THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV

EPA
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Oak Ridge National Laboratory

Verification Statement

TECHNOLOGY TYPE:	**X-RAY FLUORESCENCE**		
APPLICATION:	**MEASUREMENT OF LEAD IN DUST WIPES**		
TECHNOLOGY NAME:	**Pb-Test XRF Instrument**		
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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 six technology areas under ETV. In this verification test, ORNL evaluated the performance of lead in dust wipe measurement technologies. This verification statement provides a summary of the test results for KeyMaster Technologies' Pb-Test x-ray fluorescence (XRF) instrument.

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 Oak Ridge National Laboratory in Oak Ridge, TN, from January 7 through January 9, 2002. KeyMaster Technologies, a vendor of commercially-available, field portable x-ray fluorescence (XRF) instruments for lead detection and measurement, blindly analyzed 160 dust wipe samples containing known amounts of lead, ranging in concentration from ≤2 to 1,500 µg/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 µg/ft^2 for floors, 250 µg/ft^2 for window sills, and 400 µg/ft^2 for window troughs. The samples included wipes newly-prepared and archived from the Environmental Lead Proficiency Analytical Testing Program (ELPAT). These samples were prepared from dust collected in households in North Carolina and Wisconsin. Also, newly-prepared 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 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. 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— KeyMaster Technologies, Pb-Test X-Ray Fluorescence Instrument,* EPA/600/R-02/058.

TECHNOLOGY DESCRIPTION

The Pb-Test is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses a sealed, highly purified Cobalt-57 radioisotope source (<12 mCi) to excite a test sample's constituent elements. The Pb-Test utilizes the recently developed Cadmium Telluride (CdTe) Schottky diode detectors. The age of the detector at the time of testing was approximately 4 to 5 months. Each element produces x-rays at a unique set of energies, allowing one to non-destructively measure the elemental composition of a sample. These characteristic x-rays are continuously detected, identified, and quantified by the spectrometer during sample analysis. In other words, the energy of each x-ray identifies a particular element present in the sample and the rate at which the x-rays of a given energy are emitted allows the analyzer to determine the quantity of a particular element present in that sample. Signals from the detector are amplified, digitized, and then quantified via an integrated multichannel analyzer and data processor. Sample test results are displayed in total micrograms of lead per dust-wipe. KeyMaster did not provide a reporting limit for the instrument during the verification test.

VERIFICATION OF PERFORMANCE

The following performance characteristics of the Pb-Test XRF were observed:

Precision: Precision, based on the average percent relative standard deviation (RSD), was 18% for the ELPAT samples and 15% for the UC samples. A technology's performance is considered very precise if the average RSD is less than 10%, but acceptable as long as the average RSD is less than 20%.

Accuracy: Accuracy was assessed using the estimated concentrations of the ELPAT and UC samples. Acceptable bias falls in a range of average percent recovery values of 100% ± 25%. The average percent recovery values for the ELPAT and UC samples (excluding the "detectable blank" samples at concentrations < 2 µg/wipe that are described in more detail below) were 189% and 168%, respectively. If only those samples with concentrations between 200 and 1,500 µg/wipe are considered, the Pb-Test results were unbiased, with an average percent recovery value of 96% for ELPAT samples and 102% for the UC

samples. The Pb-Test results for samples at 800, and 1,500 µg/wipe were both negatively biased (78% and 72%, respectively), but there was not enough data to ascertain that the technology was negatively biased above 800 µg/wipe. For the NLLAP laboratory results, the average percent recovery values were 98% and 91%, respectively, for the ELPAT and UC samples. The NLLAP laboratory's negative bias for both the ELPAT and UC samples was statistically significant.

Comparability: A comparison of the average Pb-Test results and the average NLLAP-recognized laboratory results was performed for all samples (ELPAT and UC) for estimated concentrations above and below 200 µg/wipe. The correlation coefficient (r) for the $\leq$ 200 µg/wipe data set was 0.967 [slope (m) = 1.060, intercept = 66]. For the > 200 µg/wipe data, the r value was 0.989 [slope = 0.662, intercept = 121). The slopes for both data sets were statistically different from 1.00. The Pb-Test results above 200 µg/wipe indicate fair agreement with the NLLAP laboratory's results, since correlation coefficient values greater than 0.990 indicate good agreement with the laboratory data.

Detectable blanks: All twenty samples, prepared at concentrations < 2 µg/wipe, were reported as detections by the Pb-Test, with concentrations ranging from 46 to 137 µg/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 Pb-Test reported 20 of a possible 38 fp results, while the NLLAP laboratory did not report any fp results. For the ELPAT samples, the Pb-Test reported 6 of a possible 12 fp results, while the NLLAP laboratory reported two.

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 Pb-Test reported 7 of a possible 22 fn results, while the NLLAP laboratory reported 23 of a possible 30 fn results. For the ELPAT samples, the Pb-Test reported 8 of a possible 28 fn results, while the NLLAP laboratory reported 7.

Completeness: Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result is not rejected). An acceptable completeness rate is 95% or greater. The Pb-Test instrument generated results for all 160 dust wipes samples for a completeness of 100%.

Sample Throughput: Sample throughput is a measure of the number of samples that can be processed and reported by a technology in a given period of time. With two analysts, the KeyMaster team accomplished a sample throughput rate of approximately eighty samples per 10-hour day. One operator prepared the samples, while the other performed the analyses. The vendor chose to run the samples on two instruments and report the average value. The instrument can be operated by a single trained analyst.

Overall Evaluation: The overall performance was characterized as having acceptable precision, biased high for concentrations below 200 µg/wipe, and unbiased for concentrations above 200 µg/wipe. The Pb-Test results above 200 µg/wipe were also found to be in fair linear agreement with the NLLAP laboratory's results. The verification team found that the Pb-Test was simple for the trained analyst to operate in the field, requiring less than one-half 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 clearance 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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