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

**ENVIRONMENTAL TECHNOLOGY
VERIFICATION PROGRAM**

ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM
VERIFICATION STATEMENT

TECHNOLOGY TYPE:	**FIELD PORTABLE X-RAY FLUORESCENCE ANALYZER**
APPLICATION:	**MEASUREMENT OF METALS IN SOIL**
TECHNOLOGY NAME:	**TN Pb ANALYZER**
COMPANY:	**TN SPECTRACE**
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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 Spectrace TN Pb 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, Spectrace TN 9000 and TN Pb Field Portable X-ray Fluorescence Analyzers." The EPA document number for this report is EPA/600/R-97/145.

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 energy components of the excited X-ray spectrum are analyzed directly by an energy proportional response in the 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 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 TN Pb Analyzer was specifically designed to analyze for lead in a variety of matrices. It is field portable, weighing less than 20 pounds, and can be battery powered up to 8 hours. It uses a single radioactive source (cadmium-109) and a mercuric iodide semiconductor detector to analyze metals in soils using relatively short count times (60 seconds). The single radioactive source limits the number of analytes which can be detected. In this demonstration, the TN Pb Analyzer's Soils Application software was configured to report concentrations for arsenic, chromium, copper, lead, and zinc. Contaminant concentrations are computed using a fundamental parameters (FP) calibrated algorithm included in the analyzer's operations software. The FP method does not require site-specific calibration samples; however, such samples can be used to customize the calibration to a particular matrix. The TN Pb Analyzer can conduct *in situ* measurements or measure samples in cups. At the time of the demonstration, the cost of the TN Pb Analyzer was about $39,500, or it could be leased for $5,500 per month or $3,000 for 2 weeks.

VERIFICATION OF PERFORMANCE

The performance characteristics of the TN Pb 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 115 milligrams per kilogram (mg/kg) or less for arsenic, copper, lead, and zinc. Chromium was determined to be 460 mg/kg.

- **Throughput:** Average throughput was between 20 and 25 analyses per hour using a count time of 60 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.

- **Drift:** Based on a daily analysis of an SRM, which contained quantifiable levels of arsenic, copper, lead, and zinc, the drift RSD values for the mean recovery of these analytes were less than 8 percent.

- **Completeness:** The TN Pb Analyzer produced results for all of the 1,260 samples for a completeness of 100 percent.

- **Blank results:** During the demonstration, a total of 20 SiO_2 blank samples was analyzed. None of the reported analytes were detected above the method detection limits.

- **Precision:** The goal of the demonstration was to achieve relative standard deviations (RSD) of less than 20 percent at analyte concentrations of 5 to 10 times the method detection limits. The RSD values for arsenic, copper, lead, and zinc were less than 10 percent. A value for chromium was not determined due to a lack of sufficient samples in the required concentration range.

- **Accuracy:** Intramethod accuracy was assessed using site-specific PE soil samples and soil SRMs. The data showed that 20 of 28 results (71.4 percent) of the PE sample analytes had recoveries within the 80 - 120 percent quantitative acceptance range and that 100 percent (16 of 16) of the SRM analytes were within this range. Results were more accurate when the sample closely matched the matrix used to set the fundamental parameters calibration.

- **Comparability:** This demonstration showed that the TN Pb 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 0.95 for arsenic, 0.94 for copper, 0.95 for lead, and 0.92 for zinc. The value for chromium, 0.55, was derived primarily from clay soil at the RV Hopkins site.

- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the TN Pb Analyzer produced definitive level data for arsenic, copper, lead, and zinc. A value for chromium could not be assigned without adequate precision data.

The results of the demonstration show that the Spectrace TN Pb 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.