 found in the four spiked aliquots ranged from 5.0 to 6.6 mg/kg. Therefore, the percent recoveries obtained for the Aroclor 1254-spiked samples ranged from 50 to 68 percent. From these results, PRC determined that a sample containing 10 mg/kg of Aroclor 1254 can be detected with the FASP PCB Method. However, during this specificity test, all of the reported results for this Aroclor were below 7 mg/kg. One possible explanation for this low recovery is that FASP PCB Method found this sample to contain 22.9 mg/kg of Aroclor 1242. This high concentration of Aroclor 1242 may have affected the results of the Aroclor specificity test for Aroclor 1254.

Sample 021 was spiked with Aroclor 1232. The unspiked soil sample result (1232) reported by the FASP PCB Method was less than 0.4 mg/kg. The amount of Aroclor 1232 found in the four spiked aliquots ranged from 2.7 to 3.6 mg/kg. The percent recoveries obtained for these samples ranged from 27 to 36 percent. From the Aroclor specificity test results, PRC determined that a sample containing 10 mg/kg of Aroclor 1232 can be detected with the FASP PCB Method. However, during this specificity test, all of the reported results for this Aroclor were below 4 mg/kg, which is a 40 percent recovery.

Sample 012 was spiked with Aroclor 1260. The unspiked soil sample result (1260) reported by the FASP PCB Method was less than 0.4 mg/kg. The amount of Aroclor 1260 found in the four spiked aliquots ranged from 6.4 to 9.3 mglkg. Therefore, the percent recoveries obtained for these samples ranged from 64 to 93 percent. From the Aroclor specificity test results, PRC determined that a sample containing 10 mg/kg of Aroclor 1260 can be detected with the FASP PCB Method. However, during this specificity test, all of the reported results for this Aroclor were below 10 mg/kg, which is less than a 100 percent recovery.

Sample 003 was spiked with Aroclor 1221. The unspiked soil sample result (1221) reported by the FASP PCB Method was less than 0.4 mg/kg. The amount of Aroclor 1221 found in the four spiked aliquots ranged from 3.6 to 4.1 mg/kg. Therefore, the percent recoveries obtained for the Aroclor 1221-spiked aliquots ranged from 37 to 42 percent. From the Aroclor specificity test results, PRC determined that a sample containing 10 mg/kg of Aroclor 1221 can be detected with the FASP PCB Method. However, during this specificity test, all of the reported results were below 5 mg/kg, which is a 50 percent recovery.

Table 6-2 shows some statistics used to assess the results of the Aroclor specificity test. The statistics detail the mean percent recoveries and standard deviations of the Aroclor spikes in terms of percentages and concentrations.

Another measure of specificity is the ability of an operator to correctly identify Aroclors. The FASP PCB Method uses one column to determine the Aroclor, which is usually sufficient to provide a positive identification, because Aroclors are multiple-component compounds, which exhibit multiple characteristic peak

TABLE 6-2. STATISTICAL ANALYSIS OF THE AROCLOR SPECIFICITY TEST RESULTS.

Sample No.	Aroclor Spike	Mean Percent Recovery (%)	Mean Recovery (mg/kg)	Standard Deviation Recovery (%)	Standard Deviation Recovery (mg/kg)
003ARSPA1-4	AR1221	39.3	3.8	2.1	0.23
012ARSPB1-4	AR1260	79.5	2.1	13.4	1.35
021ARSPC1-4	AR1232	33.3	3.3	4.3	0.42
034ARSPDI-4	AR1 254	55.5	5.5	8.4	0.74
040ARSPE1-4	AR1 242	87.8	8.7	44.3	4.43
058ARSPF1-4	AR1 248	46.3	4.43	5.44	0.64
077ARSPG1-4	AR1016	43.0	4.3	6.22	0.62

patterns when analyzed by the GC and ECD. The characteristic peak patterns give the operator more identification information than single-component compounds. Therefore, it is possible for an experienced operator to correctly identify and quantify PCBs in most soil samples without needing second column confirmation.

