



# **Environmental Technology Verification Report**

## **Lead in Dust Wipe Measurement Technology**

### **Monitoring Technologies International PDV 5000 Trace Element Analyzer**



Oak Ridge National Laboratory

**ETV ✓ ETV ✓ ETV ✓**

THE ENVIRONMENTAL TECHNOLOGY VERIFICATION  
PROGRAM



U.S. Environmental Protection Agency



Oak Ridge National Laboratory

## Verification Statement

<b>TECHNOLOGY TYPE:</b>	<b>ANODIC STRIPPING VOLTAMMETRY</b>	
<b>APPLICATION:</b>	<b>MEASUREMENT OF LEAD IN DUST WIPES</b>	
<b>TECHNOLOGY NAME:</b>	<b>PDV 5000 Trace Element Analyzer</b>	
<b>COMPANY:</b>	<b>Monitoring Technologies International</b>	
<b>ADDRESS:</b>	<b>78 Collingwood Street Osborne Park 6017 Perth, Western Australia</b>	<b>PHONE: +61 8 9204 3600 FAX: +61 8 9204 2602</b>
<b>WEB SITE:</b>	<b>www.mti.com.au</b>	
<b>E-MAIL:</b>	<b>cgreen@colingreen.idps.co.uk</b>	

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

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

Oak Ridge National Laboratory (ORNL) is one of the verification organizations operating under the Advanced Monitoring Systems (AMS) Center. AMS, which is administered by EPA's National Exposure Research Laboratory (NERL), is one of six technology areas under ETV. In this verification test, ORNL evaluated the performance of lead in dust wipe measurement technologies. This verification statement provides a summary of the test results for Monitoring Technologies International's (MTI) PDV 5000 Trace Metal Analyzer.

## VERIFICATION TEST DESCRIPTION

This verification test was designed to evaluate technologies that detect and measure lead in dust wipes. The test was conducted at the Capitol Community Technical College in Hartford, CT, from November 5 through November 9, 2001. The vendors of commercially-available, field portable technologies blindly analyzed 160 dust wipe samples containing known amounts of lead, ranging in concentration from  $\leq 2$  to 1,500  $\mu\text{g/wipe}$ . The experimental design was particularly focused on important clearance standards, such as those identified in 40 CFR Part 745.227(e)(8)(viii) of 40  $\mu\text{g/ft}^2$  for floors, 250  $\mu\text{g/ft}^2$  for window sills, and 400  $\mu\text{g/ft}^2$  for window troughs. The samples included wipes newly-prepared and archived from the Environmental Lead Proficiency Analytical Testing Program (ELPAT). These samples were prepared from dust collected in households in North Carolina and Wisconsin. Also, newly-prepared samples were acquired from the University of Cincinnati (UC). The UC dust wipe samples were prepared from National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs). The results of the lead analyses generated by the technology were compared with results from analyses of similar samples by conventional laboratory methodology in a laboratory that was recognized as proficient by the National Lead Laboratory Accreditation Program (NLLAP) for dust testing. Details of the test, including a data summary and discussion of results, may be found in the report entitled *Environmental Technology Verification Report: Lead in Dust Wipe Detection Technology—Monitoring Technologies International, PDV 5000 Trace Metal Analyzer*, EPA/600/R-02/060.

## TECHNOLOGY DESCRIPTION

MTI's PDV 5000, a field portable instrument, is a self-contained anodic stripping analyzer. Anodic Stripping Voltammetry (ASV) works by electroplating metals in solution onto an electrode. This concentrates the metal. The metals on the electrode are then sequentially stripped off, which generates a current that can be measured. The current (milliamps) is proportional to the amount of metal being stripped off. The potential (voltage in millivolts) at which the metal is stripped off is characteristic for each metal. This means the metal can be identified as well as quantified. The PDV 5000's reporting limits during this verification test was  $< 20 \mu\text{g/wipe}$ .

## VERIFICATION OF PERFORMANCE

The following performance characteristics of the PDV 5000 were observed:

**Precision:** Precision, based on the average percent relative standard deviation (RSD), 22% for the ELPAT samples and 21% for the UC samples, excluding two outlier values. Both values are above the level of acceptable precision ( $< 20\%$  average relative standard deviation).

**Accuracy:** Accuracy was assessed using the estimated concentrations of the UC and ELPAT samples. The average percent recovery value for all samples reported above 30  $\mu\text{g/wipe}$  was 87% for the UC samples and 93% for the ELPAT samples. The range of percent recoveries values was from 35% to 137%. This negative bias is statistically significant, but the average value is within the acceptable bias range of  $100\% \pm 25\%$ . For the NLLAP laboratory results, the average percent recovery values were 91% and 98%, respectively, for the UC and ELPAT samples. The negative bias for both the UC and ELPAT samples was statistically significant.

**Comparability:** A comparison of the PDV 5000 results and the NLLAP-recognized laboratory results was performed for all samples (UC and ELPAT) that were reported above 30  $\mu\text{g/wipe}$ . The correlation coefficient ( $r$ ) for the comparison to NLLAP lab results for the UC samples was 0.999 [slope ( $m$ ) = 1.074, intercept = -14.345], and for the ELPAT samples was 0.988 [ $m$  = 0.885, intercept = 15.633]. While the slopes for both the UC and ELPAT samples were statistically different than 1.00, correlation coefficients greater than 0.990 indicate a strong linear agreement with the NLLAP laboratory data.

**Detectable blanks:** All twenty samples, prepared at concentrations  $< 2 \mu\text{g/wipe}$ , were reported correctly as  $< 20 \mu\text{g/wipe}$  by the PDV 5000. Performance was also assessed at levels near the reporting limits of  $20 \mu\text{g/wipe}$ . The instrument reported the eight samples near  $17 \mu\text{g/wipe}$  as  $< 20 \mu\text{g/wipe}$ , but the four samples around  $30 \mu\text{g/wipe}$  were reported as  $< 20$ ,  $< 20$ ,  $24$ , and  $25$ .

**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 PDV 5000 reported four of a possible 29 results as false positives, while the NLLAP laboratory did not report any false positives. For the ELPAT samples, the PDV 5000 reported three of a possible 12 fp results and the NLLAP laboratory reported two of 12.

**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 PDV 5000 reported 17 of 29 possible fn results, while the NLLAP laboratory reported 23 of 30 fn results. For the ELPAT samples, the PDV 5000 reported 12 of a possible 28 fn results, while the NLLAP laboratory reported 7 of 12.

**Completeness:** Completeness is defined as the percentage of measurements that are judged to be usable i.e., the result is not rejected). An acceptable completeness rate is 95% or greater. The PDV 5000 generated results for all 160 dust wipe samples. However, two results for UC samples were reported as non-detects for sample concentrations of 40 and  $250 \mu\text{g/wipe}$ . These were considered outliers and excluded from the data analysis. Therefore, completeness was 99%.

