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September 2003

Environmental Technology Verification Report

Lead in Dust Wipe Measurement Technology

NITON LLC
X-Ray Fluorescence Spectrum Analyzer, XLt 700 Series

Oak Ridge National Laboratory

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

ETV✓

U.S. Environmental Protection Agency

Oak Ridge National Laboratory

Verification Statement

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

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

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

The accompanying notice is an integral part of this verification statement.

VERIFICATION TEST DESCRIPTION

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

TECHNOLOGY DESCRIPTION

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

The XLt 700 analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses a low power miniature x-ray tube with a silver transmission target to excite characteristic x-rays of a test sample's constituent elements. These characteristic x-rays are continuously detected, identified, and quantified by the spectrometer during sample analysis. The energy of each x-ray detected identifies a particular element present in the sample. The rate at which x-rays of a given energy are counted provides a determination of the quantity of that element that is present in the sample. Detection of the characteristic lead x-rays is achieved using a highly-efficient, thermo-electrically cooled, solid-state detector. Signals from the detector are amplified, digitized, and then quantified via integral multichannel analysis and data processing units. Sample test results are displayed in total micrograms of lead per dust-wipe. NITON's XLt 700 Series XRF spectrum analyzer reporting limit was 10 µg/wipe during the verification test.

VERIFICATION OF PERFORMANCE

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

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

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

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

Detectable blanks: All twenty blank samples, prepared at concentrations < 2 µg/wipe, were reported correctly as less than reporting limits, with results reported by the XLt 700 as less than 10 µg/wipe.

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

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

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

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

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

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NOTICE: ETV 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.

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X-Ray Fluorescence Spectrum Analyzer, XLt 700 Series

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Notice

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Acknowledgments

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Abbreviations and Acronyms

AIHA	American Industrial Hygiene Association
AMT	Advanced Monitoring Technology Center, ETV
ASTM	American Society for Testing and Materials
CDC	Centers for Disease Control and Prevention
CL	Clearance level for lead of 40, 250, or 400 µg/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
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
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.

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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 North Chelmsford, MA, from January 6 through January 9, 2003. The performance of NITON's XLt 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 performances of NITON's XL-700 and XL-300 Series XRFs have been reported in separate verification reports.

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

In Figure 1 is portrayed the XLt 700 Series analyzer and supporting notebook computer. The sample analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses a low power miniature x-ray tube with a silver transmission window to excite characteristic x-rays of a test sample's constituent elements. These characteristic x-rays are continuously detected, identified, and quantified by the spectrometer during sample analysis. The energy of each x-ray detected identifies a particular element present in the sample. The rate at which x-rays of a given energy are counted provides a determination of the quantity of that element that is present in the sample.

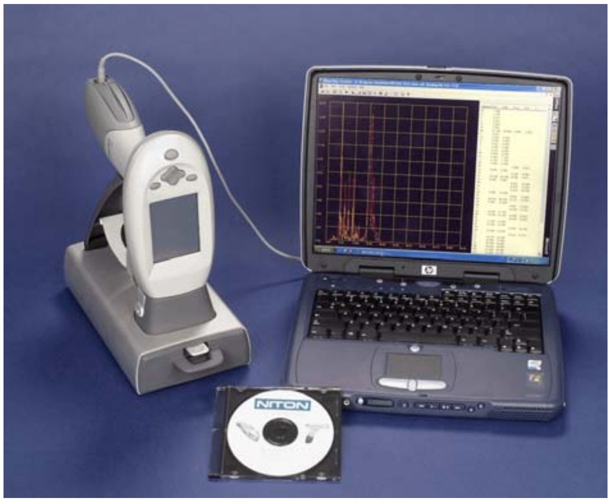


Figure 1. NITON XLt 700 series XRF spectrum analyser with optional notebook computer.

Note that the XLt 700 series instrument employs an x-ray tube source for x-ray generation, rather than a radioactive isotopic source. Each one of these approaches has its strengths and limitations. For example, tube-based x-ray sources offer a) potentially faster analysis (x-ray flux can be higher than most isotope based sources), b) can be used over a wider range of excitation energies, eliminating the need for multiple isotopic sources to produce x-rays over the entire excitation spectrum, c) no drop off in excitation power with the age of the tube, and d) fewer requirements for transporting sources outside the State in which the source is licensed. However, tube based sources are somewhat less rugged than isotopic sources, and tend to be slightly larger and heavier. In addition, they typically require more external or battery power. Most isotopic x-ray sources tend to have a diminishing flux output over their lifetime, which requires longer counting times for analysis.

Detection of the characteristic lead x-rays is achieved using a highly-efficient, thermo-electrically cooled, solid-state detector. Signals from the detector are amplified, digitized, and then quantified via integral multichannel analysis and data processing units. Sample test results are displayed in total micrograms of lead per dust wipe.

Sample Preparation

The following is the procedure that was used to prepare the samples for analysis during the verification test.

The dust wipe samples were unfolded and the dust samples were distributed across the surface of the wipe using a spatula or equivalent tool. The tool was cleaned in between each sample preparation by wiping with a damp paper towel. Next, the sample was folded five times, as specified in the schematic in Figure 2, such that it was neatly folded to the proper size (1 x 1.5 inches).

The sample was dried prior to testing, because the addition of this step has been found to improve the accuracy and precision of dust wipe measurements. Typically, moisture will reduce the readings on the instrument, i.e., elements will read lower than the actual value. In addition, the presence of water has the effect of scattering more of the x-rays (Compton scatter). The detection limit will become slightly worse as the background increases. As the moisture content varies from wipe to wipe, it is not possible to assume that the "offset" is the same for all samples, so drying provides a practical alternative. In this case, the sample was dried for 20 minutes at 250° F. in a toaster oven, and then allowed to cool at ambient conditions for 5 minutes prior to measurement. (The vendor states that, if accelerated drying is not practical, allowing the re-folded wipe to stand at ambient temperature and relative humidity overnight should result in an adequately dried wipe.)

After drying, the wipe sample was placed in a 1.5 x 2 inch, 1.5 mil thick plastic bag (NITON part number 187-471 or equivalent) and labeled. A fresh bag was used for each sample to eliminate the potential for cross-contamination of samples. Finally, 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).

Calibration

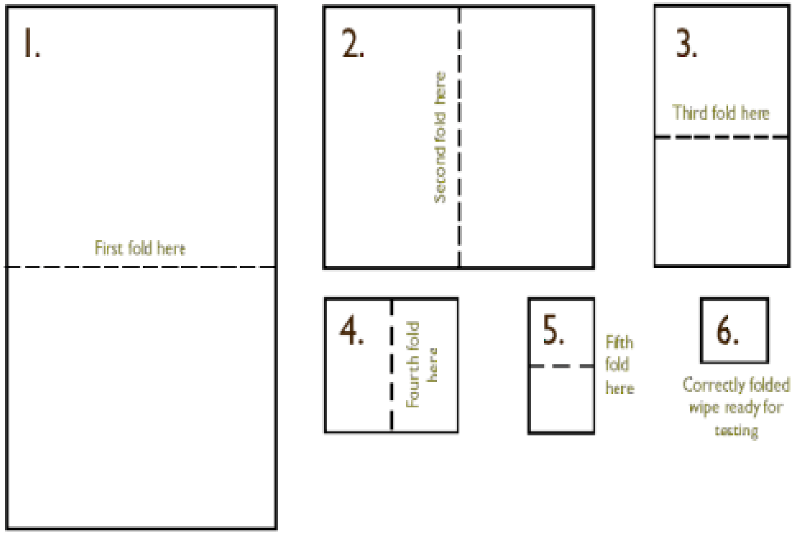
The instrument is factory calibrated. For the verification test, the instrument performance was verified at the start and end of each day's analyses by analyzing samples of known quantity.

