



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: **TN 9000**
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 9000 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 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. 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 analysis programs, a portion of the samples should be sent to a laboratory for confirmatory analyses.

The TN 9000 is a commercially available instrument that can use up to three radioactive sources and a mercuric iodide semiconductor detector for the analysis of metals in soil. It is field portable, weighing less than 20 pounds, and can be battery powered for up to 8 hours. For this demonstration, the TN 9000's Soils Application software was configured to report concentrations for chromium, iron, nickel, copper, zinc, arsenic, lead, cadmium, antimony, and barium. Contaminant concentrations are computed using a fundamental parameters (FP) calibrated algorithm included in the analyzer's operations software. The FP calibration does not require site-specific calibration standards. The TN 9000 can conduct *in situ* measurements or measure samples in cups. At the time of testing, the TN 9000 cost about \$58,000, or it could be leased for \$6,000 per month or \$3,500 for 2 weeks.

VERIFICATION OF PERFORMANCE

The performance characteristics of the TN 9000 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 100 milligrams per kilogram (mg/kg) or less for all of the reported analytes except chromium, which was determined to be 200 mg/kg using the Fe⁵⁵ source and 500 mg/kg using the Cd¹⁰⁹ source which was used for the other reported analytes. Values for iron and cadmium were not reported due to an insufficient number of samples in the required concentration range.
- **Throughput:** Average throughput was 8.5 to 10.5 analyses per hour using a source count time of 220 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 a daily analysis of an SRM which contained quantifiable levels of arsenic, barium, copper, lead, and zinc. Over the 18 days of the demonstration, the RSD values for the mean recovery of barium, copper, lead, and zinc were all less than 8 percent. The corresponding value for arsenic was 18.2 percent.
- **Completeness:** The TN 9000 produced results for 1,259 of the 1,260 samples analyzed, resulting in a completeness of 99.9 percent. The remaining sample was lost due to operator error in transferring the data.
- **Blank results:** During the demonstration, 37 blank samples were analyzed. None of the reported primary analytes were detected above the method detection limits. However, iron frequently reported a value above the MDL after analyzing samples with an iron concentration of greater than 20,000 mg/kg.
- **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 antimony,

arsenic, barium, copper, lead, and zinc were less than 8 percent. The corresponding value for chromium was 22 percent. Values for cadmium, iron, and nickel were not reported because too few samples containing the required concentrations were measured.

- **Accuracy:** Intramethod accuracy was assessed using site-specific soil PE samples and soil SRMs. The data showed that 37 of 41 or 90.2 percent of the PE sample analytes had recoveries within the quantitative acceptance range of 80 - 120 percent. A corresponding 22 of 24 or 91.7 percent of the SRM analytes were within the 80 - 120 percent recovery range. The TN 9000 gave more accurate results when the reference sample closely matched the matrix used to set the fundamental parameters calibration for the analyzer.
- **Comparability:** This demonstration showed that the TN 9000 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, 0.95 for copper, 0.96 for lead, 0.93 for zinc, 0.79 for barium, and 0.78 for chromium. Results for iron, nickel, cadmium, and antimony were not reported due to limited sample data.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, the TN 9000 produced definitive level data for arsenic, copper, lead, and zinc; quantitative level data for barium; and data of qualitative screening level for chromium. Values for iron, nickel, cadmium, and antimony could not be assigned without adequate precision or comparability data.

The results of the demonstration show that the Spectrace TN 9000 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.