



Environmental Technology Verification Report

Lead in Dust Wipe Measurement Technology

NITON Corporation X-Ray Fluorescence Spectrum Analyzer, XL-700 Series



Oak Ridge National Laboratory

ETV ✓ ETV ✓ ETV ✓

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION
PROGRAM



Verification Statement

TECHNOLOGY TYPE:	X-RAY FLUORESCENCE		
APPLICATION:	MEASUREMENT OF LEAD IN DUST WIPES		
TECHNOLOGY NAME:	XL-700 Series XRF Spectrum Analyzer		
COMPANY:	NITON Corporation		
ADDRESS:	900 Middlesex Turnpike, Bldg. 8 Billerica, MA 01821	PHONE:	+1(978) 670-7460
		FAX:	+1(978) 670-7430
WEB SITE:	www.niton.com		
E-MAIL:	sales@niton.com		

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 NITON's XL-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 Capitol Community Technical College in Hartford, CT, from November 5 through November 9, 2001. The vendors of commercially-available, field portable technologies 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 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— NITON Corporation, XL-700 Series X-Ray Fluorescence Spectrum Analyzer*, EPA/600/R-02/055. NITON's XL-300 Series XRF was also evaluated in the test and a separate report has been prepared (*Environmental Technology Verification Report: Lead in Dust Wipe Detection Technology— NITON Corporation, XL-300 Series X-Ray Fluorescence Spectrum analyzer*, EPA/600/R-02/059).

TECHNOLOGY DESCRIPTION

The XL-700 Series spectrum analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses one to three sealed, radioisotope sources 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. In this test, the exact model used was XL-723S with two sources (10 mCi Cd-109 and 14 mCi Am-241). The Cd-109 source, the source which was used for the lead analysis, was approximately 11 months old. 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 lead x-rays is achieved using a highly efficient, thermo-electrically cooled, solid-state, Big-Area Silicon PIN-diode (BASP) detector. Signals from the BASP 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 XL-700 Series XRF spectrum analyzer reporting limits were 4.9 to 6.1 $\mu\text{g}/\text{wipe}$ during the verification test.

VERIFICATION OF PERFORMANCE

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

Precision: Precision, based on the average percent relative standard deviation (RSD), was 8% for both the UC and ELPAT sample analyses. 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 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 107% for the UC samples and 119% for the ELPAT samples. The high bias was statistically significant for both the UC and ELPAT samples, but it was well within the acceptable bias range of $100\% \pm 25\%$. For the NLLAP laboratory results, the average percent recovery

values were 91% and 98%, respectively, for the UC and ELPAT samples. The negative bias for both the UC and ELPAT samples was statistically significant.

Comparability: A comparison of the XL-700 results and the NLLAP-recognized laboratory results was performed for all samples (ELPAT and UC) that were reported above 20 $\mu\text{g/wipe}$. The correlation coefficient (r) for the comparison of the UC samples was 0.999 [slope (m) = 1.206, intercept = -3.290], and for the ELPAT samples was also 0.999 [m = 1.112, intercept = 13.283]. While the slopes for both the ELPAT and UC samples were statistically different than 1.00, correlation coefficients greater than 0.990 indicate strong linear agreement with the NLLAP laboratory data.

Detectable blanks: All twenty samples, prepared at concentrations $< 2 \mu\text{g/wipe}$, were reported correctly as less than reporting limits, with results reported by the XL-700 ranging from < 4.9 to $< 6.1 \mu\text{g/wipe}$.

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 XL-700 reported 21 of a possible 34 fp results, while the NLLAP laboratory did not report any fp results. For the ELPAT samples, the XL-700 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 XL-700 reported 2 of a possible 26 fn results, while the NLLAP laboratory reported 23 of a possible 30 fn results. For the ELPAT samples, the XL-700 reported 2 of a possible 28 fn results, while the NLLAP laboratory reported seven.

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 XL-700 Series spectrum analyzer 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. A single analyst (a NITON expert) analyzed all 160 samples in four 12-hour days. Eight measurements were taken for each wipe, for a total of 320 measurements. For the first two days of testing, the analyst used a 60 second count time and analyzed 25 to 30 samples per day. For the last two days of testing, the analyst used a 30 second count time and analyzed 50 to 60 samples per day. Replicate results analyzed using the two count times were not statistically different.

Overall Evaluation: The overall performance was characterized as being biased slightly high (but within the limits of acceptable bias), very precise, and in good linear agreement to an NLLAP-laboratory results. The verification team found that the XL-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 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>.

Gary J. Foley, Ph.D.
Director
National Exposure Research Laboratory
Office of Research and Development

W. Franklin Harris, Ph.D.
Associate Laboratory Director
Biological and Environmental Sciences
Oak Ridge National Laboratory

NOTICE: EPA verifications are based on evaluations of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. EPA and ORNL make no expressed or implied warranties as to the performance of the technology and do 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. Mention of commercial product names does not imply endorsement or recommendation.

Environmental Technology Verification Report

Lead in Dust Wipe Measurement Technology

NITON Corporation X-Ray Fluorescence Spectrum Analyzer, XL-700 Series

By

Amy B. Dindal
Charles K. Bayne, Ph.D.
Roger A. Jenkins, Ph.D.
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831-6120

Eric N. Koglin
U.S. Environmental Protection Agency
Environmental Sciences Division
National Exposure Research Laboratory
Las Vegas, Nevada 89193-3478

Notice

The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development (ORD), funded and managed, through Interagency Agreement No. DW89937854 with Oak Ridge National Laboratory, the verification effort described herein. This report has been peer and administratively reviewed and has been approved for publication as an EPA document. Mention of trade names or commercial products does not constitute endorsement or recommendation for use of a specific product.

Table of Contents

Notice	ii
List of Figures	v
List of Tables	vi
Acknowledgments	vii
Abbreviations and Acronyms	viii
Section 1 — Introduction	1
Section 2 — Technology Description	2
General Technology Description	2
Sample Preparation	2
Calibration	2
Sample Analysis	2
Section 3 — Verification Test Design	4
Objective	4
Testing Location and Conditions	4
Drivers and Objectives for the Test	4
Summary of the Experimental Design	4
ELPAT and Blank Sample Description	4
University of Cincinnati Sample Description	5
Distribution and Number of Samples	6
Description of Performance Factors	6
Precision	6
Accuracy	7
Comparability	8
Detectable Blanks	8
False Positive/Negative Results	8
Completeness	9
Sample Throughput	9
Ease of Use	9
Cost	9
Miscellaneous Factors	9
Section 4 — Laboratory Analyses	10
Background	10
NLLAP Laboratory Selection	10
Laboratory Method	10
Section 5 — Technology Evaluation	17
Objective and Approach	17
Precision	17
Accuracy	17
Comparability	18
False Positive/False Negative Results	19

Completeness	22
Sample Throughput	22
Ease of Use	22
Cost Assessment	22
XL-700 Series XRF Costs	23
Equipment	23
Laboratory Costs	24
Sample Shipment	24
Labor, Equipment, and Waste Disposal	24
Cost Assessment Summary	24
Miscellaneous Factors	24
Summary of Performance	25
Section 6 — References	27
Appendix	28

List of Figures

1. NITON's XL-700 series XRF spectrum analyzer	2
2. Distribution of both UC and ELPAT sample concentrations	7
3. Plot of DataChem reported values versus estimated values, shown for concentrations less than 500 $\mu\text{g/wipe}$.	12
4. False negative probabilities for DataChem average concentrations at a target concentration level of 40 $\mu\text{g/wipe}$.	14
5. False negative probabilities for DataChem average concentrations at a target concentration level of 250 $\mu\text{g/wipe}$.	15
6. False negative probabilities for DataChem average concentrations at a target concentration level of 400 $\mu\text{g/wipe}$.	15
7. Plot of the average XL-700 concentration versus the average DataChem concentrations for all samples (n=21), shown for ELPAT and UC concentrations less than 500 $\mu\text{g/wipe}$.	19
8. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 40 $\mu\text{g/wipe}$	20
9. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 250 $\mu\text{g/wipe}$	21
10. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 400 $\mu\text{g/wipe}$	21

List of Tables

1. Summary of DataChem Pre-Test Results	11
2. Summary of DataChem Percent Recovery Values by Sample Source	12
3. Summary of DataChem Precision Estimates by Sample Source	12
4. False Positive/False Negative Results for DataChem Measurements of UC Samples	14
5. Summary of the Linear Regression Constants and Recovery Data for DataChem's Measurements Versus the Estimated Concentrations at the Clearance Levels	16
6. Precision of the XL-700	17
7. Accuracy of XL-700	17
8. Linear regression constants for the plots of the XL-700 versus the estimated values and versus the DataChem average measurements	18
9. False Positive/False Negative Error Rates for XL-700 Measurements	20
10. Summary of the Linear Regression and Recovery Data for the XL-700 Response versus the Estimated Concentrations	22
11. Estimated analytical costs for lead dust wipe samples	23
12. Performance Summary for NITON's XL-700 Series XRF Spectrum Analyzer	26

Acknowledgments

The authors wish to acknowledge the support of all those who helped plan and conduct the verification test, analyze the data, and prepare this report. In particular, we recognize: Patricia Lindsey, Capitol Community Technical College, for providing a site for the verification test; Sandy Roda (University of Cincinnati) and Laura Hodson (Research Triangle Institute) for coordination of sample preparation; the inorganic analytical laboratory in EPA/Region 1 (North Chelmsford, MA) for the analysis of quality control samples; and the expertise of the technical advisory panel, including Kenn White (American Industrial Hygiene Association), John Schwemberger, Dan Reinhart, Oksana Pozda, and Darlene Watford (EPA/Office of Pollution Prevention and Toxics), Paul Carroll (EPA/Region 1), Sharon Harper (EPA/Research Triangle Park), Peter Ashley, Warren Friedman, Gene Pinzer, and Emily Williams (U.S. Department of Housing and Urban Development); Paul Halfmann and Sharon Cameron (Massachusetts Childhood Lead Poisoning Prevention Program), Kevin Ashley (National Institute for Occupational Safety and Health); Walt Rossiter and Mary McKnight (National Institute of Standards & Technology); Bill Gutknecht (Research Triangle Institute), and Bruce Buxton (Battelle Memorial Institute). The authors would specifically like to thank Paul Halfmann and John Schwemberger for serving as peer reviewers of this report. The authors also acknowledge the participation of NITON Corporation, in particular, Mark Gardner, Debbie Schatzlein, and Jon Shein.

For more information on the Lead in Dust Wipe Measurement Technology Verification contact:

Eric N. Koglin
Project Technical Leader
Environmental Protection Agency
Environmental Sciences Division
National Exposure Research Laboratory
P.O. Box 93478
Las Vegas, Nevada 89193-3478
(702) 798-2332
koglin.eric@epa.gov

Roger A. Jenkins
Program Manager
Oak Ridge National Laboratory
Chemical Sciences Division
P.O. Box 2008
Oak Ridge, TN 37831-6120
(865) 574-4871
jenkinsra@ornl.gov

For more information on NITON's XL-700 Series XRF Spectrum Analyzer, contact:

Jonathan J. Shein
NITON Corporation
900 Middlesex Turnpike, Building 8
Billerica, MA 01821
+1 (978) 670-7460
sales@niton.com
www.niton.com

Abbreviations and Acronyms

AIHA	American Industrial Hygiene Association
AMS	Advanced Monitoring Systems Center, ETV
ASTM	American Society for Testing and Materials
BASP	Big-Area Silicon PIN-diode detector
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CL	clearance level for lead of 40, 250, or 400 $\mu\text{g/wipe}$
EDXRF	energy dispersive x-ray fluorescence
ELPAT	Environmental Lead Proficiency Analytical Testing program
EPA	U. S. Environmental Protection Agency
ETV	Environmental Technology Verification Program
ETVR	Environmental Technology Verification Report
fn	false negative result
fp	false positive result
ICP-AES	Inductively coupled plasma-atomic emission spectrometry
NERL	National Exposure Research Laboratory, U.S. EPA
NIOSH	National Institute for Occupational Safety and Health, CDC
NIST	National Institute of Standards & Technology
NLLAP	National Lead Laboratory Accreditation Program, U.S. EPA
OPPT	Office of Pollution Prevention and Toxics, U.S. EPA
ORNL	Oak Ridge National Laboratory
QA	quality assurance
QC	quality control
RSD	relative standard deviation
RTI	Research Triangle Institute
SD	standard deviation
SRM	Standard Reference Material
UC	University of Cincinnati
XRF	x-ray fluorescence spectrum analyzer

Section 1 — Introduction

The U.S. Environmental Protection Agency (EPA) 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 verification 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 (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

ETV is a voluntary program that seeks to provide objective performance information to all of the participants in the environmental marketplace and to assist them in making informed technology decisions. ETV does not rank technologies or compare their performance, label or list technologies as acceptable or unacceptable, seek to determine “best available technology,” or approve or disapprove technologies. The program does not evaluate technologies at the bench or pilot scale and does not conduct or support research. Rather, it conducts and reports on testing designed to describe

the performance of technologies under a range of environmental conditions and matrices.