**Sample Throughput:** Two analysts (one expert and one novice analyst) each operated their own instrument, with the expert running odd-numbered samples and the novice analyzing the even-numbered samples. The analysts completed the analysis of 160 samples over the course of three days. The first day was spent setting up, training the novice, and running approximately 32 samples. On the second day (a 14-hour day), 130 samples were analyzed. The data was checked and transposed onto the results sheets on the third day. The MTI team spent a total of about 18 hours analyzing the samples.

**Overall Evaluation:** The overall performance was characterized as having an acceptable amount of negative bias, larger than acceptable precision, but in good linear agreement with an NLLAP-recognized laboratory's data. The verification team found that the PDV 5000 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 where required) need to be followed. For more information on this and other verified technologies, visit the ETV web site at <http://www.epa.gov/etv>.

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

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

**NOTICE:** EPA verifications are based on evaluations of technology performance under specific, predetermined criteria and appropriate quality assurance procedures. EPA and ORNL make no expressed or implied warranties as to the performance of the technology and do not certify that a technology will always operate as verified. The end user is solely responsible for complying with any and all applicable federal, state, and local requirements. Mention of commercial product names does not imply endorsement or recommendation.

# **Environmental Technology Verification Report**

## **Lead in Dust Wipe Measurement Technology**

### **Monitoring Technologies International PDV 5000 Trace Metal Analyzer**

By

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

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

## **Notice**

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

# Table of Contents

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

False Positive/False Negative Results .....	19
Completeness .....	22
Sample Throughput .....	22
Ease of Use .....	22
Cost Assessment .....	22
PDV 5000 Costs .....	23
Labor .....	23
Laboratory Costs .....	23
Sample Shipment .....	23
Labor, Equipment, and Waste Disposal .....	24
Cost Assessment Summary .....	24
Miscellaneous Factors .....	24
Summary of Performance .....	24
Section 6 — References .....	26
Appendix .....	27

## List of Figures

1. MTI's PDV 5000 system. . . . .	2
2. Distribution of both UC and ELPAT sample concentrations. Four samples were analyzed at each concentration level . . . . .	7
3. Plot of DataChem reported values versus estimated values, shown for concentrations less than 500 $\mu\text{g/wipe}$ . . . . .	12
4. False negative probabilities for DataChem average concentrations at a target concentration level of 40 $\mu\text{g/wipe}$ . . . . .	14
5. False negative probabilities for DataChem average concentrations at a target concentration level of 250 $\mu\text{g/wipe}$ . . . . .	15
6. False negative probabilities for DataChem average concentrations at a target concentration level of 400 $\mu\text{g/wipe}$ . . . . .	15
7. Plot of the PDV 5000 average concentrations versus DataChem's average concentrations, for both UC and ELPAT samples ( $n=20$ ), shown for concentrations less than 500 $\mu\text{g/wipe}$ . . . . .	19
8. Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 40 $\mu\text{g/wipe}$ . . . . .	20
9. Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 250 $\mu\text{g/wipe}$ . . . . .	21
10. Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 400 $\mu\text{g/wipe}$ . . . . .	21

## List of Tables

1. Summary of DataChem Pre-Test Results .....	11
2. Summary of DataChem Percent Recovery Values by Sample Source .....	12
3. Summary of DataChem Precision Estimates by Sample Source .....	12
4. False Positive/False Negative Results for DataChem Measurements of UC Samples .....	14
5. Summary of the Linear Regression Constants and Recovery Data for DataChem's Measurements Versus the Estimated Concentrations at the Clearance Levels .....	16
6. Precision of the PDV 5000 Analyzer .....	17
7. Accuracy of PDV 5000 Analyzer .....	17
8. Linear regression constants for the plots of the PDV 5000 versus the estimated values and versus the DataChem average measurements .....	18
9. False Positive/False Negative Results for PDV 5000 Measurements of UC Samples .....	20
10. Summary of the Linear Regression and Recovery Data for the PDV 5000 Response versus the Estimated Concentrations .....	22
11. Estimated analytical costs for lead dust wipe samples .....	23
12. Performance Summary for the PDV 5000 System .....	25

## Acknowledgments

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

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

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

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

For more information on MTI's PDV 5000, contact:

Colin Green  
Monitoring Technologies International  
29 Chinthurst Park  
Shalford Surrey  
GU48JH United Kingdom  
phone: 441-483-564183  
[cgreen@colingreen.idps.co.uk](mailto:cgreen@colingreen.idps.co.uk)

Felecia Owen  
Owen Scientific  
1609 Ebb Drive  
Wilmington, NC 28409  
phone: 910-391-5714  
[fowen@owenscientific.com](mailto:fowen@owenscientific.com)

## Abbreviations and Acronyms

AIHA	American Industrial Hygiene Association
AMS	Advanced Monitoring Systems Center, ETV
ASTM	American Society for Testing and Materials
ASV	Anodic Stripping Voltammetry
CDC	Centers for Disease Control and Prevention
CFR	Code of Federal Regulations
CL	clearance level of 40, 250, or 400 $\mu\text{g/wipe}$
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
MTI	Monitoring Technologies International
NERL	National Exposure Research Laboratory, U.S. EPA
NIST	National Institute of Standards & Technology
NLLAP	National Lead Laboratory Accreditation Program, U.S. EPA
OPPT	Office of Pollution Prevention and Toxics, U.S. EPA
ORNL	Oak Ridge National Laboratory
QA	quality assurance
QC	quality control
RSD	relative standard deviation
RTI	Research Triangle Institute
SD	standard deviation
SRM	Standard Reference Material
UC	University of Cincinnati

## Section 1 — Introduction

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

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

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

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

The program now operates six centers covering a broad range of environmental areas. ETV began with a 5-year pilot phase (1995–2000) to test a wide range of partner and procedural alternatives in various technology areas, as well as the true market demand for and response to such a program. In these centers, EPA utilizes the expertise of partner “verification organizations” to design efficient processes for conducting performance tests of innovative technologies. These expert partners are both public and private organizations, including federal laboratories, states, industry consortia, and private sector entities. Verification organizations oversee and report verification activities based on testing and QA protocols developed with input from all major stakeholder/customer groups associated with the technology area. The verification described in this report was administered by the Advanced Monitoring Technology (AMT) 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 AMT Center is administered by EPA’s National Exposure Research Laboratory (NERL), Environmental Sciences Division, in Las Vegas, Nevada.

