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Environmental Technology Verification Program

Verification Test Plan

Evaluation of Field Portable Measurement Technologies for Lead in Dust Wipes

Oak Ridge National Laboratory

APPROVAL SIGNATURES

This document is intended to ensure that all aspects of the verification are documented, scientifically sound, and that operational procedures are conducted within quality assurance/quality control specifications and health and safety regulations.

The signatures of the individuals below indicate concurrence with, and agreement to operate compliance with, procedures specified in this document.

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November 2002

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Verification Test Plan

Evaluation of Field Portable Measurement Technologies for Lead in Dust Wipes

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EXECUTIVE SUMMARY

EPA created the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative technologies through performance verification and information dissemination. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. The ETV Program is intended to assist and inform those involved in the design, distribution, permitting, and purchase of environmental technologies. The verification study described in this test plan will be conducted by the Advanced Monitoring Systems Center (AMS), one of six Centers of the ETV program. The AMS Center is administered by the EPA's National Exposure Research Laboratory. The Oak Ridge National Laboratory (ORNL) will serve as the verification organization for the test.

This is a verification test of a commercially available x-ray fluorescence instruments (XRF) capable of measuring lead in dust wipe samples. This test will be the third round of testing for lead in dust wipe measurement technologies. In November 2001, four technologies were tested in Hartford, CT. In January 2002, one technology was tested in Oak Ridge, TN. The experimental design described in this test plan is the same as the previous two tests. The vendor will blindly analyze 160 dust wipe samples containing known amounts of lead, ranging in concentration from < 2 to 1,500 μg/wipe. The experimental design is particularly focused on important clearance standards, such as those identified in 40 CFR Part 745.227(e)(8)(viii) 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. The samples will include wipes archived from the Environmental Lead Proficiency Analytical Testing Program (ELPAT). These samples have been prepared from dust collected in households in North Carolina and Wisconsin. Also, samples were acquired from the University of Cincinnati and archived from the first round of testing. These dust wipe samples were prepared from National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs).

ABBREVIATIONS AND ACRONYMS

AIHA	American Industrial Hygiene Association
AMS	Advanced Monitoring Systems Center, ETV
ASTM	American Society for Testing and Materials
BASP	Big-Area Silicon PIN-diode detector
BMI	Battelle Memorial Institute
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CL	clearance level
EDXRF	energy dispersive x-ray fluorescence
ELPAT	Environmental Lead Proficiency Analytical Testing program
EPA	U. S. Environmental Protection Agency
ESH&Q	Environmental Safety, Health, and Quality
ETV	Environmental Technology Verification Program
ETVR	Environmental Technology Verification Report
fn	false negative result
fp	false positive result
HASP	Health and Safety Plan
ICP-AES	Inductively coupled plasma-atomic emission spectrometry
NERL	National Exposure Research Laboratory, U.S. EPA
NIOSH	National Institute for Occupational Safety and Health, CDC
NIST	National Institute of Standards & Technology
NLLAP	National Lead Laboratory Accreditation Program, U.S. EPA
OPPT	Office of Pollution Prevention and Toxics, U.S. EPA
ORNL	Oak Ridge National Laboratory
PPE	personal protective equipment
QA	quality assurance
QAPP	Quality Assurance Project Plan
QAS	ORNL Quality Assurance Specialist
QC	quality control
RSD	relative standard deviation
RTI	Research Triangle Institute
SD	standard deviation
SRM	Standard Reference Material
UC	University of Cincinnati
XRF	x-ray fluorescence instrument

1 INTRODUCTION

This chapter discusses the purpose of the verification and the verification test plan, describes the elements of the verification test plan, and provides an overview of the Environmental Technology Verification (ETV) Program and the technology verification process.

1.1 Verification Objectives

The purpose of this verification test is to evaluate the performance of commercially available field analytical technologies for analyzing dust wipe samples for lead. Specifically, this plan defines the following elements of the verification test:

- Roles and responsibilities of verification test participants;
- Procedures governing verification test activities such as sample collection, preparation, analysis, data collection, and interpretation;
- Experimental design of the verification test;
- Quality assurance (QA) and quality control (QC) procedures for conducting the verification and for assessing the quality of the data generated from the verification; and,
- Health and safety requirements for performing the verification test.

1.2 What is the Environmental Technology Verification Program?

The U.S. Environmental Protection Agency (EPA) created the Environmental Technology Verification Program (ETV) to facilitate the deployment of innovative or improved environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by substantially accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

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

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

The program now operates six Centers covering a broad range of environmental areas. ETV began with a 5-year pilot phase (1995–2000) to test a wide range of partner and procedural alternatives in various pilot areas, as well as the true market demand for and response to such a program. In the 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 test described in this plan will be 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 www.epa.gov/etv and ORNL’s web site at www.ornl.gov/etv). The AMS Center is administered by EPA’s National Exposure Research Laboratory (NERL).

1.3 Technology Verification Process

The technology verification process is intended to serve as a template for conducting technology verifications that will generate high quality data which can be used to verify technology performance. Four key steps are inherent in the process:

- Needs identification and technology selection;
- Verification test planning and implementation;
- Report preparation;
- Information distribution.

1.3.1 Needs Identification and Technology Selection

The first step in the technology verification process is to determine technology needs of the user-community (typically state and Federal regulators and the regulated community). Each Center utilizes stakeholder groups. Members of the stakeholder groups come from EPA, the Departments of Energy and Defense, industry, and state regulatory agencies. The stakeholders are invited to identify technology needs and to assist in finding technology vendors with commercially available technologies that meet the needs. Once a technology need is established, a search is conducted to identify suitable technologies. The technology search and identification process consists of reviewing responses to *Commerce Business Daily* announcements, searches of industry and trade publications, attendance at related conferences, and leads from technology vendors. The following criteria are used to determine whether a technology is a good candidate for the verification:

- Meets user needs
- May be used in the field or in a mobile laboratory
- Applicable to a variety of environmentally impacted sites
- High potential for resolving problems for which current methods are unsatisfactory
- Costs are competitive with current methods
- Performance is better than current methods in areas such as data quality, sample preparation, or analytical turnaround
- Uses techniques that are easier and safer than current methods
- Is commercially available and field-ready.