The program now operates six centers covering a broad range of environmental areas. ETV began with a 5-year pilot phase (1995–2000) to test a wide range of partner and procedural alternatives in various technology areas, as well as the true market demand for and response to such a program. In these centers, EPA utilizes the expertise of partner “verification organizations” to design efficient processes for conducting performance tests of innovative technologies. These expert partners are both public and private organizations, including federal laboratories, states, industry consortia, and private sector entities. Verification organizations oversee and report verification activities based on testing and QA protocols developed with input from all major stakeholder/customer groups associated with the technology area. The verification described in this report was administered by the Advanced Monitoring Systems (AMS) Center, with Oak Ridge National Laboratory (ORNL) serving as the verification organization. (To learn more about ETV, visit ETV’s Web site at <http://www.epa.gov/etv>.) The AMS Center is administered by EPA’s National Exposure Research Laboratory (NERL).

The verification of a field analytical technology for measurement of lead in dust wipe samples is described in this report. The verification test was conducted in Hartford, Connecticut, from November 5 through November 9, 2001. The performance of NITON’s XL-700 Series X-Ray Fluorescence (XRF) spectrum analyzer was determined under field conditions. The technology was evaluated by comparing its results to estimated concentration values and with results obtained on similar samples using a recognized laboratory analytical method. The performance of NITON’s XL-300 Series XRF has been reported in a separate verification report.

Section 2 — Technology Description

In this section, the vendor (with minimal editorial changes by ORNL) provides a description of the technology and the analytical procedure used during the verification testing activities.

General Technology Description

The XL-700 Series spectrum analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses one to three sealed, radioisotope sources to excite characteristic x-rays of a test sample's constituent elements. These characteristic x-rays are continuously detected, identified, and quantified by the



Figure 1. NITON's XL-700 series XRF spectrum analyzer.

spectrometer during sample analysis. In this test, the exact model used was XL-723S with two sources (10 mCi Cd-109 and 14 mCi Am-241), although only the Cd-109 source (~ 11 months old) was used for the analysis of lead. Stated simply, 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 lead x-rays is achieved using a highly efficient, thermoelectrically cooled, solid-state, Big-Area Silicon PIN-diode (BASP) detector. Signals from the BASP 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.

Sample Preparation

For this verification test, the dust on the wipes had to be distributed more evenly across the wipe prior to analysis. This step is only required for prepared samples where the lead loading is clumped in discrete areas. The sample was then folded five times such that it was the proper size (1" x 1.5") for the XRF window.

For this test, the samples were dried for 20 minutes at 250 °F in a toaster oven prior to testing. Alternatively, the sample could be exposed

overnight to ambient temperature and humidity, but the toaster oven was used in this case to expedite the process. After oven drying, the dried sample sat in ambient air for at least 5 minutes before reading.

The dried, folded wipe was placed in a 2" x 2" plastic bag (NITON part number 187-471 or equivalent) and labeled. (To eliminate the potential for cross-contamination of samples, plastic bags should never be re-used.) The wipe sample in its plastic bag was positioned within the frame of the metal dust wipe holder (NITON part number 180-407 or equivalent) and analyzed.

Calibration

The instrument is factory calibrated. For the verification test, the instrument performance was verified at the start and end of each day's analyses by analyzing samples of known quantity.

Sample Analysis

The following is the procedure that was used to analyze the samples during the verification test. Note that eight sample measurements were taken to insure that the entire area of the folded dust-wipe sample was properly measured by the spectrometer.

The dust wipe was placed in the sample holder at the number-one position and a 60 second (s) measurement was taken. The sample was then placed in the number two position and another 60 s reading was taken. The wipe was rotated 180 degrees (without turning the sample holder upside-down), and placed in the number one position for a third 60 s measurement. Finally, the wipe was measured in the number two position for a fourth reading. The four readings were averaged and represented the concentration measured on the "front" of the wipe. The wipe was then flipped over and the procedure repeated to obtain four measurements on the "back" of the wipe. The average concentration from the "front" was averaged with the average concentration from the "back" readings to produce the final result.

For the first two days of testing, the sample analyses were taking longer than expected (25-30 samples per day). The NITON analyst believed that the instrument could achieve adequate sensitivity with a shorter count time, so he elected to reduce the run-times to 30 seconds. Samples 1-48 were analyzed using 60 second count time, and

samples 49-160 were analyzed using 30 second count time. (See Appendix for data.)

The sensitivity of the measurements is a function of the age of the detector source, so modification of count times can be made, depending on the user's needs.

Section 3 — Verification Test Design

Objective

The purpose of this section is to describe the verification test design. It is a summary of the test plan (ORNL, 2001).

Testing Location and Conditions

The verification of field analytical technologies for lead in dust wipes was conducted at the Capitol Community Technical College in Hartford, Connecticut. The test was conducted in the basement of a classroom building. The temperature and relative humidity were monitored during field testing, but remained fairly constant. The average temperature and relative humidity over the four days of testing were 68 °F and 32%, respectively.

Drivers and Objectives for the Test

The purpose of this test was to evaluate the performance of field analytical technologies that are capable of analyzing dust wipe samples for lead contamination. This test provides information on the potential applicability of field technologies to EPA standards for dust clearance testing. The experimental design was designed around the three clearance standards 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 that are outlined in 40 CFR Part 745.227(e)(8)(viii) (CFR, 2001).

The primary objectives of this verification were to evaluate the field analytical technologies in the following areas: (1) how well each performs relative to a conventional, fixed-site, analytical method for the analysis of dust wipe samples for lead; (2) how well each performs relative to results generated in previously rounds of ELPAT testing (described in the next section), and (3) the logistical and economic resources necessary to operate the technology. Secondary objectives for this verification were to evaluate the field analytical technology in terms of its reliability, ruggedness, cost, range of usefulness, sample throughput, data quality, and ease of use. Note that this verification test does not provide an assessment of the selection of locations for dust samples in a facility or an assessment of the way that dust samples are collected. The planning for this verification test follows the guidelines established in the data quality objectives process.

Summary of the Experimental Design

All of the samples analyzed in this verification test were prepared gravimetrically. At the time of the test, both of the wipes utilized in the test (PaceWipe™ and Aramsco LeadWipe™) were on the list of wipes recommended for lead testing by the American Society for Testing and Materials (ASTM, 1996). Initial consideration was given to conducting the test in a real-world situation, where the technologies would have been deployed in a housing unit that had been evacuated due to high levels of lead contamination. In addition to the safety concern of potentially subjecting participants to lead exposure, the spatial variability of adjacent samples would have been expected to be so great that it would be much larger than the anticipated variability of these types of technologies, thereby making it difficult to separate instrument/method variability and sampling variability. The availability of well-characterized samples derived from “real-world” environments made the use of proficiency testing samples (so-called “ELPAT” samples) and other prepared samples an attractive alternative.

ELPAT and Blank Sample Description

In 1992, the American Industrial Hygiene Association (AIHA) established the Environmental Lead Proficiency Analytical Testing (ELPAT) program. The ELPAT Program is a cooperative effort of the American Industrial Hygiene Association (AIHA), and researchers at the Centers for Disease Control and Prevention (CDC), National Institute for Occupational Safety and Health (NIOSH), and the EPA Office of Pollution Prevention and Toxics (OPPT). The ELPAT program is designed to assist laboratories in improving their analytical performance, and therefore, does not specify use of a particular analytical method. Participating laboratories are sent samples to analyze on a quarterly basis. The reported values must fall within a range of acceptable values in order for the laboratory to be deemed proficient for that quarter.

Research Triangle Institute (RTI) in Research Triangle Park, NC, is contracted to prepare and distribute the lead-containing paint, soil, and dust

wipe ELPAT samples. For the rounds of testing which have occurred since 1992, archived samples are available for purchase. Some of these samples were used in this verification test. Because the samples have already been tested by hundreds of laboratories, a certified concentration value is supplied with the sample. This certified value represents a pooled measurement of all of the results submitted, with outliers excluded from the calculation.

The following description, taken from an internal RTI report, briefly outlines how the samples were prepared. RTI developed a repository of real-world housedust, collected from multiple homes in the Raleigh/Durham/Chapel Hill area, as well as from an intervention project in Wisconsin. After collection, the dust was sterilized by gamma irradiation, and sieved to 150 μm . A PaceWipe™ was prepared for receiving the dust by opening the foil pouch, removing the wet folded wipe and squeezing the excess moisture out by hand over a trash can. The wipe was then unfolded and briefly set on a Kimwipe™ to soak up excess moisture. The PaceWipe was then transferred to a flat plastic board to await the dust. After weighing a 0.1000 ± 0.0005 g portion of dust on weighing paper, the pre-weighed dust was gently tapped out onto the PaceWipe. The wipe was then folded and placed in a plastic vial, which was then capped. All vials containing the spiked wipes were stored in a cold room as a secondary means of retarding mold growth until shipment.

Before use in the ELPAT program, RTI performed a series of analyses to confirm that the samples were prepared within the quality guidelines established for the program. The data quality requirements for the ELPAT samples were: 1) the relative standard deviation of the samples analyzed by RTI must be 10% or less; 2) the measured concentrations must be within 20% of the target value that RTI was intending to prepare; and 3) analysis by an accredited laboratory must yield results within $\pm 20\%$ of the RTI result. Ten samples were analyzed by RTI and nine samples were sent to the Wisconsin Occupational Health Laboratory for independent, confirmatory analysis. All ELPAT samples used in this test met the data quality requirements described above. The estimated concentration for an ELPAT sample used in this evaluation was the certified (“consensus”) value (i.e., an analytically derived result).

RTI prepared the blank samples using the same preparation method as the ELPAT samples, but the concentration of lead was $< 2 \mu\text{g/wipe}$, well below the expected reporting limits of the participant technologies.

University of Cincinnati Sample Description

The ELPAT samples consisted of dust mounded in the center of a PaceWipe. The University of Cincinnati (UC) prepared “field QC samples” where the dust was sprinkled over the wipe, more similar to how a wipe would look when a dust wipe sample is collected in the field. In a typical scenario, UC sends these control samples to a laboratory along with actual field-collected samples as a quality check of the laboratory operations. Because the samples are visually indistinguishable from an actual field sample, are prepared on the same wipe, and are shipped in the same packaging, the laboratory blindly analyzes the control samples, which provides the user with an independent assessment of the quality of the laboratory’s data.

A cluster of twenty UC samples prepared at the key clearance levels were added to the experimental design, primarily so that an abundance of data would exist near the clearance levels, in order to assess false positive and false negative error rates. The UC samples were prepared on Aramsco Lead Wipes™ (Lakeland, FL). The UC wipe samples were prepared using National Institute of Standards & Technology (NIST) Standard Reference Materials (SRMs). NIST SRM 2711 was used to prepare the 40 $\mu\text{g/wipe}$ samples, and NIST SRM 2710 was used to prepare the 250 and 400 $\mu\text{g/wipe}$ samples. Both SRM 2711 and SRM 2710 are Montana Soil containing trace concentrations of multiple elements, including lead. Some NIST SRM materials that are spiked on dust wipes are known to have low extraction recoveries when prepared by standard analytical methods (e.g., lead silicates cannot be extracted unless hydrofluoric acid is used) (Ashley et al., 1998). These particular SRMs are not known to contain lead silicates or to give lower lead recoveries. However, it is important to note the possibility of such when using NIST SRMs for lead dust wipe analysis, since similar SRMs (e.g., Buffalo river sediment from Wyoming) do show recoveries in the low 90% range (Ashley et al., 1998).

Because accurate and precise estimated concentrations for the UC samples were imperative, ORNL imposed the following data quality requirements for the UC-prepared wipe samples: 1) each estimated concentration had to be within a $\pm 10\%$ interval of the target clearance level; 2) additional quality control (QC) samples (at least 5% of the total samples ordered) were to be prepared and analyzed by UC as a quality check prior to shipment of the samples; and 3) the relative standard deviation of the QC samples had to be $\leq 10\%$. It is important to note here the reason why the data quality requirements between the UC and ELPAT samples were different. The data quality requirements for the ELPAT samples (i.e., $\pm 20\%$ of the target value) were established by the ELPAT program. Since archived samples were being used, the ELPAT data quality requirements could not be changed.

As a quality check of the sample preparation process, UC prepared an additional 24 samples (5% of the total number ordered). UC extracted and analyzed the samples following internal procedures (nitric/hydrochloric acid extraction, followed by atomic absorption spectrometry - see EPA, 1996 for Method 3050B and Method 6010B) and provided those results to ORNL. For the 24 samples (eight at each of the three clearance levels), the average percent recovery (i.e., UC measured concentration/UC estimated concentration $\times 100\%$) was 97% (median value = 96%, standard deviation = 3%, range = 93% to 102%). Additionally, 42 randomly-selected samples (14 at each of the three clearance levels) were analyzed by the EPA Region 1 laboratory in North Chelmsford, MA, as an independent quality control check of the accuracy and precision of UC's sample preparation procedure (nitric acid digestion followed by ICP/AES analysis - EPA, 1996). The average percent recovery (EPA Region 1 reported concentration/UC estimated concentration $\times 100\%$) was 90% (median 89%, standard deviation = 2%), with a range of values from 86% to 93%. The average recovery determined from the EPA Region 1 analyses (90%) was lower than that which was determined by UC (102%), but both values were within the data quality requirement of $100 \pm 10\%$. Based on these data, ORNL determined that the UC sample preparation process met the established data quality criteria and was deemed acceptable for use in the determination of false positive/false negative error rates.