PRC evaluated the chromatograms of samples from the demonstration to determine the particular Aroclor present in each sample. The chromatograms were complex and presented some identification problems. The presence of interferants in the samples presented some problems in identifying Aroclors during this demonstration, but these problems were overcome. In some samples, other problems also affected the identification of Aroclors.

One problem was that some samples contained more than one Aroclor. The operator of the FASP PCB Method was initially unaware of this. The presence of two Aroclors in the samples was observed only after a review of the data by the PRC lead chemist. Six samples analyzed by using the FASP PCB Method were found to contain Aroclor 1242 and Aroclor 1260. These samples were 040, 047, 047D. 053, 068, and 078. Because the operator of the FASP PCB Method overlooked the presence of Aroclor 1260 in these samples, their chromatograms were misinterpreted, resulting in incorrect results. The interpretation of PCB chromatograms is a very complex and interpretive procedure, and the chromatograms for these samples were particularly difficult to interpret. It is believed that, if the operator of the FASP PCB Method had been more experienced in interpreting PCB chromatograms, the Aroclor 1260 in the samples would have been identified.

Another Aroclor pattern recognition problem observed during the demonstration was disagreement between the FASP PCB Method and confirmatory laboratory Aroclor identifications for numerous samples did not agree. The demonstration resulted in the analysis of 146 total samples, consisting of 112 samples, 32 field duplicate samples, and 2 PE samples. Of these samples, 101 were determined to contain PCBs by both the FASP PCB Method and the confirmatory laboratory.

The FASP PCB Method identified three different Aroclors in the demonstration. Two samples were found to contain Aroclor 1232, 13 samples were found to contain Aroclor 1248, and 86 samples were found to contain Aroclor 1242. Most of the samples analyzed by the confirmatory laboratory were identified as containing either Aroclor 1242 or Aroclor 1248. Seventy-three samples were identified as containing only Aroclor 1242, and 33 samples were identified as containing only Aroclor 1248. The confirmatory laboratory identified sixteen samples as containing a mix of two Aroclors. The predominant mix consisted of Aroclor 1242 and Aroclor 1248. The were found to contain this mix. Four samples were identified as containing a mix of Aroclor 1242 and Aroclor 1260. Three samples were found to contain a mix of Aroclor 1248 and Aroclor 1260. Two samples were found to contain a mix of Aroclor 1242 and Aroclor 1254.

Of the 122 samples found to contain PCBs by the confirmatory laboratory, only 101 were found to contain PCBs by the FASP PCB Method. Of the 101 samples determined by both the FASP PCB Method and the

confirmatory laboratory to contain PCBs, the two methods identified different Aroclors in 29 samples.

Intramethod Assessment

Reagent blank samples were prepared by taking reagents through all extraction, cleanup, and reaction steps of the analysis. Reagent blanks were prepared with each batch of 20 samples. Ten reagent blanks were prepared and analyzed during the demonstration. No PCBs were detected in any of the blanks, at levels above the detection limit of 0.4 mg/kg. The reagent blanks had no large broad peaks present in their chromatograms. These results indicate that no laboratory-induced contamination was present.

For this demonstration, completeness refers to the proportion of valid, acceptable data generated by using the FASP PCB Method. Seventeen of the results from the samples were reported as below the EPA Superfund FASP PCB method's detection limit. The results from these samples were coded with a "J" to indicate that the reported concentrations were below the detection limit. These points were used in the statistical evaluation of the method. This was consistent with the use of coded data for the other technologies. Completeness for the samples analyzed by the FASP PCB Method during the demonstration was 100 percent.

