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CLOR-N-SOIL PCB TEST KIT, DEXSIL CORP.

INNOVATIVE TECHNOLOGY EVALUATION REPORT

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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.

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Abstract

This innovative technology evaluation report (ITER) presents the evaluation of two field screening technologies for determining polychlorinated biphenyl (PCB) contamination in soil. The demonstration was conducted by PRC Environmental Management, Inc. (PRC), under contract to the Environmental Protection Agency's (EPA) Environmental Monitoring Systems Laboratory-Las Vegas (EMSL-LV).

The Clor-N-Soil Test Kit and the L2000 PCB/Chloride Analyzer, both developed by the Dexsil Corporation, were demonstrated in August 1992 in Kansas City, Missouri. The Clor-N-Soil Test Kit is designed to provide semiquantitative results for PCBs in soil. It provides a greater than or less than 50 milligrams per kilogram (mg/kg) result. Compounds containing organic chlorine, such as PCBs, are extracted from the soil samples; through a series of chemical steps, the chloride ions are stripped from the compound, transferred to an aqueous solution, and mixed with a reagent to induce a color change. Because the test kit reacts to all sources of organic chlorine, the presence of some compounds other, than PCBs will cause it to produce false positive results. The presence of sulfur in samples may produce the same result. During this demonstration, the test kit produced 87 correct assays, 58 false positives, and 1 false negative. Because the test kit reacts with the chlorine in PCBs, it will produce different responses to individual Aroclors, each of which contains a different percentage of chlorine. The test kit will produce false negative results for samples containing Aroclors 1016, 1221, and 1232, which contain lower percentages of chlorine than Aroclor 1260, the Aroclor the kit is designed to detect.

Like the Clor-N-Soil Test Kit, the L2000 PCB/Chloride Analyzer uses the principle of total organic chlorine detection. The analyzer, though, uses a chloride-specific electrode to measure the amount of total organic chlorine in the extract. The analyzer also is capable of electronically converting the chloride concentration to produce quantitative results for two different Aroclors. The detection limit of the analyzer is reported to be 5 mg/kg, although a detection limit of 2 mg/kg was used during this demonstration. Because the analyzer reacts with the chlorine in PCBs, it will produce various responses to individual Aroclors. The analyzer has a high likelihood of producing false negative results for samples containing Aroclors 1016, 1221, and 1232, which contain lower percentages of chlorine than the Aroclors the analyzer is set to detect. If, however, the Aroclor type is known prior to analysis, the analyzer can be set to "total chlorine" and the result divided by the appropriate factor (0.21 for Aroclor 1221, 0.32 for Aroclor 1232, and so forth.) During this demonstration the analyzer's precision was found to be acceptable after reviewing its performance on duplicate samples. To assess its accuracy, PRC used a linear regression approach to compare the analyzer's data to corresponding confirmatory laboratory data. This analysis was based on 47 matched pairs of positive sample results. For this regression analysis, the r^2 factor was 0.86, indicating that a relationship existed between the two data sets. The analysis defined a regression line with a y-intercept of 26.6 mg/kg and a slope of 0.84. These results indicate that the analyzer is not accurate, but can be corrected mathematically.

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

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List of Abbreviations and Acronyms

AICO	Abandoned Indian Creek Outfall
CCAL	continuing calibration
CLP	Contract Laboratory Program
CMS	corrective measure study
CRQL	contract required quantitation limit
DOE	Department of Energy
DQO	data quality objective
ECD	electron capture detector
EMSLLV	Environmental Monitoring Systems Laboratory-Las Vegas
EPA	Environmental Protection Agency
ERA	Environmental Research Associates
GC	gas chromatograph
ICAL	initial calibration
IDW	investigation-derived waste
ITER	Innovative Technology Evaluation Report
KCP	Kansas City Plant
LCD	liquid crystal display
µg/kg	micrograms per kilogram
meq	milliequivalents
mg	milligram
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
mL	milliliter
MMTP	Monitoring and Measurement Technologies Program
MS	mass spectrometer
MSDS	material safety data sheet
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
QAPjP	quality assurance project plan
r^2	correlation coefficient
RCRA	Resource Conservation and Recovery Act
RFI	RCRA facility investigation
RPD	relative percent difference
SARA	Superfund Amendments and Reauthorization Act of 1986

List of Abbreviations and Acronyms (Continued)

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

Acknowledgements

This demonstration and the subsequent preparation of this report required the services of numerous personnel from the Enviromnental Protection Agency, Enviromnental Monitoring Systems Laboratory (Las Vegas, Nevada); Environmental Protection Agency, Region 7 (Kansas City, Kansas); Dexsil Corporation (Hamden, Connecticut); 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.

Section 1
Executive Summary

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

The two technologies selected for this demonstration and evaluation were the Clor-N-Soil Test Kit and the L2000 PCB/Chloride Analyzer, both developed by the Dexsil Corporation. They were demonstrated and evaluated in August 1992 in Kansas City, Missouri. The demonstration and evaluation of these two innovative technologies were conducted in conjunction with the demonstration and evaluation of two other field screening methods, the EnviroGard PCB Test produced by Millipore , Inc., and the Field Analytical Screening Program PCB Method developed during the Field Investigative Team Contract under the EPA Superfund Program. The demonstration and evaluation of these two other technologies are discussed in separate ITERs.

The findings of the demonstration of the two technologies manufactured by Dexsil are summarized below.

Clor-N-Soil Test Kit

The Clor-N-Soil Test Kit is designed to quickly provide semiquantitative results for PCB concentrations in soil samples. The test kit used for this demonstration provides a greater than or less than 50 milligrams per kilogram (mg/kg) result using the principle of total organic chlorine detection. Compounds containing organic chlorine, such as PCBs, are extracted from the soil sample using an organic solvent. Then, through a series of chemical steps, the chloride ions are stripped from the PCB compound and transferred to an aqueous solution. The extract is then mixed with a reagent to induce a color change that corresponds to the number of chloride ions in the sample. Assuming that all chloride ions detected in the sample come from PCBs, it is possible to determine whether PCBs are present at concentrations above a particular level. Because the test kit reacts to all sources of organic chlorine, the presence of chlorine-containing compounds other than PCBs will cause the kit to produce false positive results. The presence of sulfur in samples may produce the same result.

The Clor-N-Soil Test Kit is portable, easy to operate, and useful under limited site conditions. Depending on how the test kit is ordered from the developer, the cost of this technology ranges from $10 to $14 per analysis. The average time required to perform one analysis during the demonstration was found to be 11 minutes.

To ensure that the test kit always produces a positive result for samples containing at least 50 mg/kg of PCBs, the kit is designed with a correction factor. This correction factor accounts for any losses of chloride during its extraction from the sample. Because of this correction factor, the test kit is likely to produce a high number of false positive results when PCBs are present at concentrations below, but near, the detection level. During this demonstration, the test kit produced 87 correct assays, 58 false positives, and 1 false negative.

The false negative occurred when the test kit determined that less than 50 mg/kg of PCBs was present in the sample, but the confirmatory laboratory indicated that the sample contained 293 mg/kg. Despite sample homogenization, though, the confirmatory laboratory indicated a level of 1.77 mg/kg in a field duplicate of that sample. This single false negative occurred from the analysis of a sample that appears as an outlier in all other technology data evaluations.

Because the Clor-N-Soil Test Kit reacts with the chlorine in PCBs, it will produce different responses to

individual Aroclors, each of which contains a different percentage of chlorine. Because of this, the test kit will produce false negative results for samples containing Aroclors 1016, 1221, and 1232, which contain lower percentages of chlorine than Aroclor 1260, the Aroclor the kit is designed to detect.

PRC evaluated the test kit's precision by analyzing duplicate samples. Thirty-two field duplicate samples were analyzed by the Clor-N-Soil Test Kit during the demonstration. The results indicated that the test kit was able to duplicate its results 81 percent of the time. A review of the results that did not match showed that the Clor-N-Soil Test Kit appeared to have more difficulty duplicating its results when the PCB concentrations were near 1 mg/kg.

To evaluate the test kit's accuracy, PRC determined whether each confirmatory laboratory result was above or below 50 mg/kg. The test kit's results and the confirmatory laboratory's results were then evaluated using a 2 by 2 contingency table and the Fisher's Test statistic. Results from this analysis indicated that there was no correlation between data from the test kit and data from the confirmatory laboratory. This suggests that the Clor-N-Soil Test Kit is not accurate. However, this absolute assessment of accuracy may not affect the test kit's usefulness. The test kit's inaccuracies were primarily the result of false positive results, and this type of inaccuracy would, at worst, result in the misidentification of clean material as contaminated. To eliminate the effects of false positive and false negative results, all critical samples should be confiied using EPA-approved methods.

L2000 PCB/Chloride Analyzer

The L2000 PCB/Chloride Analyzer is designed to quickly provide quantitative results for PCB concentrations in soil samples. Like the Clor-N-Soil Test Kit, the analyzer uses the principle of total organic chlorine detection. The principal difference between the Clor--N-Soil Test Kit and the L2000 PCB/Chloride Analyzer is the way total organic chlorine is detected after the sample is extracted. The analyzer uses a chloride--specific electrode to measure the amount of total organic chlorine in the extract and displays the results on a screen. The analyzer also is capable of electronically converting the chloride concentration to produce quantitative results for two different Aroclors. Like the Clor-N-Soil Test Kit, the L2000 PCB/Chloride Analyzer reacts to all sources of organic chlorine, and the presence of chlorine-containing compounds other than PCBs will cause the analyzer to produce false positive results.

The analyzer is very portable, although electricity is required to operate it. It is easy to operate. During this demonstration, the analyzer often needed to be re-calibrated, particularly when analyzing samples with high PCB concentrations. The analyzer is sold with enough reagents to perform 200 analyses at a cost of $3,500. Additional reagents also can be purchased. Depending on the quantity ordered, the cost of additional reagents ranges from $8 to $10 per analysis. The amount of time required to perform one complete sample analysis during the demonstration averaged nine minutes. The detection limit of the analyzer is reported by its developer to be 5 mg/kg, although a detection limit of 2 mg/kg was used during this demonstration.

Because the L2000 PCB/Chloride Analyzer reacts with the chlorine in PCBs, it will produce various responses to individual Aroclors. The analyzer has a high likelihood of producing false negative results for samples containing Aroclors 1016, 1221, and 1232, which contain lower percentages of chlorine than the Aroclors the analyzer is set to detect. If, however, the Aroclor type is known prior to analysis, the L2000 PCB/ChIoride Analyzer can be set to "total chlorine" and the result divided by the appropriate factor (0.21 for Aroclor 1221, 0.32 for Aroclor 1232, and so forth.)

To assess the analyzer's precision, PRC evaluated its performance in analyzing both laboratory and field duplicate samples. The L2000 PCB/Chloride Analyzer had 18 sample pairs in which both the sample and its duplicate had positive results. PRC used the data from the duplicate analyses to establish precision control limits. The determination of precision was based on the percentage of duplicate sample pairs that had relative percent differences (RPD) within these control limits. The precision control limits were set at 0 and 77.4 percent RPD. All but one of the 18 sample pairs' RPDs fell within the control limits. This one failure caused the analyzer's overall precision to be 94.5 percent. The goal for precision for this evaluation was between 95 and 100 percent. While the 94.5 percent is not between 95 and 100 percent, the analyzer could not have come closer to 100 percent without every sample pair falling within the control limits; therefore, the precision was considered acceptable.

To evaluate the analyzer's accuracy, PRC used a linear regression approach to compare the analyzer's data to the corresponding confiitory laboratory's data. This analysis was based on 47 matched pairs of positive sample results. For this regression analysis, the r^2 factor was 0.86, indicating that a relationship existed between the two data sets. The analysis defined a regression line

with a y-intercept of 26.6 mg/kg and a slope of 0.84. These results indicate that the analyzer is not accurate, but can be corrected mathematically. In addition to the regression approach, PRC used a nonparametric test statistic, the Wilcoxon Signed Ranks Test, to verify the regression evaluation. It also indicated, at a 95 percent confidence level, that the analyzer's data was significantly different from that of the confiiatory laboratory.

Based on these results, the L2000 PCB/Chloride Analyzer's results should not be expected to be the same as those from a confirmatory laboratory. However, if 10 to 20 percent of the samples collected also are sent to a confirmatory laboratory, then the results from the other to 90 percent can be corrected. This may result in a significant savings in analytical costs.

Section 2
Introduction

This ITER summarizes the procedures used to demonstrate the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer. It discusses the results of the demonstration and evaluates the effectiveness and possible uses of these two innovative technologies at various hazardous waste sites. The primary goal of the demonstration was to evaluate these technologies and to provide Superfund decisionmakers with adequate reliable information on their performance and cost effectiveness.

EPA's Site Program and MMTP: An Overview

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

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

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

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

The Role of Monitoring and Measurement Technologies

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

Candidate technologies may come from within the federal government or from the private sector. Through the program, developers are provided with the opportunity to rigorously evaluate the performance of their technologies. Finally, by distributing the results and recommendations of those evaluations, the market for the technologies is enhanced.

Defining the Process

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

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

Components of a Demonstration

Once a decision has been made to demonstrate technologies to meet a particular EPA need, the MMTP performs a number of activities. First, 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 insure that the technology meets the parameters of the demonstration. Then, after evaluation of the information, all respondents are told whether they have been accepted into the demonstration or not. While participants are being identified, potential sites also are identified, and basic site information is obtained. These activities complete the initial component of an MMTP demonstration.

The next component. and probably the most important component, is the development of plans that describe how various aspects of the demonstration will be conducted. A major part of the EPA's responsibility is the development of a demonstration plan, quality assurance project plan (QAPjP), and a health and safety plan. While the EPA pays for and has the primary responsibility for these plans, each is developed with input from all of the demonstration's participants. The plans define how activities will be conducted and how the technologies will be evaluated. MMTP also provides each developer with site information and often predemonstration samples so the developer can maximize the field performance of its innovative technology. Generally, the developers train demonstration personnel so that performance is not based on special expertise. This also insures that potential users have valid information on training requirements and the types of operators who typically use a technology successfully.

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

The final component of an MMTP demonstration consists of reporting the results and insuring distribution of demonstration information. The primary product of the demonstration is an ITER, like this one, which is peer-reviewed and distributed as part of the technology transfer responsibility of the MMTP. The ITER fully documents the procedures used during the field demonstration, QA/QC results, the field demonstration's results, and its conclusions. A separate QA/QC data package also is made available for those interested in evaluating the demonstration in greater depth. Two-page "Technical Briefs" are prepared to summarize the demonstration results and to insure 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 EPAdesignated operators. The MMTP does not provide any funds to developers for costs associated with preparation of equipment for demonstration or for development, and it does not cover the costs developers incur to demonstrate their products.

Demonstration Purpose, Goals, and Objectives

For this demonstration, the two innovative technologies produced by the Dexsil Corporation were evaluated for their accuracy and precision in detecting high and low levels of PCBs in soil samples, and the ef-fects, if any, of matrix interferences on the technologies. The

high and low levels of PCBs in soil samples, and the effects, if any, of matrix interferences on the technologies. The accuracy and precision of the technologies were tistically compared to the accuracy and precision attained in a conventional, fixed laboratory using standard EPA analytical methods. The technologies also were qualitatively evaluated for the length of time required for analysis, ease of use, portability, and operating cost.

Section 3
Predemonstration Activities

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

Identification of Developers

EMSL-LV identified the Clor-N-Soil PCB Test Kit and L200O PCB/Chloride Analyzer as showing promise for use in PCB field screening. After a review of available data on these technologies, EMSL-LV concluded that they warranted evaluation under the MMTP.

Site Selection

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

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

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

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

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

PCBs are the only significant contaminant at the site. Samples from 12 borings were analyzed for priority pollutants other than Only one of these borings contained non-PCB priority pollutants. This

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

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

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

Selection of Confirmatory Laboratory and Method

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

Operator Training

The Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer each was demonstrated by a PRC employee. Prior to the demonstration, the operators were trained in the use of the two technologies. This training included a review of operating procedures and instructions provided by Dexsil and informal field training conducted by Dexsil at the start of the demonstration. Training was equivalent to that recommended by Dexsil for actual site characterization projects.

Sampling and Analysis

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

This predemonstration sampling was conducted so that Dexsil could refine its technologies and revise its operating instructions, if necessary, before the demonstration. This sampling also allowed potential matrix effects or interferences to be evaluated prior to the demonstration. The principal finding from predemonstration sampling was that the soil at the AICO site was more clayey than expected which made homogenizing the samples difficult.