Distribution and Number of Samples

A total of 160 samples were analyzed in the verification test. Figure 2 is a plot containing the distribution of the sample concentrations that were analyzed in this study. Twenty samples were prepared by the University of Cincinnati at $\pm 10\%$ of each of the three clearance levels (3 test levels \times 20 samples = 60 samples total). Research Triangle Institute prepared 20 "blanks" at lead concentrations $< 2 \mu\text{g/wipe}$. These samples are noted as such in Figure 2. The remaining samples in Figure 2 are ELPAT samples. For most of the ELPAT samples, four samples were analyzed at each concentration level (16 test levels \times 4 samples each = 64 samples total). There were two concentration levels (at 49 and 565 $\mu\text{g/wipe}$) where eight samples were analyzed. While the set of samples at each concentration level were prepared using homogeneous source materials and an identical preparation procedure, ELPAT samples cannot be considered true "replicates" because each sample was prepared individually. However, these samples represent four samples prepared similarly at a specified target concentration, with an estimated value calculated from more than 100 analyses of similarly prepared samples.

Sample Randomization

The samples were packaged in 20-mL plastic scintillation vials and labeled with a sample identifier. Each participant received the same suite of samples, but in a randomized order. The samples were distributed in batches of 16. Completion of chain-of-custody forms documented sample transfer.

Description of Performance Factors

In Section 5, technology performance is described in terms of precision, accuracy, completeness, and comparability, which are indicators of data quality (EPA, 1996). False positive and negative results, sample throughput, and ease of use are also described. Each of these performance characteristics is defined in this section.

Precision

Precision is the reproducibility of measurements under a given set of conditions. Standard deviations estimated at each concentration level can be used to establish the relationship between the uncertainty and the average lead concentration. Standard deviation (SD) and relative standard deviation

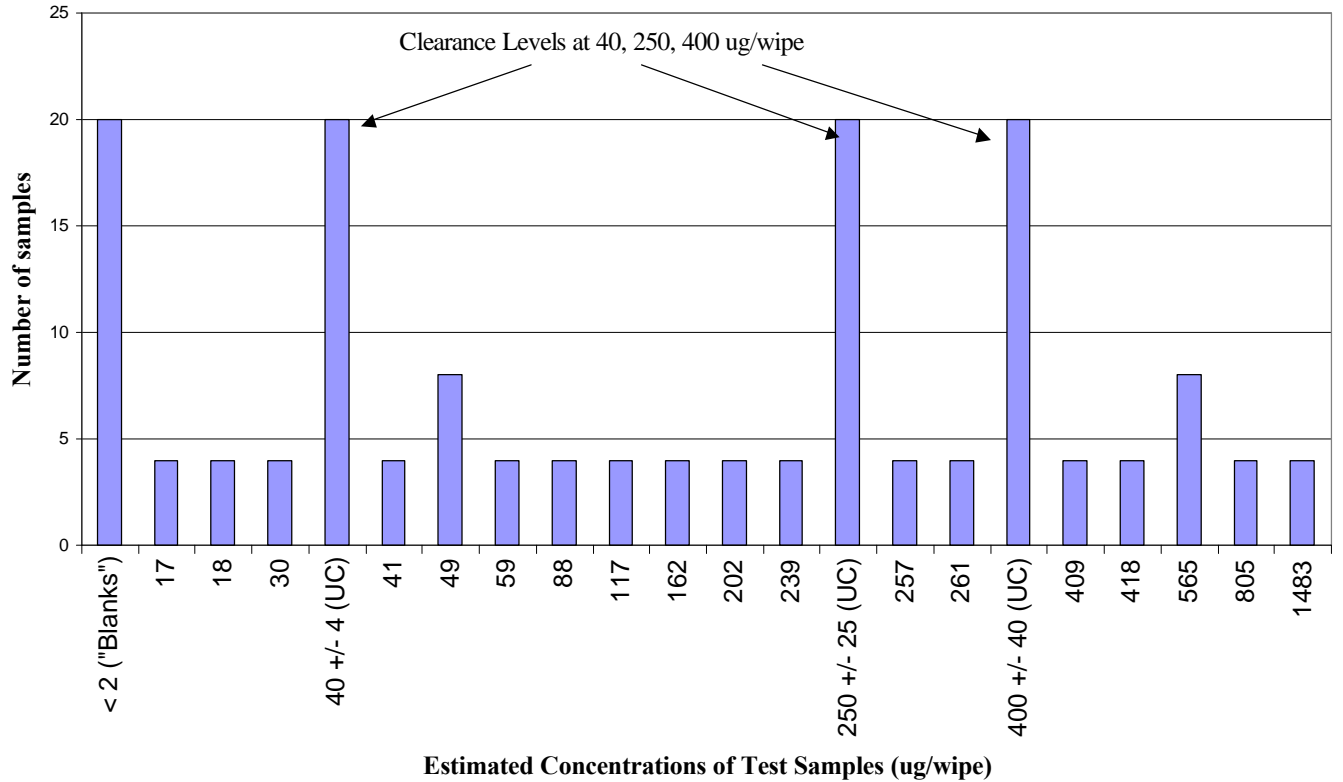


Figure 2. Distribution of concentration levels.

(RSD) for replicate results are used to assess precision, using the following equation:

$$RSD = (SD/average\ concentration) \times 100\% . \quad (Eq. 1)$$

The overall RSD is characterized by two summary values:

- mean — i.e., average;
- range — i.e., the highest and lowest RSD values that were reported.

The average RSD may not be the best representation of precision, but it is reported for convenient reference. An average RSD value less than 10% indicates that the measurements are very precise. RSDs greater than 20% should be viewed as indicators of larger variability and possibly non-normal distributions. The uncertainty in the analytical measurements will include influences from both the preparation (i.e., extraction) and measurement steps.

Accuracy

Accuracy is a measure of how close the measured lead concentrations are to estimated values of the true concentration. The estimated values for the

ELPAT samples are the certificate values that are reported on the certificate of analysis sheet provided with the samples. The ELPAT certified values represent an average concentration determined by more than 100 accredited laboratories that participated in previous rounds of ELPAT testing. The UC estimated value is the concentration reported by UC for individual samples, calculated by the amount of NIST-traceable material loaded on the dust wipes. The accuracy and precision of the UC value was assessed by an independent laboratory analyzing randomly selected QC samples. An EPA laboratory in Region 1 analyzed 10% of the total number of samples prepared by UC at each of the three concentration levels and confirmed that the process used to prepare the samples met the pre-determined data quality objective of accuracy within a $\pm 10\%$ interval of the estimated value.

Accuracy of the field technology measurements was statistically tested using t-tests or non-parametric tests at the 5% significance level. These statistical tests compared the average results with the overall estimated values using the precision of the sample measurements. Bias was quantified by computing the percent recovery for four similar samples or a single sample using the equation:

$$\text{percent recovery} = \left[\frac{\text{measured amount(s)}}{\text{estimated value}} \right] \times 100\% \quad (\text{Eq. 2})$$

Accuracy was assessed using both the ELPAT and UC estimated concentrations. The comparison to the ELPAT value represents how close the technology reported results to the consensus value, which represents the amount of “recoverable” lead in the sample. Because the UC samples were prepared gravimetrically from samples of known lead content, the comparison to the UC samples represents how close the technology reported results to an absolute lead value. Comparison to the gravimetric values reveals any bias imposed by the tested sampling and analytical method.

The optimum percent recovery value is 100%. Percent recovery values greater than 125% indicate results that are biased high, and values less than 75% indicate results that are biased low. A small but statistically significant bias may be detectable for a field technology if precision is high (i.e., low standard deviation). The field technology can still have acceptable bias with an average percent recovery in the interval of 75% to 125%. Bias within the acceptable range can usually be corrected to 100% by modification of calibration methods.

Comparability

Comparability refers to how well the field technology and conventional laboratory data agree. The difference between accuracy and comparability is that accuracy is judged relative to a known value, comparability is judged relative to the results of a laboratory procedure, which may or may not report the results accurately. Because true “replicates” were not available for use in this study, the averages from similar samples measured by the technology were compared with corresponding averages measured by the laboratory for all target concentration levels.

A correlation coefficient quantifies the linear relationship between two measurements (Draper and Smith, 1981). The correlation coefficient is denoted by the letter *r*; its value ranges from -1 to $+1$, where 0 indicates the absence of any linear relationship. The value $r = -1$ indicates a perfect negative linear relation (one measurement decreases as the second measurement increases); the value $r = +1$ indicates a perfect positive linear relation (one measurement increases as the second measurement increases). Acceptable *r* values are 0.990 or greater. The slope

of the linear regression line, denoted by the letter *m*, is related to *r*. Whereas *r* represents the linear association between the vendor and laboratory concentrations, *m* quantifies the amount of change in the vendor’s measurements relative to the laboratory’s measurements. A value of $+1$ for the slope indicates perfect agreement. Values greater than 1 indicate that the vendor results are generally higher than the laboratory, while values less than 1 indicate that the vendor results are usually lower than the laboratory.

Detectable Blanks

Twenty samples in the test were prepared at ≤ 2 $\mu\text{g/wipe}$, below the anticipated reporting limits of both the field technologies and the laboratory. Any reported lead for these samples is considered a “detectable blank”.

False Positive/Negative Results

A false positive (fp) result is one in which the technology detects lead in the sample above a clearance level when the sample actually contains lead below the clearance level (Keith et al., 1996). A false negative (fn) result is one in which the technology indicates that lead concentrations are less than the clearance level when the sample actually contains lead above the clearance level. For example, if the technology reports the sample concentration to be $35 \mu\text{g/wipe}$, and the true concentration of the sample is $45 \mu\text{g/wipe}$, the technology’s result would be considered a fn at the $40 \mu\text{g/wipe}$ clearance level. Accordingly, if the technology reports the result as $45 \mu\text{g/wipe}$ and the true concentration is $35 \mu\text{g/wipe}$, the technology’s result would be a fp at the $40 \mu\text{g/wipe}$ clearance level.

A primary objective for this verification test was to assess the performance of the technology at each of the three clearance levels of 40 , 250 , and $400 \mu\text{g/wipe}$, and estimate the probability of the field technology reporting a fp or fn result. For each clearance level, the probabilities of fn were estimated as curves that depend on a range of concentrations reported about the clearance level. These error probability curves were calculated from the results on the 60 UC samples at concentrations $\pm 10\%$ of each clearance level. In order to generate probability curves to model the likelihood of false negative results, it was assumed that the estimated concentration provided by UC was the true

concentration. However, this evaluation did not include the gravimetric preparation uncertainty in the UC estimated concentration. This error is likely to be much smaller than other sources of measurement error (e.g., extraction efficiency and analytical).

The fp/fn evaluation also included a comparison to the ELPAT sample results. The “estimated” value for the UC and ELPAT samples are defined differently. The UC value is based on weight of the NIST-traceable material, while the ELPAT estimated value is the average analytical reported value from more than 100 accredited laboratories. The UC sample estimated lead content is determined gravimetrically, which should be closer to the “true” concentration than an analytical measurement that includes preparation and instrumental errors. In contrast, determining the technology’s fp/fn error rates relative to the ELPAT estimated concentrations represents a comparison to typical laboratory values. One limitation of using the ELPAT sample is that concentrations covered a wider overall distribution of lead levels. Thus, the availability of sample concentrations that were tightly (i.e., +/- 10%) clustered about the clearance levels was limited. In order to perform a broader fp/fn analysis, the range of lead levels in the ELPAT samples that bracketed the pertinent clearance levels was extended to $\pm 25\%$ of the target concentration.

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 is 95% or greater.

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. Sample throughput is reported in Section 5 as number of

samples per day per number of analysts.

Ease of Use

A significant decision factor in purchasing an instrument is how easy the technology is to use. Several factors are evaluated and reported on in Section 5:

- What is the required operator skill level (e.g., technician or advanced degree)?
- How many operators were used during the test?
- Could the technology be run by a single person?
- How much training would be required in order to run this technology?
- How much subjective decision-making is required?

Cost

An important factor in the consideration of whether to purchase a technology is cost. Costs involved with operating the technology and a typical laboratory analyses are estimated in Section 5. To account for the variability in cost data and assumptions, the economic analysis is presented as a list of cost elements and a range of costs for sample analysis. Several factors affect the cost of analysis. Where possible, these factors are addressed so that decision makers can independently complete a site-specific economic analysis to suit their needs.

Miscellaneous Factors

Any other information that might be useful to a person who is considering purchasing the technology is documented in Section 5 under “Observations”. Examples of information that might be useful to a prospective purchaser are the amount of hazardous waste generated during the analyses, the ruggedness of the technology, the amount of electrical or battery power necessary to operate the technology, and aspects of the technology or method that make it user-friendly or user-unfriendly.