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. However, when it was observed that there was virtually no response to the wipe for lead, the vendor assumed that the test sample was a blank, and truncated the number of measurements at four.

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 60s 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.

The x-ray tube in the XLt 700 spectrum analyzer was approximately 3 months old. However, decay and half-life time corrections are not relevant factors for an x-ray tubes. Time to complete each individual measurement was 60 seconds.



Dust wipe folding. Start at top left, and proceed as shown, making 5 folds.

Figure 2. Schematic diagram of recommended folding procedure for dust wipes. (Not to scale.)

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 EPA/Region 1 Laboratories in North Chelmsford, Massachusetts. The test was conducted in one particular laboratory. 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 71 °F and 40%, 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 $\mu g/ft^2$ for floors, 250 $\mu g/ft^2$ for window sills, and 400 $\mu g/ft^2$ 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, and (3) the logistical and economic resources necessary to operate the technology. Secondary objectives for this verification are 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 samples analyzed in this verification test were archived from the original verification test in 2001. In August, 2002, a suite of 16 samples from these lots were shipped to the reference laboratory, DataChem (Cincinnati, OH), and analyzed, in order to confirm that the lead concentrations on the sample wipes had not changed. Based on the values reported by DataChem, there was no evidence to suggest that there had been any demonstrable change in the lead levels on the tested sample wipes. For this particular test, the samples prepared in the fall of 2001 had been stored at -22° C since receipt.

All of the samples used in this verification test were prepared gravimetrically. The wipes utilized in the test (PaceWipe™ and Aramsco Lead Wipe™) met the specifications of the American Society for Testing and Materials requirements for lead testing (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, therefore 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 are 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. The weighing paper containing the pre-weighed dust was then removed from the balance, and 0.1000 ± 0.0005 g portion of 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 off-site laboratory must yield results within ± 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 µg/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 µg/wipe samples, and NIST SRM 2710 was used to prepare the 250 and 400 µg/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 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., ± 20% of the target value) was 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) 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 x 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 an by 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 - see EPA 1996). The average percent recovery (EPA Region 1 reported concentration/UC estimated concentration x 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 ± 10%. Based on this 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

Figure 3 portrays the distribution of the sample concentrations that were analyzed in this study. A total of 160 samples were analyzed in the verification test. For the ELPAT samples, four samples were analyzed at each of 20 test levels (20 test levels x 4 samples each = 80 samples total). While the set of four samples were prepared using homogeneous source materials and an identical preparation procedure, they cannot be considered true "replicates" because each sample was prepared individually. However, these samples represent four samples prepared in the same batch of ELPAT samples. Twenty samples were prepared near each of the three clearance levels (3 test levels x 20 samples = 60 samples total) by UC, and RTI prepared 20 "blanks" at lead concentrations < 2 µg/wipe. In Figure 3, the clearance levels are denoted as horizontal lines.

Sample Randomization

The samples were packaged in 20-mL plastic scintillation vials and labeled with a sample identifier. The participant received the same suite of samples as used in previous tests, 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 (RSD) for replicate results are used to assess precision, using the following equation:

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

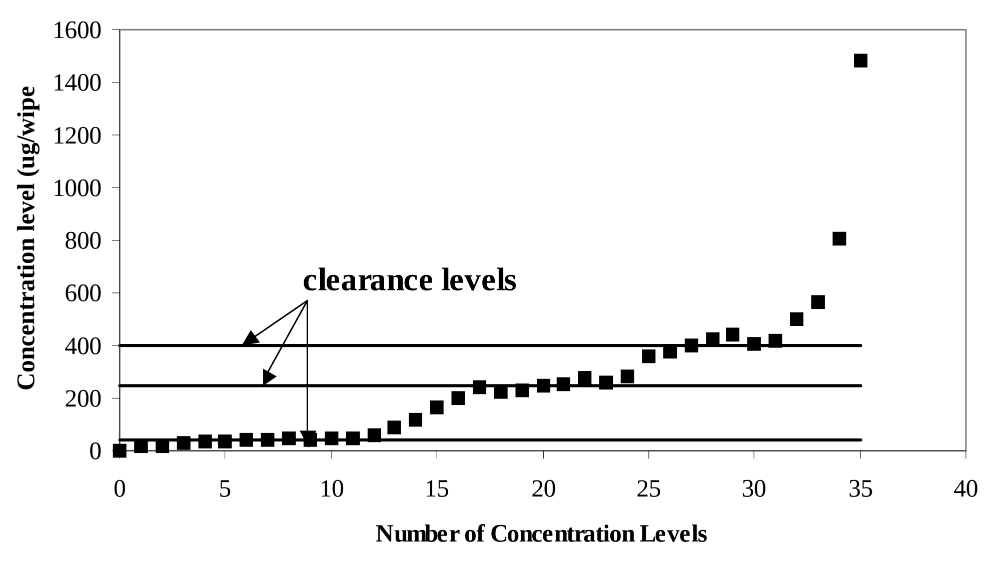


Figure 3. Distribution of both UC and ELPAT sample concentrations. Four replicates were analyzed at each concentration level.

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 certificate 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 ± 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:

percent recovery = [measured amount(s)/estimated value] × 100% (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. Such would reveal 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). Bias within the acceptable range can usually be corrected to 100% by modification of calibration methods. But the field technology can still have acceptable bias with an average percent recovery in the interval of 75% to 125%.

Comparability

Comparability refers to how well the field technology and the NLLAP-recognized 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 was 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). 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 µg/wipe, below the anticipated reporting limits of both the field technology 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 (Keith et al. 1996). For example, if the technology reports the sample concentration to be 35 µg/wipe, and the true concentration of the sample is 45 µg/wipe, the technology's result would be considered a fn. Accordingly, if the technology reports the result as 45 µg/wipe and the true concentration is 35 µg/wipe, the technology's result would be a fp.

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 µg/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 ± 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 (Recall that 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 ± 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. This 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 approximately130 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 Table1 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 (µg/wipe)	Estimated Conc (µg/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. A line fitted to the data using both data sets is shown graphically in Figure 4. The average percent recovery for the ELPAT samples was 98%, while the average for the UC samples was 91%. Both Table 2 and Figure 4 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. A regression analysis of the individual data sets shows that both the negative bias for the UC and the slightly negative bias for the ELPAT samples were statistically significant (i.e. slope not equal to 1.0). 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 µg/wipe), DataChem reported 71 (99%) within the acceptable ranges of values. (see Appendix).

