UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Research and Development
Washington, D.C. 20460

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

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VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**FIELD PORTABLE X-RAY FLUORESCENCE ANALYZER**
APPLICATION:	**MEASUREMENT OF METALS IN SOIL**
TECHNOLOGY NAME:	**X-MET 920-MP**
COMPANY:	**METOREX INC.**
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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 Metorex X-MET 920-MP.

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. The EPA's National Exposure Research Laboratory, which conducts demonstrations of field characterization and monitoring technologies, selected PRC Environmental Management, Inc., as the testing organization for the performance verification of field portable X-ray fluorescence (FPXRF) analyzers.

DEMONSTRATION DESCRIPTION

In April 1995, the performance of seven FPXRF analyzers was determined under field conditions. Each analyzer was independently evaluated by comparing field analysis results to those obtained using approved reference methods. Standard reference materials (SRM) and performance evaluation (PE) samples also were used to independently assess the accuracy and comparability of each instrument.

The demonstration was designed to detect and measure a series of inorganic analytes in soil. The primary target analytes were arsenic, barium, chromium, copper, lead, and zinc; nickel, iron, cadmium, and antimony were secondary analytes. The demonstration sites were located in Iowa (the RV Hopkins site) and Washington (the ASARCO site). These sites were chosen because they exhibit a wide range of concentrations for most of the target metals and are located in different climatological regions of the United States; combined, they exhibit three distinct soil types: sand, clay, and loam. The conditions at these sites are representative of those environments under which the technology would be expected to operate. Details of the demonstration, including a data summary and

discussion of results, may be found in the report entitled "Environmental Technology Verification Report, Field Portable X-ray Fluorescence Analyzer, Metorex X-MET 920-MP." The EPA document number for this report is EPA/600/R-97/151.

The EPA SW-846 Method 6200 was tested and validated using the data derived from this demonstration. This method may be used to support the general application of FPXRF for environmental analysis.

TECHNOLOGY DESCRIPTION

This analyzer operates on the principle of energy dispersive X-ray fluorescence spectroscopy where the characteristic energy components of the excited X-ray spectrum are analyzed directly by an energy proportional response in an X-ray detector. Energy dispersion affords a highly efficient, full-spectrum measurement which enables the use of low intensity excitation sources (such as radioisotopes) and compact battery-powered, field-portable electronics. The FPXRF instruments are designed to provide rapid analysis of metals in soil. This information allows investigation and remediation decisions to be made on-site and reduces the number of samples that need to be submitted for laboratory analysis. In the operation of these instruments, the user must be aware that FPXRF analyzers do not respond well to chromium and that field detection limits may be 5 to 10 times greater than conventional laboratory methods. As with all field collection programs, a portion of the samples should be sent to a laboratory for confirmatory analyses.

The X-MET 920-MP is designed to produce quantitative data on metals in soils, sludges, and other solids. The X-MET 920-MP consists of a laptop computer, an electronics unit, and a surface analysis probe system (SAPS). The electronics is housed in a rugged, weatherproof, self-contained case, weighing about 5 pounds that can be operated from battery power up to 8 hours. The SAPS is designed to house one excitation source (cadmium-109 for this demonstration) and a gas-filled proportional counter detector. The SAPS weighs about 3 pounds and is specifically designed for *in situ* analysis, but can be adapted for measurement of samples in cups. The single excitation source limits the number of metals that can be quantified. The X-MET 920-MP is operated and calibrated using the "X-MET" software to analyze samples with an empirical calibration. Training and field experience is necessary to successfully derive empirical calibration curves and to operate the "X-MET" software. The X-MET 920-MP reported the analytes arsenic, barium, copper, chromium, lead, nickel, and zinc for this demonstration using source count times between 30 and 180 seconds. At the time of the demonstration, the cost of the X-MET 920-MP with the SAPS probe and cadmium-109 source (including the laptop computer) was $36,325, or it could be leased for $3,633 per month.

VERIFICATION OF PERFORMANCE

The performance characteristics of the X-MET 920-MP include the following:

- **Detection limits:** Precision-based detection limits were determined by collecting 10 replicate measurements on site-specific soil samples with metals concentrations 2 to 5 times the expected MDLs. The results were less than or equal to 50 milligrams per kilogram (mg/kg) for all analytes except barium (330 mg/kg) and chromium (115 mg/kg). Barium is normally analyzed using an americium-241 source; therefore, its detection limit was expected to be high. A value for nickel could not be determined because the soil concentration of this analyte was too low.
- **Throughput:** Average throughput was found to be between 8 and 14 analyses per hour, depending on count times. This rate only represents the analysis time since different personnel were used to prepare the samples.
- **Drift:** Based on an evaluation of results from periodic analysis of a site-specific control sample, with a few exceptions, drift was -15 to +15 percent. Lead and arsenic displayed the least drift at both sites.
- **Completeness:** The X-MET 920-MP produced results for 1,168 of the 1,260 samples for a completeness of 92.7 percent. This was less than the demonstration objective of 95 percent. Operator error and computer software and hardware problems reduced completeness. None of the data loss was caused by mechanical or electronic malfunctions of the analyzer.

- **Blank results:** The X-MET 920-MP reported values for arsenic and copper above the precision-based method detection limits at the ASARCO site and values for chromium, lead, and zinc above the MDL at the RV Hopkins site. Analyzer blanks were composed of a pure lithium carbonate that was processed using the sample preparation steps.
- **Precision:** The goal of the demonstration was to achieve relative standard deviations (RSD) less than 20 percent at analyte concentrations of 5 to 10 times the method detection limit. The RSD values for the reported analytes were less than 8 percent. Chromium and nickel were not determined due to a lack of sufficient data in the specified concentration range.
- **Accuracy:** Intramethod accuracy was assessed using site-specific soil PE samples. The results showed that 7 of 32 (21.9 percent) of the PE sample analytes had recoveries within a quantitative acceptance range of the 80 - 120 percent.
- **Comparability:** This demonstration showed that the X-MET 920-MP produced data that exhibited a $\log_{10}$-$\log_{10}$ linear correlation to the reference data. The coefficient of determination (r^2) which is a measure of the degree of correlation between the reference and field data was 0.95 for arsenic, 0.88 for lead, 0.69 for copper, 0.68 for chromium, and 0.55 for zinc. Using data from the RV Hopkins clay soil produced values of 0.62 for barium and 0.32 for nickel.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the X-MET 920-MP produced definitive level data for arsenic and lead and data of qualitative screening level for copper, barium, and zinc. No recommendation regarding data quality for chromium or nickel could be made due to a lack of precision or comparability data.

The results of the demonstration show that the Metorex X-MET 920-MP can provide useful, cost-effective data for environmental problem-solving and decision-making. Undoubtedly, it will be employed in a variety of applications, ranging from serving as a complement to data generated in a fixed analytical laboratory to generating data that will stand alone in the decision-making process. As with any technology selection, the user must determine what is appropriate for the application and the project data quality objectives.

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