United States
Environmental Protection
Agency

Office of Research and
Development
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Innovative Technology Verification Report

Field Measurement Technology for Mercury in Soil and Sediment

NITON's Xli/XLt 700 Series X-Ray Fluorenscence Analyzer

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Office of Research and Development
Washington, DC 20460

MEASUREMENT AND MONITORING TECHNOLOGY PROGRAM VERIFICATION STATEMENT

TECHNOLOGY TYPE: **Field Measurement Device**

APPLICATION: **Measurement for Mercury**

TECHNOLOGY NAME: **NITON's® XLi/XLt 700™ Series Environmental Analyzers**

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VERIFICATION PROGRAM DESCRIPTION

The U.S. Environmental Protection Agency (EPA) created the Superfund Innovative Technology Evaluation (SITE) and Measurement and Monitoring Technology (MMT) Program s to facilitate deployment of innovative technologies through performance verification and information dissemination. The goal of these programs is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. These programs assist and inform those involved in design, distribution, permitting, and purchase of environmental technologies. This document summarizes results of the demonstrations of two XLi/XLt 700 Series X-ray Fluorescence Analyzers developed by NITON Inc.

PROGRAM OPERATION

Under the SIT E and MMT Programs, with the full participation of the technology developers, the EPA evaluates and documents the performance of innovative technologies by developing demonstration plans, conducting field tests, collecting and analyzing demonstration data, and preparing reports. The technologies are evaluated under rigorous quality assurance (QA) protocols to produce well-documented data of known quality. The EPA National Exposure Research Laboratory, which demonstrates field sampling, monitoring, and measurement technologies, selected Science Applications International Corporation as the verification organization to assist in field testing five field measurement devices for mercury in soil and sediment. This demonstration was funded by the SITE Program.

DEMONSTRATION DESCRIPTION

In May 2003, the EPA conducted a field demonstration of the XLi/XLt 700 Series Analyzers XLi 702 (isotope) and XLt 792 (X-ray tube) and four other field measurement devices for mercury in soil and sediment. This verification statement focuses on these two analyzers; a similar statement has been prepared for each of the other four devices. The performance of each of these two X-ray fluorescence analyzers was compared to that of an off-site laboratory using the reference method, "Test Methods for Evaluating Solid Waste" (SW -846) Method 7471B (modified). To verify a wide range of performance attributes, the demonstration had both primary and secondary objectives. The primary objectives were:

(1) Determining the instrument sensitivity with respect to the Method Detection Limit (MDL) and Practical Quantitation Limit (PQL);
(2) Determining the analytical accuracy associated with the field measurement technologies;
(3) Evaluating the precision of the field measurement technologies;
(4) Measuring the amount of time required for mobilization and setup, initial calibration, daily calibration, sample analysis, and demobilization; and
(5) Estimating the costs associated with mercury measurements for the following four categories: capital, labor, supplies, and investigation-derived waste (IDW).

Secondary objectives for the demonstration included:

(1) Documenting the ease of use, as well as the skills and training required to properly operate the devices;
(2) Documenting potential health and safety concerns associated with operating the devices;
(3) Documenting the portability of the devices;
(4) Evaluating the devices durability based on their materials of construction and engineering design; and

(5) Documenting the availability of the devices and associated spare parts.

The XLi/XLt 700 Series Analyzers analyzed 62 field soil samples, 23 field sediment samples, 42 spiked field samples, and 70 performance evaluation (PE) standard reference material (SRM) samples in the demonstration. The field samples were collected in four areas contaminated with mercury, the spiked samples were from these same locations, and the PE samples were obtained from a commercial provider.

Collectively, the field and PE samples provided the different matrix types and the different concentrations of mercury needed to perform a comprehensive evaluation of the XLi/XLt 700 Series Analyzers. A complete description of the demonstration and a summary of the results are available in the Innovative Technology Verification Report: "Field Measurement Technology for Mercury in Soil and Sediment—NITON's XLi/XLt 700 Series X-Ray Fluorescence Analyzers" (EPA/600/R-03/148).

TECHNOLOGY DESCRIPTION

The NITON XL 700 series analyzer is an energy dispersive X-ray fluorescence (EDXRF) spectrometer that uses either a Cd-109 radioactive isotope (XLi model) or a low powered miniature X-ray tube with a silver target (X Lt model) to excite characteristic X-rays of a test sample's constituent elements. These characteristic X-rays are continuously detected, identified, and quantified by the spectrometer during sample analysis. The energy of each X-ray detected identifies a particular element present in the sample, and the rate at which X-rays of a given energy are counted provides a determination of the quantity of that element that is present in the sample.