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 µg/wipe. In addition, DataChem reported seven of the eight samples with estimated concentrations of either 16.9 µg/wipe or 17.6 µg/wipe as less than their detection limit and only one was incorrectly reported as 30 µg/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)

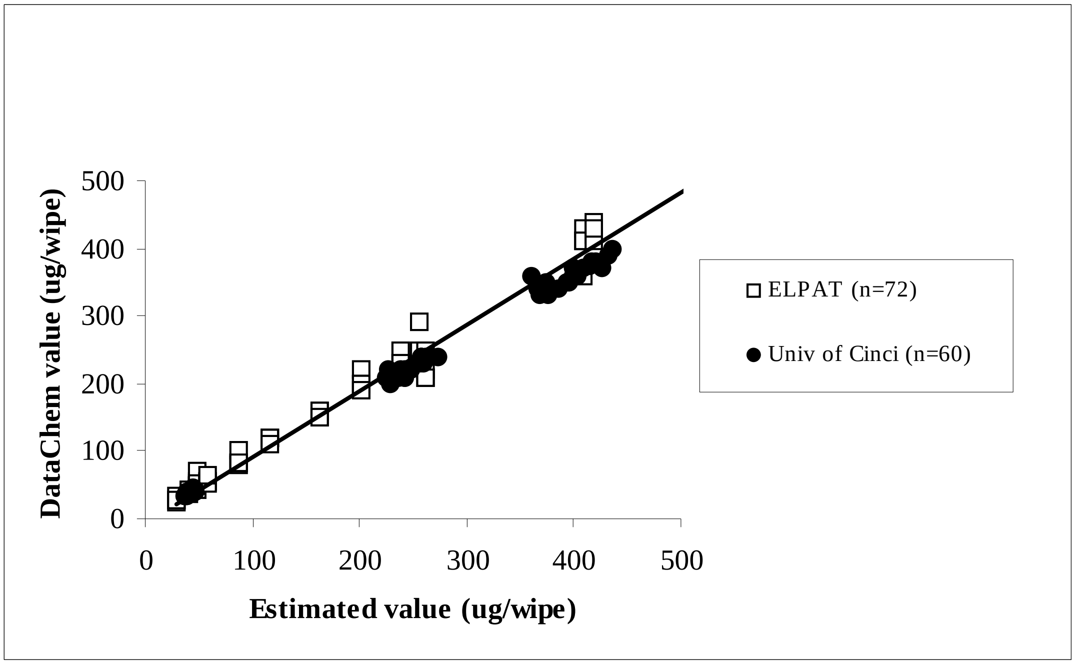


Figure 4. Plot of DataChem reported values versus estimated values, shown for concentrations less than 500 µg/wipe. This figure indicates that UC-prepared wipes were generally reported lower than ELPAT samples.

Table 3. Summary of DataChem Precision Estimates by Sample Source

Sample Source	n	average RSD	Min RSD	Max RSD
ELPAT	0.25	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 ± 10% of each clearance level of 40, 250, and 400 µg/wipe, for a total of 60 UC samples. The ELPAT samples covered a wider range of concentrations. A total of 40 ELPAT samples fell within a ±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. As shown in Table 4, 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 5, 6, and 7 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 are 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 µg/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 5, 6, and 7, 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 5, 5% FN probability (y = 0.05) corresponds to a "true" lead concentration of 46 µg/wipe (meaning if the true concentration of the sample is 46 µg/wipe, there is only a 5% chance/risk that DataChem will report the value as < 40 µg/wipe.)

When the DataChem measured values versus the estimated concentrations for each of the three CL, the equation of the linear regression lines can be calculated. 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 < CL). For example, for the UC samples, we know that a value reported by DataChem as 39 µg/wipe is biased low and will have a true concentration of > 40 (41.8 µg/wipe, using the linear regression equation in Table 5). A true concentration of 40 µg/wipe for a UC sample would correspond to a reported value rounded to the nearest whole number of 37 µg/wipe (see Table 5). For an ELPAT sample, a true concentration of 40 µg/wipe corresponds to a DataChem reported value of 40 µg/wipe, because the negative bias was so small for the ELPAT samples. Estimates of the reported concentration at the 250 and 400 µg/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 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 [a]	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] Because all eight ELPAT values were above 400 µg/wipe, no samples were available to assess fp results at this level.

CL = clearance level

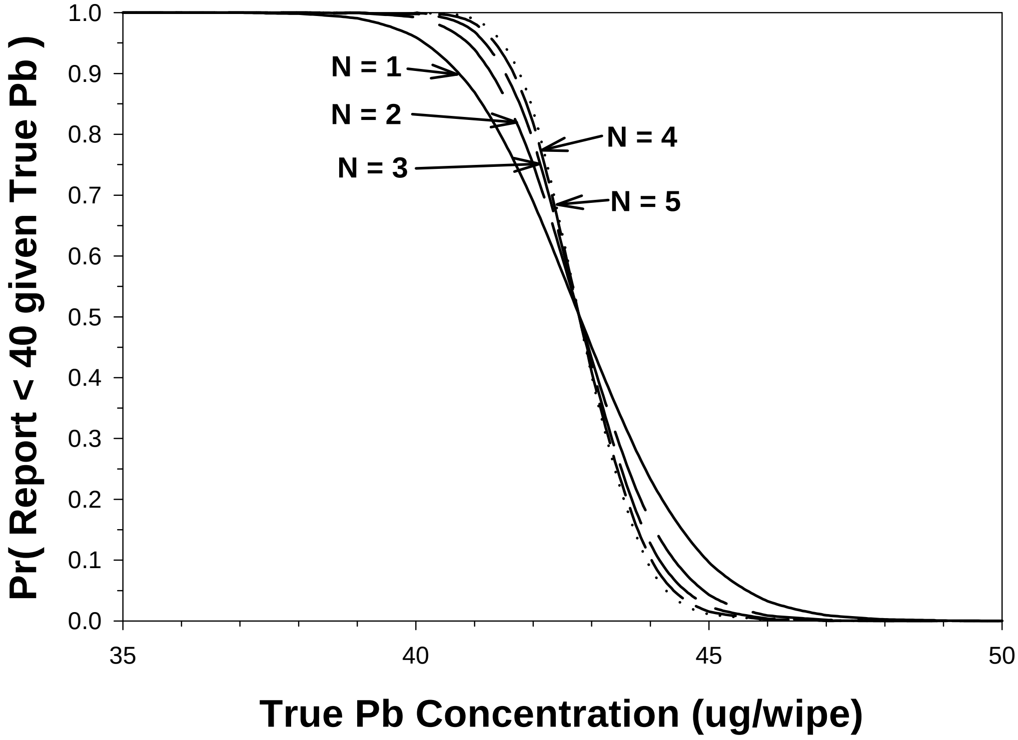


Figure 5. False negative probabilities for DataChem average concentrations at a target concentration level of 40 µg/wipe.