The verification of a field analytical technology for measurement of lead in dust wipe samples is described in this report. The verification test was conducted in Hartford, Connecticut, from November 5 through November 9, 2001. The performance of the Monitoring Technologies International (MTI)’s PDV 5000 trace metal analyzer, an anodic stripping voltammetry system, 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. For background information, additional information on anodic stripping voltammetry for dust wipe analysis can be found in other published reports (Ashley et al., 2001).

## 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

MTI's PDV5000 is a self-contained anodic stripping analyzer. Anodic stripping voltammetry (ASV) works by electroplating metals in solution onto an electrode. This concentrates the metal. The metals on the electrode are then sequentially stripped off, which generates a current that can be measured. The current (milliamps) is proportional to the amount of metal being stripped off. The potential (voltage in millivolts) at which the metal is stripped off is characteristic for each metal. This means the metal can be identified as well as quantified.

The PDV 5000 system is a new type of three-electrode device. Instead of liquid mercury as the electrode, this device uses a glassy carbon electrode that is plated with a very thin film of mercury (mercury thin film electrode, MTFE). This is carried out at the beginning of an analytical run and lasts for between 10 and 30 subsequent analyses. The mercury is contained as a salt in the supporting buffer used. This means only a very small amount of mercury is used and ensures the operator never comes into contact with liquid mercury. The amount of mercury used per analysis is measured in parts per billion. If however the analysis is for arsenic, selenium or mercury, the glassy carbon electrode is given a gold film. Ease of use has been the primary objective in the design. A simple, menu driven software allows the user to select the metal and concentration range of interest. Analysis time is dependent on the metal concentration, but ranges from a few minutes to 20 minutes for an ultra low concentration. The initial calibration can be used for many subsequent "unknown" analyses without the need for recalibration.

### Sample Preparation and Analysis

During the verification test, MTI performed the following procedure. Fifteen mL of 2M hydrochloric acid was added to each 20-mL scintillation vial which contained a dust wipe sample. The vials were sonicated in a water bath



Figure 1. MTI's PDV 5000 system.

for 20 min. After sonication, 400  $\mu\text{L}$  of sample was added to 20 mL of electrolyte solution, which was an aqueous solution of sodium chloride, sodium acetate, and acetic acid. The electrode was placed in the same, the "run" button was pushed, and the result was produced in 120 s. Every fifth analysis was a calibration check. If the calibration check standard was more than 50% different than the expected value, the calibration was re-done and the last sample run before the calibration check was reanalyzed.

### Theory of Operation

The liquid sample is added to the supporting electrolyte (buffer) to ensure the oxidation states of the metal ions are optimized for electrochemistry. This also dilutes the sample, which removes many of the potentially interfering compounds. Another component of the buffer removes any dissolved oxygen in the sample that would interfere with the analysis.

The analysis proceeds by initially plating the working electrode with mercury or gold. Several quick runs with a standard are performed to stabilize the mercury or gold film and to confirm the analyzer is working correctly. Each film lasts between 10 and 30 subsequent analyses. The diluted sample is then added to the cell and the

working electrode is given a negative potential relative to the reference electrode. The value can be varied depending on which metals are to be analyzed. The negative potential attracts the positive metal ions to it, where electrons combine with the metal ions to produce the metal. The use of the mercury film enhances the process as when the metal ion is reduced to the metallic state, it forms an amalgam with the mercury, which stabilizes it during the stripping phase. Mercury on glassy carbon also has a high over potential relative to hydrogen. This means the potential can be set that allows metals such as zinc to be plated onto the electrode, without producing hydrogen gas. Hydrogen is very reducing and will interfere with the subsequent stripping. The potential is then held for around 60 seconds (up to 300 in some applications) while the metal ions accumulate on the electrode, effectively concentrating the sample.

During the plating process, the sample is mixed at high speed. This ensures that the metal ion concentration at the electrode/sample interface is the same as the concentration in the bulk sample. This also helps prevent a capacitive buildup on the electrode where a layer of positive ions shield the negative electrode from other ions in the sample. By ensuring the negative potential is the dominant factor during the analysis, the reproducibility of the analysis is dramatically improved. An added bonus is the complex mathematical formula used to calculate the amount of metal deposited for a given time at a given potential is simplified.

The potential is then allowed to become less negative and the metals re-oxidize (or are stripped from the electrode), which generates electrons. Each metal will strip from the electrode at a specific potential, which allows for identification of a metal. The rate at which the potential is changed is called the sweep rate and is another variable that can be altered to optimize an analysis. The faster the sweep rate (mV/sec), the better the resolution. However, sensitivity is lowered because, at high sweep rates, the metals on the electrode have a much shorter time to strip off, giving less chance for the peaks to overlap. A slow sweep rate allows more metal to strip off, giving a larger signal, but conversely increases the noise on the baseline, potentially masking the metal of interest. By applying different waveforms to the sweep, stripping potentials can be shifted, which is useful when 2 metals of interest strip at a similar potential.

The generation of electrons is measured by the counter electrode as a current produced in the cell. The current in micro- or nano-amps is proportional to the metal concentration on the electrode. As each metal strips from the electrode, a graph is produced showing a series of peaks corresponding to current (metal concentration) at specific potentials. By selecting a potential “window” where a specific metal is expected to appear, ASV can be used to identify and quantify the metal concentration in the sample.

The instrument can be used to detect and quantify other metals (As, Cd, Cr, Cu, Au, Fe Mn, Hg Ni, Ag, Sn, Zn, Co), but the performance of the instrument was only verified for lead in this test.

## Section 3 — Verification Test Design

### Objective

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

### Testing Location and Conditions

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

### Drivers and Objectives for the Test

The purpose of this test was to evaluate the performance of field analytical technologies that are capable of analyzing dust wipe samples for lead contamination. This test provides information on the potential applicability of field technologies to EPA standards for dust clearance testing. The experimental design was designed around the three clearance standards of 40  $\mu\text{g}/\text{ft}^2$  for floors, 250  $\mu\text{g}/\text{ft}^2$  for window sills, and 400  $\mu\text{g}/\text{ft}^2$  for window troughs that are outlined in 40 CFR Part 745.227(e)(8)(viii) (CFR, 2001).

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

### Summary of the Experimental Design

All of the samples analyzed in this verification test were prepared gravimetrically. At the time of the test, both of the wipes utilized in the test (PaceWipe™ and Aramsco LeadWipe™) were on the list of wipes recommended for lead testing by the American Society for Testing and Materials (ASTM, 1996). Initial consideration was given to conducting the test in a real-world situation, where the technologies would have been deployed in a housing unit that had been evacuated due to high levels of lead contamination. In addition to the safety concern of 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 these types of technologies, thereby making it difficult to separate instrument/method variability and sampling variability. The availability of well-characterized samples derived from “real-world” situations 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 over one-hundred 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  $\mu\text{m}$ . 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  $0.1000 \pm 0.0005$  g portion of dust on weighing paper, the pre-weighed dust was gently tapped out onto the PaceWipe. The wipe was then folded and placed in a plastic vial, which was then capped. All vials containing the spiked wipes were stored in a cold room as a secondary means of retarding mold growth until shipment.