Detection of the characteristic mercury X-rays is achieved using a highly-efficient, therm o-electrically cooled, solid-state detector. Signals from this detector are amplified, digitized, and then quantified via integral multichannel analysis and data processing units. Results are displayed in ppm (mg/kg) of total elemental mercury.

The NITON XLt 700 Series Analyzer with X-ray tube excitation provides the user with the speed and efficiency of X-ray tube excitation, while reducing the regulatory demands typically encountered with isotope-based systems. In most cases, the X-ray tube equipped XLt 700 analyzer can be shipped between most states and countries with minimal paperwork and expense. The XLi and XLt 700 Series Analyzers offer testing m odes for soil and other bulk samples; filters, wipes and other thin sam ples; and lead-based paint. Testing applications include management of remediation projects, site assessments, and compliance testing. They provide simultaneous analysis of up to 25 elements, including all eight of the characteristic metals under the Resource Conservation and Recovery Act (RCRA). XRF analysis is nondestructive, so screened samples can be sent to an accredited laboratory for confirmation of results obtained on-site.

NITON's software corrects automatically for variations in soil m atrix and density, making it applicable for both in-situ and intrusive testing.

ACTION LIMITS

Action limits and concentrations of interest vary and are project specific. There are, however, action limits which can be considered as potential reference points. The EPA Region IX Preliminary Remedial Goals (PRGs) for mercury are 23 mg/k g in residential soil and 310 mg/k g in industrial soil.

VERIFICATION OF PERFORMANCE

To ensure data usability, data quality indicators for accuracy, precision, representativeness, completeness, comparability, and sensitivity were assessed for the reference method based on project-specific QA objectives. Key demonstration findings are summarized below for the primary objectives.

***Sensitivity*:** The two primary sensitivity evaluations performed for this demonstration were the MDL and PQL. Both will vary dependent upon whether the matrix is a soil, waste, or aqueous solution. Only soils/sediments were tested during this demonstration, and therefore, MDL calculations and PQL determinations for this evaluation are limited to those matrices. By definition, values measured below the PQL should not be considered accurate or precise and those below the MDL are not distinguishable from background noise.

Method Detection Lim it - The evaluation of an MDL requires seven different measurements of a low concentration standard or sample following the procedures established in the 40 Code of Federal Regulations (CFR) Part 136, the range of the MDL for the NITON X-ray tube instrument is between 13.9 and 69.8 mg/k g. It is likely that the MDL is closer to the lower end of this range based upon the results for sample lot 62 (referee laboratory value = 14.6 mg/kg) and sample lot 47 (SRM value = 32.4 mg/kg) which both had one of the seven results reported as below the NITON detection level indicating that these values are on the edge of the instruments detection capability. The lowest calculated MDL for the NITON Isotope instrument is 39.3 mg/kg. Based upon results presented in the report, the MDL for the NITON Isotope field instrument is close to 32 mg/kg. The equivalent calculated MDL for the referee laboratory is 0.0026 mg/kg.

Practical Quantitation Limit - The NITON X-ray PQL is somewhere between 62.9 mg/kg and 99.8 mg/kg. The %D for the 99.8 mg/kg SRM is 8.2%. The NITON Isotope PQL is also between 62.9 mg/kg and 99.8 mg/kg. The %D for the 99.8 mg/kg SRM is 9.2%. The referee laboratory PQL confirmed during the demonstration is 0.005mg/kg, with a %D

<10%.

***Accuracy*:** The results from the XLi/XLt 700 Series Analyzers were compared to the 95% prediction interval for the SRM materials and to the referee laboratory results (Method 7471B). NITON X-ray data were within SRM 95% prediction intervals 93% of the time, which suggests significant equivalence to certified standards. NITON Isotope data were within SRM 95% prediction intervals 91% of the time, which also suggest significant equivalence to certified standards.

The statistical comparison between the NITON X-ray field data and the referee laboratory results suggest that the two data sets are not the same. The statistical comparison between the NITON Isotope field data and the referee laboratory results also suggest that these two data sets are not the same. Because the NITON data compare favorably to the SRM values, the differences between NITON and the referee laboratory are likely the result of matrix interferences for field sample analysis. The number of NITON X-ray average values less than 30% different from the referee laboratory results or SRM reference values; however, was 14 of 26 different sample lots. Only 1 of 26 NITON average results have relative percent differences greater than 100% for this sam e group of sam ples. The number of NITON Isotope average values less than 30% different from the referee laboratory results or SRM reference values was 14 of 24 different sample lots. Zero of 24 NITON Isotope average results have relative percent differences greater than 100% for this same group of samples. Both NITON X-ray and NITON Isotope results; therefore, can provide a reasonable estimate of accuracy for field determination.