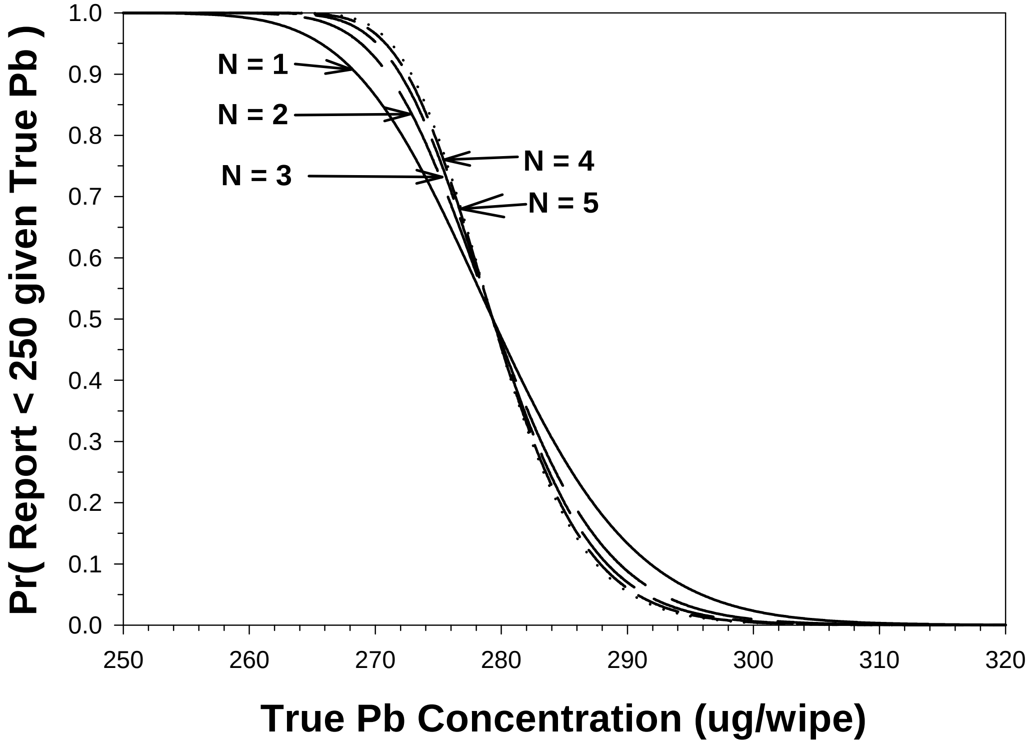


Figure 6. False negative probabilities for DataChem average concentrations at a target concentration level of 250 µg/wipe.

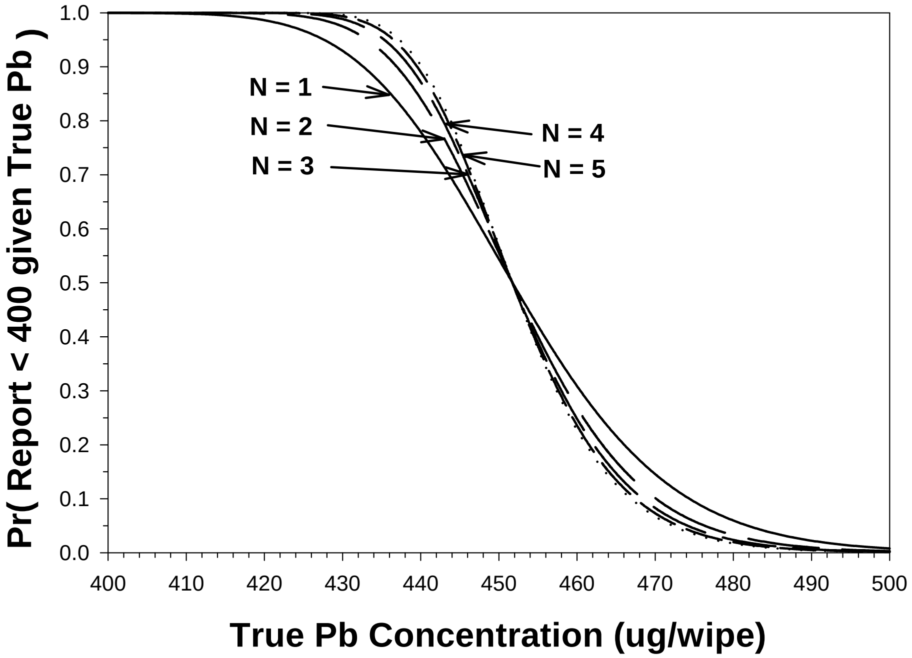


Figure 7. False negative probabilities for DataChem average concentrations at a target concentration level of 400 µg/wipe.

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	0.84	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 XLt 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 XLt 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 XLt 700 reporting limit (10 µg/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 results in Table 6 demonstrate that the XLt 700's average RSD value was 11%, with a range from 2 to 26%, indicating that the XLt 700 measurements were precise. For the UC samples, 20 samples were analyzed at three target concentration levels of 40, 250, and 400 µg/wipe. With the expectation that UC was to prepare the samples as close to the target concentrations as possible, the allowable laboratory variability was 10% RSD. As presented in Table 6, the actual laboratory variability of the UC preparation process was an average of 5% RSD. The average precision of the UC sample measurements by the XLt 700 was 11% RSD.

Accuracy

Accuracy represents the closeness of the XLt 700's measured concentrations to the estimated content of spiked samples. A practical measure of accuracy is the number of results for the ELPAT samples that were reported within the acceptance ranges that have been established for those samples. Of the 80 ELPAT samples, the XLt 700 reported 71 of the results (89% of the total) within the acceptance ranges. For those values outside of the acceptance range, 2 values were below the lower limit and 7 values were above the upper limit.

Table 6. Precision of the XLt 700

Source	No. of sample sets	RSD, %		
		Average	Min	Max
ELPAT	0.33333	11	2	26
UC	3 [b]	11	10	12
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 XLt 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. These results are presented in Table 7.

Table 7. Accuracy of XLt 700

Statistic	% Recovery	
	ELPAT	UC
n [a]	80	60
average	101	97
standard deviation	14	12
median	99	97
minimum	77	74
maximum	151	145

[a] Excludes estimated values < 10 µg/wipe

The results for the 80 ELPAT samples reported by the XLt 700 had an average percent recovery value of 101%, the range of values was from 77 to 151%, and the median value was 99%. The UC sample results were slightly lower, with an average percent recovery of 97%, and a range of values from 74% to 145%. Both average recovery values were within the range of acceptable bias (100% ± 25%).

Another way to assess accuracy is to perform a regression analysis for the results obtained with the XLt 700 versus the estimated values that are > 10 µg/wipe (without constraining the intercept of the regression to pass through zero). The linear regression constants for the line fitted to the ELPAT and the UC data are listed in Table 8. That the slopes are statistically (at the 5% significance level) less than 1.00, indicates that the results are biased slightly low. The fact that this small bias is statistically significant indicates that the precision on the slope measurement is exceptional and thus small differences from the ideal slope of 1.00 can be detected. While the performance met the criteria stated for accuracy in Section 4 (unbiased if the average percent recovery is between 75 and 125%), the XLt 700 result generally were reported significantly less than the estimated value.