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

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

### **University of Cincinnati Sample Description**

The ELPAT samples consisted of dust mounded in the center of a PaceWipe. The University of Cincinnati (UC) prepared “field QC samples” where the dust was sprinkled over the wipe, more similar to how a wipe would look when a dust wipe sample is collected in the field. In a typical scenario, UC sends these control samples to a laboratory along with actual field-collected samples as a quality check of the laboratory operations. Because the samples are visually indistinguishable from an actual field sample, are prepared on the same wipe, and are shipped in the same packaging, the laboratory blindly analyzes the control samples. This 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. For MTI, the UC samples were prepared on Aramsco LeadWipes™. The UC wipe samples were prepared using National Institute of Standards & Technology (NIST) Standard Reference Materials (SRMs). NIST SRM 2711 was used to prepare the 40  $\mu\text{g/wipe}$  samples, and NIST SRM 2710 was used to prepare the 250 and 400  $\mu\text{g/wipe}$  samples. Both SRM 2711 and SRM 2710 are Montana Soil containing trace concentrations of multiple elements, including lead. Some NIST SRM materials that are spiked on dust wipes are known to have low extraction recoveries when prepared by standard analytical methods (e.g., lead silicates cannot be extracted unless hydrofluoric acid is used) (Ashley et al., 1998). These particular SRMs are not known to contain lead silicates or to give lower lead recoveries. However, it is important to note the possibility of such when using NIST SRMs for lead dust wipe analysis, since similar SRMs (e.g., Buffalo river sediment from Wyoming) do show recoveries in the low 90% range (Ashley et al., 1998).

Because accurate and precise estimated concentrations for the UC samples were imperative, ORNL imposed the following data quality requirements for the UC-prepared wipe samples: 1) each estimated concentration had to be within a  $\pm 10\%$  interval of the target clearance level; 2) additional quality control (QC) samples (at least 5% of the total samples ordered) were to be prepared and analyzed by UC as a quality check prior to shipment of the samples; and 3) the relative standard deviation of the QC samples had to be  $\leq 10\%$ . It is important to note here the reason why the data quality requirements between the UC and ELPAT samples were different. The data quality requirements for the ELPAT samples (i.e.,  $\pm 20\%$  of the target value) were established by the ELPAT program. Since archived samples were being used, 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 for Method 3050B and Method 6010B) and provided those results to ORNL. For the 24 samples (eight at each of the three clearance levels), the average percent recovery (i.e., UC measured concentration/UC estimated concentration  $\times 100\%$ ) was 97% (median value = 96%, standard deviation = 3%, range = 93% to 102%). (102%), but both values within the data quality r Additionally, 42 randomly-selected samples (14 at each of the three clearance levels) were analyzed by the EPA Region 1 laboratory in North Chelmsford, MA, as an independent quality control check of the accuracy and precision of UC's sample preparation procedure (nitric acid digestion followed by ICP/AES analysis - see EPA, 1996). The average percent recovery (EPA Region 1 reported concentration/UC estimated concentration  $\times 100\%$ ) was 90% (median 89%, standard deviation = 2%), with a range of values from 86% to 93%. The average recovery determined from the EPA Region 1 analyses (90%) was lower than that which was determined by UC (102%), but both values met the data quality requirement of  $100 \pm 10\%$ . Based on these data, ORNL determined that

the UC sample preparation process met the established data quality criteria and was deemed acceptable for use in the determination of false positive/false negative error rates.

### **Distribution and Number of Samples**

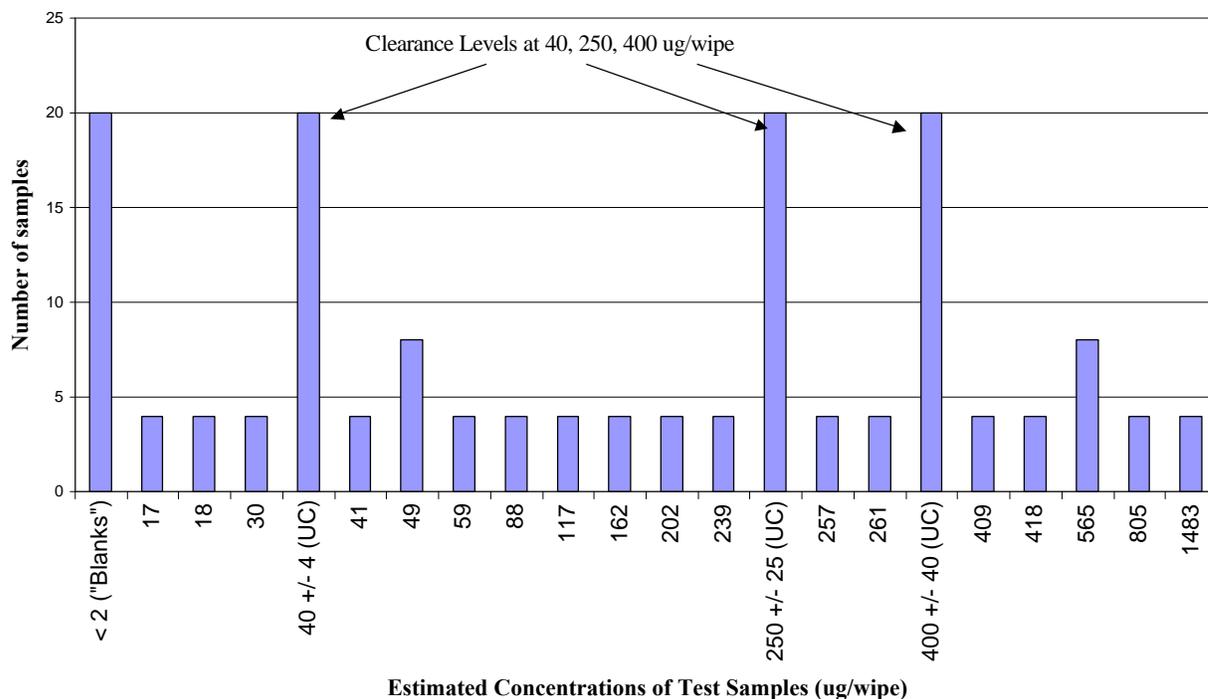
A total of 160 samples were analyzed in the verification test. Figure 2 is a plot containing the distribution of the sample concentrations that were analyzed in this study. Twenty samples were prepared by the University of Cincinnati at  $\pm 10\%$  of each of the three clearance levels (3 test levels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

### **Sample Randomization**

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

### **Description of Performance Factors**

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



**Figure 2. Distribution of concentration levels.**

### ***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:

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

The overall RSD is characterized by two summary values:

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

The average RSD may not be the best representation of precision, but it is reported for convenient reference. An average RSD value less than 10% indicates very precise measurements. RSDs greater than 20% should be viewed as indicators of larger variability and possibly non-normal distributions.