Section 4
Demonstration Design and Description

This section describes the sample collection procedures and the experimental design used to evaluate the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer. These innovative technologies were evaluated in conjunction with two other field screening technologies that also screen for PCBs in soil. The demonstration design and description, and the experimental design described in this section were common to all four evaluations. The four evaluations also shared a single demonstration plan and QAPjP. Key elements of the QAPjP (PRC 1992b), field analysis operations, and data management activities are summarized in this section.

Sample Collection

For the demonstration, 112 soil samples and 32 field duplicate samples were collected from the AICO site. Each sample was thoroughly homogenized and then split into six replicate samples. One replicate from each sample was submitted to the confirmatory laboratory for analysis using the CLP 1990 SOW method. A second replicate was submitted to EMSL-LV for separate analysis at the request of the EPA technical project manager (TPM), although the data generated by EMSL-LV was not used in this demonstration. A third replicate was analyzed in the field using the Clor-N-Soil Test Kit. A fourth was analyzed in the field using the L2000 PCB/Chloride Analyzer. The remaining replicates were analyzed in the field using the two other technologies described in separate ITERs.

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

Samples were collected from areas known to exhibit PCB concentrations ranging from not detected (at a concentration of 0.16 mg/kg) to 9,680 mg/kg Most of the samples were collected from areas previously identified as containing PCBs in the not detected to 100 mgkg range, for two reasons. First, this range encompasses typical regulatory thresholds for PCBs, such as the 10 mg/kg level for cleanups in unrestricted access areas and the 50 mg/kg level for cleanups in industrial areas. Second, most of the four field screening technologies demonstrated, including the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer, were designed primarily for operation in this range.

Twenty samples were collected from areas previously identified as containing PCBs at concentrations ranging from 100 to 1,000 mg/kg. An additional 20 samples were collected from areas previously identified as containing PCBs at concentrations between 1,000 and 10,000 mg/kg. These samples were analyzed to evaluate the abilities of the field screening technologies to monitor PCBs in higher concentrations as well as in the average range.

After collection, soil samples were placed in plastic bags and thoroughly homogenized. Samples were then split and placed in sample containers. Samples to be submitted for confirmatory laboratory analysis were placed in 8-ounce, wide-mouth glass jars with teflon-lined lids. Samples for submittal to EMSL-LV and for analysis by the field screening technologies were placed in 4-ounce, wide-mouth glass jars with teflon-lined lids.

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

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

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

To apply the matched pair Student's t-test, it was necessary to have a normally distributed data population. The differences between confirmatory laboratory samples and their respective duplicates were statistically evaluated and found to be normally distributed. Two data point outliers were noted in the frequency plot. Samples 91 and 102, respectively, were the low and high outliers. The Student's paired t-test, however, was found acceptable even when the outliers were included in the data set.

The statistical analysis indicates that the homogenization was acceptable, but even at a 95 percent confidence level, a few anomalous duplicate results can exist in a data set without the analysis being greatly affected. For example, a single pair of samples such as 102 and 102D 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 a limited number of poorly homogenized samples were included in the demonstration. The analysis of such data could produce limited cases of inaccurate data. For this reason, a large number of samples were collected and analyzed to prevent any anomalous samples from affecting the overall results.

Quality Assurance Project Plan

To ensure that all activities associated with this demonstration met the demonstration objectives, a QAPjP was prepared (PRC 1992b). The QAPjP, 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 QAPjP, and ultimately, all participants agreed to its content.

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

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

Representativeness refers to the degree to which the data accurately and precisely represents the condition or characteristic of the parameter represented by the data (Stanley and Vemer 1983). In this demonstration, representativeness was ensured by executing a consistent

sample collection, homogenization, and handling program. Representativeness also was ensured by using each technology at its optimum capability to provide results that represented the most accurate and precise measurements it was capable of achieving.

Completeness refers to the amount of data collected from a measurement process compared to the amount that was expected to be obtained (Stanley and Vemer 1983). For this demonstration, completeness refers to the proportion of valid, acceptable data generated using each of the technologies and the confiiatory laboratory. The completeness objective for each technology during this demonstration was 90 percent, which was achieved.

Comparability refers to the confidence with which one data set can be compared to another (Stanley and Verner 1983). The main focus of this demonstration was to compare data generated by the Clor-N-Soil Test Kit, the L2000 PCB/Chloride Analyzer, and the other technologies with confirmatory laboratory results. Additional QC for comparability was achieved by analyzing QC samples, blanks, and Aroclor standards, and by adhering to standard EPA analytical methods and standard operating procedures (SOP) for preparing samples and operating instruments.

Accuracy refers to the difference between the sample result and the reference or true value for the sample. Bias, a measure of the departure from complete accuracy, can be caused by variations in instrument calibration, loss of analyte in the sample extraction process, interferences, and systematic contamination or carryover of analyte from one sample to the next.

One purpose of the demonstration was to assess the accuracy of the Clor-N-Soil Test Kit and the L2000 PCB/Chloride Analyzer. The accuracy of the Clor-N-Soil Test Kit and L2000 PCB/Chloride Analyzer are detailed in Sections 6 and 7. The accuracy DQO for the confirmatory laboratory was achieved and is discussed in more detail in Section 5.

Precision refers to the degree of mutual agreement among individual measurements and provides an estimate of random error. Precision for this demonstration was measured by comparing the RPDs of samples and their duplicates to control limits established through the statistical methods detailed in Section 4. Determining the precision of the Clor-N-Soil PCB Test Kit and L2000 PCB/Chloride Analyzer was one of the objectives of this demonstration. Data on the precision of the technologies is detailed in Sections 6 and 7. The precision DQO for the confirmatory laboratory was achieved and is discussed in Section 5.

Experimental Design

The primary objective of the demonstration was to evaluate the Clor-N-Soil Test Kit, the L2000 PCB/Chloride Analyzer, and two other field screening technologies for determining PCB contamination in soil. This evaluation included defining the precision, accuracy, cost, and range of usefulness for each technology. This objective also included determining theDQOs that each technology was capable of achieving. A second objective was to evaluate the specificity of each technology to different Aroclors.

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

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

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

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

The Aroclor expected to be found at the AICO site was Aroclor 1242, which is a common mix of PCBs. However, there are other common Aroclors as well. In

the planning stages of this demonstration, interest was shown in the cross reactivity between Aroclors for each technology. To assess this factor, cross reactivity for each technology was evaluated through the use of matrix spikes for each of the seven Aroclors (1016, 1221, 1232, 1242, 1248, 1254, and 1260) typically analyzed using standard EPA analytical methods. This information was then used to determine the sensitivities of the technologies to each Aroclor.

Statistical Analysis of Results

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

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

In cases where field duplicate samples were collected, the demonstration plan stated that the results of the two duplicates would be averaged and 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 equation used was:

(4-1)

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

where

RPD = relative percent difference.

R_i = initial result.

R_d = duplicate result.

The RPDs were then compared to upper and lower control limits. Because the technologies being demonstrated were themselves being assessed, the control limits used were calculated from data provided during this investigation. To determine these control limits, the standard deviation of the RPDs was calculated for each technology. This standard deviation was then multiplied by two and added to its respective mean RPDs. This established the upper control limit for the technology. Because an RPD of zero would mean that the duplicate samples matched their respective samples perfectly, zero was used as the lower control limit. This resulted in a large range of acceptable values. Because duplicate analyses seldom match perfectly, even for established technologies, all samples that fell within the control liits were considered acceptable. PRC determined that if at least 95 percent of the duplicate samples fell within these control limits, the technology had acceptable precision.

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

Linear regression was calculated for the technologies that were capable of determining quantitative results. One of those was the L2000 PCB/Chloride Analyzer. PRC calculated this data by the method of least squares. Calculating linear regression in this way makes it possible to determine whether two sets of data are reasonably related, and if so, how closely. Calculating linear regression results in an equation that can be visually expressed as a line. Three factors are determined during calculations of linear regression. These three factors are the y-intercept, the slope of the line, and the correlation coefficient, also called an r^2. All three of these factors had to have acceptable values before a technology's accuracy was considered acceptable.

The r^2 expresses the mathematical relationship between two data sets. If the r^2 is one, then 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. This skewing may sometimes occur because technologies are often more accurate when analyzing samples in one range than when analyzing samples in another range. In particular, samples with either very high or very low levels of contamination

often skew the results. For this demonstration, the technique used to identity outliers that might have skewed the results was residual examination (Draper and Smith 1981). The computer program used for calculating the linear regression, in fact, identified most of the outliers. When outliers were identified, they were removed and linear regression was calculated again.

If the corrected data set resulted in an r^2 between 0.80 and 1, then the regression line's y-intercept and slope were examined to determine how closely the two data sets matched. A slope of one and a y-intercept of zero would mean that the results of the technology matched those of the confirmatory laboratory perfectly. Theoretically, the farther the slope and y-intercept differ from these expected values, the less accurate the technology. Still, a slope or y-intercept can differ slightly from 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, then the technology's data was determined to be 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, then 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 confiiatory laboratory. Analysis of a percentage of the samples by a confirmatory laboratory would provide a basis for determining a correction factor. Still, only in cases where the r^2, the y-intercept, and the slope were all found to be acceptable did PRC determine that the technology was accurate.

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

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

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

Two of the field screening technologies demonstrated produce semiquantitative results. One of those was the Clor-N-Soil Test Kit manufactured by Dexsil. Linear regression analysis and the Wilcoxon Signed Ranks Test cannot be used to compare semiquantitative results. Instead, PRC used a 2 by 2 contingency table and a Fisher's Test. The Fisher's Test determines whether both data sets are correlated. When used in a two-tailed manner, as it was in this case, its formula is usually conservative. Therefore, use of a modified Chi-square formula is recommended (Pearson and Hartley 1976). This formula, as used in this demonstration, is:

(4-2)

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

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

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

During this demonstration, values of 95 percent or better indicated that the precisions were statistically the same.

It should be noted that results below 95 percent do not mean that the precision of the technology was not acceptable, only that it may be different from the precision of the confirmatory laboratory. In particular, Dunnett's Test has no way of determining whether or not any difference between the two data sets actually resulted because a technology's data was more precise than the confirmatory laboratory's.

Field Analysis Operations

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

Section 5
Confirmatory Analysis Results

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

Confirmatory Laboratory Procedures

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

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

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

In addition to being generally discussed in this section, all of the confirmatory laboratory results used to assess the two innovative technologies produced by Dexsil are presented in tables in Sections 6 and 7.

Soil Sample Holding Times

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

Soil Sample Extraction

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

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

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

Initial and Continuing Calibrations

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

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

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

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

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

Sample Analysis

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

Quantitation of PCBs is performed by measuring the response of the peaks in the sample to those same peaks identified in the ICAL standard. The reported results of this calculation are based on dry weights, as required by the CLP 1990 SOW method. Because the 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 defined as 16 times the response of the Aroclor standards analyzed during the ICAL. Once a sample is diluted to within the linear range, it is analyzed again. Dilutions were performed when appropriate on the samples for this demonstration.

Detection Limits

One concentration of each Aroclor was analyzed during the ICAL. The concentration of each Aroclor standard should correspond to the Contract Required Quantitation Limit (CRQL) when corrected for the sample extraction concentration factors. The concentration used for Aroclor 1221 was 200 micrograms per kilogram (pglkg); the level used for the other six Aroclors was 100 µg/kg. This corresponds to soil sample detection limits of 67 µg/kg for Aroclor 1221 and 33 µg/kg for the other Aroclors.

Because of CLP 1990 SOW method requirements, these detection limits are based on samples that have no moisture content. Because almost all soil samples contain moisture, the detection limits stated above are raised to correct for the percent moisture present in the soil sample. However, PRC did not correct the detec-

tion 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 Dexsil PCB technologies and the other technologies were listed in mg/kg. Even when corrected to account for percent moisture, the CRQLs would be significantly below the detection limits for each technology.

Quality Control Procedures

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

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

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

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

Second Column Confirmation

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

The CLP 1990 SOW states that results from the two columns should be within 25 percent of each other. When this requirement is not met, the result for that sample must be coded to indicate that the results are estimated. For the analysis of the samples from this demonstration, 17 sample results were above the 25 percent requirement of the CLP 1990 SOW. These results were J-coded to indicate that the results were estimated, but were not validated by approved QC procedures. Finally, following the CLP 1990 SOW method 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 CLP 1990 SOW requires that when pesticides or PCBs are present in samples at sufficient quantities, they must be confirmed by GC and MS analysis. Twenty samples from this demonstration contained sufficient quantities of PCBs to require GC and MS conflation. These samples were compared to Aroclor standards. None of the 20 samples were confirmed through GC and MS analysis. Lack of GC and MS confirmation is not uncommon for Aroclors because they are a mixture of congeners, and the GC and MS analysis is better suited for identifying individual congeners. Because all 20 samples were confirmed on the second GC column, the lack of GC and MS confirmation was determined to be insignificant during the EPA Level II data review. Therefore, these samples were not coded.

Data Reporting

The data report PRC received from the EPA Region 7 Laboratory included a standard EPA Region 7 Analysis Request Report. were the only compounds reported. Results were reported on a dry weight basis, as required in the CLP 1990 SOW. PRC obtained data on the percentage of solids in the sample from the

confiiatory laboratory and used this data to convert the results to wet weight. This conversion was required because the data was to be compared to data from the two Dexsil technologies and two other technologies, all of which reported concentrations based on wet soil weight. PRC also converted the confiitory laboratory results from **µg/kg** to mg/kg.

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

Aroclors Reported by the Confirmatory Laboratory

According to RF1 and CMS results from April 1989, the only Aroclor believed to be present at the AICO site was Aroclor 1242. However, the confirmatory laboratory found three additional Aroclors in the samples collected during the demonstration. Most of the samples analyzed by the confirmatory laboratory were found to contain either Aroclor 1242 or Aroclor 1248. Seventy-three samples were found to contain only Aroclor 1242, while 33 samples were found to contain only Aroclor 1248. Sixteen samples were found to contain mixtures of two of the four Aroclors found. The predominant mixture was Aroclor 1242 and Aroclor 1248. Seven samples were found to contain this mixture. Four samples were found to contain a mixture of Aroclor 1242 and Aroclor 1260. Three samples were found to contain a mixture of Aroclor 1248 and Aroclor 1260. Two samples were found to contain a mixture of Aroclor 1242 and Aroclor 1254. In all, 122 soil samples submitted to the confiitory laboratory for this demonstration were found to contain detectable levels of PCBs. Twenty-four samples were reported as not containing PCBs above the CRQLs.

Data Quality Assessment of Confirmatory Laboratory Data

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

Accuracy

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

The two PE samples contained different concentrations, one low and one high. These samples were extracted and analyzed in exactly the same manner as the other soil samples. The confirmatory laboratory knew that the samples were PE samples, but the true concentrations and acceptance ranges of the samples were not known to the confirmatory laboratory. The true concentration of sample 047-4024-l 14 (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 confiitory laboratory was 67 mg/kg of Aroclor 1242, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 61 percent. The true value concentration of sample 047-4024-113 (the low-level sample) was 32.7 mg/kg, with an acceptance range of 12 to 43 mg/kg. The result reported by the confirmatory laboratory for this sample was 15 mg/kg, which was within the acceptance range. The percent recovery of this sample by the confirmatory laboratory was 46 percent. Based on the results of the PE samples, the accuracy of the confirmatory laboratory was acceptable.

Precision

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

The evaluation of field duplicate sample results was used to assess the precision of the analytical method. Precision can be evaluated by determining the RPDs for sample results and their respective field duplicate sample results. The RPDs for the 32 field duplicates and their respective samples averaged 31.8 percent, but this included two pairs of samples with extremely dissimilar results. Sample 102 had a result of 293 mg/kg while its duplicate, Sample 102D. had a result of 1.77 mg/kg. The RPD for the sample pair was calculated as 197.6 percent. Also, Sample 97 had a result of 1.23 mg/kg while its duplicate had a result of 0.285 mg/kg. The RPD for Sample 97 and 97D was 124.8 percent. The

other RPDs, though, had much lower percentages. Without these two samples, the mean RPD fell to 20 percent. Overall, this data shows excellent agreement between the samples and their respective field duplicates, indicating a high degree of precision by the confirmatory laboratory. The mean RPD also indicated that the method used to homogenize the samples before splitting them for analysis was highly effective.