Section 4 — Laboratory Analyses

Background

EPA regulations (40 CFR Part 745.227(e)(8)(vii)) specify that residences and child occupied facilities built before 1978 that have undergone an abatement must pass clearance testing (CFR 2001). These EPA regulations also state in 40 CFR Part 745.227(f)(2) that dust samples for clearance must be analyzed by a laboratory recognized by EPA (CFR 2001). Many EPA-authorized state and tribal lead programs have the same or similar requirements. EPA's vehicle for recognizing laboratory proficiency is the National Lead Laboratory Accreditation Program (NLLAP). Although the NLLAP was initially designed to accredit fixed site laboratories, in August 1996 the NLLAP was modified so that mobile laboratory facilities and testing firms operating portable testing technologies could also apply for accreditation. Despite this modification, the NLLAP list of accredited laboratories has almost exclusively consisted of fixed site laboratories. One possible outcome of this ETV test is that more mobile laboratory facilities and testing firms operating portable testing technologies will apply for NLLAP accreditation. In order to assess whether the field portable technologies participating in this verification test produce results that are comparable to NLLAP-recognized data, an NLLAP-recognized laboratory was selected to analyze samples concurrently with the field testing.

NLLAP Laboratory Selection

NLLAP was established by the EPA Office of Pollution Prevention and Toxics under the legislative directive of Title X, the Lead-Based Paint Hazard Reduction Act of 1992. In order for laboratories to be recognized under the NLLAP, they must successfully participate in the ELPAT Program and undergo a systems audit. The acceptable range for the ELPAT test samples is based upon the reported values from participating laboratories. Acceptable results are within three standard deviations from the consensus value. A laboratory's performance is rated as proficient if either of the following criteria are met: (1) in the last two rounds, all samples are analyzed and the results are 100% acceptable; or (2) three-fourths (75%) or more of the accumulated results over four rounds are acceptable.

The NLLAP required systems audit must include an on-site evaluation by a private or public laboratory accreditation organization recognized by NLLAP. Some of the areas evaluated in the systems audit include laboratory personnel qualifications and training, analytical instrumentation, analytical methods, quality assurance procedures, and record keeping procedures.

The list of recognized laboratories is updated monthly. ORNL obtained the list of accredited laboratories in July 2001. The list consisted of approximately 130 laboratories. Those laboratories which did not accept commercial samples and those located on the U.S. west coast were automatically eliminated as potential candidates. ORNL interviewed at random approximately ten laboratories and solicited information regarding cost, typical turnaround time, and data packaging. Based on these interviews and discussions with technical panel members who had personal experience with the potential laboratories, ORNL selected DataChem (Cincinnati, OH) as the fixed-site laboratory. As a final qualifying step, DataChem blindly analyzed 16 samples (8 ELPAT and 8 prepared by UC) in a pre-test study. As shown in Table 1 below, DataChem passed the pre-test by reporting concentrations that were within 25% of the estimated concentration for samples above the reporting limit.

Laboratory Method

The laboratory method used by DataChem was hot plate/nitric acid digestion, followed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) analysis. The preparation and analytical procedures, as supplied by DataChem, can be found in the test plan (ORNL, 2001). To summarize the procedure, the wipe was digested in 2 mL of nitric acid, heated in a hotblock for 1 hour at 95 °C, diluted to 20 mL with distilled water, and analyzed by ICP-AES. DataChem's procedures are modifications of Methods 3050B and 6010B of EPA SW-846 Method Compendium for the preparation and analysis of metals in environmental matrices (EPA, 1996). Other specific references for the preparation and analysis of dust wipes are available from the American Society for Testing and Materials (ASTM, 1998).

Table 1. Summary of DataChem Pre-Test Results

Sample Type	DataChem Reported Conc ($\mu\text{g/wipe}$)	Estimated Conc ($\mu\text{g/wipe}$)	Percent Recovery	Analysis Order
ELPAT	<20	2.12	n/a	16
ELPAT	<20	2.12	n/a	12
ELPAT	41	41.3	99%	6
ELPAT	44	41.3	107%	3
ELPAT	190	201.6	94%	15
ELPAT	210	201.6	104%	9
ELPAT	440	408.7	108%	2
ELPAT	450	408.7	110%	13
UC	<20	10.3	n/a	4
UC	<20	5.9	n/a	1
UC	25	29.9	84%	14
UC	38	44	86%	10
UC	150	172.4	87%	11
UC	200	237.5	84%	7
UC	250	327.3	76%	5
UC	310	379	82%	8

Laboratory Performance

ORNL validated all of the laboratory data according to the procedure described in the verification test plan (ORNL, 2001). During the validation, the following aspects of the data were reviewed: completeness of the data package, correctness of the data, correlation between “replicate” sample results, and evaluation of QC sample results. Each of these categories is described in detail in the verification test plan. An evaluation of the performance of the laboratory results through statistical analysis of the data was performed and is summarized below. (See Section 3 for a detailed description of how the performance factors are defined and the calculations that are involved.)

In Table 2, DataChem’s reported values are compared to the estimated values to determine percent recovery (i.e., accuracy of the DataChem results) for both the ELPAT and the UC samples. The results are also shown graphically in Figure 3. The average percent recovery for the ELPAT samples was 98%, while the average for the UC samples was 91%. Both Table 2 and Figure 3 indicate that the analytical results from the University of Cincinnati wipe samples were generally reported lower than the estimated value, while the results for the ELPAT samples were closer to the estimated value. The better agreement with the ELPAT samples is not unexpected, given that the ELPAT estimated concentrations represent analytical consensus values that include typical extraction inefficiencies and instrumental error.

The negative bias observed with the UC and the ELPAT samples was statistically significant. The cause of the negative bias for the UC samples could be related to: 1) extraction inefficiencies (due to the use of NIST SRMs that contain lead that is unrecoverable with the extraction procedure which was used) and/or, 2) typical analytical variation due to preparation and measurement errors. Another indication of accuracy is the number of individual ELPAT results which were reported within the acceptance ranges that have been established for those samples. For the 72 ELPAT samples (> 20 $\mu\text{g/wipe}$), DataChem reported 71 (99%) within the acceptable ranges of values.

The precision assessment presented in Table 3 indicates that the analyses were very precise. The average RSD for the ELPAT samples was 7%, while the average RSD for the UC samples was 8%. The variability of the UC sample preparation process, provided for reference of the minimal achievable RSD for the UC samples, was 6%. A single estimate of the ELPAT variability was not determined, since the ELPAT samples were comprised of 20 different batches of samples. DataChem reported all 20 detectable blank samples correctly as < 20 $\mu\text{g/wipe}$. In addition, DataChem reported seven of the eight samples with estimated concentrations of either 16.9 $\mu\text{g/wipe}$ or 17.6 $\mu\text{g/wipe}$ as less than their reporting limit of 20 $\mu\text{g/wipe}$ and only one was incorrectly reported as 30 $\mu\text{g/wipe}$.

Table 2. Summary of DataChem Percent Recovery Values by Sample Source

Statistic	ELPAT	UC
n ^a	72	60
average % recovery	98	91
standard deviation	9	3
minimum % recovery	81	86
maximum % recovery	143	102

^a excludes estimated values <20 µg/wipe (n=28)

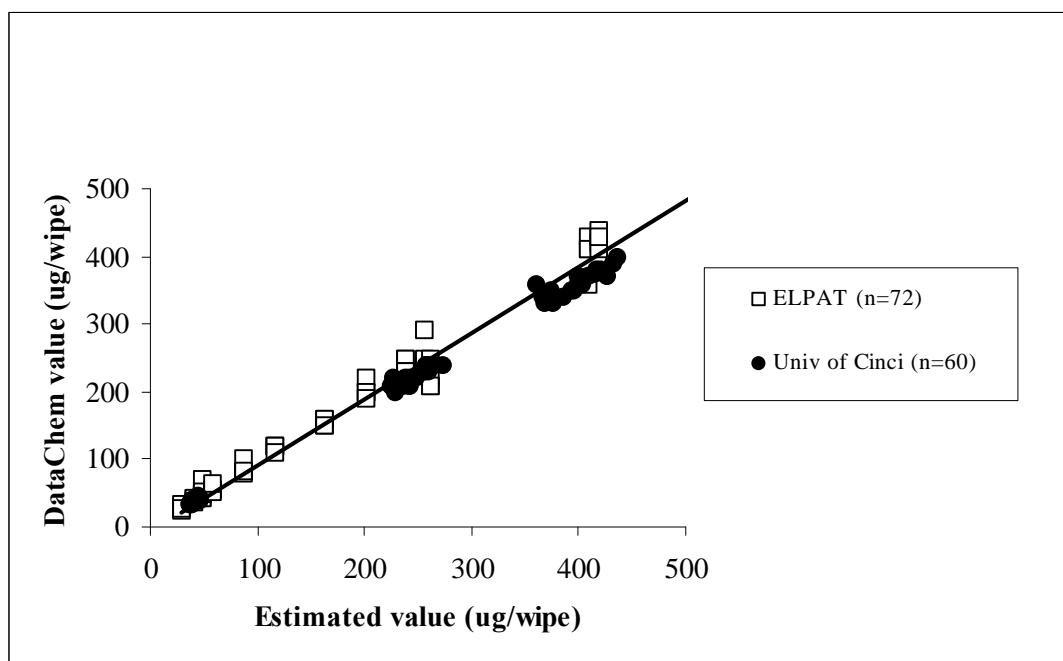


Figure 3. Plot of DataChem reported values versus estimated values, shown for concentrations less than 500 µg/wipe.

Table 3. Summary of DataChem Precision Estimates by Sample Source

Sample Source	n	average RSD	Min RSD	Max RSD
ELPAT	18 ^a	7	2	21
UC	3 ^b	8	6	9
UC preparation	3 ^c	6	6	7

^a 4 replicates in each sample set

^b 20 replicates in each sample set

^c This value represents the variability in the sample preparation process.

An important evaluation parameter for the analysis of dust wipe samples is how the method performs at the clearance levels and the method's likelihood of reporting false positive (fp) and false negative (fn) results. Recall from the experimental design that 20 UC samples were prepared at $\pm 10\%$ of each clearance level of 40, 250, and 400 $\mu\text{g/wipe}$, for a total of 60 UC samples. The ELPAT samples covered a wider range of concentrations. There was a total of 40 ELPAT samples that fell within a $\pm 25\%$ interval of the target values that could be used for the fp/fn assessment. The number of false negative and false positive results reported by DataChem relative to the UC and ELPAT estimated concentrations is summarized in Table 4. There are a specific number of possible fp and fn results. For example, if the estimated lead level on the wipe is less than the clearance level (CL), then it is not possible to produce a false negative result; only a false positive (i.e., > 40) result is possible. For the UC samples, in every case where the estimated concentration was less than the CL, DataChem reported a result for that was also less than the CL, indicating no fp results at any of the three CL. DataChem reported two fp results for the ELPAT samples out of a possible 12.

When the estimated concentration was above the clearance level, however, DataChem sometimes reported results as less than the clearance level. DataChem reported a higher rate of fn results for the UC samples than the ELPAT samples (23 of 30 vs 7 of 28 possible fn results, respectively). This finding is not surprising, since the results reported above indicated that DataChem's results were negatively biased, or reported lower than the estimated values for the UC samples. As stated in Section 3, it is important to note that in this evaluation, the estimated concentration of the UC samples is assumed to be the "true" concentration, and the uncertainty in gravimetric preparation for the UC estimated concentration is not considered in the evaluation.

Figures 4, 5, and 6 show models of the likelihood of DataChem reporting a false negative result at each of the clearance levels versus the true concentrations of the UC samples. (Note that only the UC samples must be used in generation of probability curves because these estimated values are a closer representation of the true lead concentration than the ELPAT estimated concentration. Song et al., 2001.) These figures indicate that the likelihood of DataChem reporting false negative results for the UC

samples at the exact clearance level is high, near 100% in all three cases. This means, for example, that if DataChem reported a value as exactly 250 $\mu\text{g/wipe}$, the probability that the true concentration is >250 is essentially 100%. Again, this is due to the negative bias that was observed in the measurement of the UC samples. The plots also demonstrate that, due to the relatively high level of precision of results reported by DataChem, the performance is very minimally impacted by performing replicate analyses, as the distribution of false negative probabilities is very similar whether 1 or 5 measurements (in Figures 4, 5, and 6, delineated as $N = 1$, $N = 2$, etc.) are performed. The interpretation of these curves for use in a "real-world" situation can be demonstrated by the following example. Suppose that a user decides that an acceptable level of risk for having false negative results is 5%. Using Figure 4, 5% FN probability ($y = 0.05$) corresponds to a "true" lead concentration of 46 $\mu\text{g/wipe}$ (meaning if the true concentration of the sample is 46 $\mu\text{g/wipe}$, there is only a 5% chance/risk that DataChem will report the value as < 40 $\mu\text{g/wipe}$.)