### ***Accuracy***

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

Accuracy of the field technology measurements was statistically tested using t-tests or non-parametric tests at the 5% significance level. These statistical tests compared the average results with the overall estimated values using the precision of the sample

measurements. Bias was quantified by computing the percent recovery for four similar samples or a single sample using the equation:

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

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

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

### ***Comparability***

Comparability refers to how well the field technology and 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). Acceptable *r* values are 0.990 or greater. The slope of the linear regression line, denoted by the letter *m*, is related to *r*. Whereas *r* represents the linear association between the vendor and laboratory concentrations, *m* quantifies the amount of change in the vendor’s measurements relative to the laboratory’s measurements. A value of +1 for the slope indicates perfect agreement. Values greater than 1 indicate that the vendor results are generally higher than the laboratory, while values less than 1 indicate that the vendor results are usually lower than the laboratory.

### ***Detectable Blanks***

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

### ***False Positive/Negative Results***

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

A primary objective for this verification test was to assess the performance of the technology at each of the three clearance levels of 40, 250, and 400  $\mu\text{g/wipe}$ , and estimate the probability of the field technology reporting a fp or fn result. For each clearance level, the probabilities of fn were estimated as curves that depend on a range of

concentrations reported about the clearance level. These error probability curves were calculated from the results on the 60 UC samples at concentrations  $\pm 10\%$  of each clearance level. In order to generate probability curves to model the likelihood of false negative results, it was assumed that the estimated concentration provided by UC was the true concentration. However, this evaluation did not include the gravimetric preparation uncertainty in the UC estimated concentration. This error is likely to be much smaller than other sources of measurement error (e.g., extraction efficiency and analytical).

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

### ***Completeness***

Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result is not rejected). An acceptable completeness is 95% or greater.

### ***Sample Throughput***

Sample throughput is a measure of the number of samples that can be processed and reported by a technology in a given period of time. Sample throughput is reported in Section 5 as number of samples per day per number of analysts.

### ***Ease of Use***

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

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

### ***Cost***

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

### ***Miscellaneous Factors***

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

## Section 4 — Laboratory Analyses

### Background

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

### NLLAP Laboratory Selection

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

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

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

### Laboratory Method

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

**Table 1.** Summary of DataChem Pre-Test Results

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

### Laboratory Performance

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

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

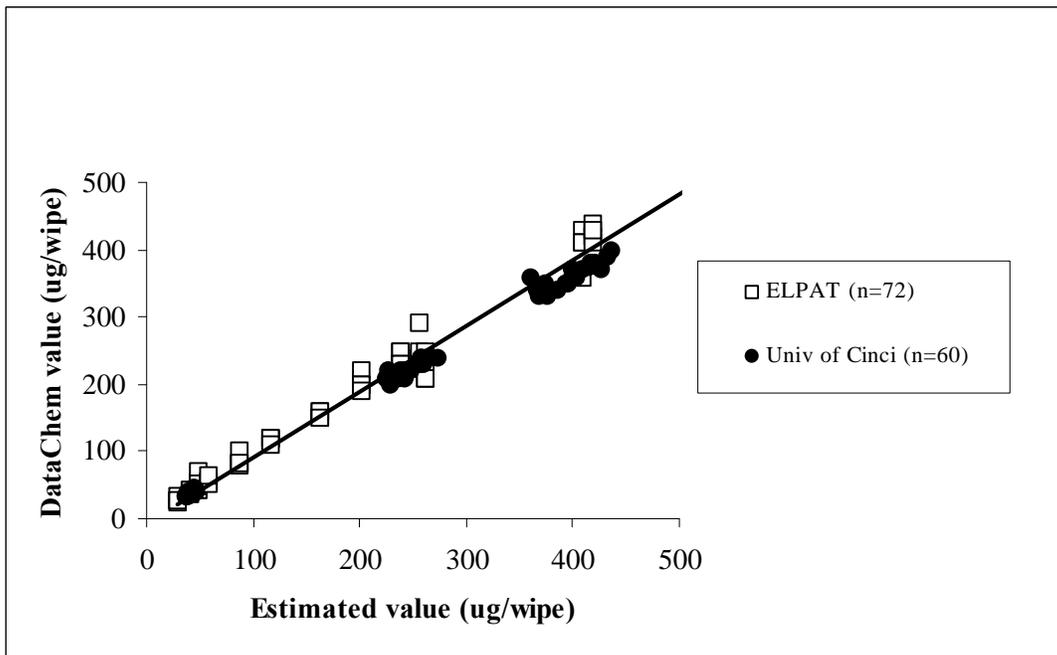
The negative bias observed with the UC and the ELPAT samples was statistically significant. The cause of the negative bias for the UC samples could be related to: 1) extraction inefficiencies (due to the use of NIST SRMs that contain lead that is unrecoverable with the extraction procedure which was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

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

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

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

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



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

**Table 3.** Summary of DataChem Precision Estimates by Sample Source

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

<sup>a</sup> 4 replicates in each sample set

<sup>b</sup> 20 replicates in each sample set

<sup>c</sup> This value represents the variability in the sample preparation process.