For this verification test of lead measurement technologies, ORNL has assembled a technical panel of the nation's experts in this field. The technical panel includes representation from the U.S. Department of Housing and Urban Development, the National Institute for Occupational Safety and Health, the National Institute of Standards and Technology, Research Triangle Institute, the American Industrial Hygiene Association, the Massachusetts Childhood Lead Poisoning and Prevention Program, and several EPA offices, including the Office of Pollution Prevention and Toxics (OPPT).

1.3.2 Verification Planning and Implementation

After a vendor agrees to participate, EPA, the Verification Organization, and the vendor meet to discuss each participants responsibilities in the verification process. In addition, the following issues are addressed:

- Site selection. Identifying sites that will provide the appropriate physical or chemical environment, including contaminated media
- Determining logistical and support requirements (for example, field equipment, power and water sources, mobile laboratory, communications network)
- Arranging analytical and sampling support
- Preparing and implementing a verification test plan that addresses the experimental design, sampling design, QA/QC, health and safety considerations, scheduling of field and laboratory operations, data analysis procedures, and reporting requirements

1.3.3 Report Preparation

Innovative technologies are evaluated independently and, when possible, against conventional technologies. The technologies being verified are operated by the vendors in the presence of independent observers. The observers are EPA staff, technical panel staff and from a independent third-party organization. The data generated during the verification test are used to evaluate the capabilities, limitations, and field applications of each technology. A data summary and detailed evaluation of each technology are published in an Environmental Technology Verification Report (ETVR). The original complete data set is available upon request.

An important component of the ETVR is the Verification Statement, which consists of three to five pages, using the performance data contained in the report, are issued by EPA and appear on the ETV Internet Web page. The Verification Statement is signed by representatives of EPA and ORNL.

1.3.4 Information Distribution

Producing the ETVR and the Verification Statement represents a first step in the ETV outreach efforts. ETV gets involved in many activities to showcase the technologies that have gone through the verification process. The Program is represented at many environmentally-related technical conferences and exhibitions. ETV representatives also participate in panel sessions at major technical conferences. ETV maintains a traveling exhibit that describes the program, displays the names of the companies that have had technologies verified, and provides literature and reports.

We have been taking advantage of the Web by making the ETVRs available for downloading to anyone interested. The ETVRs and the Verification Statements are available in Portable Document Format (.pdf) on the ETV Web site (http://www.epa.gov/etv).

1.4 Purpose of this Verification Test Plan

The purpose of the verification test plan is to describe the procedures that will be used to verify the performance goals of the technologies participating in this verification. This document incorporates the QA/QC elements needed to provide data of appropriate quality sufficient to reach a credible position regarding performance. This is not a method validation study, nor does it represent every environmental situation which may be appropriate for these technologies. But it will provide data of sufficient quality to make a judgement about the application of the technology under conditions similar to those encountered in the field under normal conditions.

This test plan was developed based on the first round of testing which occurred in November 2001 in Hartford, CT (four technologies) and the second round of testing which occurred in January 2002 in Oak Ridge, TN (one technology).

2 VERIFICATION RESPONSIBILITIES AND COMMUNICATION

This section identifies the organizations involved in this verification test and describes the primary responsibilities of each organization. It also describes the methods and frequency of communication that will be used in coordinating the verification activities.

2.1 Verification Organization and Participants

Participants in this verification are listed in Table 2-1. The specific responsibilities of each verification participant are discussed in Section 2.3 This verification test is being coordinated by the Oak Ridge National Laboratory (ORNL) under the direction of Battelle Memorial Institute (BMI) and the EPA's Office of Research and Development, National Exposure Research Laboratory. EPA and BMI's role is to administer the verification program. ORNL's role is to provide technical and administrative leadership and support in conducting the verification.

Table 2-1. Verification Participants in the Lead in Dust Field Analytical Technology Verification Test

Organization	Point(s) of Contact	Role
Oak Ridge National Laboratory P.O. Box 2008 Bethel Valley Road Bldg. 4500S, MS-6120 Oak Ridge, TN 37831-6120	**Project Manager:** Roger Jenkins phone: (865) 574-4871 fax: (865) 576-7956 jenkinsra@ornl.gov	verification organization
Battelle Memorial Institute Statistics and Data Analysis Systems Department Battelle Columbus 505 King Avenue Columbus, OH, 43201-2693	**Project Lead:** Jessica Sanford phone: (614) 424-4998 fax: (614) 424-4250 Sanford@battelle.org	BMI project management
U. S. EPA National Exposure Research Laboratory Environmental Science Division P.O. Box 93478 Las Vegas, NV 89193-3478	**Project Officer:** Eric Koglin phone: (702) 798-2332 fax: (702) 798-2107 koglin.eric@epa.gov	EPA project management
NITON, LLC 900 Middlesex Tpk., Bldg. 8 Billerica, MA 01821	**Contact:** Jonathan Shein phone: (978) 670-7460 fax: (978) 670-7430 jjshein@niton.com	technology vendor
DataChem 4388 Glendale-Milford Road Cincinnati, Ohio 45242	**Contact:** Dixie Yockey phone: (513) 733-5336 fax: (513) 733-5347 dyockey@datachemlabs.com	NLLAP-recognized laboratory
U.S. EPA Region 1 11 Technology Drive North Chelmsford, MA 01863-2431	**Contact:** Paul Carroll phone: (617) 918 8306 carroll.paulr@epa.gov	Test site host

2.2 Responsibilities

The following is a delineation of each participant's responsibilities for the verification test. In this section, the term "vendor" applies to NITON, LLC.

The Vendor, in consultation with ORNL, BMI, and EPA, is responsible for the following elements of this verification test:

- Contribute to the design and preparation of the verification test plan;
- Provide detailed procedures for using the technology;
- Prepare field-ready technology for verification;
- Operating the technology during the verification test;
- Documenting the methodology and operation of the technology during the verification;
- Furnish data in a format that can be compared to laboratory values;
- Logistical, and other support, as required.

ORNL has responsibilities for:

- Preparing the verification test plan;
- Developing a quality assurance project plan (QAPP) (Section 6 of the verification test plan);

- Preparing a health and safety plan (HASP) (Section 7 of the verification test plan) for the verification activities;
- Developing a test plan for the verification;
- Acquiring the necessary laboratory analysis data;
- Performing sample preparation activities (including purchasing, labeling, and distributing).