Completeness

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

Use of Qualified Data for Statistical Analysis

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

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

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

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

Section 6
Dexsil Corporation: Clor-N-Soil Test Kit

This section provides information on the Clor-N-Soil Test Kit, including background information, operational characteristics, performance factors, a data quality assessment, and a comparison of its results with those of the confirmatory laboratory. Observations about the technology made during the demonstration by the operator are also presented throughout this section.

Theory of Operation and Background Information

According to its developer, the Clor-N-Soil Test Kit is designed to provide quick, semiquantitative analytical results of PCB concentrations in soil samples. During this demonstration, the Clor-N-Soil Test Kit yielded a greater than or less than 50 mg/kg result for PCB concentrations in soil samples. The 50 mg/kg level of contamination is a common EPA cleanup goal for PCBs in soil.

The Clor-N-Soil Test Kit operates on the principle of total organic chlorine detection. Sample extraction is required before total organic chlorine detection is performed. PCB compounds are extracted from the soil sample using butyl diglyme. Dexsil uses this solvent to extract PCBs from soil samples because of its ability to permeate soil pores more readily than other common extraction solvents. This results in greater PCB extraction efficiency. Once the PCBs are extracted, the extract is free from sources of inorganic chlorine, water, soil particles, and some potential interferences, namely compounds containing polar chlorine.

The organic sample extract is treated with metallic sodium to strip chlorine from the biphenyl compound. This reaction is performed with the aid of a catalyst containing a mixture of naphthalene and diglyme. Chlorine exists in the form of chloride ions as a result of the stripping process. An acidic buffer solution is added to the organic sample extract to quench any unreacted sodium and to transfer the chloride into an aqueous phase. The aqueous phase extract containing the chloride is transferred to an indicator tube where total organic chlorine detection is performed.

The chloride content in the aqueous phase is measured with an indicating solution of mercuric nitrate and diphenyl carbazone, which results in a colorimetric determination of chloride content. Mercuric nitrate disassociates in the aqueous phase and binds to any free chloride. Diphenyl carbazone is then added to the aqueous phase where it reacts with any unbound mercury ions. The diphenyl carbazone and mercury complex results in a vivid purple color. The development of the purple color is inversely proportional to the chloride content of the aqueous phase. The purple color indicates an absence of chloride, therefore, the absence of PCBs in the soil sample. A yellow or clear color indicates the presence of chloride, therefore, the presence of PCBs in the soil sample. The amount of mercuric nitrate added to the aqueous phase will determine the concentration at which a color change will occur.

The amount of mercuric nitrate added to the aqueous phase extract is determined by calculating the amount of chlorine found in a soil sample containing a predetermined amount of Aroclor 1242. The Clor-N-Soil Test Kit used during this demonstration was designed to provide results greater than or less than 50 mg/kg. Kits designed to operate at this level use enough mercuric nitrate to completely react with the chlorine present in a soil sample containing 44 mg/kg of Aroclor 1242. This accounts for possible operator error and avoids the reporting of false negative results. Therefore, soil samples containing 44 mg/kg or more of Aroclor 1242 are identified as containing greater than 50 mg/kg of PCBs.

Other sources of organic chlorine also will be detected using this test kit, and may mistakenly be identified as PCBs. Sources of organic chlorine other than that are commonly found in environmental samples include chlorinated solvents, organochlorine pesticides, and chlorinated disinfectants.

Operational Characteristics

The Clor-N-Soil Test Kit is contained in a portable, 5-ounce cardboard box. This box is used to store all items needed to perform an analysis, and doubles as a test tube rack for holding the different test tubes required for the analysis. The box contains (1) a stainless-steel scoop, (2) a hand-held scale, (3) a plastic pipet bulb, (4) an extraction test tube, (5) organic extraction solvent, (6) a filter-syringe assembly, (7) a reaction test tube containing two ampules, (8) an indicator test tube containing two ampules and 7 mL of buffer solution, and (9) complete step-by-step instructions, which included a color chart for use in determining the presence or absence of PCBs.

Logistical requirements for the Clor-N-Soil Test Kit are minimal because it is primarily self-contained. No electrical power is needed to analyze samples. The glassware and reagents needed for the analysis are included in the test tubes or other containers supplied in the cardboard box.

Supplies needed, but not included with the test kit, include (1) a permanent marker for labeling the cardboard box and test tubes, (2) a table or work space area at least 8 square feet in size, (3) a refrigerator for storing samples, (4) a logbook or a report form for recording sample results, and (5) an ink pen. The refrigerator is an optional piece of equipment if all samples are analyzed on the same day they are collected. However, it is recommended for storing samples overnight or for storage until it is determined which of the samples will be transported to a formal laboratory for further analysis. A refrigerator is not required for storing reagents. Safety equipment, such as gloves, a laboratory coat, and safety glasses are also recommended.

The Clor-N-Soil Test Kit is easy to operate. Dexsil claims that the test kit can be used by anyone, including nontechnical personnel. Based on this demonstration, PRC concluded that Dexsil' s claim is true and that the test kit can be used by individuals who have little or no technical expertise. The test kit instructions provide detailed descriptions and proved to be invaluable to the operator. The operator did note, however, that a certain amount of laboratory skill, such as the ability to transfer and accurately measure liquids, is needed during one step of the analysis. This step was made easier, however, by the marked test tubes included with the test kit indicating the volume of liquid needed.

The Clor-N-Soil Test Kit is designed for use in the field. The test kit contains no instrumentation or mechanical parts. Most components are made of plastic or steel. Two exceptions are tbe organic extraction solvent vial, which is made of glass, and the crushable glass ampules contained in both the plastic reaction test tube and the plastic indicator test tube. This equipment must be stored with some care so that it is not crushed or broken.

Instrument reliability was evaluated through the number of test kits that were received in good working order. The demonstration required the use of 198 Clor-N-Soil Test Kits. Of the 198 kits used, three contained bent hand-held scales which could not be used, two contained organic extraction solvent vials without an aqueous phase component, one contained a reaction tube that did not contain the crushable ampules, and six contained reaction or indicator test tubes that leaked due to cracks or caps which did not screw down properly. Also, one reaction test tube unexpectedly foamed and overflowed when the buffer solution was added. Based on these findings, the reliability of the Clor-N-Soil Test Kit was determined to be 93 percent. It should be noted that the six test kits with the reaction and indicator test tubes that leaked, and the reaction test tube which overflowed, may have been the result of sample or operator effects. This would change the instrument reliability for the Clor-N-Soil Test Kit to 97 percent.

The Clor-N-Soil Test Kit contains seven different chemicals. The chemicals used include flammable solvents, such as naphthalene, diglyme, butyl diglyme, and ethanol. Each Clor-N-Soil Test Kit contains only small amounts of these chemicals but care should be taken when using them to prevent personal exposure and fire.

To free the chloride from the biphenyl group, metallic sodium is used. Metallic sodium can react explosively with water. The amount of metallic sodium in the Clor-N-Soil Test Kit is 50 milligrams (mg). Dexsil' s material safety data sheets (MSDS) for the test kit explained that the small amount of metallic sodium included with the test kit was not explosive and that water was suitable for extinguishing any sodium-induced fire. Tbis is probably true, unless large numbers of the test kits are stored together.

The test kit contains a florisil cleanup column. Plorisil is a fine dust and can be a skin and eye irritant. The operator observed frequent leakage from the florisil columns into the packaging material. A small amount of mercuric nitrate is included in the test kit. Mercury is used to bind free chloride ions liberated from the biphenyl group. The small amount of mercury contained in the test kit (less than 0.5 mg) should not be a major safety factor; however, adequate ventilation in the work area is recommended.

Finally, the Clor-N-Soil Test Kit requires that crushable glass ampules be broken within the confines of a plastic test tube. The operator received a small puncture wound to the thumb while crushing one of these ampules. Apparently, a piece of glass went through the plastic test tube and the operator's gloves. This was considered an uncommon occurrence. Still, care should be taken when crushing these ampules.

The operator chosen to analyze samples using the Clor-N-Soil Test Kit was Mr. Brad Helland, an employee of PRC. Mr. Helland has a B.S. degree in chemistry and some graduate work. He also spent three years as a research technician and a laboratory technician.

Mr. Helland's training in the use of the Clor-N-Soil Test Kit included one hour of hands-on training, which was conducted by the demonstration's lead chemist. Additional instruction was provided to Mr. Helland on the first day of the demonstration by Mr. Stephen Finch of Dexsil. This training included Mr. Finch overseeing Mr. Helland performing each step of the Clor-N-Soil Test Kit. Mr. Finch also provided information on filtration rates and separation techniques for troublesome sample extractions. According to Mr. Helland, some of this information was not included in the test kit instructions. After analyzing 10 demonstration samples under supervision, Mr. Helland noted that he felt confident in his ability to properly analyze soil samples using the Clor-N-Soil Test Kit. Mr. Helland was able to analyze six samples concurrently using the test kit.

Costs associated with analyzing samples using the Clor-N-Soil Test Kit include the cost of the kit, the cost of the operator, and the cost of waste disposal. Dexsil sells its test kits individually, in packs of 12, or in cases. A case contains four packs. The price for one pack is $168, which is $14 per kit. The price for one case is $576, which is $144 per pack or $12 per kit. Dexsil also offers reduced prices when larger quantities are purchased. The shelf life of each test kit is one year. Operator costs for the use of the Clor-N-Soil Test Kit will vary depending on the technical level of the operator. The Clor-N-Soil Test Kit can be used by individuals with little technical training, thereby decreasing this cost. The waste generated by using the 198 test kits used during this demonstration half-filled a 55-gallon drum. The appropriate way to dispose of this waste is through an approved PCB incinerator facility. The cost of disposing of one drum of this waste is estimated at $1,000.

Performance Factors

The following sections describe the Clor-N-Soil Test Kit's performance factors. These factors include detection limits and sensitivities, sample throughput, linear range, and drift. Specificity is another important performance factor. Due to the complexity of specificity, it is discussed separately.

Detection Limits and Sensitivity

The Clor-N-Soil Test Kit reports semiquantitative results for the presence of PCBs in soil samples. The means of determining whether PCBs are present is the color of the sample following extraction and subsequent chemical reactions. According to Dexsil, a vivid purple color indicates that the PCB concentration in the soil sample is below 50 mg/kg. A clear or yellow color indicates that the PCB concentration of the soil sample is above 50 mg/kg. A color chart is included with each Clor-N-Soil Test Kit to help the operator determine whether the soil sample contains PCBs above or below 50 mg/kg.

The operator of the Clor-N-Soil Test Kit indicated that comparing the final color of the soil sample extract to the color chart was difficult when the resulting color fell between the light purple and the yellow colors shown on the chart. When this occurred during this demonstration, the operator reported that the sample contained concentrations of PCBs above 50 mg/kg. This conservative approach reduced the likelihood of false negative results and increased the chance of false positive results.

The detection limit of 50 mg/kg is determined by the amount of mercury provided in the test kit. The mercury is in the form of mercuric nitrate. During the indicating step, the mercuric nitrate is introduced into the aqueous soil sample extract where it is liberated from the nitrate group to form a mercury ion (Hg^{++}). The mercury ions bind to the free chloride ions from the sample extraction and reaction steps. The calorimetric step of the test kit involves binding diphenyl carbazone to any mercury ions not bound to chloride ions. The diphenyl carbazone-mercury mixture results in a purple color indicating the absence of chloride ions, thus the absence of PCBs.

A discussion with Mr. Finch of Dexsil indicated that 1.65×10^{-3} milliequivalents (meq) of the mercury ion are available for reaction. The predominant form of mercury chloride formed during this reaction is

[HgCl] + because the reaction is performed under acidic conditions (Snoeyink and Jenkins 1980). One chloride ion from the PCBs in the soil sample will react with one mercury ion from the indicating step. Using this stoichiometric relationship, it will take 1.65 x 10^{-3} meq of chloride ions to bind with the 1.65 x 10^{-3} meq of mercury in the Clor-N-Soil Test Kit. When this amount of chloride is in a soil sample extract, there will be no mercury left to bind to the diphenyl carbazone. This will result in a clear or yellow color in the final sample extract, indicating the presence of PCBs.

Dexsil determined the 50 mg/kg detection limit using Aroclor 1242, the predominant Aroclor found at the AICO site. The detection limit was stoichiometrically calculated using the following equation:

(6-1)

$$[(1.69 \times 10^{-3}\ meq/0.8) \times 7/5] \times 2) \times 35.5\ mg/meq = 0.21\ mg\ of\ chloride\ needed$$

where

1.69×10^{-3} meq = chloride ions react with all mercury ions.

0.8 = 80 percent extraction efficiency correction factor.

7/5 = ratio of buffer solution added versus buffer solution used.

2 = ratio of organic extraction solvent added versus extraction solvent used.

35.5 mg/meq = milliequivalent formula for chloride.

In this case, if a l0-gram soil sample containing 50 mg/kg of Aroclor 1242 is extracted and analyzed using the Clor-N-Soil Test Kit, a positive result would be expected. This is because the IO-gram sample would contain 50 mg/kg of Aroclor 1242 or 0.5 milligrams of Aroclor 1242. Aroclor 1242 contains 42 percent chlorine by weight or 0.21 milligrams of chloride. The 0.21 mg is equivalent to 5.92 x 10^{-3} meq chloride. The lo-gram soil sample is extracted with 10 mL of solvent, but only 5 mL of the solvent is used in the reaction and indicating steps of the test kit. The amount of chloride is reduced by half or 2.96 x 10^{-3} meq. The reaction step of the Clor-N-Soil Test Kit uses 7 mL of the buffer solution to transfer the chloride to the aqueous phase. Of the 7 mL of buffer solution added, only 5 mL are used in the indicating step. This results in a reduced number of chloride ions available for reaction with the mercury ions. This five-sevenths reduction of the chloride leaves 2.11 x 10^{-3} meq of chloride.

To ensure that the Clor-N-Soil Test Kit will give a positive result in a soil sample containing 50 mg/kg of Aroclor 1242, Dexsil uses a correction factor. This factor is used to account for any losses of chloride during the extraction steps of the test kit. Dexsil uses a correction factor of 80 percent. Twenty percent was experimentally determined to be the largest amount of chloride that can be lost during the extraction step. Multiplying the number of expected chloride ions by 80 percent results in 1.69 x 10^{-3} meq of chloride should the 20 percent be lost. This amount of chloride would react completely with the 1.65 x 10^{-3} meq of mercury ion in the Clor-N-Soil Test Kit resulting in a positive test result.

FCBs were manufactured as mixtures of chlorinated biphenyls known as Aroclors. These Aroclors were given numbers for identification, such as Aroclor 1242 or Aroclor 1260. The last two numbers indicate the percentage of chloride present in the Aroclor on a weight-to-weight basis. Aroclor 1016 is the exception. Its last two identification numbers do not indicate the percentage of chlorine present in the Aroclor. Aroclor 1016 contains 41 percent chloride by weight. With this information it is possible to determine the detection limits of the Clor-N-Soil Test Kit for each of the seven major Aroclors. This can be accomplished by determining the mass of chloride needed to react with all of the mercury provided in the Clor-N-Soil Test Kit. The following equation is used:

(6-2)

$$(0.21\ mg/percent\ chloride\ of\ Aroclor) \times 0.01\ kg = detection\ limit\ for\ each\ Aroclor$$

Sample Matrix Effects

The matrix of the soil samples analyzed during the demonstration was less than ideal. Most of the samples consisted of clay, which caused some problems in extracting and analyzing them. The most common problem was that a colloidal suspension formed for some of the samples after the initial extraction. The colloidal suspension prevented the recovery of the 7 mL of organic extract needed for the florisil column cleanup. The 7 mL was the amount of organic sample extract required to recover 5 mL after the florisil column cleanup. No guidance was given in the test kit' s instructions on how to handle this problem.

Centrifugation would have been the ideal solution to this problem; however, a centrifuge was not provided or recommended with the test kit, and one was not available at the trailer. To obtain results from samples with colloidal suspension, another attempt to extract the sample was made using less of the soil sample. Instead of using the recommended 10 grams, 5 grams were used. If 2.5 mL of the sample extract was obtained, this

amount was taken through the rest of the sample analysis procedure. In some cases this did not solve the problem, so the sample was again extracted using 2.5 grams. When the amount of sample analyzed was reduced from 10 grams, the detection limit' for that sample was raised to account for the difference in the sample weight or volume extracted and analyzed. For these samples, the result was S-coded to indicate that, due to sample matrix effects, less than 10 grams of the soil sample was used for extraction and analysis. Samples that exhibited this colloidal suspension and their corresponding elevated detection levels are Sample 019 (200 mg/kg), Sample 093 (200 mg/kg), Sample 109 (200 mg/kg), Sample 109D (400 mg/kg), and Sample 110 (100 mg/kg).

