

US EPA ARCHIVE DOCUMENT



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
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**ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT**

TECHNOLOGY TYPE:	POLYCHLORINATED BIPHENYL (PCB) FIELD ANALYTICAL TECHNIQUES
APPLICATION:	MEASUREMENT OF PCBs IN SOILS AND SOLVENT EXTRACTS
TECHNOLOGY NAME:	4100 VAPOR DETECTOR
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The U.S. Environmental Protection Agency (EPA) has created a program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the Environmental Technology Verification (ETV) Program is to further environmental protection by substantially accelerating the acceptance and use of improved and more cost effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. This document summarizes the results of a demonstration of the Electronic Sensor Technology (EST) 4100 Vapor Detector.

PROGRAM OPERATION

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

DEMONSTRATION DESCRIPTION

In July 1997, the performance of six PCB field analytical techniques was determined under field conditions. Each technology was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Performance evaluation (PE) samples also were used to assess independently the accuracy and comparability of each technology.

The demonstration was designed to detect and measure PCBs in soil and solvent extracts. The demonstration was conducted at the Oak Ridge National Laboratory (ORNL) in Oak Ridge, Tennessee from July 22 through July 29. The study was conducted under two environmental conditions. The first site was outdoors, with naturally fluctuating temperature and relative humidity conditions. The second site was inside a controlled environmental chamber, with generally cooler

temperatures and lower relative humidities. Multiple soil types, collected from sites in Ohio, Kentucky, and Tennessee, were analyzed in this study. Solutions of PCBs were also analyzed to simulate extracted surface wipe samples. The results of the soil and extract analyses conducted under field conditions by the technology were compared with results from analyses of homogeneous replicate samples conducted by conventional EPA SW-846 methodology in an approved reference laboratory. Details of the demonstration, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Portable Gas Chromatograph/Surface Acoustic Wave Detector, Electronic Sensor Technology 4100 Vapor Detector*, EPA/600/R-98/114.

TECHNOLOGY DESCRIPTION

A handheld, portable (35 lbs.) chromatography system equipped with a non-specific Surface Acoustic Wave (SAW) detector is used to speciate and quantify PCBs. The SAW detector is an integrating mass detector (micro-balance) with the ability to quantify chromatographic peaks, with peak widths measured in milliseconds. Measurement speed makes the instrument well suited to rapid screening of soil samples. Early separation of those soil samples below the regulatory level from those which require laboratory validation by GC/MS reduces the cost associated with site characterization and monitoring.

A sampling pump and loop trap are used to sample and inject analyte into a GC capillary column. Speciation is based upon retention time measurements using a temperature programmed DB-5 column. Quantification is based upon the frequency shift produced by the PCB congeners as they exit the GC column. By focusing the effluent onto a specific area on the surface of a temperature controlled piezoelectric crystal, high sensitivity is achieved with a 10 second analysis time. The 4100 Vapor Detector is able to screen selectively and quantify PCB levels of Aroclors in soil and flyash.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the 4100 Vapor Detector were observed:

Detection limits: EPA defines the method detection limit (MDL) as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. Because there was a significant "site effect" inherent to the PE samples, separate MDLs were calculated for both the outdoor and chamber conditions. The MDL was calculated to be 26 ppm under outdoor conditions and 62 ppm under chamber conditions.

Throughput: Throughput was 5 to 6 samples/hour under outdoor conditions and 10 samples/hour under chamber conditions. This rate included sample preparation and analysis.

Ease of Use: Two operators were used for the demonstration due to the number of samples and working conditions, but the technology can be run by a single operator. Operators generally require several hours of training and should have a basic knowledge of gas chromatographic techniques. These methods should be used by, or under the supervision of, analysts experienced in the use of sampling techniques and gas chromatography.

Completeness: The 4100 generated results for all 232 PCB samples for a completeness of 100%.

Blank results: EST did not specify a method detection limit prior to the demonstration, therefore, any PCB concentration that was detected was considered real. PCBs were detected in all of the soil blanks, resulting in 100% false positive results. PCBs were also detected in 3 of 8 of the extract blanks or 38% false positive results. The 4100 reported 5% false negative results for soils and no false negative results for extracts.

Precision: The overall precision, based on average relative standard deviations (RSDs), was 87% for soil samples and 65% for extract samples. The 4100 was imprecise compared to the reference laboratory's precision (21% for soils and 14% for extracts).

Accuracy: Accuracy was assessed using PE soil and extract samples. The study was conducted under two experimental conditions to detect and control for "site effects." The data showed that the 4100 exhibited a significant site effect, and the results were generally biased high. The overall accuracy, based on average percent recoveries, was 177% (outdoor site) and 631% (chamber) for PE soil samples. For the extract samples, the results indicated a high bias (267% recovery) on the lower concentration samples and a low bias on the higher concentration samples (54% recovery).

Comparability: The demonstration showed that the 4100 generated data that exhibited low correlation to the reference laboratory data. The coefficient of determination (R^2) which is a measure of the degree of correlation between the reference laboratory and the 4100 data was 0.177 when all soil samples (0 to 700 ppm) were considered. For the concentration range from 0 to 125 ppm, the R^2 value was 0.115. Most of the percent difference values were greater than 100%, when the 4100 results were compared directly with the reference laboratory results. The comparability of the extract samples also exhibited low correlation.

Regulatory Decision-making: One objective of this demonstration was to assess the technology's ability to perform at regulatory decision-making levels for PCBs, specifically 50 ppm for soils and 100 $\mu\text{g}/100\text{cm}^2$ for surface wipes. For PE and environmental soil samples in the range of 40 to 60 ppm, the precision was low (72% RSD) and the accuracy was variably biased with both high and low recoveries (an average recovery of 132%). For extract samples representing surface wipe sample concentrations of 100 $\mu\text{g}/100\text{cm}^2$ and 1000 $\mu\text{g}/100\text{cm}^2$ (assuming a 100 cm^2 wipe sample), measurements were also imprecise (65% RSD) and indicated a high bias (161% recovery).

Data quality levels: The overall performance of the EST 4100 Vapor Detector was characterized as biased and imprecise. EST is working to improve the performance of the methodology.

The results of the demonstration show that certain cautions should be considered when using this technology for PCB analysis due to its bias and imprecision. This technology should be employed in well-defined applications for PCB analysis, and only in conjunction with a stringent quality assurance plan. As with any technology selection, the user must determine if the technology is appropriate for the application and the project data quality objectives. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

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