By plotting DataChem's measured values versus the estimated concentrations, the equations of the linear regression lines can be calculated for each of the three CL. The slope, intercept, and correlation coefficient for the ELPAT and UC samples are presented in Table 5. The user might like to know at what reported value (and at what associated probability) will DataChem be likely to report a "clean" sample (i.e., there is a high probability that the true concentration is $< \text{CL}$). For example, for the UC samples, we know that a value reported by DataChem as 39 $\mu\text{g/wipe}$ is biased low and will have a true concentration of > 40 (41.8 $\mu\text{g/wipe}$, using the linear regression equation in Table 5). A true concentration of 40 $\mu\text{g/wipe}$ for a UC sample would correspond to a reported value rounded to the nearest whole number of 37 $\mu\text{g/wipe}$ (see Table 5). For an ELPAT sample, a true concentration of 40 $\mu\text{g/wipe}$ corresponds to a DataChem reported value of 40 $\mu\text{g/wipe}$, because the negative bias was not as large for the ELPAT samples. Estimates of the reported concentration at the 250 and 400 $\mu\text{g/wipe}$ levels are reported in Table 5. In both cases, the reported concentrations for the ELPAT samples are higher (i.e., closer to the clearance level) than those of the UC samples.

The user is reminded that the data obtained during this verification test represent performance at one

point in time. The data produced by DataChem at some other time after the writing of this report may or may not be similar to what has been produced here. To understand a method's performance at critical clearance levels, it is recommended that the

user perform their own assessment of the method's performance by including samples of known concentration (at or near the clearance levels) along with the analysis of "real-world" samples.

Table 4. False Positive/False Negative Results for DataChem Measurements of UC Samples

Evaluation Parameter	Sample Source	Number of Samples			Total
		40 µg/wipe	250 µg/wipe	400 µg/wipe	
fp: # samples where DataChem reported the result as > CL ^a of the # samples where the estimated concentration was < CL	UC	0 of 9	0 of 11	0 of 10	0 of 30
	ELPAT	0 of 4	2 of 8	0 of 0 ^b	2 of 12
fn: # samples where DataChem reported the result as < CL of the # samples where the estimated concentration was > CL	UC	5 of 11	9 of 9	9 of 10	23 of 30
	ELPAT	1 of 12	5 of 8	1 of 8	7 of 28

^a CL = clearance level

^b Because all eight ELPAT values were above 400 µg/wipe, no samples were available to assess fp results at this level.

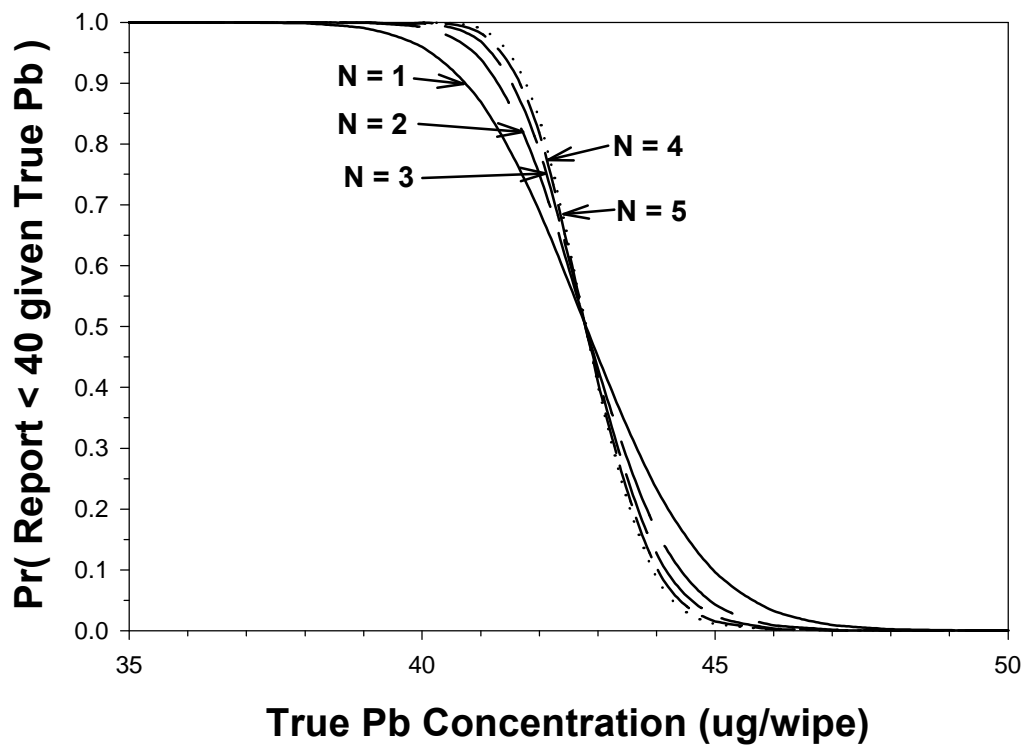


Figure 4. False negative probabilities for DataChem average concentrations at a target concentration level of 40 µg/wipe.

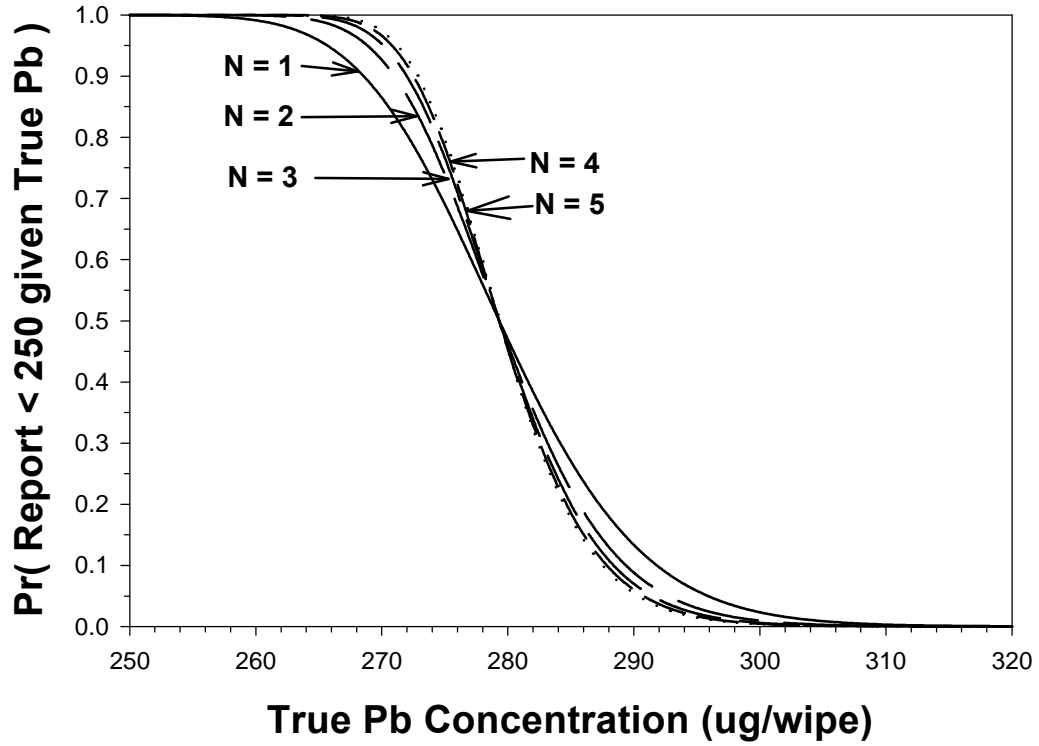


Figure 5. False negative probabilities for DataChem average concentrations at a target concentration level of 250 $\mu\text{g/wipe}$.

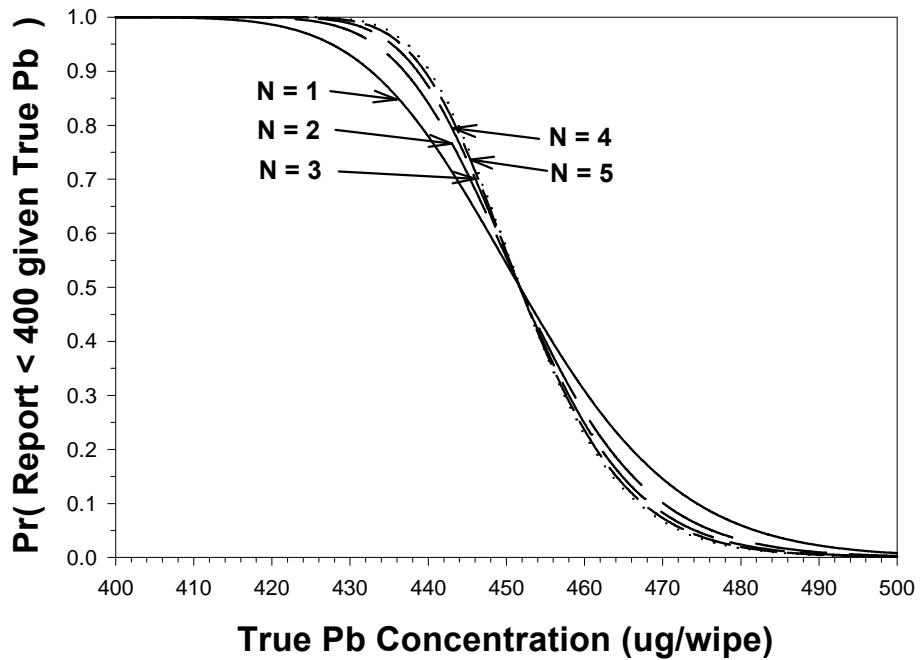


Figure 6. False negative probabilities for DataChem average concentrations at a target concentration level of 400 $\mu\text{g/wipe}$.

Table 5. Summary of the Linear Regression Constants and Recovery Data for DataChem's Measurements Versus the Estimated Concentrations at the Clearance Levels

Evaluation Parameter	40 µg/wipe		250 µg/wipe		400 µg/wipe	
	UC	ELPAT	UC	ELPAT	UC	ELPAT
n	20	16	20	16	20	8
slope	1.021	1.612	0.829	0.578	0.736	2.394
intercept	-3.673	-6.182	18.557	90.826	67.649	-575.771
correlation coefficient	0.884	0.840	0.879	0.549	0.861	0.492
average % recovery	93%	101%	90%	96%	91%	100%
SD of % recovery	4%	13%	3%	9%	3%	5%
Reported concentration at the CL	37 µg/wipe	40 µg/wipe	226 µg/wipe	234 µg/wipe	362 µg/wipe	382 µg/wipe

Section 5 — Technology Evaluation

Objective and Approach

The purpose of this section is to present a statistical evaluation of the XL-700 XRF data and determine the technology's ability to measure lead in dust wipe samples. This section includes an evaluation of comparability through a one-to-one comparison with NLLAP-recognized laboratory data. Other aspects of the technology (such as accuracy, precision, cost, sample throughput, hazardous waste generation, and logistical operation) are also evaluated in this section. The Appendix contains the raw data provided by the vendor during the verification test that were used to assess the performance of the XL-700.

Precision

Precision is the reproducibility of measurements under a given set of conditions. Precision was determined by examining the results of blind analyses for replicate samples with estimated concentrations greater than the XL-700 reporting limits (4.9 to 6.1 $\mu\text{g}/\text{wipe}$). For the ELPAT samples, precision was measured on each set of four samples from a particular round of archived samples. For the 20 sets of ELPAT samples, the XL-700's average RSD value was 8%, with a range from 1 to 20%, indicating that the XL-700 measurements were very precise (Table 6). For the UC samples, 20 samples were analyzed at three target concentration levels of 40, 250, and 400 $\mu\text{g}/\text{wipe}$. The average precision of the UC sample measurements by the XL-700 was 8% RSD. With the expectation that UC was to prepare the samples as close to the target concentrations as possible, the allowable variability was 10% RSD. The actual variability of the UC preparation process was an average of 5% RSD.

Accuracy

Accuracy represents the closeness of the XL-700's measured concentrations to the estimated content of spiked samples. One measure of accuracy is the number of results for the ELPAT samples that were reported within the individual acceptance ranges that have been established for those samples. Of the 80 ELPAT samples, the XL-700 reported 67 of the results (84% of the total) within the acceptance ranges. All 13 that were outside the range were above the upper limit.

Table 6. Precision of the XL-700

Source	No. of sample sets	RSD, %		
		Average	Min	Max
ELPAT	20 ^a	8	1	20
UC	3 ^b	8	7	9
UC prep ^c	3	5	4	6

^a 4 replicates in each sample set

^b 20 replicates in each sample set

^c precision of UC sample preparation process

The results reported by the XL-700 can also be compared to the ELPAT certificate value, i.e., the average concentration reported by 100+ laboratories who participated in previous rounds of ELPAT testing. The results for the 80 ELPAT samples reported by the XL-700 were biased high (Table 7). The average percent recovery value of 119%, the range of values was from 95 to 155%, and the median value of 116%. The UC sample results were lower, with an average percent recovery of 107%, and a range of values from 89% to 122%. While biased high, both average recovery values were within the range of acceptable bias (100% \pm 25%).