Another sample matrix effect observed during the demonstration was a difference between some samples and their respective field duplicates. The operator of the Clor-N-Soil Test Kit Observed physical differences in the soil matrix between sample 082 and its field duplicate, 082D. This problem also was noted during the pre-demonstration activities, and steps were taken to correct it. Among the steps taken were a more thorough homogenization of the samples, the use of fluorescein to enable the sampling personnel to visually inspect the effectiveness of the homogenization technique, and increasing the number of field duplicate samples. Although these steps improved the homogeneity of the samples, it should be noted that some differences between duplicate sample results must be expected due to the nonhomogeneity inherent in soil samples. It was, however, the purpose of this demonstration to test this field screening technology under normal field conditions. Nonhomogeneity and less than ideal matrices are common problems with field soil samples. The samples analyzed during this demonstration are believed to be typical of those found in normal field operations.

Sample Throughput and Linear Range

Sample throughput evaluates the amount of time required to extract and analyze one soil sample and the number of samples analyzed in one work day. Dexsil claims that complete analysis time is about 10 minutes per sample. Using this information, the number of samples that could be analyzed in one 8-hour day would be 48 samples. The operator of the Clor-N-Soil Test Kit determined that the average time needed to perform a sample analysis was 11 minutes. This did not include the time required for sample handling, data documentation, difficult extractions, or the preparation of QC samples. The additional time needed to perform these tasks prevented the operator from being able to complete the analysis of the reported 48 samples per day. The largest number of samples analyzed in one day during the demonstration was 35 samples. The average number of samples analyzed was 19 samples a day.

The Clor-N-Soil Test Kit does not exhibit a linear range. The test kit was designed to give a positive or a negative result as to whether a soil sample contains 50 mg/kg of PCBs. According to Dexsil, the intensity of purple color in a negative sample may give some indication of PCB concentration. However, Dexsil does not recommend using the test kit quantitatively.

Specificity

The discussion of specificity is divided into two sections. The first section discusses some potential interference problems that may be encountered when using this test kit. The second section details the results of an Aroclor specificity test that was conducted during the demonstration.

Potential Interferences

The Clor-N-Soil Test Kit operates on the principle of total organic chlorine detection. It is responsive to chloride, particularly when it is found in an organic form. The test kit is less responsive to inorganic forms of chloride, such as salt, for two reasons. First, inorganic forms of chloride are not very soluble in the organic solvent used for extraction. Second, the use of the florisil column removes most sources of inorganic chloride.

Organic sources of chloride in soil samples will give false positive results when analyzed with this test kit. Common sources of organic chloride in soil samples include chlorinated solvents and chlorinated pesticides. Another source of chloride would be trichlorobenzenes contained in transformer oils. Transformer oils are a common soil contaminant at many PCB-contamination sites.

It seems logical that other halogenated compounds would react to the Clor-N-Soil Test Kit in a similar manner as chloride compounds. Organic compounds containing bromine, fluorine, and iodine can be extracted from soil samples with the organic solvent and can pass through the florisil column. If these halogenated ions are liberated from the parent compounds through the sodium reaction, they may bind to the mercury ions in the indicating step, which would lead to false positive results. Common sources of these halogenated compounds include solvents and pesticides.

Another source of interference is sulfur. Sulfur is commonly found in soil samples and is a common interferant during analysis of PCBs by GC using an

ECD. Sulfur can be extracted from soil samples with the organic solvent used with this test kit and can pass through the florisil column if the sulfur is present in sufficient concentrations. If sulfur survives the vigorous sodium reaction, then it may bind to the mercury ions in the indicating step. Again, this type of interference would result in an increase in false positive results.

Soil samples containing sources of organic mercury may result in false negative results if PCBs are also present. Organic sources of mercury include many types of organomercury pesticides and explosives. This type of mercury can be extracted from soil samples with the organic solvent used with this test kit and can pass through the florisil column. If the mercury is separated from the parent compound and survives the sodium reaction, it may bind with any of the diphenyl carbazone present in the final indicating step. If PCBs are also present in the soil sample, the test kit may not be able to determine PCB concentrations above 50 mg/kg if sufficient mercury is in the extract and it binds with the diphenyl carbazone to produce a purple color. The chance of this occurring is small, but it is possible to find both PCBs and organic mercury in the same sample.

Aroclor Specificity Results

The concentration of PCBs needed to result in a positive identification using the Clor-N-Soil Test Kit depends on the Aroclor and its concentration. The Aroclors and the concentrations of each that calculations indicate are needed to produce correct results are: Aroclor 1016 (51.2 mg/kg), Aroclor 1221 (100.0 mg/kg), Aroclor 1232 (65.6 mg/kg), Aroclor 1242 (50.0 mg/kg), Aroclor 1248 (43.8 mg/kg), Aroclor 1254 (38.9 mg/kg), and Aroclor 1260 (35.0 mg/kg).

The specificity of the Clor-N-Soil Test Kit toward each of these Aroclors was measured during the demonstration. Seven soil samples were chosen to be spiked with known amounts of each Aroclor. Each sample was spiked with a different Aroclor. First, each sample was divided into four aliquots. Two aliquots were then spiked with about 30 mg/kg of the Aroclor, and two were spiked with concentrations of about 70 mg/kg The results of the Aroclor specificity test are tabulated in Table 6-1.

To ensure that results of the assessment were unbiased by operator effects, the operator did not know which Aroclor was used for spiking or the concentration of the Aroclor in the samples. At the time that the Aroclor spikes were prepared, the concentrations of the PCBs in the original samples were not known. Initial indications from the Dexsil Clor-N-Soil Test Kit were available, but the results had not been final&d. Two of the original samples used for the Aroclor specificity test were later found to contain PCBs at concentrations above the 50 mg/kg detection limit. This voided the results for these samples. Positive results would be expected for all spikes of these samples because the sample already contained more than 50 mg/kg of PCB. The two samples affected had been spiked with Aroclors 1242 and 1260. However, the test kit indicated that one of the aliquots spiked with Aroclor 1260 contained less than 50 mg/kg of PCBs. This sample result was thought to be an experimental error. All of the other samples spiked with Aroclors 1242 and 1260 were greater than the 50 mg/kg level, as expected.

Sample 003 was spiked with Aroclor 1221. All results for Aroclor 1221 spikes were reported as less than the 50 mg/kg detection limit. At least two of these spikes, therefore, resulted in false negative results. The expected detection limit was 100 mg/kg for Aroclor 1221. Sample 077 was spiked with Aroclor 1016 at the two concentrations. The results for all Aroclor 1016 spikes also were reported at less than the 50 mg/kg detection limit. This did not agree with the expected detection limit for this Aroclor, which was 51.2 mg/kg. The 70 mg/kg spike samples should have given positive results.

Sample 058 was spiked with Aroclor 1248. The results for the Aroclor 1248 spikes were reported as greater than 50 mg/kg The expected detection limit was 43.8 mg/kg. The 30 mg/kg spiked samples gave a positive result using this test kit. The confirmatory laboratory results indicate that the sample contained less than 1 mg/kg of PCB before being spiked. Therefore, these results did not agree with the expected detection limit of the Clor-N-Soil Test Kit for Aroclor 1248.

Sample 034 was spiked with Aroclor 1254. The results of the Aroclor 1254 spikes were reported at greater than 50 mg/kg The expected detection limit was 38.9 mg/kg . The 30 mg/kg spiked samples gave a positive result using the test kit. The confirmatory laboratory results for sample 034, though, indicate a PCB level of 34 mg/kg before the sample was spiked. Because of the presence of PCBs in the original sample, even the total PCBs in the samples spiked with 30 mg/kg were above both 50 mgikg and the expected detection limit of 38.9 mg/kg.

The Aroclor spike results for Aroclor 1232 were reported as both greater than and less than the 50 mg/kg detection limit. Sample 021 was spiked with Aroclor 1232. The original sample, 021, was found to contain less than 50 mg/kg of PCBs. The 30 mg/kg Aroclor 1232 spikes were reported at less than the 50 mg/kg detection limit, while the 70 mg/kg Aroclor 1232 spikes

TABLE 6-1. AROCLOR SPECIFICITY TEST RESULTS: Clor-N-Soil Test Kit

Sample No.	Soil Sample Result (mg/kg)	Aroclor Spike	Spike Amount (mg/kg)	Spiked Sample Result (mg/kg)
003ARSPAl	< 50	AR1221	69.7	< 50
003ARSPA2	< 50	AR1221	29.6	< 50
003ARSPA3	< 50	AR1221	69.6	< 50
003ARSPA4	< 50	AR1221	29.8	< 50
012ARSPBl	> 50	AR1260	39.9	> 50
012ARSPB2	> 50	AR1260	59.8	> 50
012ARSPB3	> 50	AR1260	39.9	< 50
012ARSPB4	> 50	AR1260	59.9	> 50
021ARSPCl	< 50	AR1232	29.7	< 50
021ARSPC2	< 50	AR1232	**69.0**	> 50
02lARSPC3	<50	AR1232	29.8	< 50
021ARSPC4	< 50	AR1232	69.7	> 50
034ARSPDl	< 50	AR1254	29.6	> 50
034ARSPD2	< 50	AR1254	29.5	> 50
034ARSPD3	< 50	AR1254	69.4	> 50
034ARSPD4	< 50	AR1254	68.8	> 50
040ARSPEl	> 50	AR1242	29.5	> 50
040ARSPE2	> 50	AR1242	69.2	> 50
040ARSPE3	> 50	AR1 242	68.8	> 50
040ARSPE4	> 50	AR1242	29.7	> 50
058ARSPFl	< 50	AR1248	30.0	>50
058ARSPF2	< 50	AR1248	29.9	> 50
058ARSPF3	< 50	AR1248	68.9	> 50
058ARSPF4	< 50	AR1248	69.9	> 50
077ARSPGl	< 50	AR1016	69.0	< 50
077ARSPG2	< 50	AR1016	29.6	< 50
077ARSPG3	< 50	AR1016	29.9	< 50
077ARSPG4	< 50	AR1016	69.7	< 50

were reported as greater than 50 mg/kg. These results agreed with the expected detection limit of 65.6 mg/kg.

Intramethod Assessment

Specific QC measures were used during the demonstration for analyzing soil samples using the Clor-N-Soil Test Kit. Laboratory contamination and the extraction and analysis efficiency of this technology were evaluated. Other QC measures were used to evaluate the ability of the test kit to find expected results and to reproduce the results it found. Although the types of QA/QC samples analyzed are often used to evaluate operator effects, all of these samples were intended to evaluate the technology. Operator effects were controlled and uncontrollable operator errors were assumed to be indistinguishable from errors inherent in the technology. Because the technology was designed for use by nontechnical personnel in field conditions, but was operated during this demonstration by a trained chemist, operator effects in this demonstration were expected to be minimal. The QA/QC samples analyzed and the parameters monitored are discussed in the following paragraphs.

Reagent blanks were used to evaluate laboratory-induced contamination. Eight reagent blanks were

analyzed during this demonstration. All reagent blank results were less than 50 mg/kg of PCBs. The reagent blanks analyzed by the Clor-N-Soil Test Kit during the demonstration indicated that there was no problem with laboratory-induced contamination.

For this demonstration, completeness refers to the proportion of valid, acceptable data generated using the Clor-N-Soil Test Kit. Completeness for the samples analyzed by the Clor-N-Soil Test Kit during this demonstration was 100 percent.

Intramethod accuracy was assessed for the Clor-N-Soil Test Kit by analyzing PE samples and matrix spike and matrix spike duplicate samples. Accuracy also was assessed by comparing the results from the test kit to those from the confirmatory laboratory. Two PE samples were analyzed by the Clor-N-Soil Test Kit during the demonstration. One PE sample had a low concentration of PCBs; the other, high. The operator knew that the samples were PE samples, but did not know the true concentration, the acceptance range, nor which sample had the high or low concentration.

The true concentration for sample 047424-l 13 (the low-level sample) was 32.7 mg/kg, with an acceptance range of 12 to 43 mg/kg The actual reported result for this sample analyzed by the Clor-N-Soil Test Kit was greater than 50 mg/kg This value was determined to be outside the acceptance range. The test kit was actually designed to give a positive result for samples containing Aroclor 1242 above 44 mg/kg, which is close to the upper limit of the acceptance range for this PE sample. Still, the true value of the PE sample was more than 11 mg/kg below the cushion that was designed into the Clor-N-Soil Test Kit. The result, therefore, is still considered unacceptable. The true concentration for sample 047-4024-l 14 (the high-level sample) was 110 mg/kg, with an acceptance range of 41 to 150 mg/kg. The result for this sample when analyzed with the Clor-N-Soil Test Kit was greater than 50 mg/kg, which was acceptable.

Matrix spike samples were used to evaluate the test kit' s extraction and analysis efficiency. Matrix spike samples were prepared by adding a known quantity of PCBs to an actual sample. The PCB used was Aroclor 1242 and enough of a concentrated Aroclor 1242 standard was added to a lo-gram soil sample to produce a matrix spike concentration of 50 mg/kg. The spiked sample was duplicated to produce a matrix spike duplicate sample. The recovery results of the matrix spike and matrix spike duplicate samples are listed in Table 6-2. Six samples were used for the matrix spike samples. Each was reported to contain less than 50 mg/kg of PCBs for the original sample result.

TABLE 6-2. MATRIX SPIKE AND MATRIX SPIKE DUPLICATE RESULTS.

Sample No.	Sample Result (mg/kg)	Matrix Spike Result (mg/kg)	Matrix Spike Duplicate Result (mg/kg)
047-4024-013	< 50	> 50	> 50
047-4024-021	< 50	> 50	> 50
047-4024-049	< 50	> 50	> 50
047-4024-070	< 50	< 50	> 50
047-4024-092	< 50	> 50	> 50
047-4024-I 02	< 50	> 50	> 50

TABLE 6-3. LABORATORY DUPLICATE SAMPLE RESULTS.

Sample No.	Sample Result (mg/kg)	Duplicate Result (mg/kg)
047-4024-008	> 50	< 50
047-4024-012	> 50	< 50
047-4024-026	< 50	> 50
047-4024-047	< 50	< 50
047-4024-074	> 50	> 50
047-4024-095	> 50	< 50

In all, six matrix spikes and six matrix spike duplicates were analyzed. Eleven of the results were found to contain greater than 50 mg/kg of PCBs. One matrix spike result was found to contain less than 50 mg/kg of PCBs. The extraction and analysis efficiency for the samples analyzed by the Clor-N-Soil Test Kit during the demonstration was 92 percent based on the matrix spike recovery data.

Precision for this technology was assessed by comparing each of the results obtained from duplicate samples. The results for the laboratory duplicate samples for this demonstration are listed in Table 6-3. Three types of precision data were generated: data from laboratory duplicate samples, data from matrix spike duplicate samples, and data from field duplicate samples. Laboratory duplicate samples are two analyses performed on a single sample brought to the laboratory. These samples are used to monitor the precision of the

procedures and technology used for the analysis. For this demonstration, laboratory duplicate samples were analyzed with each set of 20 samples submitted for analysis. Six laboratory duplicate samples were analyzed with the Clor-N-Soil Test Kit. The original goal was to analyze laboratory duplicate samples determined to contain greater than 50 mg/kg of PCBs. This goal later was modified. Four of the samples used for laboratory duplicate samples had original sample results of greater than 50 mg/kg of PCBs Two of the samples used for laboratory duplicate samples had original sample results of less than 50 mg/kg of PCBs It should be noted that the four samples with results of greater than 50 mg/kg were selected from among those samples that the operator had difficulty reading.

Of the six laboratory duplicate sample results obtained, only two agreed with the result obtained from the original sample. One of these samples had an original sample result of less than 50 mg/kg of PCBs; the other had an original sample result of greater than 50 mg/kg of PCBs. The other four laboratory duplicate samples did not agree with the results from their respective original samples.