***Precision*:** The precision of the NITON X-ray and NITON Isotope field instruments is better then the referee laboratory precision. The overall average RSD is 20.0% for the referee laboratory, compared to the NITON X-ray overall average RSD of 13.1% and the NITON Isotope overall average RSD of 14.4%. Both the laboratory and NITON precision goals are within the predicted 25% RSD objective for precision expected from both analytical and sampling variance.

***Measurement Time*:** From the time of sample receipt, NITON required 17.5 hours (35 man hours) to prepare a draft data package of mercury results for 197 samples for both devices. Two technicians performed all setup, sample preparation and analysis, and equipment demobilization. Individual measurements took approximately 120 seconds each (after sample preparation), but the total time per analysis averaged 5.3 minutes when all field activities and data package preparation were included and only one technician per device is included in the calculation.

***Measurement Costs*:** The cost per analysis based upon 197 samples, when renting the XLi 702, is $39.52 per sample. The cost per analysis for the 197 samples, excluding rental fee, is $13.18 per sample. Based on the 3-day field demonstration, the total cost for equipment rental and necessary supplies is estimated at $7,786. The cost breakout by category is: capital costs, 66.7%; supplies, 3.6%; support equipment, 3.5%; labor, 7.7%; and IDW, 18.5%.

The cost per analysis based upon 197 samples, when renting the XLt 792, is $47.69 per sample. The cost per analysis for the 197 samples, excluding rental fee, is $13.18 per sample. Based on the 3-day field demonstration, the total cost for equipment rental and necessary supplies is estimated at $9,396. The cost breakout by category is: capital costs, 72.4%; supplies, 3.0%; support equipment, 2.9%; labor, 6.4%; and IDW, 15.3%.

Key demonstration findings are summarized below for the secondary objectives.

***Ease of Use*:** Based on observations made during the demonstration, the XLi/XLt 700 Series Analyzers are very easy to operate, requiring one field technician with a high school education. A free 8-hour training course on instrument operation and radiation safety is mandatory prior to operating the instruments. The analyzers contain an integrated touch-screen display with an advanced and intuitive user interface.

***Potential Health and Safety Concerns*:** No significant health and safety concerns were noted during the demonstration. Potential exposure to radiation from the excitation sources (Cd-109, Am-241, Fe-55 and X-ray tube) was the only health and safety concern during the demonstration. The analyzers should never be pointed at anyone while the sources are exposed. No solvents or acids are used for sample preparation. According to NITON, the sources are designed to remain secure even under extreme conditions, so that even if the instrument is broken, crushed or burned there should be no leakage of radioactive material.

Portability: The XLi/XLt 700 Series Analyzers are handheld portable single piece units weigh only 0.8 kg (XLi 702) and 1.4 kg (XLt 792). There are no cables and no separate processing units. The analyzers have an attractive ergonomic form. During the demonstration, the analyzers each operated on 1 battery pack that lasted for 4-8 hours.

Durability: Based on observations during the demonstration, the analyzers were well constructed, field-rugged and durable. They are constructed of high-strength injection molded plastic. During the three days in which the instrument was observed, there was no downtime, maintenance or repairs. The equipment apparently was not affected by the almost continuous rain.

Availability of the Devices: The XLi/XLt 700 Series Analyzers are readily available for lease or purchase. During most of the year, NITON is typically able to rent an analyzer to a customer in 10-14 days (10 isotope rentals and 3 X-ray tube rentals). There are also radiation licensing requirements for these devices. NITON offers over 100 user/radiation training classes to help expedite the process. Supplies not provided by NITON are readily available from supply firms.

PERFORMANCE SUMMARY

In summary, during the demonstration, the XLi 702 and XLt 792 exhibited the following desirable characteristics of a field mercury measurement device: (1) good accuracy compared to standard reference materials, (2) good precision, (3) high sample throughput, (4) low measurement costs, and (5) ease of use. During the demonstration the XLi 702 and XLt 792 were found to have the following limitations: (1) a PQL that exceeds the residential soil PRG action lim it. The XLi/XLt 700 Series Analyzers are handheld devices for rapid field measurements of mercury in soil and sediment.

NOTICE: EPA verifications are based on an evaluation of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. The EPA makes no expressed or implied warranties as to the performance of the technology and does not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements.