



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, D.C. 20460



ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM VERIFICATION STATEMENT

TECHNOLOGY TYPE: **FIELD PORTABLE X-RAY FLUORESCENCE ANALYZER**
APPLICATION: **MEASUREMENT OF METALS IN SOIL**
TECHNOLOGY NAME: **XL SPECTRUM ANALYZER**
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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 Niton XL Spectrum Analyzer.

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, Niton XL Spectrum Analyzer." The EPA document number for this report is EPA/600/R-97/150.

The EPA 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 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 that enables the use of low intensity excitation sources (such as radioisotopes) and compact battery-powered, field-portable electronics. 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 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 Niton XL Spectrum Analyzer was originally designed to produce quantitative data for lead in painted surfaces. This demonstration found that it could also provide quantitative data for metals contamination in soil. It is a hand-held instrument, weighing less than 3 pounds, and can be battery powered up to 8 hours. It uses a single radioactive source (cadmium-109) and a silicon pin-diode detector for the analysis of metals in soil using relatively short count times (60 seconds). The single radioactive source limits the number of analytes that can be detected. For the purposes of this demonstration, the XL Spectrum Analyzer's "SOILAIR" software was configured to report concentrations of arsenic, chromium, copper, lead, and zinc in soil samples. The XL Spectrum Analyzer was initially calibrated by the developer using the Compton normalization method to quantitate metals. The XL Spectrum Analyzer can conduct *in situ* measurements or measure samples in cups. The cost of the Niton XL Spectrum Analyzer was \$11,990 at the time of the demonstration, or it could be leased for \$2,200 per month.

VERIFICATION OF PERFORMANCE

The performance characteristics of the Niton XL Spectrum Analyzer 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 130 milligrams per kilogram (mg/kg) or less for all of the reported target analytes except chromium, which was determined to be 900 mg/kg.
- **Throughput:** Average throughput was 20 - 25 analyses per hour using a live count time of 60 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.
- **Drift:** This was evaluated using the results of an analysis of an SRM calibration check sample which contained quantifiable levels of arsenic, copper, lead, zinc, and iron. Over the course of the demonstration, this sample was analyzed approximately 100 times. The mean recovery for these analytes was between 85 and 140 percent. The drift RSD for the mean recovery of these analytes was less than 8 percent.
- **Completeness:** The XL Spectrum Analyzer produced results for 1,258 of the 1,260 samples for a completeness of 99.8 percent. The two lost data points were a consequence of operator error.
- **Blank results:** More than 100 lithium carbonate blanks were analyzed during the demonstration. None of the reported analytes were observed above the method detection limits.
- **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 limits. The RSD value for arsenic was

9.2 percent, 13.2 percent for copper, 6.5 percent for lead, and 11.2 percent for zinc. Chromium was not reported due in part to the short 60 live-second count time.

- **Accuracy:** Intramethod accuracy was assessed using site-specific soil PE samples and soil SRMs. The data showed that 18 of 28 or 64.2 percent of the PE sample analytes had recoveries within the quantitative acceptance range of 80 - 120 percent. For the soil SRMs, 11 of 16 (68.7 percent) of the results were within the 80 - 120 percent recovery range.
- **Comparability:** This demonstration showed that the XL Spectrum Analyzer 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 0.82 for arsenic, 0.50 for chromium, 0.92 for copper, 0.96 for lead, and 0.89 for zinc.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the XL Spectrum Analyzer produced definitive level data for lead and data of quantitative screening level for arsenic, copper, and zinc. Since a precision RSD value was not determined for chromium, no data quality level can be assigned.

The results of the demonstration show that the Niton XL Spectrum Analyzer 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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NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and the appropriate quality assurance procedures. EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always, under circumstances other than those tested, operate at the levels verified. The end user is solely responsible for complying with any and all applicable Federal, State, and Local requirements.