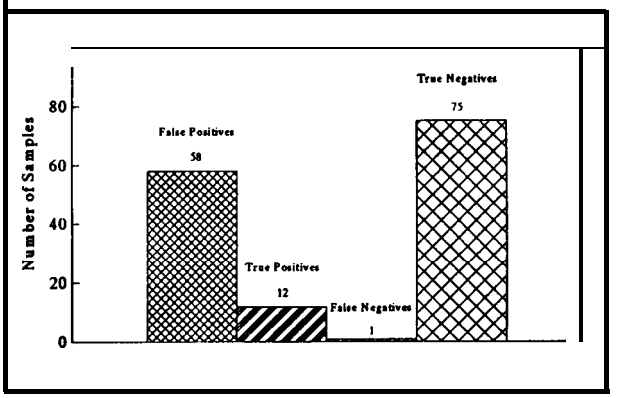
Six matrix spike duplicate samples were prepared and their results were compared to the results of their respective matrix spike samples in this precision evaluation. Five of the matrix spike duplicate sample results matched the matrix spike sample results. One matrix spike duplicate sample result (047-4024-070MSD) did not match the matrix spike sample result.

Thirty-two field duplicate samples were analyzed by the Clor-N-Soil Test Kit during the demonstration. One pair of samples produced incomparable data. In six out of the remaining 31 pairs of results, which is 19 percent, one result was positive and one was negative. Though the test kit produces only semiquantitative results, it only was able to duplicate its results 81 percent of the time. A review of the results that did not match seem to show that the Clor-N-Soil Test Kit had more difficulty duplicating its results when the PCB concentrations were near 1 mg/kg. Five out of the six pairs that did not match had concentrations in this range. The other pair of samples had confirmatory laboratory results of 17.5 mg/kg and 3 1.2 mg/kg.

Comparison of Results to Confirmatory Results

This section compares data generated by the Clor-N-Soil Test Kit to the data generated by the confirmatory laboratory. The confiiatory laboratory' s data is considered correct, and its accuracy and precision are considered within acceptable limits. The results of the

FIGURE 6-1.



Clor-N-Soil Test Kit analyses and the confirmatory laboratory analyses are summarized in Table 6-4. The assessment of the Clor-N-Soil Test Kit analyses are presented on Figure 6-l.

Accuracy

The Clor-N-Soil Test Kit is designed and promoted as a semiquantitative test kit for determining whether a soil sample contains PCBs above 50 mg/kg. Because it does not produce quantitative results, PRC determined whether each confirmatory laboratory result was above or below 50 mg/kg. The test kit' s results and the confirmatory results were then presented on a 2 by 2 contingency table and a Fisher' s Test was used to determined whether a correlation existed between the two sets of data.

The Fisher' s Test was based on 146 pairs of matched data. The Clor-N-Soil Test Kit and the confirmatory laboratory gave the same result 87 times and did not give the same result 59 times. The calculated Fisher' s Test value for the comparison of the two sets of data was 56.21. The **Chi-square$_{crit}$** to which it WAS compared was 3.84 at a 95 percent confidence level with 1 degree of freedom. These results indicate that there is no correlation between data from the test kit and the data from the confirmatory laboratory. This suggests that the Clor-N-Soil Test Kit is not accurate.

As discussed earlier, the Clor-N-Soil Test Kit is designed to do conservative PCB field analysis. While it is marketed to determine whether a sample contains PCBs above 50 mg/kg, it actually is designed to give positive results for samples containing 44 of Aroclor 1242, the main Aroclor at the AICO site. Also as described earlier, the operator of this technology had difficulty determining the color of some analysis extracts and in these cases determined that the sample had a positive result. These factors, though, apparently did not contribute greatly to the large number of incorrect

TABLE 6-4. SUMMARY OF CLOR-N-SOIL TEST KIT DATA.

Sample No.	Clor-N-Soil Test Kit (50 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Technology Accuracy	Sample No.	Clor-N-Soil Test Kit (50 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Technology Accuracy
001	< 50	0.593	Correct	035D	< 50	ND	Correct
002	< 50	1.50	Correct	036	> 50	816	Correct
003	< 50	0.114	Correct	037	> 50	0.055J	FP
004	> 50	6.71J	FP	037D	< 50	0.040J	Correct
005	< 50	1.37	Correct	038	> 50	1,030J	Correct
006	< 50	0.679	Correct	039	< 50	0.676	Correct
007	< 50	0.552	Correct	040	> 50	4.25	FP
008	> 50	2.00	FP	041	< 50	ND	Correct
009	< 50	1.30J	Correct	042	< 50	0.517	Correct
010	< 50	0.172J	Correct	042D	< 50	0.462J	Correct
011	< 50	1.15J	Correct	043	< 50	1.69J	Correct
012	> 50	ND	FP	043D	< 50	1.74	Correct
013	< 50	1.13	Correct	044	< 50	0.592J	Correct
014	< 50	0.18	Correct	045	< 50	ND	Correct
015	> 50	9.13	FP	046	< 50	ND	Correct
015D	> 50	9.84	FP	046D	< 50	ND	Correct
016	> 50	2,110	Correct	047	< 50	0.094J	Correct
017	> 50	2.55	FP	047D	> 50	0.098J	FP
018	> 50	45.4	FP	048	< 50	ND	Correct
019	> 200S	6.70	FP	049	< 50	ND	Correct
020	> 50	0.068J	FP	050	> 50	3.60	FP
021	< 50	0.063	Correct	050D	< 50	4.41	Correct
022	> 50	0.535	FP	051	< 50	ND	Correct
022D	> 50	0.718	FP	052	> 50	4.21	FP
023	> 50	20.8	FP	053	> 50	0.958	FP
024	> 50	0.055	FP	054	> 50	0.516J	FP
024D	< 50	0.049	Correct	055	< 50	2.40	Correct
025	< 50	11.7	Correct	056	< 50	0.505	Correct
026	> 50	1.96	FP	057	> 50	ND	FP
027	< 50	0.057	Correct	058	< 50	0.681	Correct
028	< 50	0.216	Correct	059	> 50	7.86	FP
028D	< 50	0.224J	Correct	060	> 50	0.624J	FP
029	< 50	0.229J	Correct	060D	> 50	0.577	FP
030	> 50	1.15	FP	061	> 50	580	Correct
031	> 50	0.263	FP	062	> 50	2.35	FP
032	> 50	47.6	FP	063	> 50	0.092J	FP
033	< 50	6.00J	Correct	063D	< 50	0.154J	Correct
034	< 50	34.0	Correct	064	> 50	19.0	FP
035	< 50	ND	Correct	065	< 50	3.08	Correct

TABLE 6-4. (CONTINUED)

Sample No.	Clor-N-Soil Test Kit (50 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Technology Accuracy	Sample No.	Clor-N-Soil Test Kit (50 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Technology Accuracy
066	> 50	1.98	FP	090	< 50	1.01	Correct
067	< 50	0.081	Correct	090D	> 50	1.40	FP
068	> 50	0.504J	FP	091	> 50	1,630	Correct
069	> 50	ND	FP	091D	> 50	1,704	Correct
069D	> 50	ND	FP	092	< 50	1.21	Correct
070	< 50	ND	Correct	092D	< 50	ND	Correct
071	< 50	0.052J	Correct	093	> 200S	0.295	FP
071D	< 50	ND	Correct	094	> 50	0.362J	FP
072	> 50	0.035J	FP	095	> 50	17.5	FP
073	> 50	15.8	FP	095D	< 50	31.2	Correct
074	> 50	13.3	FP	096	< 50	0.059J	Correct
075	> 50	23.0	FP	097	< 50	1.23	Correct
076	> 50	46.7	FP	097D	< 50	0.285	Correct
077	< 50	ND	Correct	098	< 50	1.17	Correct
078	> 50	2.27	FP	098D	< 50	0.825	Correct
079	> 50	42.8	FP	099	< 50	ND	Correct
080	< 50	3.77	Correct	100	> 50	177	Correct
081	> 50	0.687	FP	100D	> 50	167	Correct
081D	> 50	0.450	FP	101	> 50	1.21	FP
082	< 50	ND	Correct	102	< 50	293	FN
082D	> 50	0.244	FP	102D	< 50	1.77	Correct
083	< 50	0.484	Correct	103	> 50	40.3	FP
083D	< 50	0.413	Correct	104	< 50	7.66	Correct
084	> 50	1.16	FP	105	< 50	0.210	Correct
084D	> 50	1.08	FP	106	< 50	2.50	Correct
085	> 50	428	Correct	107	> 50	14.1J	FP
085D	> 50	465	Correct	108	> 50	3.84J	FP
086	< 50	1.42	Correct	109	> 200S	ND	FP
086D	< 50	1.25	Correct	109D	< 400	ND	Correct
087	< 50	0.076	Correct	110	> 100S	ND	FP
087D	< 50	ND	Correct	111	< 50	ND	Correct
088	< 50	2.70	Correct	112	> 50	315	Correct
088D	< 50	1.77	Correct	113	> 50	14.9	FP
089	> 50	45.0	FP	114	> 50	66.3	Correct

Notes:

[a] Detection limit.

J Reported amount is below detected limit or not valid by approved QC procedures.

FP False positive.

FN False negative.

ND PCBs not detected above detection limit.

S Sample matrix effects raised detection limits.

results. A review of the actual confirmatory laboratory values for these samples shows that only six of the 59 samples had levels of PCBs within a wide borderline range of 30 to 80 mg/kg. Any incorrect results due to the test kit's conservative design or due to the operator's difficulty in determining the color of some extracts should have fallen within this range. That 53 of the inaccurate results are outside this range indicates that the test kit's accuracy problem is due to other factors.

The Clor-N-Soil Test Kit's semiquantitative results would be of primary use during a site characterization or during removal of contaminated soil at a hazardous waste site where it would be particularly important not to have false negative results. Of the 59 inaccurate results produced by the test kit, only one was a false negative. While the test kit indicated that the sample contained less than 50 mg/kg of PCB, the actual confirmatory laboratory value was 293 mg/kg. The confirmatory laboratory's duplicate of this sample, though, had a result of only 1.77 mg/kg. The false negative rate for the test kit was 1 percent, well within the 95 percent confidence level used to evaluate significance in this demonstration. At sites similar to that used for this demonstration, the test kit should not result in soil contaminated above the EPA action level being treated as if it were not contaminated at that level.

False positive results also can cause the costly disposal of material that is not really contaminated above EPA action levels, which is in opposition to EPA's policy of waste minimization. Of the 59 results that did not match their respective confirmatory laboratory results, 58 were false positives. Overall, this is a 40 percent false positive rate. A review of the actual values as determined by the confiiatory laboratory shows that only six of these samples had PCB levels between 40 and 50 mg/kg. In 44 of these cases, the test kit indicated the levels of PCBs were above 50 mg/kg when they were actually below 10 mg/kg. And in 25 of the cases, the test kit indicated levels above 50 mg/kg when the values from the confirmatory laboratory indicated they were below 1 mg/kg.

In a review of this ITER, Dexsil commented that its directions state that all samples found to have positive results should be tested by a PCB-specific method; however, this would add considerable cost and lost time. Thus, the decision to use this test kit must be made on the basis of the site-specific potential impact of false positives.

Precision

The precision of the Clor-N-Soil Test Kit was not compared to the precision of the confirmatory laboratory because the test kit produces only semiquantitative results.

Section 7
Dexsil Corporation: L2000 PCB/Chloride Analyzer

This section provides information on the L2000 PCB/Chloride Analyzer, including background information, operational characteristics, performance factors, a data quality assessment, and a comparison of its results with those of the confirmatory laboratory. Observations about the technology made during the demonstration by the operator are presented throughout this section.

Theory of Operation and Background Information

The L2000 PCB/Chloride Analyzer is designed and promoted to provide quick, quantitative results for PCB concentrations in soil samples. The analyzer uses a method similar to the Clor-N-Soil Test Kit described in Section 6, which also was developed by Dexsil.

The analyzer operates on the principle of total organic chlorine detection. Sample extraction is required before total organic chlorine detection is performed. PCBs are extracted from soil samples using butyl diglyme, an organic solvent. Then, the L2000 PCB/Chloride Analyzer uses a chloride-specific electrode to measure the amount of total organic chlorine present in the sample. Before samples are analyzed, the analyzer is calibrated by analyzing a standard containing a 50 mg/kg solution of chloride. The analyzer displays a result for this standard on its display screen. The readout on the display is then adjusted to read "50.0" using the calibration knob located on the analyzer.

The L2000 PCB/Chloride Analyzer is able to electronically convert the amount of chloride detected by the analyzer into results for various Aroclors. A "range" knob on the analyzer allows the operator to select the Aroclor believed to be present in the sample, and the analyzer will then convert chloride results into results for that Aroclor. For this demonstration, the soil sample results were taken from the Aroclor 1242 analysis range because results from previous sampling at the AICO site show that Aroclor 1242 is the principal Aroclor at the site.

The L2000 PCB/Chloride Analyzer is specific to sources of organic chlorine. If sources' of organic chlorine other than PCBs are present in samples analyzed using this technology, it will indicate higher PCB concentrations than are actually present or will produce false positive results. Common non-PCB sources of organic chlorine in environmental samples include chlorinated solvents, organochlorine pesticides, and chlorinated disinfectants.

Operational Characteristics

The L2000 PCB/Chloride Analyzer is contained in a portable carrying case, approximately l&inches-long by l&inches-wide by lo-inches-high. The weight of the carrying case containing all the instrumentation needed to analyze samples is 15 pounds. The instrumentation and equipment stored in the carrying case includes the electronic L2000 PCB/Chloride Analyzer, a step-down transformer, a portable balance, a 5-mL pipettor, a vial rack, and a timer.

Necessary reagents and equipment are provided separately in a 12-cubic-foot cardboard box. This box contains enough equipment and reagents to perform 200 soil sample analyses. The equipment and reagents shipped in the cardboard box include (1) stainless-steel spatulas, (2) disposable pipets, (3) 50 mL of electrode filling solution, (4) 250 mL of rinse solution, (5) extraction test tubes, (6) 200 vials (10 mL each) of soil extraction solvent, (7) lo-cubic-centimeter syringes, (8) disposable filters, (9) reaction test tubes containing two ampules, (10) 1 liter of extract solution, (11) drying tubes, 20-mL glass vials, (12) 250 mL of calibration solution, (13) four boxes of laboratory wipes, (14) a marking pen, and (15) operating instructions.

The primary logistical requirement for using the

L2000 PCB/Chloride Analyzer is electricity. Electricity to operate the analyzer during this demonstration was provided through a ll0-volt circuit in the trailer. A second logistical requirement is a battery to operate the portable balance supplied with the analyzer. Replacement batteries are needed periodically when the analyzer is used over an extended period of time.

Supplies not included with the L2000 PCB/Chloride Analyzer, but required for the analysis of samples, include a table or a work space area at least 8-square-feet, a refrigerator for storing samples, a logbook or report form for recording sample results, and an ink pen. The refrigerator is optional if all samples are analyzed on the same day they are collected. However, it is recommended for storing samples overnight or for storing samples until it is determined which samples will be transported to a formal laboratory for further analysis. Reagents used with the L2000 PCB/Chloride Analyzer do not require refrigeration.

The L2000 PCB/Chloride Analyzer is easy to operate. The analyzer can be used by persons with little analytical laboratory experience. The operator noted that the steps for preparing and analyzing the samples were simple and straightforward.

The L2000 PCB/Chloride Analyzer is designed for use as a portable field instrument. However, it does require special care and handling in the field to avoid damage. A mechanical pipettor and a portable electronic balance are provided with the analyzer. These items must be carefully handled to avoid spilling reagents or water on the analyzer. The analyzer itself must be handled carefully to avoid damage to the electronics. The most sensitive part of the analyzer is the chloride electrode. This electrode is made of epoxy and is sensitive to shock. The electrode must be well maintained for the analyzer to provide accurate, consistent results.

This was monitored by periodically reanalyzing the Aroclor standard during sample analysis. The analyzer is equipped with a warning light to inform the operator when recalibration is needed. This warning light functioned on a timed interval, rather than as a function of the electrode response. Instrument calibration was monitored periodically throughout sample analysis and whenever a sample was found to contain more than 100 mg/kg of PCBs.

The operator noted that after analyzing a sample containing more than 100 mg/kg of PCBs, the instrument would frequently lose its calibration. Based on this observation, the reliability of the analyzer' s calibration was found to be low after analyzing samples containing greater than 100 mg/kg PCBs. Another factor that may affect the reliability of the L2000 PCB/Chloride Analyzer is that a number of sample extraction and reaction test tubes frequently leaked during. sample preparation. It should be noted that this may have been due to sample or operator effects. One possible explanation of the sample extraction test tube leaks is that when samples were weighed, a small amount of the soil sample may have been inadvertently left on the test tube threads. If this occurred, it may have prevented a good seal of the test tube cap and resulted in leakage. This would not explain the leakage from the reaction test tubes, however, because soil particles do not come into contact with these tubes. The leakage problems noted by the operator may have a significant impact on the reliability of the L2000 PCB/Chloride Analyzer.