Table 7. Accuracy of XL-700

Statistic	% Recovery	
	ELPAT	UC
n ^a	80	60
average	119	107
standard deviation	14	7
minimum	95	89
maximum	155	122

^a Excludes estimated values < 10 $\mu\text{g}/\text{wipe}$

Another way to assess accuracy is to plot the results obtained with the XL-700 versus the estimated values that are > 10 $\mu\text{g}/\text{wipe}$. The linear regression constants for the plot of the ELPAT and the UC data

are listed in Table 8. As expected, the slopes indicate that the results are biased high, similar to the above conclusions regarding the percent recovery calculations. While the performance met the criteria stated in accuracy in Section 4 is met (unbiased if the average percent recovery is between 75 and 125%), the XL-700 result generally was reported higher than the estimated value.

Comparability

Comparability refers to how well the XL-700 and the NLLAP-recognized laboratory data agreed. In this evaluation, the laboratory results are not presumed to be the “correct” answers. Rather, these results represent what a typical fixed laboratory would report for these types of samples. A direct comparison of the XL-700 results and the laboratory results was performed for all ELPAT and UC samples that were reported above 20 µg/wipe. (Note: Data was reported to 6 µg/wipe for the XL-700, but the lab only reported to 20 µg/wipe.) Because each wipe was prepared individually, a true one-to-one

matching of XL-700 and laboratory results can not be performed. However, the average concentrations of the samples prepared at specific levels can be compared for the XL-700 and laboratory results. The difference between the regression slopes ($m = 1.206$ for ELPAT and $m = 1.112$ for UC) and a slope with a perfect agreement line ($m = 1.000$) is statistically significant, but the correlation coefficients ($r = 0.999$ for both ELPAT and UC) show a strong linear relationship between DataChem and XL-700 results (Table 8). To illustrate the strong linear agreement between the XL-700 and NLLAP laboratory results, Figure 7 is a plot of the average XL-700 results versus the average DataChem results for both ELPAT and UC data. For clarity, only those values < 500 µg/wipe are shown.

Detectable Blanks

Of the samples that were prepared at < 2 µg/wipe, the XL-700 correctly reported all 20 as less than the reporting limits. Reporting limits ranged from 4.9 to 6.1 µg/wipe, so no detectable blanks were reported.

Table 8. Linear regression constants for the plots of the XL-700 versus the estimated values and versus the DataChem average measurements

Statistic	versus estimated values		versus DataChem average concentrations	
	UC	ELPAT	UC	ELPAT
n	60	80	3	18
slope (standard error)	1.085 (0.015)	1.099 (0.009)	1.206 (0.021)	1.112 (0.011)
intercept (standard error)	0.150 (3.962)	9.698 (3.918)	-3.290 (5.195)	13.283 (5.257)
<i>r</i>	0.995	0.998	0.999	0.999

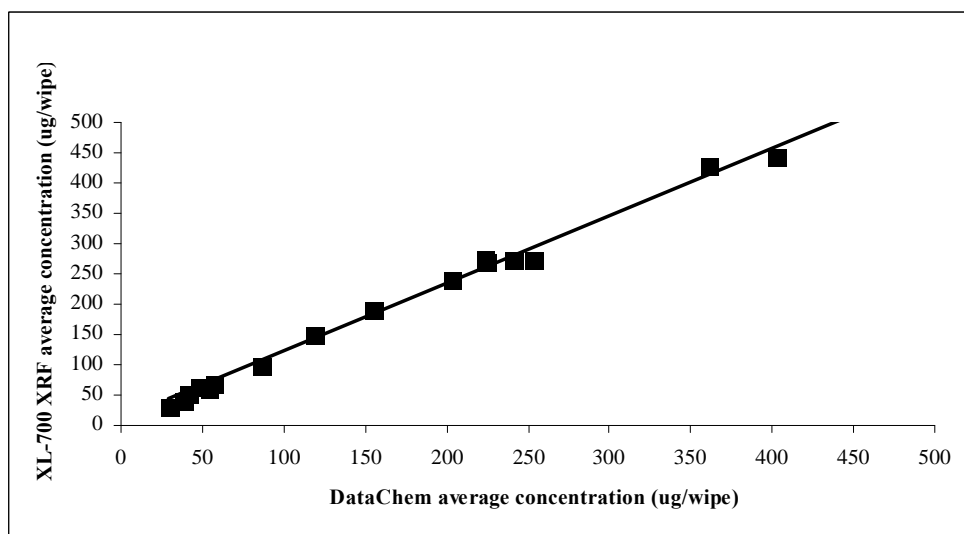


Figure 7. Plot of the average XL-700 concentration versus the average DataChem concentrations for all samples (n=21), shown for ELPAT and UC concentrations less than 500 $\mu\text{g/wipe}$.

False Positive/False Negative Results

Similar to the evaluation described and presented in Section 4 for DataChem, the number of false negative and false positive results reported by the XL-700 relative to the estimated concentrations for both the ELPAT and UC samples is summarized in Table 9. For many cases where the estimated concentration was less than the clearance level (CL), the XL-700 reported a result that was greater than the CL (21 of 34 possible fp results for the UC samples, and 6 of 12 fp results for the ELPAT samples at the three CLs). When the estimated concentration was equal to or greater than the clearance level, however, the XL-700 reported few as less than the CL and nearly all of the results as greater than the CL (2 of 26 possible fn results for UC samples and 2 of 28 fn results for ELPAT samples). These findings are not surprising, since the accuracy results reported above indicated that the XL-700 results were positively biased, or reported more than the estimated values.

In Figures 8, 9, and 10, the false negative probabilities at the three clearance levels are compared for the DataChem and XL-700 results. In these figures, the two-sided 90% confidence intervals (not shown in the figures for clarity) are used to express uncertainty on the false negative curves. These confidence intervals overlap for XL-700 and DataChem at the 40 $\mu\text{g/wipe}$ clearance levels (Figure 8) over the range of true lead concentrations. The overlapping confidence

intervals indicate the two methods are comparable when considering their uncertainty. In Figures 9 and 10, the 90% confidence intervals for the two methods only overlap for part of the true lead concentration ranges (greater than 268 $\mu\text{g/wipe}$ for the 250 $\mu\text{g/wipe}$ clearance level and greater than 440 $\mu\text{g/wipe}$ for the 400 $\mu\text{g/wipe}$ clearance level). These results indicate that the XL-700 appears to be less prone to false negatives even when considering the uncertainty of the two methods at the 250 $\mu\text{g/wipe}$ and the 400 $\mu\text{g/wipe}$ clearance levels.

Table 10 describes the linear regression constants for XL-700 measured concentration versus estimated concentration for the three CLs, average percent recovery values and standard deviations, and an estimate of the XL-700 reported concentration at each clearance level. The average recoveries indicate that the XL-700 results were positively biased for all three clearance levels for both ELPAT and UC samples. This is also apparent in the estimated concentration that a user might require from the XL-700 in order to be reasonably confident that the true result is below the clearance level. For example, for the DataChem results, the values should be 37, 226, and 362 $\mu\text{g/wipe}$ in order to be confident that the result is below 40, 250, and 400 $\mu\text{g/wipe}$, respectively. Since the XL-700 is positively biased, a result produced at 42, 276, and 431 $\mu\text{g/wipe}$ would indicate levels that are very likely to be below the respective CL. These levels are about 5-10% above the clearance levels, so to be conservative, the user

could feel relatively confident that a number produced by the XL-700 at the actual clearance level of 40, 250, and 400 $\mu\text{g/wipe}$ will have a true lead level below the CL.

Once again, the reader is reminded that the fp/fn evaluation reported herein is based on the instrument's performance during this verification test. Results produced under different conditions and

with different samples may or may not be similar. Regardless of analytical technique, there is some uncertainty in assessing false positive and false negative error rates around critical action levels due to "normal" levels of variability (Song et al., 2001). Analytical values falling near the level of interest should be interpreted with care for both fixed-laboratory and field-based analytical methods.

Table 9. False Positive/False Negative Error Rates for XL-700 Measurements

Evaluation Parameter	Sample Source	Number of Samples			Total
		40 $\mu\text{g/wipe}$	250 $\mu\text{g/wipe}$	400 $\mu\text{g/wipe}$	
fp: # samples where XL-700 reported the result as $> \text{CL}^a$ of the # samples where the estimated concentration was $< \text{CL}$	UC	4 of 12	10 of 12	7 of 10	21 of 34
	ELPAT	0 of 4	6 of 8	0 of 0 ^b	6 of 12
fn: # samples where XL-700 reported the result as $< \text{CL}$ of the # samples where the estimated concentration was $> \text{CL}$	UC	1 of 8	1 of 8	0 of 10	2 of 26
	ELPAT	0 of 12	2 of 8	0 of 8	2 of 28

^a CL = clearance level

^b Because all eight ELPAT values were above 400 $\mu\text{g/wipe}$, no samples were available to assess fp results at this level.

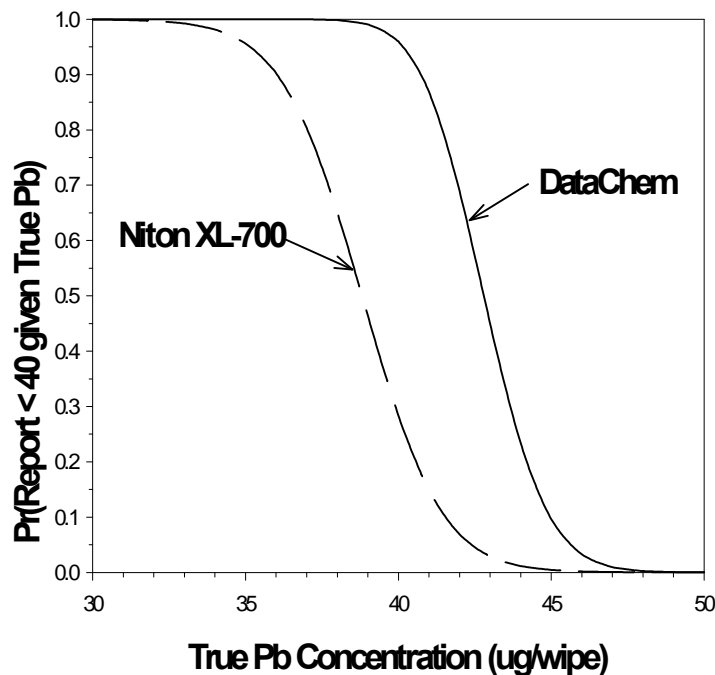


Figure 8. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 40 $\mu\text{g/wipe}$.

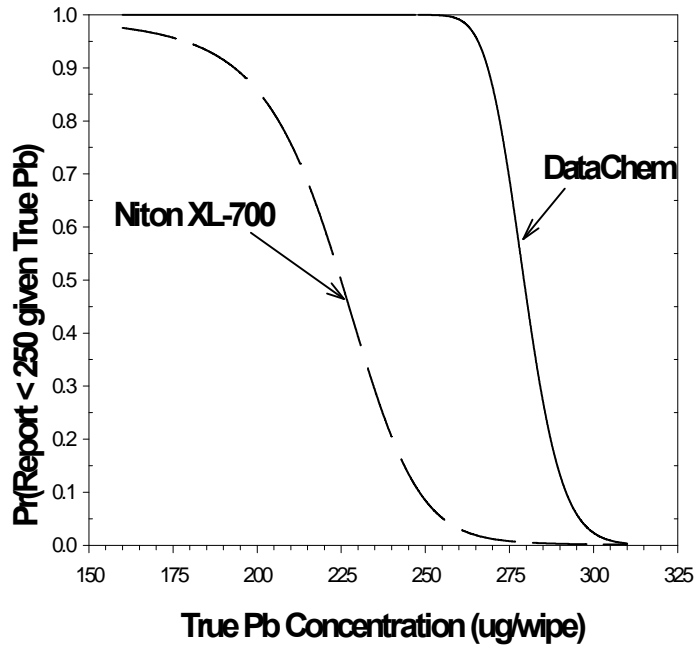


Figure 9. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 250 $\mu\text{g/wipe}$.

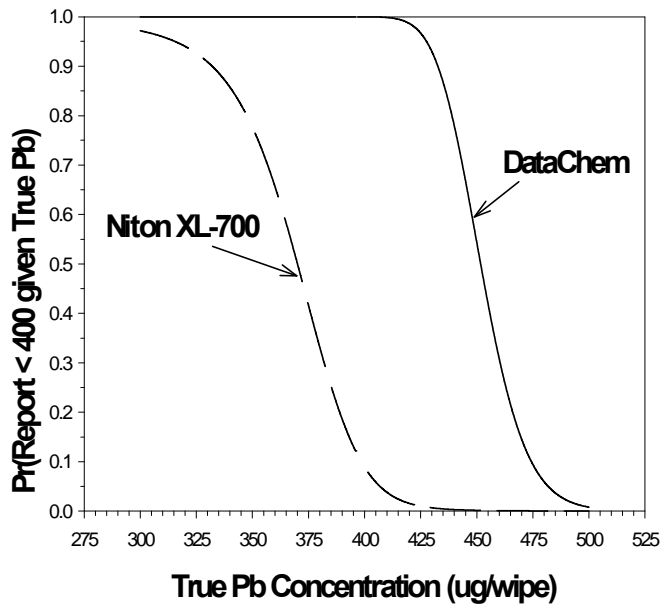


Figure 10. Comparison of the false negative probabilities for the NITON XL-700 and DataChem average concentrations at a target concentration level of 400 $\mu\text{g/wipe}$.