Surrogate standards are used to determine the extraction efficiency of the FASP PCB Method. Surrogates are added to all standards, blanks, samples, and matrix spikes performed. The surrogate standard used for the FASP PCB Method is decachloro-biphenyl. Decachloro-biphenyl is a PCB; however, it is not a major PCB peak in any of the seven Aroclors analyzed by using standard EPA-approved methods. Surrogate standards evaluated were taken from the original sample extracts. When samples with high PCB concentrations were analyzed, dilutions were often needed. No surrogate recovery was calculated for samples that required dilution. Because of the dilution, the original sample extract could not be analyzed. In all, 187 surrogate recovery values were obtained. The average surrogate recovery was 109 percent. The standard deviation of the surrogate recovery was 4 percent. Under guidelines outlined in the EPA SW-846 Manual Method 8000, control limits for surrogate standards are defined as **f** 3 standard deviations from the mean. For the surrogate standards analyzed during this demonstration, the calculated control limits are from 97 to 121 percent recovery. Two of the 187 surrogate standard recoveries were outside of these control limits. The surrogate standard control limits determined during the demonstration were very narrow. This indicates that the extraction and analysis efficiencies of the method were very good, as determined from the surrogate recoveries. However, these control limits appear too narrow for use during most projects. The FASP PCB Method lists control limits of 50 to 150 percent. These limits may be more reasonable for use on most projects than the limits determined from this demonstration. None of the surrogate standard recoveries during this demonstration exceeded these more typical control limits.

Intramethod accuracy for the FASP PCB Method was assessed through the use of PE samples and matrix spike and matrix spike duplicate samples. Intermethod accuracy was also determined by comparing the results of the method with those of the confiiatory laboratory.

During the demonstration, two PE samples were analyzed by the FASP PCB Method. These samples were extracted and analyzed in exactly the same manner as were all other soil samples. The operator knew that the samples were PE samples but did not know their true values or their ranges. The true result for sample 047-4024-114 (the high-level sample) was 110 mg/kg, with an acceptance range of 41 to 150 mg/kg. The quantitative result for the PE sample, determined by the FASP PCB Method, was 112 mg/kg. This is only 2 mg/kg above the true value and is well within the acceptance range of the PE sample. The percent recovery of this analysis was 102 percent. The true result for sample 047-4024-113 (the low-level sample) was 32.7 mg/kg. It had an acceptance range of 12 to 43 mg/kg. The result for the PE sample, when analyzed by the FASP PCB Method, was 20.3 mg/kg. This value is well within the acceptance range. The percent recovery of this analysis was 62 percent.

Matrix spike samples, prepared by adding a known quantity of PCB Aroclor 1242 to an actual sample, were used to evaluate the extraction and analysis efficiency of the technology. They were also used to determine accuracy. Matrix spike samples were prepared by adding a known quantity of PCBs to a sample. The PCB used for the matrix spike samples was Aroclor 1242. Enough of a concentrated Aroclor 1242 standard was added to a l0-gram soil sample to produce a matrix spike concentration of 2 mg/kg. The spiked sample was also duplicated to produce a matrix spike duplicate sample. Six matrix spike samples and six matrix spike duplicate samples were extracted and analyzed by using the FASP PCB Method. The recoveries for these samples ranged from 0 to 214 percent. Two of the six soil samples used as matrix spikes contained PCBs at concentrations of 6.1 and 10.4 mg/kg. Though these are low concentrations of PCBs, they are significant when compared to the 2 mg/kg concentration used to spike the samples. Therefore, these original PCB concentrations

may have affected the matrix spike recoveries observed. In the case of Sample 102, which contained 10.4 mg/kg of PCBs before being spiked, one of the two percent recovery values obtained was 0 percent. Using this result in the statistical evaluation of these data would have significantly widened the range of acceptable recoveries. For this reason, this value was not used in the statistical evaluation. The other 11 results were used for the statistical evaluation. The average recovery for these sample results was 122 percent, or 2.44 mg/kg. The standard deviation was 48 percent, or 0.96 mg/kg. Based on these data, control limits for matrix spike recovery samples were established. Under guidelines outlined in the EPA SW-846 Manual Method 8000, control limits can be established as ± 2 standard deviations from the mean percent recovery. For the matrix spike samples analyzed during this demonstration, the calculated control limits are from 26 to 218 percent recovery. All matrix spike samples analyzed-except Sample 102-fell within these control limits.

PRC assessed precision for this method by comparing the two results obtained. For this demonstration, three types of precision data were generated: data from laboratory duplicate samples, data from field duplicate samples, and data from matrix spike duplicate samples. Table 6-3 provides matrix spike duplicate results. Table 6-4 presents laboratory and field duplicate sample results.