Chemicals used include flammable solvents, such as naphthalene, diglyme, and butyl diglyme. These chemicals must be handled carefully to avoid exposure and fire.

To free the chloride ions from the biphenyl group, 50 mg of metallic sodium is used. Metallic sodium can react explosively with water, making this reaction a dangerous one. Dexsil' s MSDS form for the L2000 PCB/Chloride Analyzer explains that if a fire occurs, it should be treated as a sodium fire and water should not be used to extinguish the fire. However, because the amount of metallic sodium included in each reaction test tube is small, water would be a suitable medium for extinguishing a fire that involved fewer than five reaction test tubes. A dry chemical fire extinguisher would be an appropriate method of extinguishing a fire involving more than five of the reaction test tubes.

The analyzer contains a florisil cleanup column. Plorisil is a fine dust and can be a skin and eye irritant. The rinse, extract, and Aroclor standards used with the L2000 PCB/Chloride Analyzer contain sulfuric acid. The rinse, extract, and Aroclor standards also contain nickel nitrate which is an oxidizer. Chemical resistant clothing and safety glasses should be worn when opening the packaging material, when handling the florisil column, and when handling solutions.

The L2000 PCB/Chloride Analyzer requires that crushable glass ampules be broken within the confines of a plastic test tube. Care should be taken when crushing these ampules.

The operator of the L2000 PCB/Chloride Analyzer was Mr. Keith Brown, an employee of PRC who has a B .G. S. degree in environmental science, 2 years of experience in conducting preliminary site assessments and investigations at hazardous waste sites and who has

performed hydrogeologic investigations at similar sites. Mr. Brown's training in the use of the L2000 PCB/Chloride Analyzer included one hour of hands-on training. Further training was provided to a PRC chemist at the start of the demonstration by Mr. Finch of Dexsil. This chemist was available to assist Mr. Brown as required. Mr. Finch provided information regarding filtration rates and separation techniques for troublesome sample extractions. According to the operator, some of this information was not included in the analyzer's instructions. Mr. Brown then analyzed five samples using the L2000 PCB/Chloride Analyzer under the supervision of the lead chemist. Mr. Brown noted that he felt comfortable with his ability to properly analyze soil samples with the analyzer after analyzing these samples.

Costs include the costs of the analyzer and reagents, the operator, and waste disposal. The L2000 PCB/Chloride Analyzer can be purchased from Dexsil for $3,500. This cost covers all instrumentation included with the carrying case and enough reagents and equipment to analyze 100 soil samples. Additional reagents can be purchased from Dexsil. The cost of additional reagents depends on the number of individual tests required. A test is equivalent to one soil sample analysis. The cost of reagents to perform 40 tests is $400, which is $10 per test. If more reagents are needed, Dexsil offers a package containing enough reagents to perform 200 tests for $1,600, which is $8.00 per test. According to Dexsil, the shelf-life of the reagents is one year from the date of purchase. The shelf-life allows users of the analyzer to maintain a stock of reagents, which can reduce the response time for PCB analysis of samples. The L2000 PCB/Chloride Analyzer can also be rented from Dexsil for $500 per month. A one-time charge of $230 is also applied for use of the electrode. Operator costs for using the L2000 PCB/ChIoride Analyzer will vary depending on the technical knowledge of the operator. The waste generated by these analyses filled half a 55-gallon drum. The appropriate way to dispose of this waste is through an approved PCB incinerator facility. The cost for disposal of one drum of this waste is estimated at $1,000.

Performance Factors

The following paragraphs describe the L2000 PCBKhloride Analyzer's performance factors including detection limits and sensitivities, sample throughput, linear range, and drift. Specificity, due to its complexity, is discussed separately.

Detection Limits and Sensitivity

The detection limit for the L2000 PCB/Chloride Analyzer is reported by Dexsil to be 5 **mg/kg.** The detection limit for the samples analyzed during the demonstration was 2 mg/kg. The following paragraphs explain the reason for the different detection limits.

After analyzing the predemonstration samples, Dexsil reported two results for each of the samples analyzed. The first result for each sample is the actual concentration of PCBs detected in the sample. The second result was calculated by subtracting the concentration of PCBs detected in the reagent blank from the concentration detected in the sample. This second set of results includes three results that were below the 5 mg/kg detection limit reported by Dexsil. PRC discussed the discrepancy between the stated detection limit for the analyzer and the actual results reported by Dexsil during the predemonstration with Mr. Finch of Dexsil. In this discussion, Mr. Finch stated that the detection limit of the L2000 PCB/Chloride Analyzer is 5 mg/kg. However, this detection limit is based on results that have not been adjusted by subtracting the reagent blank result from the sample result. Subtracting the reagent blank result can result in a lower detection limit. The instructions provided with the analyzer do not discuss this issue or state that analysis of reagent blanks is necessary. According to Mr. Finch, most people using the L2000 PCB/Chloride Analyzer to analyze soil samples do not use reagent blanks.

PRC did analyze reagent blank samples with the L2000 PCB/Chloride Analyzer to evaluate the response of the analyzer to the reagents used. Every reagent blank analyzed resulted in positive results. The lead chemist and the operator determined that reagent blank results should be subtracted from positive sample results to reduce effects caused by the analyzer responding to reagents used for analyzing samples. After the demonstration, Mr. Finch was consulted about the decision to subtract reagent blank results from sample results. Mr. Finch commented that this would produce better results. He also noted that the detection limit for the L2000 PCB/Chloride Analyzer could be lowered from 5 mg/kg to 2 mg/kg when using this approach. Mr. Finch was confident in the ability of the analyzer to reach this lower detection limit and stated that this limit could be used for the samples analyzed during the demonstration. The detection limit was then lowered for the demonstration from 5 mg/kg to 2 mg/kg only after the reagent blank samples were analyzed and these results were subtracted from sample results.

The sensitivity of the L2000 PCB/Chloride Analyzer, which is established by calibration, is dependent on the amount of chloride extracted from a soil sample. Before the analyzer is calibrated, the chloride-specific electrode must be filled with solution. The electrode is then checked for proper response by monitoring the millivolt readout that results from the electrode being placed in a vial of clean rinse solution. The electrode must give a readout of above 140 millivolts before analysis continues. After proper response of the electrode is verified, the analyzer is calibrated. Calibration is performed by placing the electrode into a vial of Aroclor standard containing 50 mg/L of chloride solution. The readout of the analyzer is adjusted to read 50.0 mg/kg with the analysis range set in the "CAL" mode. After successfully completing the calibration, sample analysis can begin.

The L2000 PCB/Chloride Analyzer has five "analytical ranges." These ranges, or readouts, include: millivolt, chloride, Aroclor 1242, Aroclor 1260, and Askarel A. The Aroclor 1242 readout was used during this demonstration because this Aroclor is the primary contaminant at the AICO site. The Aroclor 1260 range also was monitored for samples that gave a response of 10.0 mg/kg or greater in the Aroclor 1242 range; however, this data was not used for reporting purposes.

The L2000 PCB/Chloride Analyzer electronics automatically convert the readout of the electrode signal to appropriate amounts of Aroclor 1242. This conversion is based on the fact that Aroclor 1242 contains 42 percent chlorine by weight. The conversion used was a simple factor in which the amount of chloride detected by the electrode is divided by the percentage of chloride present in the Aroclor 1242, which is 42 percent. This value is then multiplied by two to correct for the loss of PCBs that occurs during sample extraction. Loss of PCBs during extraction occurs because 10 grams of sample are extracted into 10 mL of organic extraction solvent, but only 5 mL of the extraction solvent is used for the reaction and analysis steps. Through these automatic conversions, the liquid crystal display (LCD) readout of the L2000 PCB/Chloride Analyzer calculates and displays the concentration of Aroclor 1242 in the sample.

The sensitivity of the L2 000 PCB/Chloride Analyzer to PCBs is dependent on the amount of chloride present in the biphenyl compound. Standard EPA analytical methods for analyzing PCBs target the seven most common Aroclors. These Aroclors differ in the amount of chloride present in the biphenyl group. Because the amount of chloride present in each Aroclor is known, it is possible to evaluate the sensitivity of the analyzer to each Aroclor. Although PRC used a 2 mg/kg detection limit to evaluate accuracy and precision, it used Dexsil's stated 5 mg/kg detection limit to evaluate sensitivity.

The sensitivity of the chloride-specific electrode to Aroclor 1242 can be determined by measuring the amount of chloride present in a lo-gram sample containing 5 mg/kg of Aroclor 1242. A l0-gram sample containing 5 mg/kg of Aroclor 1242 contains 0.05 mg of Aroclor 1242. Following the instructions for the analyzer, the PCBs are extracted into 10 mL of organic solvent. Five mLs of this extract are then taken through the remaining steps of the analysis. Because only 5 mLs of extract is used, the amount of Aroclor 1242 in the extract would be 0.025 mg. Because Aroclor 1242 contains 42 percent chloride by weight, the amount of chloride present in the 0.025 mg of Aroclor 1242 can be calculated by multiplying 0.025 mg of Aroclor 1242 by 0.42. This equals 0.0105 mg of chloride. This is the minimum amount of chloride that can be detected by the L2000 PCB/Chloride Analyzer according to the stated detection limit.

The process also can be expressed through the following equation:

(7-1)

$$(0.05\ mg\ /\ 10/5)\ x\ 0.42 = 0.0105\ mg\ Cl^-$$

where

0.05 *mg = amount of Aroclor 1242 in a 10-gram sample containing a 5 mg/kg concentration.*

10/5 *= ratio of solvent used for the extraction divided by amount used for analysis.*

0.42 *= percent chlorine present in Aroclor 1242.*

0.0105 *mg* Cl^- *= amount of chloride in a 10-gram sample containing 5 mg/kg of Aroclor 1242.*

Just as the sensitivity of the analyzer to Aroclor 1242 can be determined as discussed above, the sensitivity of the analyzer to the other six Aroclors can be determined when the analyzer is operated in the Aroclor 1242 range. These sensitivities can be determined by performing the calculations described above in reverse order, or the following equation can be used:

$$((0.0105\ mg\ Cl^-\ /\ \%Cl)\ x\ 10/5)\ /\ 0.01\ kg = sensitivity\ of\ the\ Aroclor$$

where

0.0105 Cl^- *= minimum amount of chloride detected with the chloride-specific electrode.*

%Cl = percent chloride present in the Aroclor.
10/5 = ratio of solvent used for the extraction divided by amount used for analysis.
0.01 = weight of soil sample used for analysis.

Using these calculations, the minimum amount of each Aroclor that can be detected by the analyzer in the Aroclor 1242 range was determined. The results for each Aroclor determined through this calculation are the following: Aroclor 1016 (5.1 mg/kg), Aroclor 1221 (10.0 mg/kg), Aroclor 1232 (6.6 mg/kg), Aroclor 1242 (5.0 mg/kg), Aroclor 1248 (4.4 mg/kg), Aroclor 1254 (3.9 mg/kg), and Aroclor 1260 (3.5 mg/kg).

Sample Matrix Effects

The matrix of the samples analyzed during the demonstration was problematical due to clay and lack of a centrifuge. The most common problem was that a colloidal suspension formed for nine of the samples after the initial sample extraction. To obtain results from the samples with colloidal suspension, another attempt to extract the sample was made using less of the soil sample. Instead of using the recommended 10 grams of soil sample, 5 grams were used. In seven of the samples this did not solve the problem, so the samples were again extracted using 2.5 grams. In four of the samples this still did not solve the problem, so these samples were again extracted using 1.3 grams. When this occurred, the detection level for the affected sample was raised to account for the difference in the sample weight extracted and analyzed.

Data reported for the nine samples that exhibited this problem were S-coded to indicate that, due to sample matrix effects, less than 10 grams of the sample were used for extraction and analysis. The detection limits for these samples also were raised. Samples which exhibited this colloidal suspension and their corresponding detection limits are: Sample 019 (16 mg/kg), Sample 020 (16 mg/kg), Sample 037 (4 mg/kg) Sample 037D (4 mg/kg), Sample 093 (8 mg/kg), Sample 096 (16 mg/kg), Sample 109 (8 mg/kg), Sample 109D (8 mg/kg), and Sample 111 (16 mg/kg).

Another sample matrix effect observed was a physical difference between some samples and their respective field duplicates. As with the Clor-N-Soil Test Kit, thorough homogenization of the samples, the use of fluorescein, and increasing the number of field duplicate samples was used to limit the effects of this problem.

Sample Throughput

Sample throughput with the L2000 PCB/Chloride Analyzer was determined by evaluating the amount of time required to extract and analyze one soil sample and the number of samples analyzed in one work day. Dexsil claims that a soil sample can be prepared in 10 minutes and that several samples can be prepared concurrently. Once the samples have been prepared, Dexsil claims that the actual analysis time for each sample is less than one minute. According to Dexsil, multiple samples can be prepared and then analyzed any time afterwards. Dexsil claims that one operator can complete 100 soil tests in an 8-hour day.

The operator of the L2000 PCB/Chloride Analyzer determined that the amount of time required to perform one complete sample analysis was 9 minutes. The operator reported that the 9-minute sample analysis time did not include the time required for sample handling, data documentation, difficult extractions, or the preparation of QC samples. The time required by the operator to perform these tasks prevented him from completing analysis of 100 samples per day. The largest number of samples analyzed in one 8-hour day during the demonstration was 50 samples. The average number of samples analyzed in one day was 35. To achieve this, the operator extracted a number of samples concurrently, and then analyzed them at the end of the day. This was the procedure recommended by Dexsil.

Linear Range

Dexsil states that the linear range of the L2000 PCB/Chloride Analyzer is 5 to 2,000 mg/kg; however, the lower end was changed to 2 mg/kg and this concentration was used during the precision and accuracy evaluation. This linear range is dependent on a single-point calibration of the analyzer using a 50 mg/kg chloride solution. The analyzer is operated in the normal range during all sample analyses; however, if the PCB concentration of a sample is found to be above 200 mg/kg, a "1" is displayed on the analyzer's display screen. The instructions for the analyzer state that when this occurs, the operator should switch the analyzer into high range by pushing the "high range" button. If the sample contains between 200 and 2,000 mg/kg of PCBs, the concentration can then be read from the instrument's display screen. If the sample contains above 2,000 mg/kg of PCBs, a "1" will again appear on the display screen, indicating that it cannot provide a quantitative result for the sample. Only one sample analyzed during the demonstration exceeded the upper limit of the L2000 PCB/Chloride Analyzer's linear range. Sample 036 was found to contain greater than 2,000 mg/kg of PCBs.

Drift

The drift of the L2000 PCB/Chloride Analyzer was not quantitatively measured during this demonstration, but it was periodically checked by the operator by examining the deviation of the calibration from the chloride standard. Dexsil recommends that the calibration of the analyzer be checked and, if necessary, corrected after analyzing every 10 samples. Dexsil also recommends that the analyzer's electrode be placed in a rinse solution between measurements to help maintain calibration. The operator followed these recommended practices during the demonstration sample analysis. It was found that a significant amount of drift occurred after analyzing samples containing greater than 100 mg/kg of PCBs. Whenever this occurred, the operator recalibrated the instrument using the 50 mg/kg chloride standard. The operator also noted that the calibration warning light on the analyzer would come on at regular intervals to remind the operator to recalibrate. However, the warning light runs on a timer and does not actually indicate when the instrument is out of calibration. The effect of drift on the quality of data produced by the L2000 PCB/Chloride Analyzer can be reduced by frequently checking the calibration of the analyzer.

Specificity

The L2000 PCB/Chloride Analyzer is responsive to chloride, especially in an organic form. Inorganic forms of chloride, such as salts, are not very soluble in the organic solvent used for extracting chloride when samples are being prepared. The use of the florisil column also will remove most sources of inorganic chloride. If sources of organic chlorine other than PCBs are present in soil samples at mg/kg concentrations, the analyzer may give false positive results. Common sources of organic chloride in soil samples include chlorinated solvents, chlorinated pesticides, and trichlorobenzenes contained in transformer oils. Transformer oils are commonly found at PCB-contamination sites. If samples are suspected to contain sources of organic chloride other than PCBs, the L2000 PCB/Chloride Analyzer may not provide valid data.