Table 10. Summary of the Linear Regression and Recovery Data for the XL-700 Response versus the Estimated Concentrations

Evaluation Parameter	40 µg/wipe		250 µg/wipe		400 µg/wipe	
	UC	ELPAT	UC	ELPAT	UC	ELPAT
n	20	16	20	16	20	8
slope	1.234	1.5373	1.041	0.560	1.043	8.431
intercept	-7.897	-12.083	15.827	125.062	13.973	-3000.69
correlation coefficient	0.771	0.869	0.587	0.606	0.618	0.969
average % recovery	103%	124%	111%	112%	108%	117%
SD of % recovery	6%	16%	7%	10%	6%	9%
Reported concentration at the CL	42 µg/wipe	49 µg/wipe	276 µg/wipe	272 µg/wipe	431 µg/wipe	372 µg/wipe

Completeness

Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result was not rejected). Valid results were obtained by the technology for all 160 dust wipe samples. Therefore, completeness was 100%.

Sample Throughput

Sample throughput is representative of the estimated amount of time required to prepare and analyze the sample and perform the data analysis. Operating in the field, a single analyst (a NITON expert) analyzed all 160 samples in four 12-hour days. Eight measurements were taken for each wipe, for a total of 320 measurements. For the first two days of testing, the analyst used a 60 second count time, and analyzed 25 to 30 samples per day. For the last two days of testing, the analyst used a 30 second count time, and analyzed 50 to 60 samples per day. The change in procedure did not appear to affect the results.

Ease of Use

The technology can be operated by a single person. Users unfamiliar with the technology should attend a one-day training course provided by NITON. No particular level of educational training is required for the operator. The analyst who operated the instrument during the verification test was a NITON expert.

Cost Assessment

The purpose of this economic analysis is to estimate the range of costs for analysis lead in dust wipe

samples using the NITON XL-700 Series XRF and a conventional analytical laboratory method. The analysis was based on the results and experience gained from this verification test, costs provided by NITON, and representative costs provided by the laboratory to analyze the samples. To account for the variability in cost data and assumptions, the economic analysis is presented as a list of cost elements and a range of costs for sample analysis by the XRF spectrum analyzer and by the laboratory. Costs were prepared at the time this report was written and are subject to change.

Several factors affected the cost of analysis. Where possible, these factors were addressed so that decision makers can complete a site-specific economic analysis to suit their needs. The following categories are considered in the estimate:

- sample shipment costs,
- labor costs, and
- equipment costs.

Each of these cost factors is defined and discussed and serves as the basis for the estimated cost ranges presented in Table 11. This analysis assumed that the individuals performing the analyses were fully trained to operate the technology. Costs for sample acquisition and pre-analytical sample preparation, tasks common to both methods, were not included in this assessment.

Table 11. Estimated analytical costs for lead dust wipe samples

Analysis method:	XL-700 Series XRF	Analysis method:	EPA SW846 6010b
Analyst/manufacturer:	NITON	NLLAP Laboratory:	DataChem
Sample throughput:	40 samples/day	Actual turnaround:	18 working days
Cost category	Cost (\$)	Cost category	Cost (\$)
Sample shipment	0	Sample shipment	
		Labor	100–200
		Overnight shipping	50–100
Labor		Labor	
Rate	50–100/h per analyst	Rate	30 per sample
Equipment		Equipment	Included ^a
Instrument purchase price	28,995		
Instrument lease price	1,980 per week 5,190 per month		
Reagents/supplies	0.10 per sample		
Waste Disposal	0 ^b	Waste Disposal	Included

^a“Included” indicates that the cost is included in the labor rate.

^b There was no cost to dispose of hazardous waste from the verification test because NITON elected to keep the “used” wipes since the analysis is non-destructive. However, in a real-world situation, the user would have the option of sending the wipes to an NLLAP laboratory for secondary analysis, archiving the samples, or disposing of the wipes as waste.

XL-700 Series XRF Costs

The costs associated with using the spectrum analyzer included labor and equipment costs. No sample shipment charges were associated with the cost of operating the spectrum analyzer because the samples were analyzed on site.

Labor

Labor costs included on-site labor to perform the analyses. The cost of the on-site labor was estimated at a rate of \$50–100/h, depending on the required expertise level of the analyst. This cost element included the labor involved during the entire analytical process, comprising sample preparation, sample management, analysis, and reporting. If the user would have to travel to the site, the cost of mobilization and demobilization, travel, and per diem expenses should also be considered. However, in a typical application where the XL-700 might be used, the analysis would usually be carried out by a person located on site.

Equipment

Equipment costs included purchase of equipment, and the reagents and other consumable supplies necessary to complete the analysis.

- *Radioactive Source and Re-Sourcing Description* (provided by NITON): For the analysis of lead, the instrument is supplied with a sealed Cd-109 source of between 10-40mCi strength. The half life of Cd-109 is 1.27 years. This means that after 15 months, the source strength is half of what it was at the beginning. NITON recommends a source replacement every 2 years. This recommendation is made solely for the benefit of speeding up the analysis, as performance otherwise is not affected. The unit uses a standard source decay algorithm to determine the source activity and compensates for the reduced activity by increasing the analysis time accordingly. Thus, when the instrument is first supplied, instrument time = real time. At the end of 1.27 years instrument time = 2 * real time. This has the benefit to the end user, that as long as they always use the same instrument analysis time, they will obtain the same instrument performance in terms of accuracy and precision. For the test, the Cd-109 source used in the XL-700 instrument was 11 months old.

- *Instrument purchase* (provided by NITON): The spectrum analyzer can be purchased for \$28,995. The spectrum analyzer can be calibrated for thin sample, bulk sample, or both. In the simplest version (XL-701S), the spectrum analyzer can quantify lead as well as Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Hg, Rb, Zr, and Mo. It can also be upgraded for analysis of soil/bulk samples with the same element suite and/or upgraded for additional elements (Cd, Ba, Ag, Sn, Sb, K, Ti, Sc, V). The purchase price includes shipping and handling, two 8-hour NiMH (Nickel Metal Hydride) battery packs; 110/220 VAC battery charger with 12 volt DC adapter; waterproof, crush-resistant carrying case with locks, Windows™ compatible reporting software; and an RS-232 cable for downloading data. The spectrum analyzer configured as described above can be rented from NITON for \$1,980 per week or \$5,190 per month, subject to availability, security deposit, and completed rental agreement. Longer-term leases are available for periods ranging from one through five years via NITON Financial Services. NITON provides training for all users, regardless of whether they rent or purchase the instruments. The full-day (8-hr) class covers both radiation safety and instrument operation and is conducted in metropolitan areas across the country on an ongoing basis. This training is offered at no charge, and is available for both customers, as well as those interested in learning more about the technology. Additional on-site operational training is available at no charge for customers from local Sales Offices upon instrument delivery. NITON offers several options that influence both the frequency and cost of re-sourcing. These options will be explained with instrument purchase.
- *Reagents and supplies.* The only consumable supply is the sample baggie, which NITON sells for \$0.10 each. NITON used a toaster oven (< \$100) in the test to dry the samples.
- *Labor.* This cost element included all of the tasks associated with shipping the samples to the NLLAP laboratory. Tasks included packing the shipping coolers, completing the chain-of-custody documentation, and completing the shipping forms. The estimate to complete this task ranged from 2 to 4 h, at \$50 per hour.
- *Overnight shipping.* The overnight express shipping service cost was estimated to be \$50 - 100 for two boxes of samples.

Labor, Equipment, and Waste Disposal

The labor quotes from commercial analytical laboratories that offered to perform the analysis for this verification test ranged from \$20 to \$30 per sample, with turnaround time estimates ranging from 7 to 14 days. Some laboratories can provide a 1-2 day turnaround, but the quick turnaround was not necessary for this test. The quotes were dependent on many factors, including the perceived difficulty of the sample matrix, the current workload of the laboratory, data packaging, and the competitiveness of the market. This rate was a fully loaded analytical cost that included equipment, labor, waste disposal, and report preparation. The cost for DataChem to analyze samples for this verification test was \$30 per sample, with a turnaround time of 18 working days.

Cost Assessment Summary

An overall cost estimate for use of the XL-700 Series spectrum analyzer versus use of the NLLAP-laboratory was not made because of the extent of variation in the different cost factors, as outlined in Table 11. The overall costs for the application of any technology would be based on the number of samples requiring analysis, the sample type, and the site location and characteristics. Decision-making factors, such as turnaround time for results, must also be weighed against the cost estimate to determine the value of the field technology's providing immediate answers versus the laboratory's provision of reporting data within 18 days of receipt of samples.

Miscellaneous Factors

The following are general observations regarding the field operation and performance of the XL-700 Series XRF spectrum analyzer:

- The spectrum analyzer required no electrical power, as it was battery-operated. Two (maximum 8-hour usage) batteries were used throughout the 12-hour workday and recharged.

Laboratory Costs

Sample Shipment

The costs of shipping samples to the laboratory included overnight shipping charges as well as labor charges associated with the various organizations involved in the shipping process.

- The NITON analyst was ready for the first set of samples within 30 min of arriving on site.
- The NITON analyst took four readings on the front and back of each dust wipe, for a total of eight readings per wipe. The average concentration from the four readings on the front of the wipe were averaged with the average of the four readings on the back of a wipe to give the final result. NITON recommends performing all eight readings for the most accurate results.
- On the first two days of testing, the sample analyses were taking longer than expected (25-30 samples per day). The radioactive source in the XL-700 spectrum analyzer was approximately 11 months old. The NITON analyst believed that the instrument could achieve adequate sensitivity with a shorter count time, so he elected to reduce the run-times to 30 seconds. (See Appendix. Samples 1-48 were analyzed using 60 second count time, and samples 49-160 were analyzed using 30 second count time.) Replicate results analyzed using the two count times were not statistically different (see Appendix for data).
- This spectrum analyzer can be used to detect and quantify multiple metals, although the spectrum analyzer's performance for lead is the only metal that was verified in this test.
- The NITON analyst ran QC samples at the start and periodically throughout the day to confirm that the spectrum analyzer was working properly.
- The dust in the ELPAT sample was smoothed out to a thin layer on the wipe. (This was not done for the UC samples because the dust was sprinkled rather than mounded.) All wipes were folded so that it fit the size of the XRF window and such that it was as flat as possible for the most accurate reading.
- On the last day of testing, 15 potential users attending a nearby conference on lead-safe housing observed the technology in operation and completed a survey about its user friendliness. In general, the observers reported that the NITON XRF spectrum analyzer seemed to be a very practical approach to lead testing.

Many of the observers had used or currently use the spectrum analyzer for lead-based paint testing, so the observers surmised that learning to operate the spectrum analyzer for dust wipes would be easily accomplished. The majority of the participants (n=9) stated they would consider purchasing or using this spectrum analyzer based on their observations and felt a new user could be trained in 2 to 4 hours.

- The spectrum analyzer contains a radioactive source. Licensing requirements vary by state and NITON offers contact information and guidance with spectrum analyzer purchase or lease.
- It is recommended that NITON be contacted with any specific questions (such as transportation issues) that a user might have.

Summary of Performance

A summary of performance is presented in Table 12. Note that performance is based on the specific protocols employed for this verification test. If different testing protocols are used, different performance results may be obtained.

The verification test found that the XL-700 spectrum analyzer was relatively simple for a trained analyst to operate in the field, requiring less than an hour for initial setup. The sample throughput of the XL-700 was forty samples per day with a single operator.

The overall performance of the XL-700 for the analysis of lead in dust wipe samples was characterized as biased high but within acceptable levels, very precise, and in good linear agreement with the NLLAP laboratory data.

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.