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

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

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

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

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

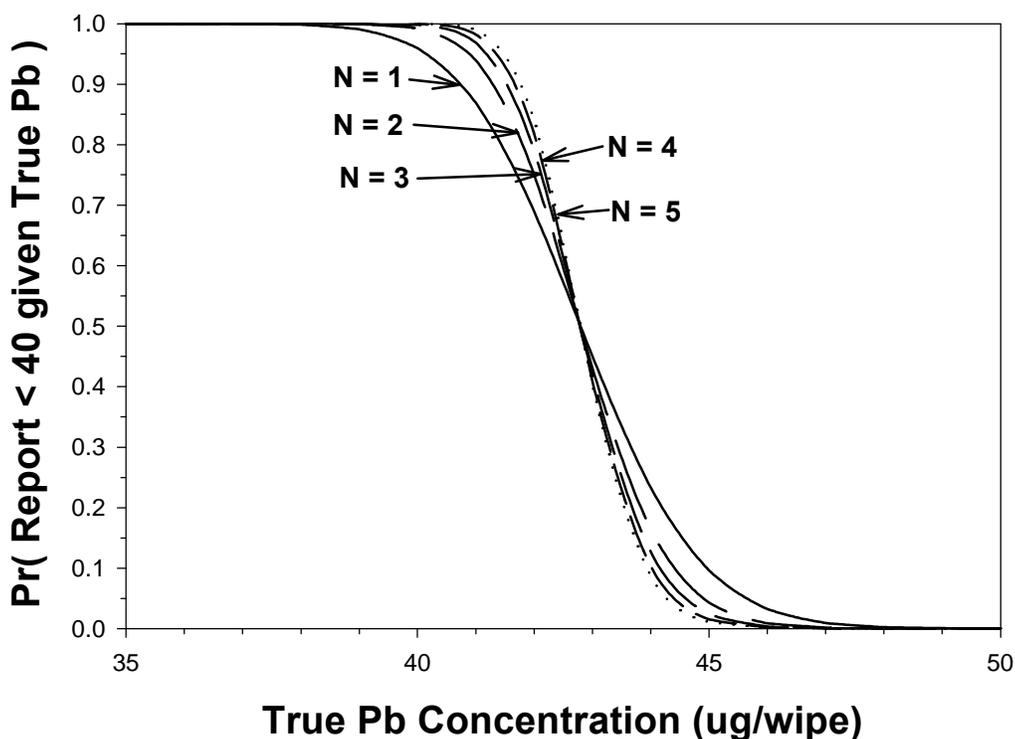
user perform their own assessment of the method's performance by including samples of known concentration (at or near the clearance levels) along with the analysis of "real-world" samples.

**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 <sup>a</sup> 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 <sup>b</sup>	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

<sup>a</sup> CL = clearance level

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



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

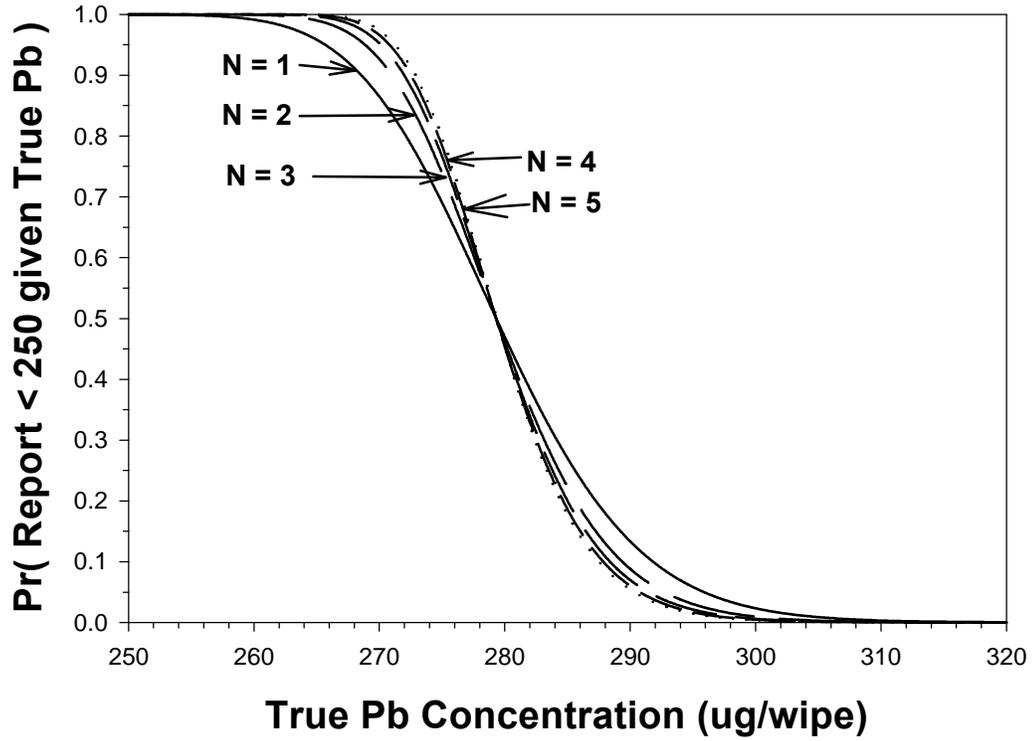


Figure 5. False negative probabilities for DataChem average concentrations at a target concentration level of 250 µg/wipe.

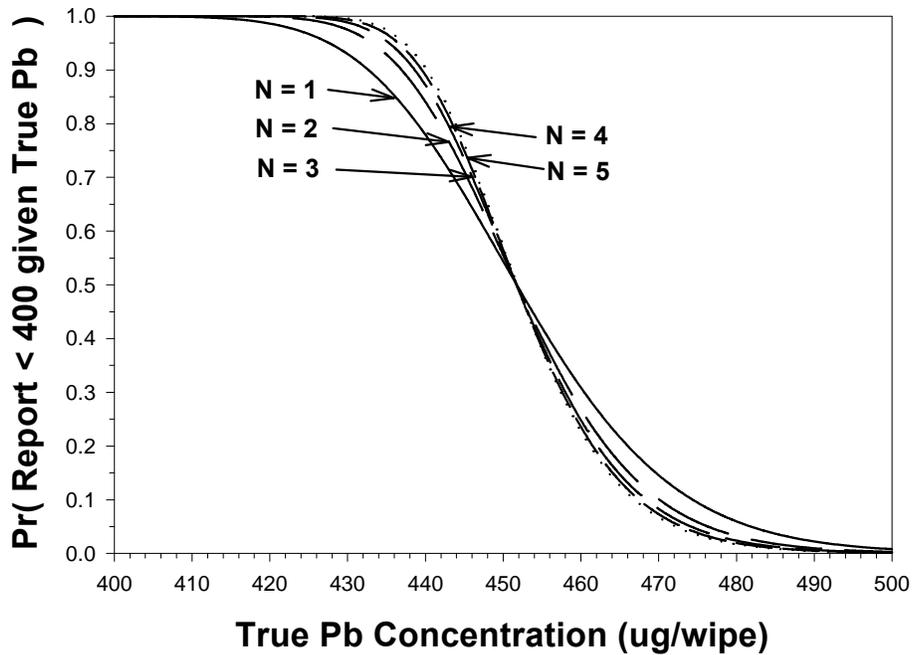


Figure 6. False negative probabilities for DataChem average concentrations at a target concentration level of 400 µg/wipe.

**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	0.840	0.879	0.549	0.861	0.492
average % recovery	93%	101%	90%	96%	91%	100%
SD of % recovery	4%	13%	3%	9%	3%	5%
Reported concentration at the CL	37 µg/wipe	40 µg/wipe	226 µg/wipe	234 µg/wipe	362 µg/wipe	382 µg/wipe

## Section 5 — Technology Evaluation

### Objective and Approach

The purpose of this section is to present a statistical evaluation of the PDV 5000 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 PDV5000.