Other halogenated compounds also may cause false positives in the L2000 PCB/Chloride Analyzer if they are present in significant concentrations. The electrode will respond to halogenated compounds, as well as the chloride. The electrode will respond to halogens less strongly than it responds to chloride, probably by orders of magnitude. Organic compounds containing bromine, fluorine, and iodine may be extracted from soil samples with the organic solvent used by this technology and may pass through the florisil column. If these halogenated ions are liberated from their parent compounds through the sodium reaction and are present in sufficient concentrations, they may be detected with the electrode. Common sources of these halogenated compounds include solvents and pesticides.

The specificity of the L2000 PCB/Cbloride Analyzer to each of the seven Aroclors was measured during the demonstration by spiking seven soil samples with the Aroclors. First, each of the seven samples was divided into four aliquots. All four aliquots were then spiked with a particular Aroclor at a concentration of about 10 mg/kg. This level was chosen because it is a common cleanup goal at PCB-contaminated sites. To ensure the results of the assessment were unbiased by operator effects, the operator did not know which Aroclor was used for spiking nor the concentration of the Aroclor in the samples. The results of the Aroclor specificity test are tabulated in Table 7-1.

All of the Aroclor spikes were analyzed in the 1242 range. The readouts of the analyzer were not an accurate indication of the particular Aroclor present. To obtain an accurate indication, the readout results must be converted by multiplying them by a factor determined by dividing the Aroclor's percent chlorination by the percent chlorination of Aroclor 1242. The readouts expected when the analyzer is set on the Aroclor 1242 setting and a soil sample spiked to 10 mglkg for each of the seven Aroclors is: Aroclor 1016 (9.8 mg/kg), Aroclor 1221 (5.0 mg/kg), ***Aroclor*** 1232 (7.6 mg/kg), Aroclor 1242 (10.0 mg/kg), Aroclor 1248 (11.4 mg/kg), Aroclor 1254 (12.9 mg/kg), and Aroclor 1260 (14.3 mg/kg).

For this demonstration, PRC did not convert its data using the factors mentioned above. Such conversions would not be performed under normal field analysis conditions. An operator in the field often would not know which of the Aroclors were present and, therefore, would not know which conversion factor to use. This demonstration was to be performed under normal field conditions and, for this reason, PRC did not convert its data. However, comments are made in the following paragraphs concerning these conversions, and the specificity results for each Aroclor can be compared to the above list to determine whether the analyzer was able to accurately analyze each Aroclor.

All Aroclor spike samples were extracted and analyzed the same way as the other samples collected during the demonstration. Reagent blank results were subtracted from the analyzer's readouts for these samples on a daily basis as they were for the other samples.Three spiked sample results fell between 1 and 2

TABLE 7-1. AROCLOR SPECIFICITY TEST RESULTS: L2000 PCB/Chloride Analyzer.

Sample No.	Soil Sample Result (mg/kg)	Aroclor Spike	Spike Amount (mg/kg)	Spiked Sample Result (mg/kg)	Percent Recovery (%)
003ARSPA1	ND	AR1221	9.97	< 1	< 10
003ARSPA2	ND	AR1221	9.83	< 1	< 10
003ARSPA3	ND	AR1221	9.83	1.7	17
003ARSPA4	ND	AR1221	9.95	< 1	< 10
012ARSPB1	ND	AR1260	9.99	11.8	118
012ARSPB2	ND	AR1260	9.83	4.0	41
012ARSPB3	ND	AR1260	10.00	11.3	113
012ARSPB4	ND	AR1260	9.91	11.3	114
021ARSPC1	ND	AR1232	9.79	2.9	30
021ARSPC2	ND	AR1232	9.92	2.8	28
021ARSPC3	ND	AR1232	9.96	1.5	15
021ARSPC4	ND	AR1232	9.96	1.5	15
034ARSPD1	14.4	AR1254	9.88	22.5	82
034ARSPD2	14.4	AR1254	9.93	49.1	349
034ARSPD3	14.4	AR1254	9.84	40.2	262
034ARSPD4	14.4	AR1254	9.92	47.2	331
040ARSPE1	5.7	AR1242	9.91	8.5	28
040ARSPE2	5.7	AR1242	10.00	19.8	141
040ARSPE3	5.7	AR1242	9.88	6.9	12
040ARSPE4	5.7	AR1242	9.90	10.8	52
058ARSPF1	ND	AR1248	9.97	4.2	42
058ARSPF2	ND	AR1248	9.93	4.2	42
058ARSPF3	ND	AR1248	10.00	3.0	30
058ARSPF4	ND	AR1248	9.92	4.7	47
077ARSPG1	ND	AR1016	9.94	6.9	69
077ARSPG2	ND	AR1016	9.86	3.1	31
077ARSPG3	ND	AR1016	9.83	2.6	26
077ARSPG4	ND	AR1016	9.94	2.5	25

Note:
ND Not detected above the 2 mg/kg detection limit

These data points were still used in the specificity evaluation, however, because PRC felt that reducing the size of the data set by removing these results would result in a reduction in the quality of the evaluation.

When the Aroclor spikes were prepared, the trations of the PCBs in the original samples were not known. Initial indications from the L2000 PCB/Chloride Analyzer were available, but the results had not been

finalized All but two of the original samples chosen for the Aroclor specificity test were found to contain less than 2 mg/kg of PCBs as determined by the analyzer. Sample 034 was found by the analyzer to contain 14.4 mg/kg of PCBs. The confiiatory laboratory result for thii sample was 34 mg/kg, which is significantly higher. The results of the Aroclor specificity test for Sample 034 were significantly affected by this discrepancy and do not provide an accurate assessment of the analyzer' s sensitivity to the Aroclor with which it was spiked, Aroclor 1254. Of the four spiked samples analyzed, only one resulted in an acceptable recovery when compared to its respective soil sample result. That spiked sample had a recovery of 82 percent. The other three spiked samples exhibited extremely high recoveries 349, 262, and 331 percent, respectively, when compared to the analyzer' s soil sample results. These recoveries are much closer to those expected when compared to the confirmatory laboratory result. This discrepancy may be attributed to the nonhomogeneity inherent in the soil samples.

Sample 040 was found to contain 5.7 mg/kg of PCBs by the analyzer. For the specificity evaluation, this sample was spiked with Aroclor 1242. The recoveries obtained for the four spiked samples used in the specificity evaluation were 12, 28, 52, and 141 percent, respectively. The L2000 PCB/Chloride Analyzer reported results for all four aliquots above the 2 mg/kg detection limit; however, assuming that the amount of PCB in the sample was consistent from aliquot to aliquot, the analyzer underestimated the amount of PCB in the spiked samples three out of four times. The confirmatory laboratory result for the sample was 4.2 mg/kg, which was close to the analyzer' s result. No conversion factor was needed for Aroclor 1242 because the analyzer was set at the Aroclor 1242 setting.

When analyzed with the L2000 PCB/Chloride Analyzer, three of the four aliquots spiked with Aroclor 1221 had results below 1 mg/kg after the reagent blank results were subtracted. These three spiked samples were reported at less than a 10 percent recovery. The fourth sample result was 1.7 mg/kg, resulting in a recovery of 17 percent. All four spiked sample results were below the 2 mg/kg detection limit. This data indicates that Aroclor 1221 cannot be accurately quantified by the analyzer on the 1242 setting at levels of 10 mg/kg in soil samples. This means that Aroclor 1221 could be present in soil samples at levels of 10 mg/kg and the analyzer would report it as below its detection limit of 2 mg/kg. The results for these samples were significantly below 5 mg/kg, the result expected based on the conversion list.

When analyzed with the L2000 PCB/Chloride Analyzer, two of the four aliquots spiked with Aroclor 1232 had results below 2 mg/kg after the reagent blank results were subtracted. The percent recovery values for these four spiked samples ranged from 15 to 30 percent. These results indicate that 50 percent of the time the analyzer did not identify that Aroclor 1232 had been spiked into a sample above its detection limit, even though that spike was at a concentration of 10 mg/kg. Also, the results for all Aroclor 1232 spike sample results were significantly below 7.6 mg/kg, the result expected for these samples based on the conversion list.

All of the samples spiked with Aroclor 1016 resulted in readouts above the 2 mg/kg detection limit after the reagent blank results were subtracted. The percent recovery values for these samples ranged from 25 to 69 percent. Three of the four sample results were significantly below 9.8 mg/kg, the expected result based on the conversion list. These results indicate that the analyzer was able to detect Aroclor 1016, but had difficulty accurately determining the concentration that had been spiked into the samples.

All of the samples spiked with Aroclor 1248 resulted in readouts above the 2 mg/kg detection limit after the reagent blank results were subtracted. The percent recovery values for these samples ranged from 30 to 47 percent. The results for these samples were significantly below 11.4 mg/kg, the expected result for this Aroclor based on the conversion list shown above. These results again indicate that the analyzer was able to detect the Aroclor 1248, but that it underestimated the concentration that had been spiked into the samples.

All of the samples spiked with Aroclor 1260 resulted in readouts above the 2 mg/kg detection limit after the reagent blank results were subtracted. The percent recoveries for three of the four samples were 113, 114, 118 percent. The percent recovery for the other sample was 41 percent. Only one of these sample results differed significantly from 14.3 mg/kg, the expected result based on the conversion list shown above. These results indicate that the analyzer was able to quantify the expected concentration of Aroclor 1260 fairly accurately .

Intramethod Assessment

Reagent blank samples were prepared by taking reagents through all extraction, cleanup, and reaction steps of the analysis. Ten reagent blanks were analyzed during the analysis of samples at the demonstration. Each time, the analysis of these reagent blanks produced

TABLE 7-2. REAGENT BLANK RESULTS.

Reagent Blank Sample No.	Analytical Result (mg/kg)
RBLK 8/ I7/I	3.7
RBLK 8/ I7/2	4.3
RBLK 8/ I8/I	4.6
RBLK 8/18 / 2	3.9
RBLK 8/ I8 /3	4.4
RBLK 8/20/1	3.5
RBLK 8/21/1	5.3
RBLK 8/24/1	7.2
RBLK 8/24/2	4.1
RBLK 8/26/1	5.0

Note:
Sample number translates to: RBLK = Reagent blank; 8/4/I = Month/day/number of blank run

positive results with the **L2000** PCB/Chloride Analyzer. These results ranged from 3.5 to 7.2 mg/kg. Overall, the analysis of reagent blanks suggests that the electrode used with the analyzer causes it to read between 3.5 and 7.2 mg/kg, even when PCBs are not present. Reagent blank results are presented in Table 7-2.

Completeness for the samples analyzed by the L2000 PCB/Chloride Analyzer was 99 percent, well above the objective of 90 percent.

Intramethod accuracy was assessed for the L2000 PCB/Chloride Analyzer through the use of PE samples and matrix spike and matrix spike duplicate samples. Accuracy was also assessed by comparing the results from the analyzer to those from the confirmatory laboratory.

Two PE samples were analyzed by the L2000 PCB/Chloride Analyzer during the demonstration. The operator knew that the samples were PE samples, but did not know the acceptance ranges or which was the high or low concentration sample. The true concentration for sample 047-4024-l 14 (the high-level sample) was 110 mg/kg, with an acceptance range of 41 to 150 mg/kg. The actual reported result for this sample after it was analyzed by the L2000 PCB/Chloride Analyzer was 107 mg/kg. This value was within the acceptance range. Its percent recovery was 97 percent. The true concentration for sample 047-4024-113 (the low-level sample) was 32.7 mg/kg, with an acceptance range of 12 to 43 mg/kg. The actual reported result for this sample after it was analyzed by the L2000 PCB/Chloride Analyzer was 21.8 mg/kg. This value was within the acceptance range. The percent recovery for the low-level PE sample was 67 percent.

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

Six soil samples were used for matrix spike samples. In five of these samples, the L2000 PCB/Chloride Analyzer detected no PCBs before the sample was spiked. In the sixth sample, only 2.7 mg/kg of PCBs was detected before it was spiked. The average recovery of the matrix spike samples and their duplicates was 102 percent or 25.5 mg/kg This is very close to the 25 mg/kg actually added to the samples. The standard deviation of the matrix spike samples was 19.5 percent or 4.9 mg/kg. Control limits are defined as ± 2 standard deviations from the mean, when following guidelines outlined in SW-846 Manual Method 8000. For the matrix spike samples analyzed during the demonstration, the calculated control limits ranged from 63 to 141 percent recovery. All matrix spike samples analyzed fell within these control limits.

Precision for the analyzer was assessed by comparing the results obtained on duplicate samples. Three types of precision data were generated: data from laboratory duplicate samples, data from field duplicate samples, and data from matrix spike duplicate samples. Results for these types of duplicate samples are provided in Tables 7-3 and 7-4.

Seven pairs of laboratory duplicate samples and their respective soil samples were analyzed with the L2000 PCB/Chloride Analyzer. The original results obtained for these samples ranged from 25.7 to 778 mg/kg. When the analysis was duplicated, the results ranged from 11.6 to 624 mg/kg. Field duplicate samples also were analyzed. PRC collected 32 field duplicate samples. Each sample and its duplicate were analyzed using each technology and by the confirmatory laboratory.

PRC was tasked with determining the precision of the technology and attempted to control factors 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 one operator per technology. Variance in that operator' s laboratory techniques would be the same for each sample, and therefore, statistically insignificant. For the field duplicates, PRC put each sample through a homogenization process designed to ensure there was little difference between the

TABLE 7-3. MATRIX SPIKE AND MATRIX SPIKE DUPLICATE RESULTS.

Sample No.	Soil Sample Result (mg/kg)	Spike Amount (mg/kg)	Matrix Spike Recovery (%)	Matrix Spike Duplicate Recovery (%)	Relative Percent Difference (%)
047-4024-011	2.7	25	123	116	6
047-4024-024	ND	25	72	68	6
047-4024-039	ND	25	96	96	0
047-4024-065	ND	25	109	136	22
047-4024-082	ND	25	110	119	8
047-4024-I 06	ND	25	96	87	10

Note:
ND Not detected above the 2 mg/kg detection limit.

tion in a sample and its duplicate. Confirmatory laboratory data on the field duplicates and their respective samples indicate that, overall, this process worked. PRC then used the laboratory and field duplicates together to determine each technology's precision. Even the best technology can not 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, all sample pairs that did not produce two positive results were removed from the data population. Then, the RPD for each pair was calculated and the mean RPD and population standard deviation were determined. The lower control limit was set at zero because this would mean that the results from a control limit were set by multiplying the standard deviation by two and adding it to the mean RPD. The RPD of each sample pair was then compared to these control limits, Each sample pair RPD was expected to fall within the control limits. If greater than 95 percent fell within this range, the technology's precision was considered adequate. If fewer than 95 percent of them fell within this range, the data was reviewed, and if no explanation could be found, the technology's precision was considered inadequate.

The L2000 PCB/Chloride Analyzer had 18 sample pairs in which both a sample and its duplicate had positive results. The data from these 18 pairs had a mean RPD of 29.6 percent and a standard deviation of 23.9. The control limits were, therefore, set at 0 and 77.4 percent. All but one of the 18 sample pairs' RPDs fell within the control limits. The sample pair that was outside the control limits had results of 47.3 and 111 mg/kg, respectively. That sample pair had an RPD of 80 percent. Still, 94.5 percent of the sample pairs had RPDs within the control limits. While this is not between 95 and 100 percent, the technology could not have come closer to 100 percent without every pair falling within the control limits; therefore, the precision was considered acceptable.

Matrix spike duplicate samples were used to further evaluate the precision of this technology. Six matrix spike duplicate samples were analyzed and their results were compared to the results of their respective matrix spike samples.

Precision of the matrix spike duplicate samples was evaluated through the RPD of the matrix spike result and the matrix spike duplicate result. RPD values for the six sets of matrix spike samples ranged from 0 to 22 percent. The mean RPD value from these six samples was 9 percent, and the sample standard deviation was 7 percent. If an upper control limit of two times the standard deviation is used, the upper control limit for RPD determined for samples analyzed during the demonstration was 23 percent. All RPD values for the matrix spike duplicate samples fell within this range.

Comparison of Results to Confirmatory Results

The following sections compare the accuracy and precision of the data from analyses using with the L2000 PCB/Chloride Analyzer to that of the confirmatory laboratory. The results from the confirmatory laboratory are considered accurate and its precision is considered acceptable. The comparison is summarized on Table 7-5 and on Figure 7- 1.

Accuracy

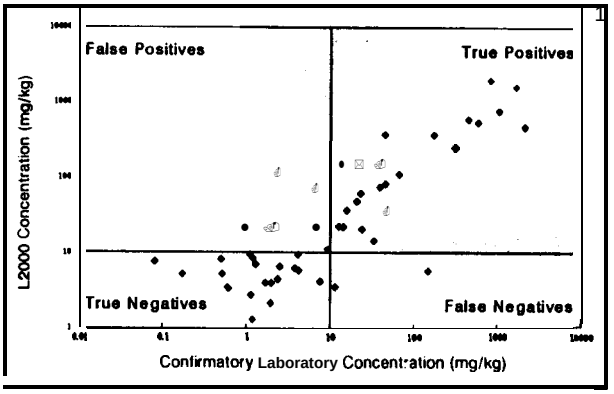
To measure the accuracy of the L2000 PCB/Chloride Analyzer, PRC compared its data to that of the confirmatory laboratory using linear regression.

TABLE 74. DUPLICATE RESULTS.

Sample No.	Soil Sample Result (mg/kg)	Duplicate Sample Result (mg/kg)	Relative Percent Difference (%)
015 FD	9.40	12.5	28
016 LD	484	347	33
022 FD	ND	ND	NA
023 LD	48.8	32.7	40
024 FD	ND	ND	NA
028 FD	ND	ND	NA
035 FD	ND	ND	NA
037 FD	< 4.00	< 4.00	NA
038 LD	778	624	22
042 FD	ND	ND	NA
043 FD	4.10	3.60	13
046 FD	ND	ND	NA
047 FD	ND	ND	NA
050 FD	ND	ND	NA
053 LD	25.7	11.6	76
060 FD	2.30	4.40	63
062 LD	111	47.3	80
063 FD	ND	ND	NA
069 FD	5.80	4.40	27
071 FD	ND	ND	NA
081 FD	ND	ND	NA
082 FD	ND	ND	NA
083 FD	ND	ND	NA
084 FD	7.60	10.9	36
085 FD	593	596	1
085 LD	593	420	34
086 FD	ND	ND	NA
087 FD	ND	ND	NA
088 FD	ND	ND	NA
090 FD	2.00	ND	NA
091 FD	1651	1608	3
092 FD	3.10	3.40	9
095 FD	20.60	20.1	2
097 FD	ND	ND	NA
098 FD	ND	ND	NA
100 FD	384	363	6
100 LD	384	264	37
102 FD	6.30	5.00	23
109 FD	<8.00	10.3	NA

Notes:
FD Field duplicate.
LD Laboratory duplicate.
ND Not detected above the 2 mg/kg detection limit.
NA Not analyzed.

FIGURE 7-I.



Generally, the regression produces a correlation coefficient, also called an r^2, that expresses whether two sets of data are related. If the two sets are related perfectly, then the r^2 would equal 1.0. For this demonstration, r^2 values between 0.80 and 1.0 were considered acceptable. The linear regression also results in an equation that expresses the relationship between two sets of results. That equation can be expressed as a line on a graph where the results of one set of data would be expected if the other set of data were given. Because, ideally, the results from the analyzer 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. All three of these factors, the slope, the y-intercept, and the r^2, had to be acceptable before a technology was considered accurate.

PRC used a normal deviate test statistic to determine whether the slope and y-intercept varied, at a two-tailed 95 percent confidence level, from what was expected. If the slope or y-intercept of the regression line varied greatly, then the two sets of data were considered comparable, yet different. In other words, the analyzer' s data was not accurate, yet there was a relationship between it and the confiitory laboratory' s data. This relationship would enable the analyzer' s results to be corrected mathematically if a certain number of samples were sent to a confiitory laboratory.

The linear regression for the L2000 PCB/Chloride Analyzer was based on results from 51 samples. The other results indicated that no PCBs were detected above the detection limit. The r^2 value for the regression was 0.522. This indicates that little or no relationship existed between the L2000 PCB/Chloride Analyzer' s results and those of the confiitory laboratory. This means that the analyzer' s results were not accurate. Figure 7-1 depicts the occurrence of correct and incorrect measurements by this technology relative to an action level of 10 mg/kg.

TABLE 7-5. COMPARISON OF L2000 PCB/CHLORIDE ANALYZER AND CONFIRMATORY DATA.

Sample No.	L2000 PCB/ Chloride Analyzer (2.0 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Difference	RPD	Sample No.	L2000 PCB/ Chloride Analyzer (2.0 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Difference	RPD
001	ND	0.593	NA	NA	035	ND	ND	ND	NA
002	ND	1.50	NA	NA	035D	ND	ND	ND	NA
003	ND	0.114	NA	NA	036	>2,000	816	NA	84.1
004	23.6	6.71J	16.9	111	037	< 4.0S	0.055J	NA	NA
005	ND	1.37	NA	NA	037D	< 4.0S	0.040J	NA	NA
006	ND	0.679	NA	NA	038	778	1030J	252	27.9
007	ND	0.552	NA	NA	039	ND	0.676	NA	NA
008	3.9	2.00	1.9	64.4	040	5.7	4.25	1.5	29
009	6.9	1.30J	5.6	136	041	ND	ND	ND	NA
010	5.1	0.172J	4.9	187	042	ND	0.517	NA	NA
011	2.7	1.15J	1.6	80.5	042D	ND	0.462J	NA	NA
012	ND	ND	ND	NA	043	4.1	1.69J	2.4	83
013	ND	1.13	NA	NA	043D	3.6	1.74	1.9	70
014	ND	0.18	NA	NA	044	ND	0.592J	NA	NA
015	9.4	9.13	0.27	2.9	045	ND	ND	ND	NA
015D	12.5	9.84	2.66	23.8	046	ND	ND	ND	NA
016	484	2,110	1626	125	046D	ND	ND	ND	NA
017	6.5	2.55	4.0	87.3	047	ND	0.094J	NA	NA
018	382	45.4	337	157	047D	ND	0.098J	NA	NA
019	71.1S	6.70	64.4	165	048	ND	ND	ND	NA
020	< 16S	0.068J	NA	NA	049	ND	ND	ND	NA
021	ND	0.063	NA	NA	050	ND	3.60	NA	NA
022	ND	0.535	NA	NA	050D	ND	4.41	NA	NA
022D	ND	0.718	NA	NA	051	ND	ND	ND	NA
023	48.8	20.8	28.0	80.5	052	9.3	4.21	5.1	75
024	ND	0.055	NA	NA	053	25.7	0.958	24.7	186
024D	ND	0.049	NA	NA	054	5.1	0.516J	4.584	163
025	3.5	11.7	8.2	35.0	055	4.4	2.40	2.0	59
026	ND	1.96	NA	NA	056	ND	0.505	NA	NA
027	ND	0.057	NA	NA	057	ND	ND	ND	NA
028	ND	0.216	NA	NA	058	ND	0.681	NA	NA
028D	ND	0.224J	NA	NA	059	ND	7.86	NA	NA
029	ND	0.229J	NA	NA	060	2.3	0.624J	1.7	115
030	ND	1.15	NA	NA	060D	4.4	0.577	3.8	154
031	ND	0.263	NA	NA	061	549	580	31	5.5
032	36.0	47.6	11.6	27.8	062	111	2.35	109	192
033	ND	6.00J	NA	NA	063	ND	0.092J	NA	NA
034	14.4	34.0	19.6	81.0	063D	ND	0.154J	NA	NA

TABLE 7-5. (CONTINUED)

Sample No.	L2000 PCB/ Chloride Analyzer (2.0 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Difference	RPD	Sample No.	L2000 PCB/ Chloride Analyzer (2.0 mg/kg)[a]	Confirmatory Laboratory (0.033 mg/kg)[a]	Difference	RPD
064	172	19.0	153	160	089	ND	45.0	NA	NA
065	ND	3.08	NA	NA	090	2.0	1.01	0.99	66
066	2.1	1.98	.12	5.9	090D	ND	1.40	NA	NA
067	7.5	0.081	7.4	196	091	1,650	1,630	20	1.1
068	8.0	0.504J	7.5	176	091D	1,608	1,704	96	5.8
069	5.8	ND	NA	NA	092	3.14	1.21	1.93	87.7
069D	4.4	ND	NA	NA	092D	3.4	ND	NA	NA
070	ND	ND	ND	NA	093	< 80.S	0.295	NA	NA
071	ND	0.052J	NA	NA	094	ND	0.362J	NA	NA
071D	ND	ND	ND	NA	095	20.6	17.5	3.1	16
072	ND	0.035J	NA	NA	095D	20.1	31.2	11.1	43.3
073	37.0	15.8	21.2	80.3	096	< 16.S	0.059J	NA	NA
074	22.0	13.3	8.7	49	097	ND	1.23	NA	NA
075	61.0	23.0	38.0	90.5	097D	ND	0.285	NA	NA
076	82.0	46.7	35.3	54.9	098	ND	1.17	NA	NA
077	ND	ND	ND	NA	098D	ND	0.825	NA	NA
078	21.0	2.27	18.7	161	099	ND	ND	ND	NA
079	148	42.8	105	110	100	384	177	207	73.8
080	ND	3.77	NA	NA	100D	363	167	196	74.0
081	ND	0.687	NA	NA	101	8.3	1.21	7.1	149
081D	ND	0.450	NA	NA	102	6.3	293	286.7	192
082	ND	ND	ND	NA	102D	5.0	1.77	3.2	95
082D	ND	0.244	NA	NA	103	75.2	40.3	34.9	60.4
083	ND	0.484	NA	NA	104	4.1	7.66	3.6	61
083D	ND	0.413	NA	NA	105	ND	0.210	NA	NA
084	7.6	1.16	6.4	147	106	ND	2.50	NA	NA
084D	10.9	1.08	9.82	164	107	161	14.1J	146.9	168
085	593	428	165	32.3	108	6.1	3.84J	2.3	46
085D	596	465	131	24.7	109	< 80.S	ND	NA	NA
086	ND	1.42	NA	NA	109D	10.3S	ND	NA	NA
086D	ND	1.25	NA	NA	110	ND	ND	ND	NA
087	ND	0.076	NA	NA	111	20.0S	ND	NA	NA
087D	ND	ND	ND	NA	112	240	315	75	27
088	ND	2.70	NA	NA	113	21.8	14.9	6.9	38
088D	ND	1.77	NA	NA	114	107	66.3	40.7	47

Notes:

[a] Detection limit.

ND Not detected above the detection limit.

NA Either the technology, the confirmatory laboratory, or both analyses produced nondetects.

J Reported amount is below the detection limit or not valid by approved quality control procedures.

S Sample matrix effects raised detection limit.

A residual analysis of the data identified that the r^2 was greatly influenced by the results of Sample 16, Sample 18, Sample 36 and Sample 91. PRC removed these four points as outliers and recalculated the linear regression. The second analysis was calculated on 47 sample results. This time, the r^2 factor was 0.86, indicating that a relationship existed between the two data sets. The analysis defined a regression line with a y-intercept of 26.6 mg/kg and a slope of 0.84. The standard deviation of the slope was 0.051, indicating relatively little variance around the regression line. The normal deviate test statistic indicated that the slope of 0.84 and the y-intercept of 26.6 mg/kg were significantly different from their expected values. This means that the results are not accurate, but can be corrected mathematically. In addition, the Wilcoxon Signed Ranks Test was used to verify these results and indicated, at a 95 percent confidence level, that the analyzer's data was significantly different from that of the confirmatory laboratory. This confirms that the results are not accurate.

Based on these results, the L2000 PCB/Chloride Analyzer's results should not be expected to be the same as those from a confirmatory laboratory. However, if 10 to 20 percent of the samples collected are also sent to a confirmatory laboratory, then the results from the other 80 to 90 percent could be corrected. This may result in a significant savings in analytical costs.

Precision

To compare the precision of the L2000 PCB/Chloride Analyzer's results to the precision of the confirmatory laboratory's results, a Dunnett's Test was performed on the RPDs determined from the field duplicates and their respective samples. Dunnett's Test determines, in a percentage, the probability that the data sets on which it is based are the same. If the RPDs from the confirmatory laboratory and those from the technology are the same, then it can be assumed that the precisions are also similar. For this demonstration, probabilities above 95 percent indicate that the precision of the technology and that of the confirmatory laboratory are the same. Lower probabilities indicate that one cannot be sure they are the same. This does not mean that the technology's precision is worse than that of the confirmatory laboratory, only that there is a greater probability that the precision of the two are different.

When Dunnett's Test compared the RPDs between the L2000 PCB/Chloride Analyzer's data and the confirmatory laboratory's data, a probability of 74 percent resulted. This indicates that the precision may be different from that of the confirmatory laboratory.

Section 8
Applications Assessment

This section summarizes the advantages and limitations of the two field screening technologies discussed in this ITER. It includes a discussion on how each technology' s characteristics might affect its use at hazardous waste sites and an assessment of how each technology might be used in the field.

Clor-N-Soil Test Kit

The principal advantages of the Clor-N-Soil Test Kit are that it is inexpensive and simple to operate, even for nontechnical personnel with little training. It is highly portable and can easily be used outdoors under limited site conditions. The test kit has a high sample throughput and is capable of quickly providing results.

One limitation of the Clor-N-Soil Test Kit is that it can produce a high number of false positive results for Aroclor 1242, which it was designed to detect. The test kit also is susceptible to reporting false positive results for samples containing interferants such as halogenated organic compounds. A second limitation is that results of the Aroclor specificity test performed during this demonstration indicated that there is a significant possibility that the Clor-N-Soil Test Kit will produce false negative results at sites where the contaminant is an Aroclor other than 1242. The test kit has substantially lower sensitivities to Aroclors 1016, 1221, and 1232, indicating that false negatives are likely at sites where these Aroclors are present. The test kit also is likely to produce a higher number of false positive results when Aroclors 1254 or 1260 are present. False negative results also are possible at sites where mercury and PCBs are both present in samples. All critical samples should be confirmed using EPA-approved methods.

The Clor-N-Soil Test Kit is most suitable for use at sites where the Aroclor of concern is positively known and where interferants such as halogenated organics which can produce false positives, or mercury, which can produce false negatives, are known not to be present. The test kit also would be useful at sites where a high number of false positive results would not represent a significant problem. An ideal use for the Clor-N-Soil Test Kit would be cleanups of transformer spills for example. At such a site, the Aroclor of concern could be identified with a high degree of certainty and the chance that interferants would be present would be small. Also, because only a small area would be affected, the cost of disposing of soil incorrectly identified as being contaminated would be low enough that the money saved by using the inexpensive Clor-N-Soil Test Kit might offset any extra disposal costs.

L2000 PCB/Chloride Analyzer

The L2000 PCB/Chloride Analyzer is inexpensive and easy to operate. It is portable, although electricity is required to operate it. It has a high sample throughput and is capable of quickly providing results. It has the additional advantage of being capable of providing quantitative results. During this demonstration, the quantitative results of the analyzer were not found to be particularly accurate, but it did produce linear results, which can easily be corrected by comparing them to results from a percentage of samples analyzed by a confirmatory laboratory. The analyzer is susceptible to reporting false positive results when interferants such as halogenated organic compounds are present in the samples. It also does not maintain its calibration well and must be recalibrated frequently, particularly when analyzing samples containing PCBs at concentrations above 100 mg/kg.

The results of the Aroclor specificity test performed during this demonstration indicated that there is a significant possibility that the L2000 Analyzer will produce false negative results at sites where the contaminant is an Aroclor other than 1242. In particular, the analyzer has substantially lower sensitivities to Aroclors 1016, 1221, and 1232, indicating that false negatives are likely at sites where these Aroclors are present. A higher number of false positive results will occur when Aroclor 1254 or 1260 is present.

The L2000 PCB /Chloride Analyzer is best suited for use at sites where the Aroclor of concern is positively known and where interferants such as halogenated organics which can produce false positives, are known not to be present. If the Aroclor is known and is one of those detectable by the analyzer, and if no interferants are suspected to be present, it would be useful at sites where quantitative results are needed quickly. However, the quantitative results reported by the analyzer must be corrected for them to be accurate. The correction factor must be determined by submitting 10 to 20 percent of the samples collected to a confirmatory laboratory for analysis using standard EPA analytical methods. This will increase the cost and lengthen the time required to obtain accurate, quantified results.

Section 9
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