Comparability

Comparability refers to how well the XLt 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 XLt 700 results and the laboratory results was performed for all ELPAT and UC samples that were reported above 20 µg/wipe. (Note: Data were reported to 10 µg/wipe for the XLt 700, but the lab only reported to 20 µg/wipe.) Because each wipe was prepared individually, a true one-to-one matching of XLt 700 and laboratory results can not be performed. However, the average concentrations of the samples prepared at specific levels can be compared for the XLt 700 and laboratory results. In Table 8, the regression constants for the average XLt 700 results versus the average DataChem results for both the ELPAT and UC values are presented. The differences between the regression slopes (m = 0.977 for ELPAT and m = 0.995 for UC) and a slope with a perfect agreement line (m = 1.000) are <u>not</u> statistically significant, and the correlation coefficients (r = 0.999 for both ELPAT and UC) show a strong linear relationship between DataChem and XLt 700 results. To illustrate the strong linear agreement between the XLt 700 and NLLAP laboratory results, Figure 8 is a plot of the average XLt 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 XLt 700 correctly reported all 20 as less than the reporting limits. The reporting limit was < 10 µg/wipe, so no detectable blanks were reported.

Table 8. Linear regression constants for the plots of the XLt 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)	0.909 (0.019)	0.963 (0.012)	0.995 (0.010)	0.977 (0.011)
intercept (standard error)	5.817 (5.111)	1.434 (5.516)	4.775 (2.478)	3.076 (5.000)
r	0.988	0.994	0.999	0.999

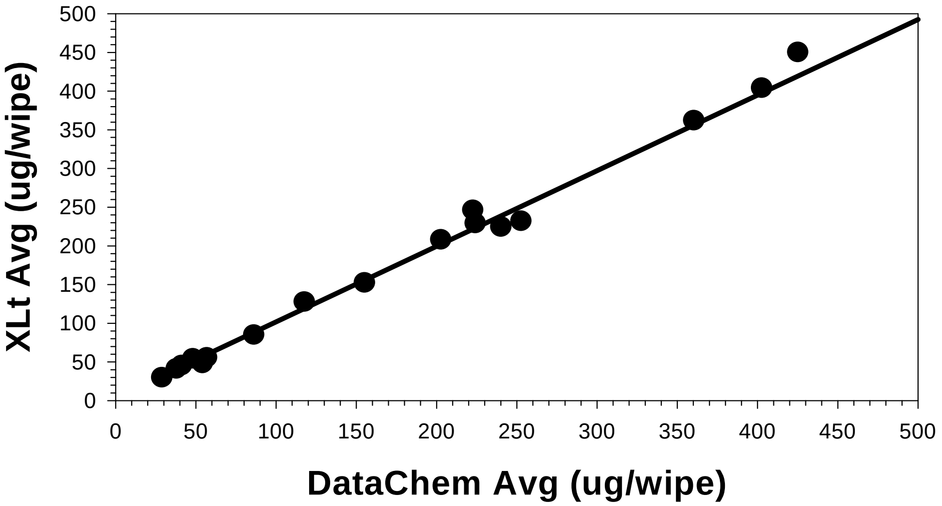


Figure 8. Plot of the average XLt 700 concentration versus the average DataChem concentration for all samples (n=21), shown for ELPAT and UC concentrations less than 500 µg/wipe.

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 XLt 700 relative to the estimated concentrations for both the ELPAT and UC samples are summarized in Table 9. For many cases where the estimated concentration was equal to or greater than the clearance level (CL), the XLt 700 reported a result that was less than the CL (10 of 23 possible fn results for the UC samples, and 8 of 28 fn results for the ELPAT samples at the three CLs). When the estimated concentration was less than the clearance level, however, the XLt 700 reported a lower percentage as greater than the CL (8 of 37 possible fp results for UC samples and 1 of 12 fp results for ELPAT samples). These findings are not surprising, since the accuracy results reported above indicated that the XLt 700 results were slightly negative biased, or reported less than the estimated values.

In Figures 9, 10, and 11, the false negative probabilities at the three clearance levels are compared for the DataChem and XLt 700 results. Ideally, a symmetric distribution of results about the clearance level would give a fn probability of 0.5. The XLt 700 results are closer to this ideal for all clearance levels but have a larger spread over the true concentration range. In these figures, the two-sided 90% confidence intervals (not shown for clarity) are used to expressed uncertainty on the false negative curves. These confidence intervals overlap for the XLt 700 and DataChem for the 40 µg/wipe clearance levels over the range of 38 to 55 µg/wipe shown in the Figure 9. The overlapping confidence intervals indicate the two methods are comparable when considering their uncertainty. In Figures 10 and 11, the 90% confidence intervals for the two methods overlap in the ranges of 243 to 355 µg/wipe and 379 to 500 µg/wipe for the 250 µg/wipe and 400 µg/wipe clearance levels, respectively. Again, the overlapping confidence intervals indicate the two methods are comparable when considering their uncertainty.

Table 10 describes the linear regression constants for XLt 700 measured concentration versus estimated concentration for the three CLs, average percent recovery values and standard deviations, and an estimate of the XLt 700 reported concentration at each clearance level. The average recoveries in Table 10 indicate that the XLt 700 results were negatively biased for the two highest clearance levels for both ELPAT and UC samples. This is also apparent in the estimated concentration that a user might require from the XLt 700 in order to be reasonably confident that the true result is below the clearance level. For samples with actual CL concentrations, the expected DataChem results would have to be 37, 226, and 362 µg/wipe, respectively, to provide confidence that sample concentrations with reported results at or above the CL concentrations are really greater than 40, 250, and 400 µg/wipe. The XLt 700 is also negatively biased for samples with the two highest CL concentrations with expected reporting results of 232, and 371 µg/wipe. Reported XLt 700 results at or greater than the two highest CL concentrations would likely indicate sample values greater than the respective CL. For the 40 µg/wipe CL, the expected XLt 700 result is 42 µg/wipe. Therefore, reported results below 40 µg/wipe (e.g., 36 to 39 µg/wipe) may indicate samples with concentrations at or greater than 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 XLt 700 Measurements

Evaluation Parameter	Sample Source	Number of Samples			Total
		40 µg/wipe	250 µg/wipe	400 µg/wipe	
fp: # samples where XLt 700 reported the result as > CL of the # samples where the estimated concentration was < CL	UC	6 of 12	2 of 14	0 of 11	8 of 37
	ELPAT	0 of 4	1 of 8	0 of 0 [a]	1 of 12
fn: # samples where XLt 700 reported the result as < CL of the # samples where the estimated concentration was > CL	UC	2 of 8	3 of 6	5 of 9	10 of 23
	ELPAT	1 of 12	5 of 8	2 of 8	8 of 28

[a] Because all eight ELPAT values were above 400 µg/wipe, no samples were available to assess fp results at this level.
CL = clearance level

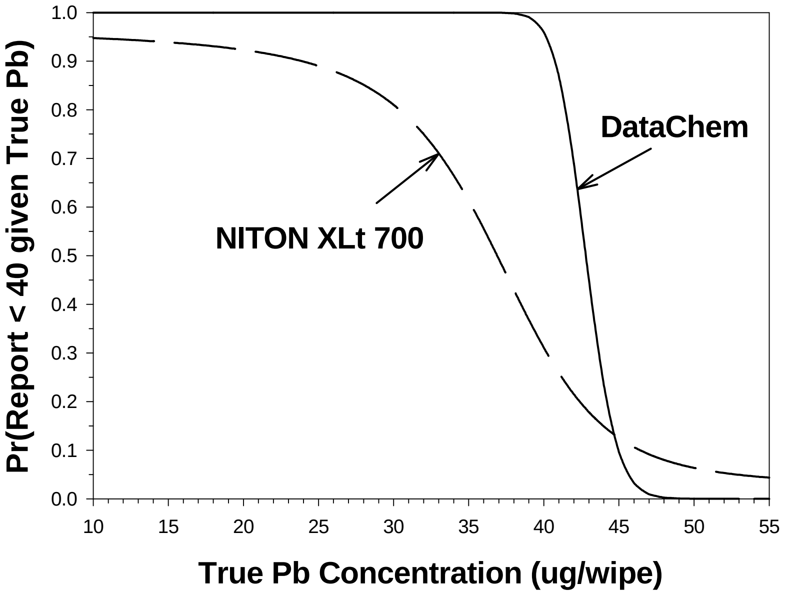


Figure 9. Comparison of false negative probabilities for the XLt 700 and DataChem average concentrations at a target concentration of 40 µg/ wipe.

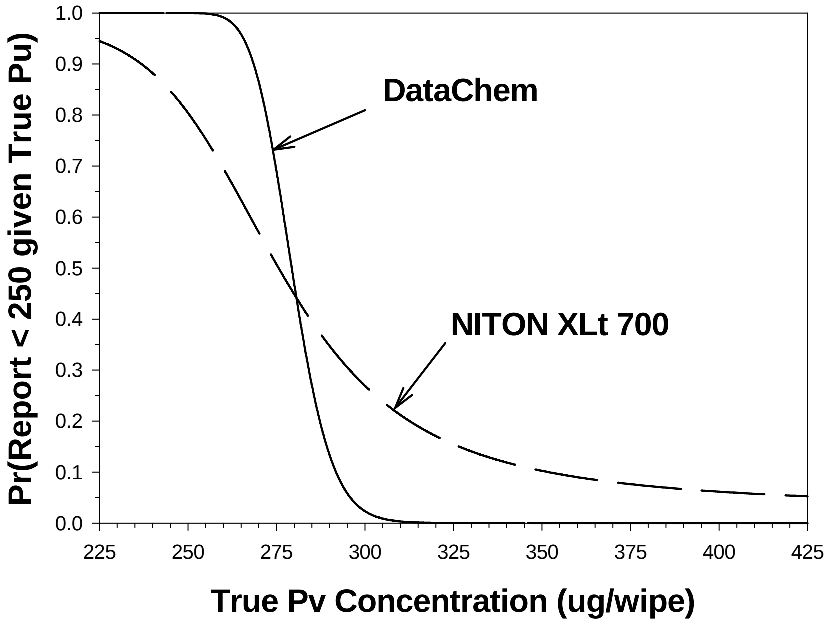


Figure 10. Comparison of the false negative probablilites for the XLt 700 and DataChem average concentrations at a target concentration of 250 μg/wipe.

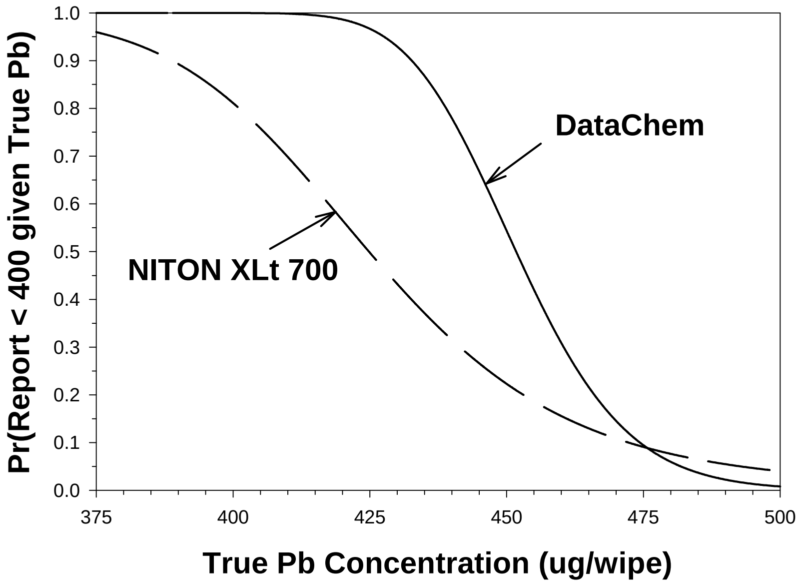


Figure 11. Comparison of the false negative probabilities for the XLt 700 and DataChem average concentrations at a target concentration level of 400 μg/wipe.

Table 10. Summary of the Linear Regression and Recovery Data for the XLt 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	0.76	1.1	0.705	0.557	1.157	4.92
intercept	11.964	-1.704	55.608	94.643	-91.326	-1606.641
correlation coefficient	0.406	0.81	0.434	0.567	0.616	0.834
average % recovery	107%	106%	93%	96%	92%	103%
SD of % recovery	12%	15%	8%	9%	85	6%
Reported concentration at the CL	42 µg/wipe	42 µg/wipe	232 µg/wipe	234 µg/wipe	371 µg/wipe	361 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, two analysts (NITON experts) analyzed all 160 samples in 32.75 hours over 3.5 calendar days. Eight measurements were taken for each sample wipe and four for each blank, for a total of 1,200 measurements. One analyst prepared the sample and the other analyst measured and recorded the data.

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 analysts who operated the instrument during the verification test were NITON experts.

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 XLt 700 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:	XLt 700 XRF	**Analysis method:**	EPA SW846 6010b
Analyst/manufacturer:	NITON	**NLLAP Laboratory:**	DataChem
Sample throughput:	45 - 50 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	$20,000 - $40,000		
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 ORNL 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.

XLt 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 and/or local 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 XLt 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.

- *X-ray tube Description* (provided by NITON): For the analysis of lead, the instrument is supplied with a low power miniature x-ray tube that utilizes a silver anode transmission window. A tube-excited instrument does not experience the loss of measurement speed over time that is common with most radioisotope-based instruments, so the measurement time is equal to the real time for the life of the x-ray tube. This means that the end user will always experience the same instrument performance in terms of precision and accuracy in the same amount of time. Miniature x-ray tubes have been in commercial use for only about a year at the time of this evaluation, so no data exists to confirm the life expectancy of them in a portable analyzer. The use of low power x-ray tubes in bench-top analyzers has shown a life expectancy of 3-5 years under less rugged conditions, but reliability data must be generated over time to make similar predictions for portable analyzers. Replacement of the x-ray tube and associated power supply is $5000, and includes the software updates, re-calibration, and replacement of the internal battery.

- *Instrument purchase* (provided by NITON): NITON instruments, with the capability of the XLt 700 series unit used in this evaluation (XLt 791), are available for dust wipe analysis in various configurations. Pricing ranges from approximately $20,000 to $40,000 with the differences between analyzers existing in both the range of elements and the matrices they are capable of analyzing. The spectrum analyzer can be calibrated for thin sample, bulk sample, or both. In the simplest version, the spectrum analyzer can quantify lead as well as Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se, Hg, Rb, Sr, Y, Zr, and Mo. It can also be upgraded for analysis of soil/bulk samples with the same element suite. The purchase price, irregardless of configuration, includes the following:
    - Shipping and handling
    - Test stand and holder for simple and convenient dust wipe measurement
    - Two rechargeable lithium-ion battery packs with 6-8 hour use (maximum depends on platform and duty cycle)
    - 110/220 VAC battery charger
    - Waterproof, crush-resistant carrying case with locks
    - Windows™ compatible NDT reporting software with an RS-232 cable for downloading data and spectra and remote control of instrument.

  Long 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 lease 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 various extended warranty options that can be discussed with NITON or your local NITON representative at the time of the 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.

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.

- *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 data report is limited and the cost is usually higher.) 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.

Cost Assessment Summary

An overall cost estimate for use of the XLt 700 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 XLt 700 XRF spectrum analyzer:

- The spectrum analyzer is provided with two rechargeable lithium-ion batteries. The batteries were swapped every 4 hours and recharging the battery took approximately 2 hours.
- 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. Fewer measurements may be sufficient for initial screening if time is an issue. However, such was not verified in this round of testing.
- 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.
- While the x-ray tube in the XLt 700 spectrum analyzer was approximately 3 months old, decay and half-life time corrections are not relevant factors for an x-ray tubes. Time to complete each measurement was 60 seconds.
- The NITON analyst ran QC samples at the start and periodically throughout the day to confirm that the spectrum analyzer was working within normal parameters. The samples were part of a suite provided by ORNL as part of the pre-test. They were the same samples that were used as test samples in the study and can be obtained directly from the American Industrial Hygiene Association (AIHA), for a price of $200 per group of four samples. More information can be obtained by contacting the ELPAT Program, Laboratory Accreditation Department, at AIHA; (703) 849-8888
- The dust in all samples was smoothed out to a thin layer on the wipe. All wipes were folded and dried (toaster oven at 250° F for 20 minutes) so that they fit the size of the sample holder and such that it was a flat as possible for the most accurate reading.
- The spectrum analyzer contains an x-ray tube. Though regulatory demands are reduced as compared to those encountered with isotope-based systems, x-ray source registration is still typically required and varies by state. NITON offers contact information and guidance with spectrum analyzer purchase or lease.

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 XLt 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 XLt 700 was 45 - 50 samples per day with two operators. The overall performance of the XLt 700 for the analysis of lead in dust wipe samples was characterized as slightly biased low but within acceptable levels, 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 samples where required) need to be followed.

Table 12. Performance Summary for NITON's XLt 700 XRF Spectrum Analyzer

Feature/parameter		**Performance summary**			
		UC Samples[a]		**ELPAT Samples**	
Precision : average RSD		11%		11%	
Accuracy: average % recovery		97%		101%	
Positive results on "detectable blank" samples (< 2 µg/wipe)		n/a		0 of 20 samples	
False positive results		DataChem 0 of 30 (0%)	XLt 700 8 of 37 (22%)	DataChem 2 of 12 (17%)	XLt 700 1 of 12 (8%)
False negative results		DataChem 23 of 30 (77%)	XLt 700 10 of 23 (43%)	DataChem 7 of 28 (25%)	XLt 700 8 of 28 (29%)
Comparison with NLLAP-recognized laboratory results (excluding < 25 µg/wipe samples)	slope	0.995		0.977	
	intercept	4.775		3.076	
	correlation coefficient	0.999		0.999	
Overall evaluation		- Statistically significant negative bias but within the acceptable bias range - Precise - Strong linear relationship to the NLLAP lab results - Higher number of fn results - Few fp results		- Statistically significant negative bias but within the acceptable bias range - Precise - Strong linear relationship to the NLLAP lab results - Higher number of fn results - Few fp results	
Completeness		100% of 160 dust wipe samples			
Size and Weight		9.75" x 10.5" x 3.75"; 3.0 lbs			
Sample throughput (2 analysts)		45 - 50 samples/day			
Power requirements		7 V rechargeable battery (Lithium-ion)			
Training requirements		One day instrument-specific training including radiation safety.			
Cost		Purchase: \$20,000 - \$40,000 Reagents/Supplies: \$0.10 per sample			
Waste generated		none			

(a) DataChem Laboratories and XLt 700 measured two different sets of UC samples. Both sets have 20 samples for each of the three target levels of 40, 250 and 400 µg/wipe with sample concentrations distributed above and below the target levels. The concentration distributions are different for the DataChem and Xlt 700 analyzed samples.

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 XLt 700 XRF Results Compared with Laboratory Results

Sample Analysis Order	Source	Rep	NITON XLt 700 XRF		DataChem	
			Result µg/wipe	Estimated µg/wipe	Result µg/wipe	Estimated µg/wipe
106	ELPAT	1.234e+31	<10.0	1.3	<20	1.3
138	ELPAT		<10.0	1.3	<20	1.3
101	ELPAT		<10.0	1.3	<20	1.3
25	ELPAT		<10.0	1.3	<20	1.3
129	ELPAT		<10.0	1.3	<20	1.3
100	ELPAT		<10.0	1.3	<20	1.3
137	ELPAT		<10.0	1.3	<20	1.3
84	ELPAT		<10.0	1.3	<20	1.3
72	ELPAT		<10.0	1.3	<20	1.3
111	ELPAT		<10.0	1.3	<20	1.3
91	ELPAT		<10.0	1.3	<20	1.3
121	ELPAT		<10.0	1.3	<20	1.3
32	ELPAT		<10.0	1.3	<20	1.3
102	ELPAT		<10.0	1.3	<20	1.3
125	ELPAT		<10.0	1.3	<20	1.3
132	ELPAT		<10.0	1.3	<20	1.3
55	ELPAT		<10.0	1.3	<20	1.3
54	ELPAT		<10.0	1.3	<20	1.3
35	ELPAT		<10.0	1.3	<20	1.3
150	ELPAT		<10.0	1.3	<20	1.3
110	ELPAT		21.3	16.9	<20	16.9
82	ELPAT		16.3	16.9	<20	16.9
145	ELPAT		24.3	16.9	<20	16.9
73	ELPAT		17.7	16.9	<20	16.9
124	ELPAT		14.6	17.6	30	17.6
8	ELPAT		26.5	17.6	<20	17.6
61	ELPAT		16.9	17.6	<20	17.6
40	ELPAT		20.3	17.6	<20	17.6
65	ELPAT		30.8	29.8	33	29.8
148	ELPAT		34.0	29.8	26	29.8
11	ELPAT		25.3	29.8	28	29.8
159	ELPAT		30.5	29.8	28	29.8

Sample Analysis Order	Source	Rep	NITON XLt 700 XRF		DataChem	
			Result µg/wipe	Estimated µg/wipe	Result µg/wipe	Estimated µg/wipe
156	UC LAB	1.234e+35	42.3	37.9	3.941e+71	40.4
107	UC LAB		44.0	42.2		44.3
47	UC LAB		38.7	36.8		43.1
45	UC LAB		39.8	40.6		38.3
87	UC LAB		38.4	41.4		37.8
58	UC LAB		37.1	36.4		38.7
51	UC LAB		41.5	37.9		38.9
86	UC LAB		32.7	36.8		38.9
67	UC LAB		37.1	35.2		42.3
13	UC LAB		50.8	35.1		36.4
60	UC LAB		48.4	39.6		36.8
135	UC LAB		33.4	35.4		41.4
139	UC LAB		35.3	37.6		38.2
6	UC LAB		44.0	41.3		45.8
41	UC LAB		47.7	39.9		45.7
77	UC LAB		43.2	41.6		41.1
109	UC LAB		44.2	41.6		41.1
152	UC LAB		45.4	40.4		44.3
154	UC LAB		42.2	37.2		40.2
10	UC LAB		44.9	44.0		38.1
2	ELPAT		33.1	41.3		41.3
14	ELPAT		53.7	41.3		41.3
5	ELPAT		50.4	41.3		41.3
93	ELPAT		46.6	41.3		41.3
20	ELPAT		46.4	49.0		49.0
66	ELPAT		50.5	49.0		49.0
142	ELPAT		64.5	49.0		49.0
134	ELPAT		56.9	49.0		49.0
43	ELPAT		43.8	49.1		49.1
105	ELPAT		50.6	49.1		49.1
76	ELPAT		46.2	49.1		49.1
21	ELPAT		53.7	49.1		49.1
28	ELPAT		57.9	58.6		58.6
89	ELPAT		55.0	58.6		58.6
23	ELPAT		50.3	58.6		58.6
143	ELPAT		60.3	58.6		58.6

Sample Analysis Order	Source	Rep	NITON XLt 700 XRF		DataChem	
			Result µg/wipe	Estimated µg/wipe	Result µg/wipe	Estimated µg/wipe
26	ELPAT	1.234e+39	90.3	88.0	82	88.0
122	ELPAT		86.8	88.0	83	88.0
78	ELPAT		75.6	88.0	79	88.0
15	ELPAT		88.7	88.0	100	88.0
119	ELPAT		139.0	117.0	120	117.0
56	ELPAT		122.0	117.0	120	117.0
146	ELPAT		112.0	117.0	120	117.0
3	ELPAT		139.0	117.0	110	117.0
92	ELPAT		152.0	162.3	150	162.3
114	ELPAT		159.0	162.3	160	162.3
71	ELPAT		142.0	162.3	150	162.3
147	ELPAT		158.0	162.3	160	162.3
127	ELPAT		208.0	201.6	200	201.6
96	ELPAT		204.0	201.6	190	201.6
75	ELPAT		221.0	201.6	200	201.6
12	ELPAT		200.0	201.6	220	201.6
97	ELPAT		217.0	239.0	230	239.0
64	ELPAT		244.0	239.0	250	239.0
36	ELPAT		188.0	239.0	250	239.0
59	ELPAT		251.0	239.0	230	239.0
80	UC LAB		251.0	265.5	230	256.7
95	UC LAB		259.0	260.6	220	226.8
44	UC LAB		208.0	234.6	220	237.9
33	UC LAB		237.0	272.7	210	242.9
103	UC LAB		243.0	249.5	230	255.6
90	UC LAB		218.0	265.5	230	260.0
136	UC LAB		228.0	237.9	220	242.3
34	UC LAB		240.0	236.2	240	262.2
88	UC LAB		188.0	252.8	240	258.3
48	UC LAB		189.0	226.3	210	236.8
52	UC LAB		227.0	242.3	210	225.2
9	UC LAB		222.0	248.9	220	247.3
16	UC LAB		265.0	244.0	210	232.9
85	UC LAB		225.0	242.3	240	266.1
42	UC LAB		209.0	234.6	210	242.3
115	UC LAB		265.0	270.5	240	273.8
151	UC LAB		250.0	233.5	240	258.3
27	UC LAB		231.0	237.9	240	258.3
70	UC LAB		214.0	234.0	220	246.7
50	UC LAB		223.0	244.5	200	228.5

Sample Analysis Order	Source	Rep	NITON XLt 700 XRF		DataChem	
			Result µg/wipe	Estimated µg/wipe	Result µg/wipe	Estimated µg/wipe
37	ELPAT	1.234e+35	257.0	256.7	2.90e+107	256.7
131	ELPAT		227.0	256.7		256.7
1	ELPAT		217.0	256.7		256.7
158	ELPAT		228.0	256.7		256.7
144	ELPAT		239.0	260.8		260.8
62	ELPAT		218.0	260.8		260.8
157	ELPAT		253.0	260.8		260.8
68	ELPAT		276.0	260.8		260.8
30	UC LAB		430.0	411.0		414.9
113	UC LAB		313.0	370.6		399.4
141	UC LAB		359.0	422.1		432.0
79	UC LAB		423.0	428.2		435.9
118	UC LAB		335.0	403.8		374.0
108	UC LAB		337.0	366.8		392.8
22	UC LAB		316.0	366.8		369.0
53	UC LAB		347.0	391.1		400.0
57	UC LAB		333.0	415.5		365.7
4	UC LAB		412.0	408.8		386.1
38	UC LAB		323.0	377.3		403.3
155	UC LAB		386.0	383.9		400.5
130	UC LAB		381.0	414.9		427.1
29	UC LAB		336.0	370.6		377.3
153	UC LAB		370.0	385.0		359.6
149	UC LAB		392.0	402.2		417.1
39	UC LAB		412.0	406.0		419.9
7	UC LAB		383.0	384.5		408.8
112	UC LAB		297.0	373.4		375.6
49	UC LAB		360.0	361.2		395.0
81	ELPAT		392.0	408.7		408.7
128	ELPAT		409.0	408.7		408.7
17	ELPAT		435.0	408.7		408.7
63	ELPAT		381.0	408.7		408.7
83	ELPAT		445.0	418.1		418.1
31	ELPAT		463.0	418.1		418.1
133	ELPAT		446.0	418.1		418.1
104	ELPAT		448.0	418.1		418.1

Sample Analysis Order	Source	Rep	NITON XLt 700 XRF		DataChem	
			Result µg/wipe	Estimated µg/wipe	Result µg/wipe	Estimated µg/wipe
116	ELPAT	1.234e+15	629.0	561.9	580	561.9
98	ELPAT		491.0	561.9	540	561.9
140	ELPAT		513.0	561.9	560	561.9
69	ELPAT		506.0	561.9	540	561.9
117	ELPAT		562.0	564.7	560	564.7
18	ELPAT		447.0	564.7	560	564.7
120	ELPAT		607.0	564.7	570	564.7
19	ELPAT		451.0	564.7	530	564.7
99	ELPAT		708.0	805.1	760	805.1
123	ELPAT		797.0	805.1	770	805.1
126	ELPAT		623.0	805.1	760	805.1
46	ELPAT		770.0	805.1	740	805.1
94	ELPAT		1387.0	1482.6	1500	1482.6
160	ELPAT		1545.0	1482.6	1500	1482.6
24	ELPAT		1377.0	1482.6	1500	1482.6
74	ELPAT		1523.0	1482.6	1400	1482.6