Laboratory duplicate samples are two analyses performed on a single sample delivered to a laboratory. Originally, laboratory duplicate samples were to be analyzed with each set of 20 samples submitted for analysis. However, additional laboratory duplicates were analyzed for the purpose of evaluating whether the homogenization of the samples was complete. In all, 14 pairs of laboratory duplicate samples and their respective soil samples were analyzed by using the FASP PCB Method. The initial analyses of the duplicate samples ranged from 0.14 to 1,790 mg/kg. When the analysis was duplicated, the results ranged from 0.07 to 2,030 mg/kg. Field duplicate samples were also analyzed during this demonstration. Field duplicates are two samples collected together but brought to the laboratory with separate sample numbers. PRC collected 32 field duplicate samples during this demonstration. Each sample and its duplicate was analyzed by each 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. The laboratory duplicates would be compared to a window of acceptable values; if one fell outside that window, the laboratory would take corrective action. Field duplicates would be used to (1) ensure that samples were not being contaminated during sample collection, and (2) set boundaries of variance resulting from heterogeneity inherent in soil.

However, PRC was tasked not with determining the precision of those collecting the samples or of the laboratory, but with determining the precision of the technology. 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 and its duplicate. To control the problems usually detected by laboratory duplicates, PRC used only one operator for the technology. PRC assumed that any variance in that operator's laboratory techniques would be the same for each sample and, therefore, statistically insignificant, as discussed in Section 4. 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 soil samples indicate that, overall, this technique worked as discussed in Section 4. The homogenization appears to have been incomplete in only a very few samples.

The 46 duplicate pairs were used together to evaluate intramethod precision. Even the best technology that determines results quantitatively cannot reproduce its results every time. PRC established control limits 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, PRC removed all sample pairs that did not produce two positive results from the data population. The RPD for each sample pair was then calculated, and the mean RPD and population standard deviation were determined. The lower control limit was set at zero, indicating the results from a duplicate and its sample matched perfectly. The upper control limit was set by multiplying the standard deviation by two and adding the product to the mean RPD. The RPD of each sample pair was then compared to these control limits. Each was expected to fall within them. If greater than 95 percent fell within this range, the technology's precision was considered adequate. If fewer than 95 percent of them fell within this range, the data were reviewed; if no explanation was found, the technology's precision was considered inadequate.

The FASP PCB Method detected PCBs in both the sample and its duplicate in 34 of the 46 sample pairs. The data from these 34 pairs had a mean RPD of 34 percent and a standard deviation of 29. Therefore, the RPDs for the duplicate pairs fell within the control

TABLE 6-3. 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 (%)	Relative Percent Difference (%)
047-4024-003	ND	2	73	86	16
047-4024-026	0.43	2	168	156	7
047-4024-042	0.73	2	89	93	4
047-4024-062	6.1	2	175	214	20
047-4024-086	0.43	2	114	121	6
047-4024-102	10.4	2	0	51	200

Note:

ND Not detected above the 0.4 mg/kg detection limit.

limits. Therefore, the technology's precision was considered acceptable.

Matrix spike duplicate samples were used to further evaluate the precision of this method. The matrix spike duplicate results were compared to the matrix spike results for this precision evaluation. 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 values for the six sets of matrix spike samples ranged from 4 to 200 percent. The RPD results for Sample 102 were removed from the precision evaluation. As discussed previously, the RPD from this matrix spike sample and its duplicate may have been affected by the original amount of PCB in the sample. Including it in the statistical analysis would have skewed the acceptable range for the RPDs. The RPDs for the remaining five matrix spike samples and their duplicates ranged from 4 to 20 percent. The mean RPD for these samples was 11 percent, and the standard deviation was 7 percent. Using an upper control limit of two times the standard deviation resulted in an upper control limit of 25 percent. All RPD values for the matrix spike duplicate samples—except that of Sample 102—fell within this range.

Comparison of Results to Confirmatory Results

The following subsections compare the accuracy and precision of the data from analyses conducted by using both the FASP PCB Method and the confirmatory laboratory. The results from the confirmatory laboratory are considered accurate, and its precision is considered acceptable. Table 6-5 and Figure 6-1 summarize the results.

