

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION
PROGRAM



U.S. Environmental Protection Agency



Oak Ridge National Laboratory

Verification Statement

TECHNOLOGY TYPE:	X-RAY FLUORESCENCE		
APPLICATION:	MEASUREMENT OF LEAD IN DUST WIPES		
TECHNOLOGY NAME:	XLt 700 Series XRF Spectrum Analyzer		
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The U.S. Environmental Protection Agency (EPA) has created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized standards and testing organizations and stakeholder groups consisting of regulators, buyers, and vendor organizations, with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

Oak Ridge National Laboratory (ORNL) is one of the verification organizations operating under the Advanced Monitoring Systems (AMS) Center. AMS, which is administered by EPA's National Exposure Research Laboratory (NERL), is one of seven technology areas under ETV. In this verification test, ORNL evaluated the performance of lead in a dust wipe measurement technology. This verification statement provides a summary of the test results for NITON's XLt 700 Series x-ray fluorescence (XRF) spectrum analyzer.

VERIFICATION TEST DESCRIPTION

This verification test was designed to evaluate technologies that detect and measure lead in dust wipes. The test was conducted at the EPA Region 1 Laboratories in North Chelmsford, MA, from January 6 through January 9, 2003. This ETV performance test was an extension of those conducted in 2001 and 2002 using the same experimental design. The vendor of this commercially-available, field portable technology blindly analyzed 160 dust wipe samples containing known amounts of lead, ranging in concentration from ≤ 2 to 1,500 $\mu\text{g}/\text{wipe}$. The experimental design was particularly focused on important clearance standards, such as those identified in 40 CFR Part 745.227(e)(8)(viii) of 40 $\mu\text{g}/\text{ft}^2$ for floors, 250 $\mu\text{g}/\text{ft}^2$ for window sills, and 400 $\mu\text{g}/\text{ft}^2$ for window troughs. The samples analyzed in this verification test were archived from the original test in 2001. The test included samples from the Environmental Lead Proficiency Analytical Testing Program (ELPAT), which were prepared from dust collected in households in North Carolina and Wisconsin. Also, samples were acquired from the University of Cincinnati (UC). The UC dust wipe samples were prepared from National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs). The integrity of the archived samples was confirmed by independent analysis prior to the tests described here. The results of the lead analyses generated by the technology were compared with results from analyses of similar samples by conventional laboratory methodology in a laboratory that was recognized as proficient by the National Lead Laboratory Accreditation Program (NLLAP) for dust testing. Quality assurance (QA) oversight of verification testing was provided by ORNL and EPA. EPA and ORNL QA staff reviewed and approved the test plan. ORNL staff conducted a data quality audit of 100% of the test data (both laboratory and vendor), and a technical systems audit of the procedures used during this verification. In addition, ORNL QA staff also conducted an independent technical systems audit at the test site. Details of the test, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Lead in Dust Wipe Detection Technology—NITON LLC, XLt 700 Series X-Ray Fluorescence Spectrum Analyzer*, EPA/600/R-03/087.

TECHNOLOGY DESCRIPTION

The following description of the XLt 700 was provided in part by the vendor (NITON) and does not represent verified information.

The XLt 700 analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses a low power miniature x-ray tube with a silver transmission target 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. 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 lead x-rays is achieved using a highly-efficient, thermo-electrically cooled, solid-state detector. Signals from the detector are amplified, digitized, and then quantified via integral multichannel analysis and data processing units. Sample test results are displayed in total micrograms of lead per dust-wipe. NITON's XLt 700 Series XRF spectrum analyzer reporting limit was 10 $\mu\text{g}/\text{wipe}$ during the verification test.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the XLt 700 Series XRF were observed:

Precision: Precision—based on the average percent relative standard deviation—was 11% for the UC samples and was 11% for the ELPAT samples.

Accuracy: Accuracy was assessed using the estimated concentrations of the UC and ELPAT samples. The average percent recovery value for all samples reported above the upper bound of the reporting limits observed in this evaluation was 97% for the UC samples and 101% for the ELPAT samples. A regression line fitted to the XLt 700 results versus the estimated concentrations showed slopes with slightly low biases that were statistically significant for both the UC and ELPAT samples. However, the average percent recovery values were well within the acceptable bias range of $100\% \pm 25\%$. In contrast, for results from the NLLAP laboratory-based analysis of these same samples, the average percent recovery values were 91% and 98%, respectively, for the UC and ELPAT samples. The regression analyses also exhibited slopes that were negative biases for both the UC and ELPAT samples and were statistically significant.

Comparability: A comparison of all samples (ELPAT and UC) was performed for cases where both the XLt 700 and the NLLAP-recognized laboratory results were above $20 \mu\text{g/wipe}$ (the reporting limit for the laboratory). The correlation coefficient (r) for the comparison of the UC samples was 0.999 [slope (m) = 0.995, intercept = 4.775], and for the ELPAT samples was also 0.999 [m = 0.977, intercept = 3.076]. Although the slope for the ELPAT samples (but not UC samples) was statistically different than 1.00, both sample sets have correlation coefficients that show a strong linear agreement with the NLLAP laboratory data.

Detectable blanks: All twenty blank samples, prepared at concentrations $< 2 \mu\text{g/wipe}$, were reported correctly as less than reporting limits, with results reported by the XLt 700 as less than $10 \mu\text{g/wipe}$.

False positive results: A false positive (fp) result is one in which the technology reports a result that is above the clearance level when the true (or estimated) concentration is actually below. For the UC samples, the XLt 700 reported 8 of a possible 37 fp results, while the NLLAP laboratory did not report any fp results. For the ELPAT samples, the XLt 700 reported 1 of a possible 12 fp results, while the NLLAP laboratory reported 2 of 12 fp results.

False negative results: A false negative (fn) result is one in which the technology reports a result that is below the clearance level when the true (or estimated) concentration is actually above. For the UC samples, the XLt 700 reported 10 of a possible 23 fn results, while the NLLAP laboratory reported 23 of a possible 30 fn results. For the ELPAT samples, the XLt 700 reported 8 of a possible 28 fn results, while the NLLAP laboratory reported 7 of 28 fn results.

Completeness: The XLt 700 Series spectrum analyzer generated results for all 160 dust wipes samples, for a completeness of 100%.

Sample Throughput: Two analysts (NITON experts) analyzed all 160 samples in 32.75 hours over 3.5 calendar days. Eight measurements were taken for sample wipe, unless the vendor, based on an initial evaluation of a non-detectable instrument response, believed the test sample to be a blank. In such cases, the vendor performed four measurements on those samples, for a total of 1200 measurements.

Overall Evaluation: The overall performance was characterized as being biased slightly low (but within the limits of acceptable bias), precise, and in good linear agreement to an NLLAP-laboratory results. The verification team found that the XLt 700 was relatively simple for the trained analyst to operate in the field, requiring less than an hour for initial setup. As with any technology selection, the user must determine if this technology is appropriate for the application and the project data quality objectives. Additionally, ORNL and ETV remind the reader that, while the ETV test provides valuable information in the form of a snapshot of performance, state, tribal, or federal requirements regarding the use of the technologies (such as NLLAP recognition for analysis of samples where required) need to be followed. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

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