ORNL, BMI, and EPA have coordination and oversight responsibilities for:

- Providing needed logistical support, establishing a communication network, and scheduling and coordinating the activities of all verification participants, including the technical panel;
- Auditing the on-site sampling activities;
- Managing, evaluating, interpreting, and reporting on data generated by the verification;
- Evaluating and reporting on the performance of the technologies;
- Other logistical information and support needed to coordinate access to the site for the field portion of the verification, such as waste disposal.

3 TECHNOLOGY DESCRIPTION

This section provides description of the technology participating in the verification test. The description was provided by the vendor, with minimal editing by ORNL.

3.1 NITON Corporation

3.1.1 General Description

The sample analyzer is an energy dispersive x-ray fluorescence (EDXRF) spectrometer that uses a low power miniature x-ray tube with a silver target tube 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, known as the Big-Area Silicon PIN-diode (BASP). Signals from the BASP 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.

3.1.2 Product Description

The NITON XLt series sample analyzer provides the user with the speed and efficiency of x-ray tube excitation, while greatly reducing the regulatory demands typically encountered with isotope-based systems. In most cases, the XLt can be shipped from state to state and country to country with minimal paperwork and expense.

As with the previous generation XL isotope-based series, the XLt series can be equipped for dust wipe analysis with both a metal dust wipe holder and a thin sample test stand. The thin sample test stand (see Figure 3-1) offers both ease of use and optimum safety as the reading cannot be initiated until the sample drawer is closed and locked into position. The sample drawer actuates the proximity sensor and permits a reading to be taken.

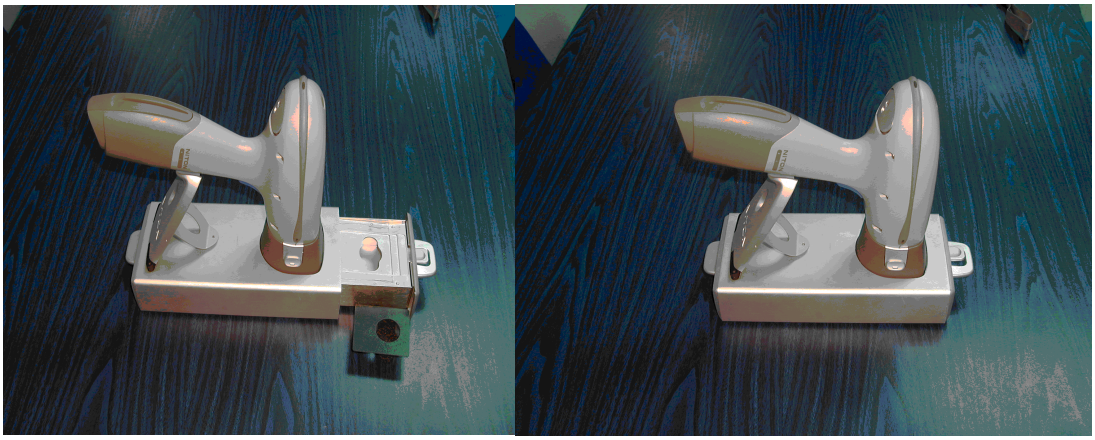

Figure 3-1. XLt with open sample drawer (left) and with closed sample drawer (right), ready for analysis.

3.1.3 Sample Preparation

1. For ELPAT samples, unfold and distribute the sample across the surface of the wipe using a spatula or equivalent tool. The tool must be cleaned in between each sample preparation.
2. Fold the sample five times, as specified in the schematic below, such that it is neatly folded to the proper size (1 x 1.5 inches) - see Figure 3-2.
3. Dry the sample prior to testing: The addition of this step has been found to improve the accuracy and precisions of dust-wipe measurements. For example, dry for 20 minutes at 250° F. in a toaster oven, or expose the sample overnight to ambient temperature and humidity. After oven drying, allow the dried sample to sit in ambient air for 5 minutes.
4. Bag the wipe sample in a 2 x 2 inch plastic bag (NITON part number 187-471 or equivalent) and label. To eliminate the potential for cross-contamination of samples, never reuse plastic bags.
5. Position the wipe sample in its plastic bag within the frame of the metal dust wipe holder (NITON part number 180-407 or equivalent).

3.1.4 Sample Analysis

1. Position the metal dust wipe holder at the number-one position on the thin sample test stand and take the first of four measurements (for 60 seconds). Note that the following procedure using four sample measurements has been designed to insure that the entire area of the folded dust-wipe sample is properly measured by the spectrometer.
2. Place the metal dust wipe holder at the number-two position on the test stand and take the second measurement (for 60 seconds).
3. Rotate the dust wipe holder 180 degrees (<u>without</u> turning the sample holder upside-down).
4. Place the metal dust wipe holder at the number-one position on the test stand and take the third measurement (for 60 seconds).
5. Place the metal dust wipe holder at the number-two position on the test stand and take the fourth measurement (for 60 seconds).

At the end of the fourth run, the instrument will display an average of the four individual readings. At this point, the bag should be turned over and steps 1-5 should be repeated for the back side of the wipe. The average concentration from the four readings on the front of the wipe will be averaged with the average of the four readings on the back of the wipe to give the final result.

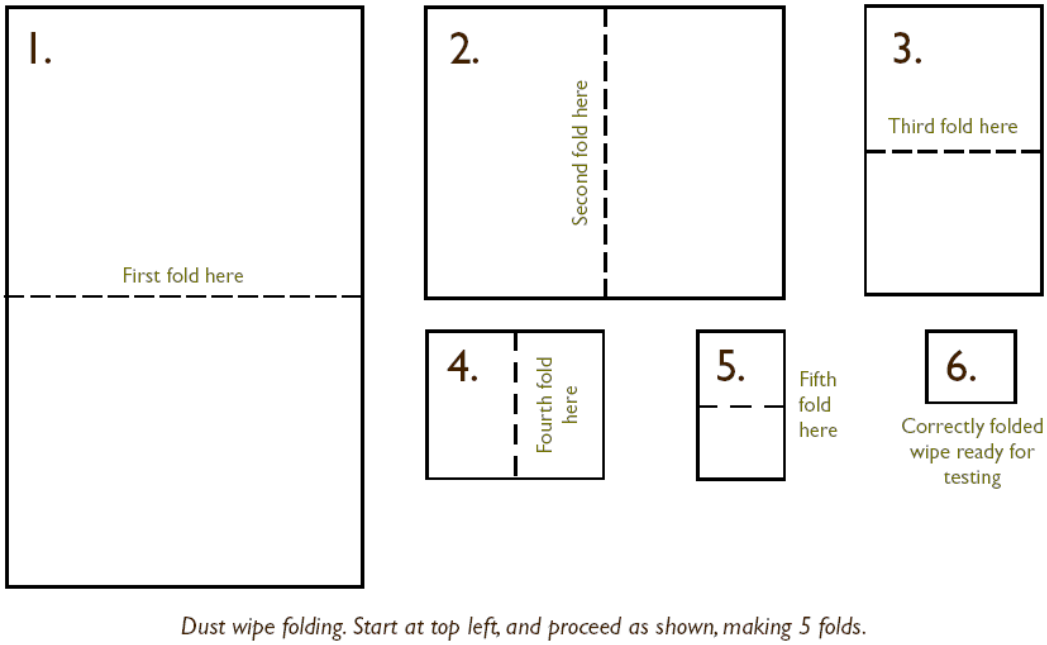

Figure 3-2. NITON folding procedure.

3.1.5 Calibration and System Verification

The instrument is factory calibrated. During the test, instrument performance will be verified by placing verification samples in the metal dust wipe holder and follow steps one through five of the "Sample Analysis" procedure above. The verification samples will be at nominal concentrations of 40 µg/wipe, 250 µg/wipe, and 400 µg/wipe. These verification samples will be previously characterized ELPAT or University of Cincinnati dust wipes samples that have been prepared in the same procedure as detailed above. These verification samples will be run at the beginning of the day and periodically throughout the day (between batches of samples or every 2-3 hours) to ensure instrument stability.

4 VERIFICATION TEST DESIGN

This section discusses the objectives and design of the verification test, factors that must be considered to meet the performance objectives, and the information that ORNL, BMI, and EPA will use to evaluate the results of the verification.

4.1 Drivers and Objectives of the Verification Test

The purpose of this test is to evaluate the performance of field analytical technologies that are capable of analyzing dust wipe samples for lead contamination. This test will provide information on the potential applicability of field technologies for clearance testing. The experimental design is 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 [1].

The primary objectives of this verification test are 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 (see ELPAT described below), 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. The planning for this verification test follows the guidelines established in the data quality objectives process.

4.2 Summary of the Experimental Design

All of the samples analyzed in this verification test were archived from the previous round of ETV testing in November 2001. Prior to the test, 16 archived samples, similar in concentration and storage conditions to those that will be used in the test, were analyzed by DataChem to confirm that the sample concentrations had not changed significantly. The results indicated that the measured values of the 16 samples were all between 87% and 105% of the estimated values, indicating that the sample concentrations had not degraded. Sample loss when stored in a freezer was not expected to be an issue, since the ELPAT program archives dust wipes for years.

All of the wipes utilized in this test (PaceWipe™ and Aramsco Lead Wipe™) were on the list of wipes recommended for lead testing by the American Society for Testing and Materials requirements [2]. 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 lead contamination. In addition to the safety concern of subjecting participants to lead exposure, the spatial variability of adjacent samples would have been so great that it would be much larger than the expected variability of this type of technology, therefore making it difficult to separate instrument/method variability and sampling variability. The availability of well-characterized samples derived from "real-world" situations made the use of proficiency testing samples (so-called "ELPAT" samples) and other prepared samples an attractive alternative.

4.2.1 ELPAT and Blank Sample Description

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

Research Triangle Institute (RTI) in Research Triangle Park, NC, is contracted to prepare and distribute the lead-containing paint, soil, and dust wipe ELPAT samples. For the rounds of testing which have occurred since 1992, archived samples are available for purchase. Some of these samples were used in this verification test. Because the samples have already been tested by hundreds of laboratories, a certified concentration value is supplied with the sample. This certified value represents a pooled measurement of all of the results submitted, with outliers excluded from the calculation.

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

Before use in the ELPAT program, RTI performed a series of analyses to confirm that the samples were prepared within the quality guidelines established for the program. The data quality requirements for the ELPAT samples were: 1) the relative standard deviation of the samples analyzed by RTI must be 10% or less; 2) the measured concentrations must be within 20% of the target value that RTI was intending to prepare; and 3) analysis by an 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.

4.2.2 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. The sample was prepared by weighing, so the concentrations can be estimated. 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) [3]. 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 [3].

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 ≤ 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, those 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 calculated from the UC data (102%), but both values 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.

4.2.3 Distribution and Number of Samples

A total of 160 samples will be analyzed in the verification test. Figure 4-1 is a plot containing the distribution of the sample concentrations that will be analyzed in this study. Twenty samples were prepared by the University of Cincinnati at +/- 10% of each of the three clearance levels (3 test levels x 20 samples = 60 samples total). Research Triangle Institute prepared 20 "blanks" at lead concentrations < 2 µg/wipe. These samples are noted as such in Figure 4-1. The remaining samples in Figure 4-1 are ELPAT samples. For most of the ELPAT samples, four samples will be analyzed at each concentration level (16 test levels x 4 samples each = 64 samples total). There are two concentration levels (at 49 and 565 µg/wipe) where eight samples will be analyzed. While the set of samples at each concentration level were prepared using homogeneous source materials and an identical preparation procedure, ELPAT samples cannot be considered true "replicates" because each sample was prepared individually. However, these samples represent four samples prepared similarly at a specified target concentration, with an estimated value calculated from more than 100 analyses of similarly prepared samples.

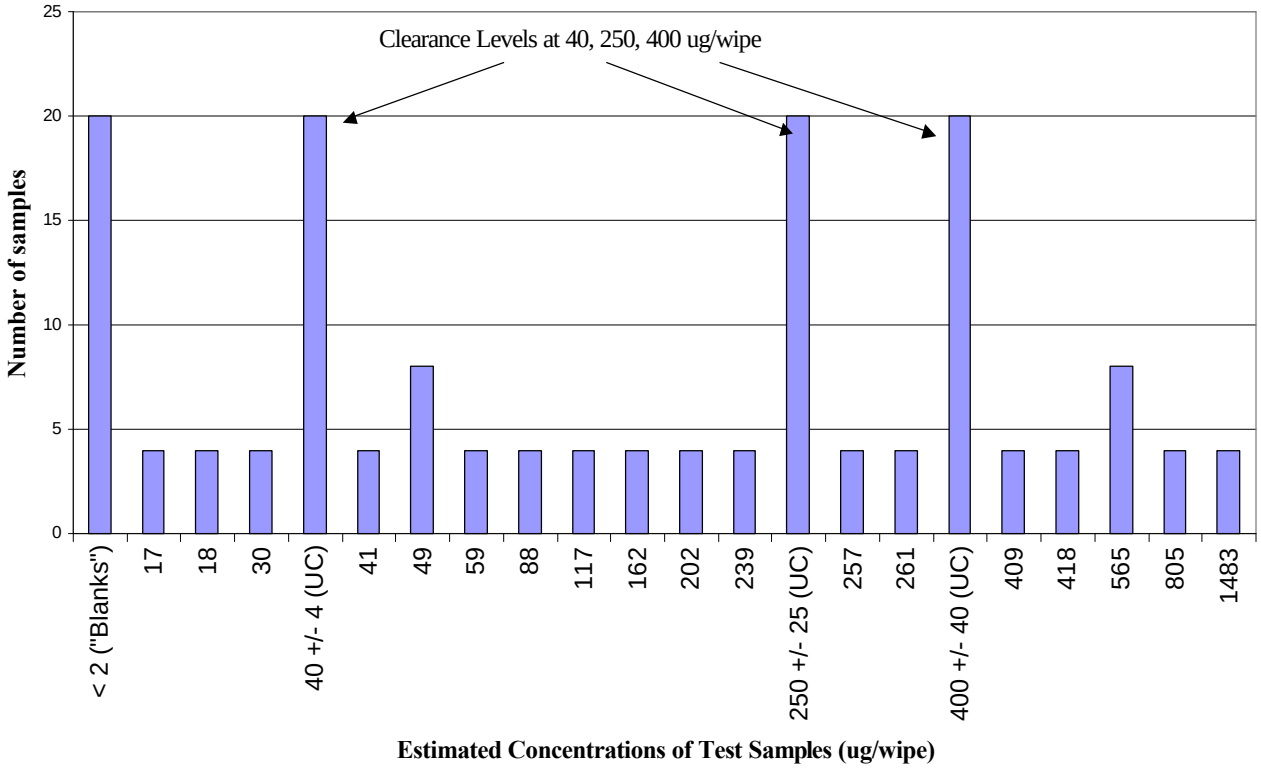

Figure 4-1. Distribution of concentration levels.

4.3 Comparison of Field Technology Results to an NLLAP-Recognized Laboratory's Results

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 [1]. 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 [1]. 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.

4.3.1 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 Table4-1 below, DataChem passed the pre-test by reporting concentrations that were within 25% of the estimated concentration for samples above the reporting limit.

4.3.2 Description of Method

The laboratory method used in this study 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 Appendix A. DataChem's procedures are modification of Methods 3050B and 6010B of EPA SW-846 Method Compendium for the preparation and analysis of metals in environmental matrices [4,5]. Other specific references for the preparation and analysis of dust wipes are available from the American Society for Testing and Materials (ASTM) [6].

Table 4-1. 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

5 EXECUTION OF THE VERIFICATION TEST

5.1 Summary of Verification Activities

This verification test will be conducted in a laboratory at EPA Region 1, in North Chelmsford, MA, from November 18 through 22, 2002. The vendor, who will operate their own equipment, must analyze all 160 samples on-site and submit results prior to departure in order to complete the verification test. The samples evaluated during the verification will consist of (1) ELPAT samples prepared from housedust collected from multiple homes in North Carolina and Wisconsin, ranging in concentration from 15 to 1,500 μg/wipe, (2) UC-prepared samples from NIST SRMs on Aramsco LeadWipes, near the three clearance levels of 40, 250, and 400 μg/wipe, and (3) low level samples called "detectable blanks", with concentrations (< 2 μg lead/wipe) below typical detection levels for field technologies, prepared by RTI using the same procedure as the ELPAT samples.

5.2 Sample Distribution

ORNL will be responsible for sample distribution. The samples will be packaged in 20-mL plastic scintillation vials and labeled with a sample identifier. The vendor will receive the suite of samples in a randomized order. All samples will be prepared for distribution at the start of the verification. The vendor will go to a sample distribution table to pick-up the samples. The samples will be distributed in batches of 16. Completion of chains-of-custody forms will document sample transfer.

5.3 Submission of Results

The vendor will provide the results to ORNL**.** The vendor will be responsible for reducing the raw data into a presentation format consistent with the evaluation requirements. At the end of the verification test, the vendor will submit all final results and raw data to ORNL. After the conclusion of the test, the vendor will have one week to review their data and make revisions to their results. These revisions will not involve re-analysis of any sample. The revisions will be limited to correcting for calculation and transcription errors.

5.4 Verification Performance Factors

The following are the logistical and technical performance verification factors that will be verified for each technology.

- Accuracy: closeness of technology result to an estimated known value (i.e., ELPAT certificate value and UC estimated value);
- Precision: reproducibility of technology's results for set of four samples prepared at a specific concentration level;
- Comparability: performance relative to the NLLAP-recognized laboratory;
- Detectable blanks: number of samples where lead is reported above reporting limits for samples which are prepared at low levels (< 2 μg/wipe);
- Probability of false positive results: relative to all three clearance levels of 40, 250, and 400 $\mu g/ft^2$. For example, number of samples where the field technology reports a result as ≥ 40 μg and the estimated concentration is less than 40 μg.
- Probability of false negative results: relative to all three clearance levels of 40, 250, and 400 $\mu g/ft^2$. For example, number of samples where the field technology reports a result as < 40 μg and the estimated concentration is ≥ 40 μg.
- Sample throughput: number of samples per day per number of analysts
- Ease of use: user friendliness of the technology; amount of training required to operate independently.

These factors and the anticipated statistical analyses are further discussed in Section 6.

6 QUALITY ASSURANCE PROJECT PLAN (QAPP)

The QAPP for this verification test specifies procedures that will be used to ensure data quality and integrity. Careful adherence to these procedures will ensure that data generated from the verification will meet the desired performance objectives and will provide sound analytical results.

6.1 Purpose and Scope

The primary purpose of this section is to outline steps that will be taken to ensure that data resulting from this verification is of known quality and that a sufficient number of critical measurements are taken. This section is written in compliance with ORNL's ETV Quality Management Plan [7].

6.2 Quality Assurance Responsibilities

The implementation of the verification test plan must be consistent with the requirements of the study and routine operation of the technology. The ORNL project manager will ensure that the QAPP is implemented during all verification activities and for its approval by EPA and BMI. ORNL's QA specialist (QAS) will review and approve the QAPP and will provide QA oversight of the verification activities. The ORNL statistician will primarily be responsible for the reduction of the vendor data. The EPA program manager and EPA QA manager will review and approve this plan.

6.3 Field Operations

6.3.1 Site Training

Preliminary site training will be provided to the vendor on the first day of testing. This will be required before initiation of the field study. This training will be conducted by the ORNL project manager or his designee. It will entail an overview of the test site, safety information, emergency procedures, and logistical information regarding the verification test.

6.3.2 Communication and Documentation

Successful field operations require detailed planning and extensive communication. ORNL will communicate regularly with the verification participants to coordinate all field activities associated with this verification and to resolve any logistical, technical, or QA issues that may arise as the verification progresses. Pertinent vendor and ORNL field activities will be thoroughly documented. Field documentation will include field logbooks, photographs, field data sheets, and chain-of-custody forms.

The ORNL project manager will be responsible for maintaining all field documentation. Field notes will be kept in a bound logbook. Each page will be sequentially numbered and labeled with the project name and number. Completed pages will be signed and dated by the individual responsible for the entries. Errors will

have one line drawn through them and this line will be initialed and dated. Any deviations from the approved final verification test plan will be thoroughly documented in the field logbook and provided to the ORNL. Photographs will be taken with a digital camera.

6.4 Performance and System Audits

The following audits will be performed during this verification.

6.4.1 Technical Systems Audit

Because the verification test will be conducted in Massachusetts, the ORNL QAS will not be able to perform an on-site surveillance during the test. However, the ORNL QAS will remotely provide oversight of the verification activities through four mechanisms: a management assessment checklist (to be completed by the ORNL project manager); email interviews with the project statistician that must be completed with 24 hours of receipt; survey for vendors to complete; and review of digital pictures of the verification activities that will be posted in near real-time on the ORNL ETV web site (www.ornl.gov/etv). This plan for remotely assessing the verification activities allows for inputs for multiple sources, so that the QAS will have an unbiased picture of how the study was conducted. The use of email will allow for spontaneous responses and follow-up questions.

6.4.2 Data quality audit of the laboratory

One of the requirements to become an NLLAP-recognized laboratory is routine quality audits. ORNL audited the laboratory during the analyses of the samples and found that the lab was proficient in following its procedures.

6.4.3 Surveillance of Technology Performance

During verification testing, ORNL staff will observe the operation of the field technology, such as observing the vendor operations, photo-documenting the test site activities, surveying calibration procedures, and reviewing sample data. The observations will be documented in a laboratory notebook. The verification report will contain the exact protocols used by the vendor during testing.

6.5 Quality Assurance Reports

QA reports provide the necessary information to monitor data quality effectively. It is anticipated that the following types of QA reports will be prepared as part of this verification.

6.5.1 QC Reports of Sample Preparation

As described in Sections 4.2.1 and 4.2.2, both RTI and UC analyzed a portion of the prepared samples to confirm the accuracy and precision of the sample preparation. The concentrations of the samples prepared by RTI were through independent confirmation through the ELPAT proficiency testing 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%). (102%), but both values within the data quality r Additionally, 42 randomly-selected samples (14 at each of the three clearance levels) were analyzed an by EPA Region 1 laboratory, 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%.

6.5.2 QAS Surveillance Report

The QAS will prepare a comprehensive report of the verification activities, based on her remote observations.

6.5.3 Status Reports

ORNL will regularly inform the EPA and BMI project managers of the status of the verification. Project progress, problems and associated corrective actions, and future scheduled activities associated with the verification test will be discussed. When problems occur, the vendor and ORNL will discuss them, estimate the type and degree of impact, describe the corrective actions taken to mitigate the impact and to prevent a recurrence of the problems, and discuss with BMI/EPA, as necessary. Major problems will be documented in the field logbook.

6.5.4 Audit Reports

Any additional QA audits or inspections, such as those conducted by interested visitors, that take place while the verification test is being conducted will be formally reported by the auditors to the ORNL project manager, who will forward them to the BMI project lead. Informal reporting of audit results will be reported immediately to BMI through a phone call, personal communication, or email.

6.6 Corrective Actions

Routine corrective action may result from common monitoring activities, such as:

- Performance evaluation audits
- Technical systems audits
- Calibration procedures

If the problem identified is technical in nature, the individual vendor will be responsible for seeing that the problem is resolved. If the issue is one that is identified by ORNL, the identifying party will be responsible for seeing that the issue is properly resolved. All corrective actions will be documented. Any occurrence that causes discrepancies from the verification test plan will be noted in the technology verification report.

6.7 Laboratory Quality Control Checks

Internal quality control (QC) samples were analyzed by DataChem to indicate whether or not the samples were analyzed properly. A summary of QC samples include: initial calibration, continuing calibration verification, and analysis of known samples. This data was reviewed by ORNL as part of the data validation process. No discrepancies were noted in the data validation records.

6.8 Data Management

The vendor, ORNL, BMI, and EPA each have distinct responsibilities for managing and analyzing verification data. The vendor is responsible for obtaining, reducing, interpreting, validating, and reporting the data associated with their technology's performance. These data should be reported on the chain-of-custody. Vendor results will be due to ORNL at the conclusion of a day's field activities. The vendor's final report will be due to ORNL one week after the verification. Any discrepancies between the originally reported result and the final result must be described. ORNL is responsible for managing all the data and information generated during the verification test. BMI and ORNL are responsible for analysis and verification of the data. EPA will review the data in the verification report.

6.9 Data Reporting, Validation, and Analysis

To maintain good data quality, specific procedures will be followed during data reduction, review, and reporting. These procedures are detailed below.

6.9.1 Data Reporting

Data reduction refers to the process of converting the raw results into a concentration which will be used for evaluation of performance. The procedures to be used will be technology dependent, but the following is required for data reporting:

- The concentration unit will be μg of lead/wipe.
- If no lead is detected, the concentration will be reported as less than the reporting limits of the technology, with the reporting limits stated (e.g., < 20 μg/wipe). A result reported as “0" will not be accepted.

6.9.2 Data Validation

Validation determines the quality of the results relative to the end use of the data. ORNL was responsible for validating the laboratory data. (Note that the vendor is responsible for validating its own data prior to final submission.) Several aspects of the data (listed below) that were reviewed. The findings of the review are documented in the validation records.

6.9.2.1 Completeness of Laboratory Records

This qualitative review ensures that all of the samples that were sent to the laboratory were analyzed, and that all of the applicable records and relevant results are included in the data package.

6.9.2.2 Holding Times

The dust wipe samples will not require refrigeration or other preservation techniques. The method requirement is that the samples be prepared within 6 months of collection, which was met.

6.9.2.3 Correctness of Data

So as not to bias the assessment of the technology’s performance, errors in the laboratory data will be corrected as necessary. Corrections may be made to data that has transcription errors, calculation errors, and interpretation errors. These changes will be made conservatively, and will be based on the guidelines provided in the method used. The changes will be justified and documented in the validation records. No changes were made to the laboratory data.

6.9.2.4 Correlation Between Samples within a Concentration Set

Normally, one would not know if a single sample result was “suspect” unless (a) the sample was a spiked sample, where the concentration is known or (b) a result was reported and flagged by the laboratory as suspect for some obvious reason (e.g., no quantitative result was determined). The experimental design implemented in this verification study will provide an additional indication of the abnormality of data through the inspection of the set of four results for samples prepared at a specific concentration. Criteria has been established to determine if data is suspect. Data sets will be considered suspect if the percent relative standard deviation for a set of four similarly-prepared samples was greater than 50%, because this criteria would indicate imprecision. These data would be flagged so as not to bias the assessment of the technology’s performance. Precision and accuracy evaluations may be made with and without these suspect values to represent the best and worst case scenarios. If both the laboratory and the vendor report erratic results, the data may be discarded if it is suspected that the erratic results are due to a sample preparation error.

6.9.2.5 Evaluation of QC Results

QC samples were analyzed by the NLLAP-laboratory with every batch of samples to indicate whether or not the samples were analyzed properly. Performance on these samples was reviewed and no major findings were noted in the validation records.

6.9.2.6 Evaluation of Spiked Sample Data

Spiked samples are samples containing known concentrations of analyte(s). For this verification test, all of the samples are considered spiked samples.

6.9.3 Data Analysis for Verification Factors

This section contains a list of the six primary performance verification factors to be evaluated for both the field technology and the NLLAP-recognized laboratory.

6.9.3.1 Precision

Precision, in general, refers to the degree of mutual agreement among measurements of the same materials and contaminants. Environmental applications often involve situations where "measurements of the same materials" can take on a number of interpretations. In environmental applications, precision is often best specified as a percentage of contaminant concentration. The following lists several possible interpretations of precision for environmental applications.

1) The precision involved in repeated measurements of the same sample without adjusting the test equipment.

2) The precision involved in repeated measurements of the same sample after reset, repositioning, or re-calibration of the test equipment or when using different equipment of the same technology.

3) The precision of measurements due to spatial variability of dust samples from adjacent locations.

4) The precision characteristics of a specific technology in determining contamination at a specific site or at an arbitrary site.

In general, users of the technology will want to be assured that measurement variability in 1) and 2) is small. Measurement variability due to spatial variability described in 3) is likely to be site specific and is minimized in this verification by using samples prepared under homogeneous conditions. The measurement variability discussed in 4) is perhaps of most interest as it includes measurement variability resulting from possible differences in the design activities and effects of environmental conditions such as temperature that would vary from one site characterization to another as well as site and technology specific sources.

The strength of this verification's experimental design is that since an equal number of similar samples will be selected from a homogeneous population at every concentration level, an equal number of precision comparisons can be made.

Precision for this verification will be estimated by the variance, or standard deviation from the measured data. If "n" lead concentration measurements are represented by $Y_1, Y_2, ..., Y_n$, the estimated variance about their average value "$\overline{Y}$" is calculated by:

$$S^2 = \frac{1}{n-1} \sum_{k=1}^{n} (Y_k - \overline{Y})^2 \quad .$$

The standard deviation is the square root of S^2 and will be analyzed to see if the precision values are a function of lead concentration levels. The estimated S^2 values will also be compared by F-tests to those values reported on the ELPAT certificate and by UC. To express the reproducibility relative to the average lead concentration, percent relative standard deviation (RSD) is used to quantify precision, according to the following equation:

RSD = (standard deviation / average concentration) x 100%

Standard deviations estimated at each concentration level can be used to establish the relationship between the uncertainty and the average lead concentration. 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 (such as extraction) and measurement steps.

6.9.3.2 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 (see Appendix B for an example). 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 technology measurements will be statistically tested using t-tests or non-parametric tests at the 5% significance level. These statistical tests will compare the average results with the overall estimated values using the precision of the sample measurements. Bias will then be quantified by computing the percent recovery for four similar samples or a single sample using the equation:

$$\textit{percent recovery} = [\textit{measured amount(s)/estimated value}] \times 100\%$$

Accuracy will be assessed using both the ELPAT and UC estimated concentrations, with the results reported separately. 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 estimated values are the gravimetric values, the comparison to the UC samples represents how close the technology reported results to an absolute lead value. The UC analysis will 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%.

6.9.3.3 Detectable Blanks

Twenty samples in the study were prepared at < 2 μg/wipe, below the anticipated reporting limits of both the field technologies and the laboratory. Any reported lead for these samples will be considered a "detectable blank".

6.9.3.4 False Positive/False 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 [8]. 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 [8]. 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 is 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 will be estimated as curves that depend on a range of concentrations reported about the clearance level. These error probability curves will be 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 will be assumed that the estimated concentration provided by UC is the true concentration. However, this evaluation does 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 will also include 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 will be extended to ± 25% of the target concentration.

6.9.3.5 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. Comparing averages from similar samples measured by the technology with corresponding averages measured by the laboratory will be performed for all target concentration levels.

A correlation coefficient quantifies the linear relationship between two measurements [9]. 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.

6.9.3.6 Completeness

Completeness refers to the amount of data collected from a measurement process expressed as a percentage of the data that would be obtained using an ideal process under ideal conditions. The completeness objective for data generated during this verification is 95% or better.

There are many instances which might cause the sample analysis to be incomplete. Some of these are:

- Instrument failure;
- Calibration requirements not being met;
- Elevated analyte levels in the method blank.

7 HEALTH AND SAFETY PLAN

This section describes the specific health and safety procedures that will be used during the field work at the EPA Region 1 laboratory in North Chelmsford, MA.