Accuracy

To measure the accuracy of the FASP PCB Method, PRC compared the data from this method with the data from the confirmatory laboratory by using linear regression techniques and the Wilcoxon Signed Ranks Test (detailed in Section 4). Linear regression produces a coefficient of determination, also called an r^2, which defines whether a relationship exists between two sets of data. The best relationship possible would be expressed as an r^2 of 1.0. For this demonstration, r^2 values of 0.80 to 1.0 were considered acceptable.

If a relationship exists, linear regression also defines an equation that shows the relationship between the two sets of results. That equation can be expressed as a line on a graph. This line represents where the results of one set of data would be expected if the other set of data was given. Because the results from the technology and the results from the confirmatory laboratory were expected to match, that line should have a slope of 1 and a y-intercept of zero.

PRC used a normal deviate test statistic to determine whether either the slope or the y-intercept varied, at a two-tailed 95 percent confidence level, from those expected. If the slope or y-intercept of the regression line varied greatly, the two sets of data were considered comparable but not the same. That is, the technology's data were not accurate, but there was a relationship between the technology's data and the confirmatory laboratory's data. This relationship would enable PRC

TABLE 6-4. 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 (%)
047-4024-004 LD	2.8	7.2	88	047-4024-063 FD	0.27J	0.11J	84
047-4024-012 LD	ND	ND	NA	047-4024-063 LD	0.27J	0.29J	7
047-4024-013 LD	0.18J	0.38J	71	047-4024-069 FD	ND	ND	NA
047-4024-014 LD	0.33J	0.24J	32	047-4024-071 FD	ND	ND	NA
047-4024-015 FD	19.0	8.9	72	047-4024-081 FD	0.75	0.40	61
047-4024-015 LD	19.0	14.2	29	047-4024-082 FD	ND	ND	NA
047-4024-016 LD	737	816	10	047-4024-083 FD	0.24J	0.22J	9
047-4024-017 LD	2.3	2.0	14	047-4024-084 FD	8.1	3.2	87
047-4024-018 LD	163	188	14	047-4024-085 FD	501	468	7
047-4024-019 LD	8.9	12.9	37	047-4024-085 LD	501	449	11
047-4024-022 FD	ND	ND	NA	047-4024-086 FD	0.43	1.0	80
047-4024-024 FD	ND	ND	NA	047-4024-087 FD	0.20J	0.19J	5
047-4024-028 FD	0.17J	0.15J	13	047-4024-088 FD	1.6	1.6	0
047-4024-033 LD	5.1	7.1	33	047-4024-090 FD	0.81	0.84	4
047-4024-035 FD	ND	ND	NA	047-4024-091 FD	1790	2030	13
047-4024-037 FD	ND	ND	NA	047-4024-092 FD	0.50	0.19	90
047-4024-042 FD	0.73	0.78	7	047-4024-095 FD	35.9	47.5	28
047-4024-043 FD	25.3	28.7	13	047-4024-097 FD	0.14J	0.07J	67
047-4024-046 FD	ND	ND	NA	047-4024-098 FD	2.4	1.9	23
047-4024-047 FD	ND	ND	NA	047-4024-100 FD	669	1,000	40
047-4024-048 LD	ND	ND	NA	047-4024-101 LD	2.3	1.9	19
047-4024-050 FD	1.3	2.5	63	047-4024-102 FD	10.3	12.6	20
047-4024-060 FD	1.5	1.6	6	047-4024-109 FD	ND	ND	NA

Notes:

FD	Field duplicate
J	Reported amount is below detection limit.
LD	Laboratory duplicate
NA	Not analyzed
ND	Not detected above the 0.4 mg/kg detection limit

to correct the technology's results mathematically if a specified number of samples were sent to a confirmatory laboratory.

All three of these linear regression factors-the slope, the y-intercept, and the r^2 factor-had to be considered acceptable before a technology would be considered accurate.

The linear regression for the FASP PCB Method was based on results from 81 samples. The other results indicated that no PCBs were detected at levels above the

TABLE 6-5. COMPARISON OF DATA OBTAINED BY FASP PCB METHOD AND CONFIRMATORY LABORATORY.

Sample No.	FASP PCB Method 0.40 mglkga	Confirmatory Laboratory 0.033 mg/kga	Difference	Relative Percent Difference	Sample No.	FASP PCB Method 0.40 mg/kg[a]	Confirmatory Laboratory 0.033 mglkga	Difference	Relative Percent Difference
001	5.98	0.593	5.39	164	033	5.10	6.OOJ	-0.90	16.2
002	1.27	1.50	-0.23	16.6	034	22.9	34.0	-11.1	39.0
003	ND	0.114	NA	NA	035	ND	ND	NA	NA
004	2.77	6.71 J	-3.94	83.1	D35D	ND	ND	NA	NA
005	1.80	1.37	0.43	27.1	036	5890	816	5074	151
006	0.37J	0.679	-0.31	58.9	037	ND	0.055J	NA	NA
007	0.12J	0.552	-0.43	130	037D	ND	0.04OJ	NA	NA
008	3.27	2.00	1.27	48.2	038	1210	103OJ	180	16.1
009	2.37	1.3OJ	1.07	58.3	039	0.53	0.676	-0.15	24.2
010	1.13	0.17J	0.96	147	040	4.63	4.25	.38	8.7
011	1.05	1.15J	-0.10	9.1	041	ND	ND	NA	NA
012	ND	ND	NA	NA	042	0.73	0.517	0.21	34.2
013	0.18J	1.13	-0.95	145	042D	0.78	0.462J	0.32	51.2
014	ND	0.18	NA	NA	043	25.3	1.69J	23.6	175
015	19.0	9.13	9.87	70.2	043D	28.7	1.74	27.0	177
015D	8.89	9.84	-0.95	10.1	044	0.16J	0.592J	0.43	115
016	737	2,110	-1373	96.5	045	ND	ND	NA	NA
017	2.32	2.55	-0.23	9.4	046	ND	ND	NA	NA
018	163	45.4	118	113	046D	ND	ND	NA	NA
019	8.90	6.70	2.20	28.2	047	ND	0.094J	NA	NA
020	ND	0.07J	NA	NA	047D	ND	0.098J	NA	NA
021	ND	0.063	NA	NA	048	ND	ND	NA	NA
022	ND	0.535	NA	NA	049	ND	ND	NA	NA
0220	ND	0.718	NA	NA	050	1.29	3.60	-2.31	94.5
023	18.2	20.8	-2.6	13.3	0500	2.52	4.41	-1.89	54.5
024	ND	0.055	NA	NA	051	ND	ND	NA	NA
024D	ND	0.049	NA	NA	052	1.99	4.21	-2.22	71.6
025	12.8	11.7	1.1	9.0	053	1.20	0.958	0.24	22.4
026	0.43	1.96	-1.53	128	054	1.04	0.516J	0.52	67.4
027	ND	0.057	NA	NA	055	2.32	2.40	-0.08	3.2
028	0.17J	0.216	-0.05	23.8	056	0.52	0.505	0.02	2.9
028D	0.15J	0.224J	-0.07	39.6	057	ND	ND	NA	NA
029	ND	0.229J	NA	NA	058	0.58	0.681	-0.10	16.0
030	1.0l	1.15	-0.014	13.0	059	9.59	7.86	1.73	19.8
031	0.14J	0.263	-0.12	61.0	060	1.47	0.624J	0.85	80.8
032	18.9	47.6	-28.7	86.3	060D	1.55	0.577	0.97	91.5

TABLE 6-5 (Continued). COMPARISON OF DATA OBTAINED BY FASP PCB METHOD AND CONFIRMATORY LABORATORY.