### 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 30 µg/wipe (see “*Detectable Blanks*” section below for explanation of why sample concentrations below 30 µg/wipe were not included). For the ELPAT samples, precision was measured on each set of four samples from a particular round of archived samples. For the 17 sets of samples, the PDV 5000's average RSD value was 22%, with a range from 6 to 44%, indicating that the PDV 5000's precision was higher than acceptable levels of variability of < 20% stated in Section 3. For the UC samples, 20 samples were analyzed at each of three target concentration levels of 40, 250, and 400 µg/wipe. The average precision of the UC sample measurements by the PDV 5000 was 21% RSD (excluding two outliers where MTI reported a 40 and 250 µg/wipe samples as non-detects). With the expectation that UC was to prepare the samples as close to the target concentrations as possible, the allowable variability was 10% RSD. As presented in Table 6, the actual variability of the UC preparation process was an average of 6% RSD.

### Accuracy

Accuracy represents the closeness of the PDV 5000's measured concentrations to the estimated content of spiked samples. One measure of accuracy is the number of ELPAT results which were reported within the acceptance ranges that have been established for those samples. For the 68 ELPAT samples above 30 µg/wipe, the PDV 5000 reported

**Table 6.** Precision of the PDV 5000 Analyzer

Source	No. of sample sets	% RSD		
		average	min	max
ELPAT	17 <sup>a</sup>	22	6	44
UC	3 <sup>b</sup>	21	19	24
UC prep <sup>c</sup>	3	6	6	6

<sup>a</sup> 4 replicates in each sample set

<sup>b</sup> 19 or 20 replicates in each sample set

<sup>c</sup> precision of UC sample preparation process

54 results (79%) within the acceptance ranges (Table 7). The results reported by the PDV 5000 can also be compared to the ELPAT certificate value, i.e., the average concentration reported by 100+ laboratories who participated in previous rounds of ELPAT testing. The average percent recovery for the 68 ELPAT samples reported by the PDV 5000 was 93%, although the range of values was quite large, from 39 to 134%. The UC sample results were lower, with an average percent recovery of 87%. The possible explanations for this difference in performance include: 1) that ELPAT “estimated” values are, in fact, consensus values from a large number of laboratories that may be similar in performance to DataChem and to the PDV 5000, and 2) the reference material used to prepare the UC samples may be more challenging than the ELPAT reference material.

**Table 7.** Accuracy of PDV 5000 Analyzer

Statistic	% recovery	
	ELPAT	UC
n <sup>a</sup>	68	58
average	93	87
standard deviation	22	20
minimum	39	35
maximum	134	137

<sup>a</sup> Excludes estimated values < 30 µg/wipe and two erroneous MTI non-detect values for the UC samples.

Another way to assess accuracy is to plot the PDV 5000 results versus the estimated values that are > 30 µg/wipe. The linear regression constants for the plot of the ELPAT and UC data are listed in Table 8. As expected, the conclusions gained from this assessment are similar to the above conclusions regarding the percent recovery calculations. The samples were generally biased low, as evidenced by the slope values < 1.0 (0.964 for the UC samples and 0.875 for the ELPAT samples.). The r values (0.936 and 0.951 for the UC and ELPAT samples, respectively) indicate that the PDV 5000 results were in fair agreement with the estimated values.

### Comparability

Comparability refers to how well the PDV 5000 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 PDV 5000 results and the laboratory results was performed for all ELPAT (> 30 µg/wipe) and UC samples. Because each wipe was prepared individually, a true one-to-one matching of PDV 5000 and laboratory results could not be performed. However, the average concentrations of the samples prepared at specific levels was compared for the PDV 5000 and laboratory results. In Table 8, the regression constants for the average PDV 5000 results versus

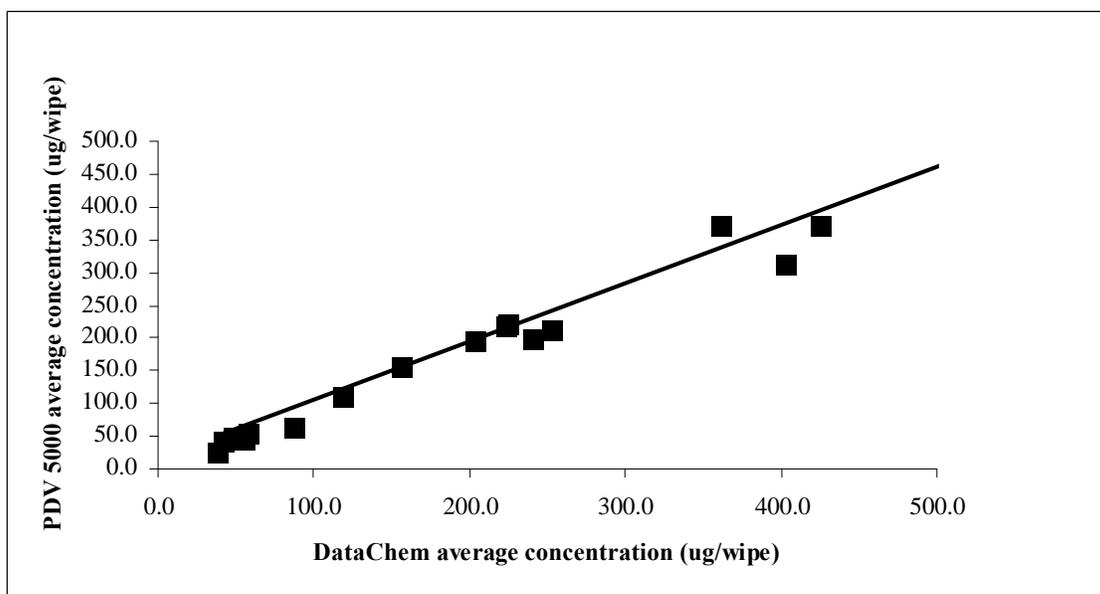
the average DataChem results for both the ELPAT and UC values are presented. The difference between the regression slopes ( $m = 0.885$  for ELPAT and  $m = 1.074$  for UC) and a slope with a perfect agreement line ( $m = 1.000$ ) is statistically significant, but the correlation coefficients ( $r = 0.988$  for ELPAT and  $r = 0.999$  for UC) show a strong linear relationship between DataChem and PDV 5000 average results. To illustrate the strong linear agreement between the PDV 5000 and NLLAP laboratory results, Figure 7 is a plot of the average PDV 5000 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 PDV 5000 correctly reported all 20 as < 20 µg/wipe, so no detectable blanks were reported. Two UC samples at 40 and 250 µg/wipe were also reported as < 20 µg/wipe. These were presumed to be sample preparation errors and excluded from the analysis as outliers. The instrument reported the eight samples near 17 µg/wipe as < 20 µg/wipe, but the four samples around 30 µg/wipe were reported as < 20, < 20, 24, and 25, indicating the reporting limits might have been closer to 30 µg/wipe. For this reason, much of the data analysis in this section considers only the data that is greater than 30 µg/wipe rather than 20 µg/wipe.