7.1 Contact Information

The ORNL project manager will be Roger Jenkins, (865) 574-4871.
The ORNL project statistician will be Chuck Bayne, (865) 574-3134.
The ES&H Coordinator will be Fred Smith, (865) 574-4945.
The ORNL Quality Assurance Specialist (QAS) will be Janet Wagner, (865) 576-8335.
The US EPA Region 1 site contact will be Paul Carroll, (617) 918-8306.

7.2 Health and Safety Plan Enforcement

ORNL project manager and the ES&H Coordinator were responsible for developing the health and safety plan. The ORNL project manager will ultimately be responsible for ensuring that all verification participants understand and abide by the requirements of this HASP.

7.3 Site Access

Site training will be provided to the vendor prior to testing. The training will include a review of this health and safety plan. Because the test will be conducted in an EPA laboratory, standard procedures for the laboratory (such as use of safety glasses) will be followed, as required.

7.4 Waste Generation

The EPA Region 1 site contact will be responsible for ensuring that the chemical waste generated during the test is handled properly. Because the vendor has an x-ray fluorescence technology which does not require the use of chemicals for sample preparation, no hazardous waste should be generated. The used (i.e., analyzed) dust wipe samples will be shipped back to ORNL after the test.

7.5 Hazard Evaluation

The technology vendor must provide their own personal protective equipment (PPE), based on the hazards associated with the operation of their technology. Although unlikely to be necessary, visitors will be provided with PPE if warranted. The hazard information provided below was gathered from the ORNL Material Safety Data Sheet (MSDS) web page and serves as a general guideline for the hazards likely to be encountered during this field test.

Lead will be the most prevalent chemical hazard at the verification test. Exposure to lead can cause eye, skin, and gastrointestinal irritation. If inhaled, it may cause a respiratory tract irritation. The highest concentration of lead in the dust samples will be 1,500 μg, and most of the sample concentrations will be well below that level.

7.6 Personal Protection

PPE is appropriate to protect against known and potential health hazards encountered during routine operation of the technology systems. For this verification, Level D PPE is required. Level D provides minimal protection against chemical hazards. Level D PPE will be supplied by the individual technology vendor. It consists only as a work uniform, with gloves worn, where necessary. The only requirement for this verification test is appropriate work clothes, with no shorts or open-toed shoes. ORNL will provide visitors with PPE if necessary. If site conditions indicate that additional hazards are present, ORNL may recommend different or additional PPE to the vendor.

7.7 Physical Hazards

Physical hazards associated with field activities present a potential threat to on-site personnel. Dangers are posed by unseen obstacles, noise, and poor illumination. Injuries may result from the following:

- Accidents due to slipping, tripping, or falling
- Improper lifting techniques
- Moving or rotating equipment
- Improperly maintained equipment

Injuries resulting from physical hazards can be avoided by adopting safe work practices and by using caution when working with machinery.

7.8 Fire

The following specific actions will be taken to reduce the potential for fire during site activities:

- No smoking in the building.
- Fire extinguishers will be maintained on-site.
- All personnel will be trained on the location and operation of the portable fire

extinguishers.
• All personnel will be trained on the location of the phones and the number to call the fire department.

7.9 Mechanical, Electrical, Noise Hazards

Some technology-specific hazards may be identified once the vendor sets up their equipment. Proper hazards controls (i.e., guarding or markings) or PPE (i.e., ear plugs for noise hazards) will be implemented as necessary.

Electrical cables represent a potential tripping hazards. When practical, cables will be placed in areas of low pedestrian travel. If necessary, in high pedestrian travel areas, covers will be installed over cables.

7.10 Medical Support

Once on-site, ORNL will discuss medical options with the EPA Region 1 site contact and provide the information to the vendor during the site training.

7.11 Environmental Surveillance

The ORNL project manager will be responsible for surveying the site before, during, and after the verification test. Appropriate personnel (e.g., ES&H Coordinator, EPO, etc.) will be contacted to assist with any health or safety concerns.

7.12 Safe Work Practices

The vendor will provide the required training and equipment for their personnel to meet safe operating practice and procedures. The individual technology vendor and their company are ultimately responsible for the safety of their workers.

The following safe work practices will be implemented at the site for worker safety:

- Eating, drinking, chewing tobacco, and smoking will be permitted only in designated areas;
- Wash facilities will be utilized by all personnel before eating, drinking, or toilet facility use;
- PPE requirements (See Section 7.6) will be followed.

7.13 Complaints

All complaints should be filed with the ORNL project manager. All complaints will be treated on an individual basis and investigated accordingly. Complaints will be documented and reported to BMI.

REFERENCES

[1] Code of Federal Regulations. 2001. "*Identification of Dangerous Levels of Lead*", Final Rule, 40 CFR Part 745, January.

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[3] 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.

[4] 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.

[5] 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.

[6] 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.* West Conshohocken, PA.

[7] 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.

[8] 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.

[9] Draper, N. R., and H. Smith. 1981. *Applied Regression Analysis.* 2nd ed. John Wiley & Sons, New York.

APPENDIX A

LABORATORY STANDARD OPERATING PROCEDURES

Supplied by: DataChem (Cincinnati, Ohio)

APPENDIX B

ELPAT CERTIFICATE OF ANALYSIS SHEET

Supplied by: American Industrial Hygiene Association

ELPAT ROUND 36

ENVIRONMENTAL LEAD PROFICIENCY ANALYTICAL TESTING PROGRAM

CERTIFICATE OF ANALYSIS

	Sample Number	Reference Value	STD	RSD%	Lower Limit	Upper Limit
PAINT CHIPS (%)	1	1.5576	.094	6.0	1.2763	1.8389
	2	3.2953	.219	6.6	2.6385	3.9521
	3	0.0598	.006	9.4	0.0429	0.0767
	4	0.2851	.016	5.6	0.2373	0.3329
SOIL (mg/kg)	1	113.1	12.3	10.8	76.3	150
	2	141.9	12.6	8.9	104.1	179.8
	3	791.7	47.9	6.1	647.9	935.5
	4	289.5	24.6	8.5	215.7	363.3
DUST WIPES (*u*g)	1	162.3	14.3	8.8	119.2	205.3
	2	17.6	3.39	19.3	7.4	27.9
	3	418.1	30.7	7.3	326	510.3
	4	49	5.88	12.0	31.3	66.7