Table 12. Performance Summary for NITON’s XL-700 Series XRF Spectrum Analyzer

Feature/parameter		Performance summary			
		UC Samples		ELPAT Samples	
Precision : average RSD		8%		8%	
Accuracy: average % recovery		107%		119%	
Positive results on “detectable blank” samples (< 2 µg/wipe)		n/a		0 of 20 samples	
False positive results		<u>DataChem</u>	<u>XL-700</u>	<u>DataChem</u>	<u>XL-700</u>
		0 of 30	21 of 34	2 of 12	6 of 12
False negative results		<u>DataChem</u>	<u>XL-700</u>	<u>DataChem</u>	<u>XL-700</u>
		23 of 30	2 of 26	7 of 28	2 of 28
Comparison with NLLAP-recognized laboratory results (excluding < 25 µg/wipe samples)	slope	1.206		1.112	
	intercept	-3.290		13.283	
	correlation coefficient	0.999		0.999	
Overall evaluation		<ul style="list-style-type: none"> - Statistically significant positive bias but within the acceptable bias range - Very Precise - Strong linear relationship to the NLLAP lab results - Higher number of fp results - Few fn results 		<ul style="list-style-type: none"> - Statistically significant positive bias but within the acceptable bias range - Very Precise - Strong linear relationship to the NLLAP lab results - Higher number of fp results - Few fn results 	
Completeness		100% of 160 dust wipe samples			
Size and Weight		8.25" x 3" x 1.875"; 2.5 lbs			
Sample throughput (1 analyst)		Day #1 and 2: 25-30 samples/12-hr day Day# 3 and 4: 50-60 samples/12-hr day			
Power requirements		12 V rechargeable battery (nickel metal hydride)			
Training requirements		One (8-hr) day instrument-specific training			
Cost		Purchase: \$28,995 Lease: \$1,980 per week; \$5,190 per month Reagents/Supplies: \$0.10 per sample			
Waste generated		none			

Section 6 — References

- American Society for Testing and Materials. 1996. "Specification E1792-96a: Standard Specification for Wipe Sampling Materials for Lead in Surface Dust" in *ASTM Standards on Lead Hazards Associated with Buildings*. ASTM: West Conshohocken, PA.
- American Society for Testing and Materials. 1998. "Practice E1644: Standard Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead" in *ASTM Standards on Lead Hazards Associated with Buildings*. ASTM: West Conshohocken, PA.
- Ashley, K., K. Mapp, and M. Millson. 1998. "Ultrasonic Extraction and Field-Portable Anodic Stripping Voltammetry for the Determination of Lead in Workplace Air Samples." *AIHA Journal*. 59(10), 671-679.
- Code of Federal Regulations. 2001. "*Identification of Dangerous Levels of Lead*", Final Rule, 40 CFR Part 745, January.
- Draper, N. R., and H. Smith. 1981. *Applied Regression Analysis*. 2nd ed. John Wiley & Sons, New York.
- EPA (U.S. Environmental Protection Agency). 1996. "Method 3050B-1: Acid Digestion of Sediment, Sludge, and Soils." In *Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods, Update II*. SW-846. U.S. Environmental Protection Agency, Washington, D.C., December.
- EPA (U.S. Environmental Protection Agency). 1996. "Method 6010B-1: Inductively Coupled Plasma-Atomic Emission Spectrometry." In *Test Methods for Evaluating Solid Waste: Physical/ Chemical Methods, Update II*. SW-846. U.S. Environmental Protection Agency, Washington, D.C., December.
- Keith, L.H., G. L. Patton, D.L. Lewis and P.G. Edwards. 1996. *Chapter 1: Determining What Kinds of Samples and How Many Samples to Analyze*, pp. 19. In Principles of Environmental Sampling, Second Edition, Edited by L. H. Keith, ACS Professional Reference Book, American Chemical Society, Washington, DC.
- ORNL (Oak Ridge National Laboratory). 1998. *Quality Management Plan for the Environmental Technology Verification Program's Site Characterization and Monitoring Technologies Pilot*. QMP-X-98-CASD-001, Rev. 0. Oak Ridge National Laboratory, Oak Ridge, Tenn., November.
- ORNL (Oak Ridge National Laboratory). 2001. *Technology Verification Test Plan: Evaluation of Field Portable Measurement Technologies for Lead in Dust Wipes*. Chemical Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tenn., November.
- Song, R., P. Schlecht, and K. Ashley. 2001. "Field Screening Test Methods: performance criteria and performance characteristics." *Journal of Hazardous Materials*. 83, 29-39.

Appendix

Niton's XL-700 Series XRF Results Compared with Laboratory Results

Sample Analysis Order	Source	Rep	NITON XL-700 Series		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
94	ELPAT	1	<5.7	1.3	<20	1.3
51	ELPAT	2	<5.5	1.3	<20	1.3
134	ELPAT	3	<5.8	1.3	<20	1.3
102	ELPAT	4	<5.5	1.3	<20	1.3
60	ELPAT	1	<5.7	1.3	<20	1.3
84	ELPAT	2	<6.1	1.3	<20	1.3
28	ELPAT	3	<5.0	1.3	<20	1.3
114	ELPAT	4	<5.8	1.3	<20	1.3
109	ELPAT	1	<6.0	1.3	<20	1.3
78	ELPAT	2	<6.0	1.3	<20	1.3
67	ELPAT	3	<5.7	1.3	<20	1.3
4	ELPAT	4	<5.5	1.3	<20	1.3
6	ELPAT	1	<4.9	1.3	<20	1.3
105	ELPAT	2	<6.0	1.3	<20	1.3
155	ELPAT	3	<5.8	1.3	<20	1.3
118	ELPAT	4	<5.7	1.3	<20	1.3
106	ELPAT	1	<6.0	1.3	<20	1.3
80	ELPAT	2	<6.1	1.3	<20	1.3
36	ELPAT	3	<6.0	1.3	<20	1.3
112	ELPAT	4	<6.1	1.3	<20	1.3
99	ELPAT	1	25.7	16.9	<20	16.9
76	ELPAT	2	22.9	16.9	<20	16.9
147	ELPAT	3	23.9	16.9	<20	16.9
19	ELPAT	4	16.7	16.9	<20	16.9
39	ELPAT	1	20.4	17.6	30	17.6
66	ELPAT	2	22.3	17.6	<20	17.6
101	ELPAT	3	27.1	17.6	<20	17.6
71	ELPAT	4	25.1	17.6	<20	17.6
5	ELPAT	1	34.5	29.8	33	29.8
149	ELPAT	2	33.2	29.8	26	29.8
7	ELPAT	3	32.6	29.8	28	29.8
83	ELPAT	4	34.3	29.8	28	29.8

Sample Analysis Order	Source	Rep	NITON XL-700 Series		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
25	UC LAB	1	46.6	42.1	33	35.4
55	UC LAB	2	42.3	39.5	32	35.7
98	UC LAB	3	42.3	41.1	31	38.5
96	UC LAB	4	37.5	39.0	29	36.4
62	UC LAB	1	43.4	40.3	32	35.1
141	UC LAB	2	36.0	37.5	38	40.7
52	UC LAB	3	39.1	37.8	37	39.4
107	UC LAB	4	41.3	40.0	36	41.0
88	UC LAB	1	45.2	37.2	37	41.0
47	UC LAB	2	41.1	39.2	37	38.8
10	UC LAB	3	37.9	38.0	33	39.3
104	UC LAB	4	35.1	35.2	41	44.7
143	UC LAB	1	39.5	40.8	32	36.0
136	UC LAB	2	41.2	41.0	38	44.7
38	UC LAB	3	49.4	45.9	30	39.9
128	UC LAB	4	38.3	36.1	35	37.5
122	UC LAB	1	42.5	40.9	36	37.4
8	UC LAB	2	35.4	37.3	31	36.7
93	UC LAB	3	41.4	38.6	34	35.8
20	UC LAB	4	36.2	38.3	34	39.7
138	ELPAT	1	44.2	41.3	37	41.3
33	ELPAT	2	50.1	41.3	42	41.3
87	ELPAT	3	55.7	41.3	44	41.3
23	ELPAT	4	56.3	41.3	41	41.3
131	ELPAT	1	60.2	49.0	43	49.0
150	ELPAT	2	75.3	49.0	52	49.0
160	ELPAT	3	57.7	49.0	49	49.0
130	ELPAT	4	67.3	49.0	48	49.0
73	ELPAT	1	75.9	49.1	70	49.1
117	ELPAT	2	56.6	49.1	54	49.1
75	ELPAT	3	65.7	49.1	48	49.1
120	ELPAT	4	47.5	49.1	44	49.1
154	ELPAT	1	68.7	58.6	64	58.6
15	ELPAT	2	66.8	58.6	55	58.6
14	ELPAT	3	81.1	58.6	56	58.6
37	ELPAT	4	69.5	58.6	52	58.6

Sample Analysis Order	Source	Rep	NITON XL-700 Series		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
54	ELPAT	1	102	88.0	82	88.0
11	ELPAT	2	89.7	88.0	83	88.0
125	ELPAT	3	93	88.0	79	88.0
48	ELPAT	4	111	88.0	100	88.0
35	ELPAT	1	141	117.0	120	117.0
44	ELPAT	2	148	117.0	120	117.0
100	ELPAT	3	165	117.0	120	117.0
82	ELPAT	4	149	117.0	110	117.0
115	ELPAT	1	178	162.3	150	162.3
77	ELPAT	2	193	162.3	160	162.3
92	ELPAT	3	191	162.3	150	162.3
127	ELPAT	4	197	162.3	160	162.3
59	ELPAT	1	261	201.6	200	201.6
145	ELPAT	2	227	201.6	190	201.6
40	ELPAT	3	251	201.6	200	201.6
53	ELPAT	4	227	201.6	220	201.6
156	ELPAT	1	273	239.0	230	239.0
151	ELPAT	2	254	239.0	250	239.0
72	ELPAT	3	270	239.0	250	239.0
79	ELPAT	4	294	239.0	230	239.0
26	UC LAB	1	289	263.3	210	244.0
30	UC LAB	2	278	236.8	250	274.4
17	UC LAB	3	272	252.3	230	252.8
3	UC LAB	4	255	230.1	230	258.9
57	UC LAB	1	263	244.5	200	241.7
31	UC LAB	2	261	226.8	240	274.9
126	UC LAB	3	287	241.2	210	244.5
81	UC LAB	4	275	241.7	210	236.2
32	UC LAB	1	275	230.1	220	244.0
140	UC LAB	2	255	244.0	220	242.3
41	UC LAB	3	315	267.7	230	260.0
146	UC LAB	4	281	254.5	170	228.5
86	UC LAB	1	304	269.4	190	242.3
65	UC LAB	2	247	240.6	210	267.2
29	UC LAB	3	243	231.8	210	236.2
121	UC LAB	4	223	250.0	250	275.5
144	UC LAB	1	261	236.2	220	262.2
152	UC LAB	2	303	252.8	210	226.3
58	UC LAB	3	265	251.2	210	227.4
111	UC LAB	4	265	234.6	220	243.4

Sample Analysis Order	Source	Rep	NITON XL-700 Series		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
22	ELPAT	1	298	256.7	290	256.7
9	ELPAT	2	245	256.7	240	256.7
43	ELPAT	3	262	256.7	230	256.7
16	ELPAT	4	294	256.7	250	256.7
46	ELPAT	1	277	260.8	220	260.8
2	ELPAT	2	248	260.8	250	260.8
116	ELPAT	3	280	260.8	210	260.8
90	ELPAT	4	299	260.8	210	260.8
27	UC LAB	1	467	402.7	320	377.8
142	UC LAB	2	389	382.8	360	395.0
63	UC LAB	3	472	398.9	350	399.4
133	UC LAB	4	431	395.5	340	385.0
69	UC LAB	1	447	427.6	350	395.5
50	UC LAB	2	412	382.3	340	382.8
113	UC LAB	3	425	402.2	370	413.8
85	UC LAB	4	451	392.2	340	374.0
68	UC LAB	1	390	380.6	370	426.5
21	UC LAB	2	474	433.7	340	378.9
119	UC LAB	3	407	402.2	370	401.1
110	UC LAB	4	424	405.5	390	423.2
123	UC LAB	1	414	407.2	330	372.9
64	UC LAB	2	473	408.8	320	362.9
108	UC LAB	3	409	385.6	330	384.5
132	UC LAB	4	423	386.7	360	411.0
139	UC LAB	1	439	409.4	340	397.2
124	UC LAB	2	385	369.5	360	393.3
74	UC LAB	3	431	375.1	390	437.6
137	UC LAB	4	417	411.6	330	375.1
61	ELPAT	1	450	408.7	360	408.7
49	ELPAT	2	449	408.7	430	408.7
159	ELPAT	3	446	408.7	410	408.7
103	ELPAT	4	435	408.7	410	408.7
70	ELPAT	1	541	418.1	440	418.1
148	ELPAT	2	513	418.1	410	418.1
13	ELPAT	3	510	418.1	430	418.1
42	ELPAT	4	533	418.1	420	418.1

Sample Analysis Order	Source	Rep	NITON XL-700 Series		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
91	ELPAT	1	629	561.9	580	561.9
135	ELPAT	2	665	561.9	540	561.9
1	ELPAT	3	690	561.9	560	561.9
56	ELPAT	4	565	561.9	540	561.9
24	ELPAT	1	709	564.7	560	564.7
157	ELPAT	2	587	564.7	560	564.7
18	ELPAT	3	654	564.7	570	564.7
158	ELPAT	4	669	564.7	530	564.7
12	ELPAT	1	803	805.1	760	805.1
129	ELPAT	2	855	805.1	770	805.1
97	ELPAT	3	925	805.1	760	805.1
45	ELPAT	4	906	805.1	740	805.1
89	ELPAT	1	1645	1482.6	1500	1482.6
95	ELPAT	2	1625	1482.6	1500	1482.6
34	ELPAT	3	1620	1482.6	1500	1482.6
153	ELPAT	4	1630	1482.6	1400	1482.6