Sample No.	FASP PCB Method 0.40 mg/kg[a]	Confirmatory Laboratory 0.033 mg/kg[a]	Difference	Relative Percent Difference	Sample No.	FASP PCB Method 0.40 mg/kg[a]	Confirmatory Laboratory 0.033 mg/kg[a]	Difference	Relative Percent Difference
061	745	580	165	24.9	085D	468	465	3	0.6
062	6.12	2.35	3.77	89.2	086	0.43	1.42	-0.99	107
063	0.27J	0.092J	0.18	98.3	086D	1.04	1.25	-0.21	18.3
063D	0.11J	0.154J	-0.04	33.3	087	0.20J	0.076	0.12	NA
064	94.4	19.0	75.4	133	087D	0.19J	ND	NA	NA
065	3.39	3.08	0.31	9.6	088	1.59	2.70	-1.11	51.7
066	2.59	1.98	0.61	26.7	088D	1.58	1.77	-0.19	11.3
067	ND	0.081	NA	NA	089	46.6	45.0	1.6	3.5
068	0.26J	0.504J	-0.24	63.9	090	0.81	1.01	-0.20	22.0
069	ND	ND	NA	NA	090D	0.84	1.40	-0.56	50.0
069D	ND	ND	NA	NA	091	1790	1630	160	9.2
070	ND	ND	NA	NA	091D	2030	1704	326	17.5
071	ND	0.052J	NA	NA	092	0.50	1.21	-0.71	83.0
071D	ND	ND	NA	NA	092D	0.19J	ND	NA	NA
072	ND	0.035J	NA	NA	093	ND	0.295	NA	NA
073	42.9	15.8	27.1	92.3	094	0.42	0.362J	0.06	14.8
074	9.50	13.3	-3.8	33.3	095	35.9	17.5	18.4	68.9
075	15.8	23.0	-7.2	37.1	095D	47.5	31.2	16.3	41.4
076	38.3	46.7	-8.4	19.8	096	ND	0.059J	NA	NA
077	ND	ND	NA	NA	097	0.14J	1.23	-1.09	159
078	2.25	2.27	-0.02	0.7	097D	0.07J	0.285	0.22	121
079	**81.1**	**42.8**	**38.3**	**61.8**	098	**2.41**	**1.17**	**1.24**	**69.3**
080	**2.00**	**3.77**	**-1.77**	**61.4**	098D	**1.85**	**0.825**	**1.03**	**76.6**
081	**0.75**	**0.687**	0.06	**8.8**	099	**ND**	**ND**	**NA**	**NA**
081D	**0.40**	**0.450**	**-0.05**	**11.8**	100	**669**	**177**	**492**	**116**
082	ND	ND	NA	NA	100D	1000	167	833	143
082D	ND	0.244	NA	NA	101	2.33	1.21	1.12	63.3
083	0.24J	0.484	-0.24	67.4	102	10.3	293	-283	186
083D	0.22J	0.413	-0.19	61.0	102D	12.6	1.77	10.8	151
084	8.09	1.16	6.93	150	103	207	40.3	167	135
084D	3.15	1.08	2.07	97.9	104	12.4	7.66	4.7	47.3
085	501	428	73	15.7	105	ND	0.210	NA	NA

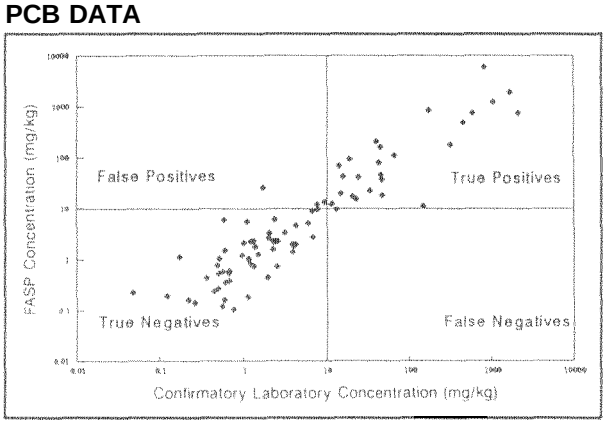
TABLE 6-5 (Continued). COMPARISON OF DATA OBTAINED BY FASP PCB METHOD AND CONFIRMATORY LABORATORY.

