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Environmental Protection
Agency

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EPA

Innovative Technology Verification Report

Field Portable X-ray Fluorescence Analyzer

HNU Systems SEFA-P

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: **SEFA-P 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

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

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, HNU Systems SEFA-P." The EPA document number for this report is EPA/600/R-971 144.

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 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 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 SEFA-P Analyzer can use up to three radioactive sources with a lithium-drifted silicon detector to analyze a large number of metals in a variety of matrices. The SEFA-P is a transportable (weighs about SO pounds) analyzer that operates in the intrusive mode (it only measures samples in cups). The SEFA-P Analyzer was able to measure all 10 target analytes for this demonstration. Most of the sample analysis data were collected after the actual demonstration. The SEFA-P Analyzer supplied by the developer experienced a malfunction when the source holder locked into place, not allowing sample exposure and analysis. This incident occurred at the start of the demonstration at the ASARCO site. HNU was unable to supply a replacement unit in time to continue the demonstration; an EPA-owned SEFA-P Analyzer was subsequently used to analyze a subset of 100 demonstration samples, The samples chosen represented all three soil types, a wide range of concentrations from each of the sites, and included all of the PE and SRM samples from the demonstration. The SEFA-P Analyzer with three sources cost $49,000 at the time of the demonstration.

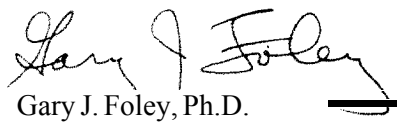
VERIFICATION OF PERFORMANCE

The performance characteristics of the SEFA-P 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 120 milligrams per kilogram (mg/kg) for antimony and lead, 225 mg/kg for copper, and 360 mg/kg for arsenic. No values were reported for cadmium, chromium, or nickel due to an insufficient number of samples in the target concentration range. Values for iron, zinc, and barium were 900,990, and 1150 mg/kg, respectively.
- **Throughput:** Average throughput was found to be 7 to 8 analyses per hour using a live count time of 240 seconds. This rate only represents the analysis time since different personnel were used to prepare the samples.
- **Drift:** A quantitative assessment of drift was performed using a calibration check standard which was analyzed at the beginning and end of each day. The drift RSD values for the mean recovery of the target analytes ranged from 0 to 35 percent.
- **Completeness:** The SEFA-P Analyzer produced results for 100 of the 100 samples for a completeness of 100 percent. However, prior to the mechanical failure in the field, the SEFA-P Analyzer was to have analyzed 630 samples.
- **Blank results:** None of the target analytes were reported above the precision-based method detection limits in the lithium carbonate blanks.

- **Precision:** The goal of the demonstration was to achieve relative standard deviations (RSD) less than 20 percent at analyte concentrations 5 to 10 times the method detection limits. The RSD values for antimony, barium, copper, and lead were less than 8 percent. The number of samples limited a complete assessment of this parameter; no values were reported for arsenic, cadmium, chromium, iron, nickel, or zinc.
- **Accuracy**: Accuracy was assessed using site-specific soil PE samples and soil SRMs. The SEFA-P Analyzer reported 7 of 33 or 21.2 percent of ail analytes in the site-specific soil PE samples and 3 of 18 or 16.7 percent of the soil SRMs within the quantitative acceptance range of 80 - 120 percent.
- **Comparability**: This demonstration showed that the SEFA-P Analyzer produced data that exhibited a log,, $\log_{10}$ linear correlation to the reference data. The coefficient of determination (1-2) measured using a Compton calibration represents the degree of correlation between the reference and field data. For this demonstration, the coefficient of determination was 0.89 for antimony, 0.95 for arsenic, 0.73 for barium, 0.64 for cadmium, 0.35 for chromium, 0.92 for copper, 0.76 for iron, 0.97 for lead, and 0.89 for zinc. No value was reported for nickel due to limited sample data.
- **Data quality levels:** Using the demonstration derived precision RSD results and the coefficient of determination as the primary qualifiers, using data generated from the Compton ratio calibrations, the SEFA-P Analyzer produced definitive level data for copper, antimony, and lead; data of quantitative screening level were produced for barium, Without adequate precision or comparability data, levels for the other six analytes could not be assigned.

The results of the demonstration show that the HNU SEFA-P portable X-ray fluorescence 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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