Sample No.	FASP PCB Method 0.40 mg/kg[a]	Confirmatory Laboratory 0.033 mg/kg[a]	Difference	Relative Percent Difference	Sample No.	FASP PCB Method 0.40 mg/kg[a]	Confirmatory Laboratory 0.033 mg/kg[a]	Difference	Relative Percent Difference
106	0.71	2.50	-1.79	112	110	ND	ND	NA	NA
107	69.3	14.1J	55.2	132	111	ND	ND	NA	NA
108	1.41	3.84J	-2.43	92.6	112	177	315	-138	56.1
109	ND	ND	NA	NA	113	20.3	14.9	5.4	30.7
109D	ND	ND	NA	NA	114	112	66.3	45.7	51.3

Notes:

[a] Detection limit.
J Reported amount is below detection limit or not valid by approved QC procedures.
NA Not analyzed.
ND PCBs not detected above the detection limit.

FIGURE 6-1. CONFIRMATORY DATA VS. FASP PCB DATA



detection limit. The r^2 value for the regression was 0.31, indicating that there was no relationship between the FASP PCB Method's results and those from the confirmatory laboratory. However, an analysis of regression residuals identified that the r^2 was greatly influenced by Samples 16, 36, 38, 91, and 100. All of these samples show high levels of contamination. PRC removed these five sample results as outliers and recalculated the linear regression. The second analysis, calculated on the remaining 76 sample results, defined an r^2 factor of 0.86, indicating a relationship between the two sets of data. It defined a regression line with a y-intercept of 3.57 mg/kg and a slope of 1.09. The normal deviate test statistic indicated that these results are not statistically different from those expected. This means that linear regression of me FASP PCB Method's results, after the outliers had been removed, produced an acceptable r^2, slope, and y-intercept. Therefore, it is accurate. No correction of the method's data would be needed. The FASP PCB Method's results can be expected to be similar to those of a confirmatory laboratory.

The Wilcoxon Signed Ranks Test was used to verify these results, It indicated, at a 95 percent confidence level, that the data from the FASP PCB Method were not significantly different than data from the confirmatory laboratory. This also indicates that the EPA Superfund FASP Method's data are accurate.

Precision

To compare the precision of the FASP PCB Method's results to the precision of the confirmatory laboratory's results, PRC performed a Dunnett's Test on the RPDs determined from the field duplicates and their respective soil 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, it can be assumed that the precisions are also similar. The 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 were considered the same. Lower probabilities indicate that one cannot be sure that the precisions are the same. However, this does not mean that the technology's precision is worse than that

of the confirmatory laboratory, only that there was a greater probability that they were different.

When the Dunnett's Test compared the RPDs between the FASP PCB Method's data and the confirmatory laboratory's data, a probability of 91.0 percent resulted. This indicates that the FASP PCB Method's precision and the confirmatory laboratory's precision may not be the same.

Section 7
Applications Assessment

The FASP PCB Method produces accurate and precise quantitative results. It is capable of identifying each specific Aroclor and is not affected by interferants under most circumstances. The method produces very few false positive or negative results. It is relatively portable and can be set up at most hazardous waste sites. However, the necessary equipment must be operated indoors, and electricity and refrigeration are required. One to 2 days may be required for equipment set-up and calibration before analysis of samples can begin.

The FASP PCB Method requires the one-time purchase of analytical equipment. After this equipment has been purchased, the method is less expensive than formal laboratory analysis, and is capable of providing higher sample throughput and quicker turnaround time than formal laboratories. The method can produce high-quality data within 1 to a few hours of sample collection.

The method is similar to standard EPA analytical methods used by formal laboratories. For this reason, experienced, highly trained personnel are required to operate it. The linear range of the method is not as great as that of other PCB field-screening technologies, and dilutions must be used to produce quantitative results for samples with high concentrations of PCBs.

The results of the Aroclor specificity test indicated that the FASP PCB Method is capable of accurately and precisely quantifying different Aroclors.

The FASP PCB Method would be useful at any PCB-contaminated site at which quantitative results were needed quickly. The method would be particularly useful in characterizing large potentially contaminated areas in which false positives could significantly affect the costs of a project. The method is also particularly useful at sites at which the presence of interferants is known or suspected or at which the Aroclors of concern are not positively known.

Section 8
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