**Table 8.** Linear regression constants for the plots of the PDV 5000 versus the estimated values and versus the DataChem average measurements

Statistic	versus estimated values		versus DataChem average concentrations	
	UC	ELPAT	UC	ELPAT
n	58	68	3	17
slope (standard error)	0.964 (0.048)	0.875 (0.035)	1.074 (0.008)	0.885 (0.036)
intercept (standard error)	-7.371 (13.331)	12.790 (17.132)	-14.354 (2.068)	15.633 (17.242)
<i>r</i>	0.936	0.951	0.999	0.988



**Figure 7. Plot of the PDV 5000 average concentrations versus DataChem’s average concentrations, for both UC and ELPAT samples (n=20), shown for concentrations less than 500  $\mu\text{g/wipe}$ .**

### False Positive/False Negative Results

Similar to the evaluation described and presented in Section 4 for DataChem, the number of false negative and false positive results reported by the PDV 5000 relative to the estimated concentrations of both UC and ELPAT samples are summarized in Table 9. For the cases where the estimated concentration was less than the clearance level (CL), the PDV 5000 reported a result that was more than the CL in four of 29 UC samples and three of 12 ELPAT samples. When the estimated concentration was equal to or above the clearance level, the PDV 5000 reported many of the results as less than the clearance level (17 of 29 possible fn results for UC samples and 12 of 28 possible fn results for ELPAT samples). This finding is not surprising, since the accuracy results reported above indicated that the PDV 5000 results were negatively biased, or reported lower than the estimated values.

The distribution of the PDV 5000's fn results, and the comparison to DataChem’s fn performance, can be examined more closely using Figures 8, 9, and 10. In these figures, the two-sided 90% confidence intervals (not shown for clarity) are used to express uncertainty on the false negative curves. In Figure 8, the likelihood of false negative results for the PDV 5000 and DataChem are comparable, because both curves have probability values near 1.0

at 40  $\mu\text{g/wipe}$ . In Figure 9, it is shown that, at exactly 250  $\mu\text{g/wipe}$ , the PDV 5000 has a much lower probability of reporting a fn result (0.5 or 50% probability) than DataChem (1.0 or nearly 100%). However, the likelihood of fn results over a wide range of concentrations is much greater for the PDV 5000 than for DataChem, due to the higher variability observed in the PDV 5000 measurements. At the 400  $\mu\text{g/wipe}$  clearance level, a trend similar to the 250  $\mu\text{g/wipe}$  level is observed (Figure 10). These conclusions are further substantiated in Table 10 which contains the linear regression constants for the PDV 5000 measured concentration versus estimated concentration for the three CLs, average percent recovery values, standard deviations, and estimates of the reported PDV 5000 concentrations at the clearance levels. The PDV 5000 reported concentrations at the clearance levels using the linear regression constants are 29, 240, and 375  $\mu\text{g/wipe}$  for the UC samples. For example, this would indicate that if the PDV 5000 reported a value for a UC sample at 29  $\mu\text{g/wipe}$ , the true concentration is probably near 40  $\mu\text{g/wipe}$ . As shown in Table 5, DataChem results for the UC samples of 37, 226, and 362  $\mu\text{g/wipe}$  would correspond to true values at the CLs. These data concur with the conclusions above for the UC samples, where at a true concentration exactly at the 40  $\mu\text{g/wipe}$  CL, the PDV 5000 is more likely to produce fn results than

the NLLAP laboratory, but is less likely to do so at the upper two clearance levels. The PDV 5000 ELPAT sample results less negatively biased at the 40 µg/wipe CL, but more negatively biased at the upper two CLs.

uncertainty in assessing false positive and false negative error rates around critical action levels due to “normal” levels of variability (see 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.

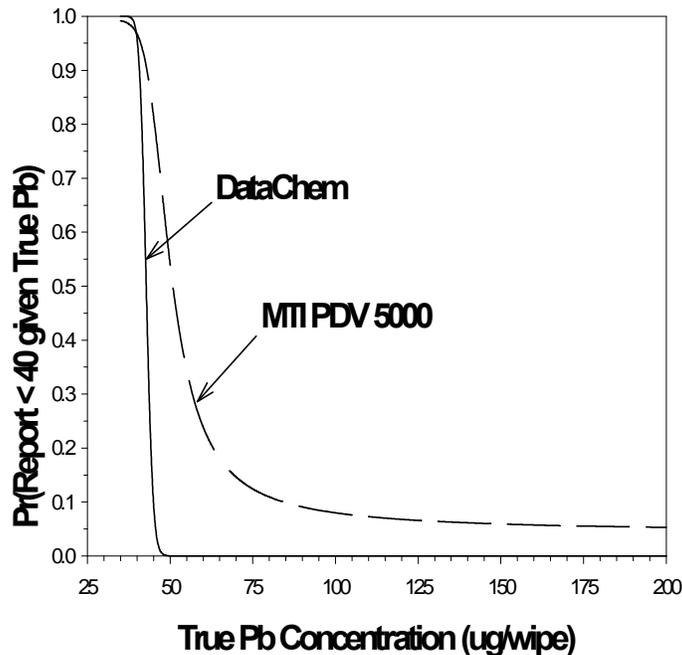
Regardless of analytical technique, there is some

**Table 9.** False Positive/False Negative Results for PDV 5000 Measurements of UC Samples

Evaluation Parameter	Sample Source	Number of Samples			Total
		40 µg/wipe	250 µg/wipe	400 µg/wipe	
fp: # samples where PDV 5000 reported the result as > CL <sup>a</sup> of the # samples where the estimated concentration was < CL	UC	0 of 10	2 of 9	2 of 10	4 of 29
	ELPAT	0 of 4	3 of 8	0 of 0 <sup>b</sup>	3 of 12
fn: # samples where PDV 5000 reported the result as < CL of the # samples where the estimated concentration was > CL	UC	8 of 9	5 of 10	4 of 10	17 of 29
	ELPAT	2 of 12	5 of 8	5 of 8	12 of 28

<sup>a</sup> CL = clearance level

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



**Figure 8.** Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 40 µg/wipe.

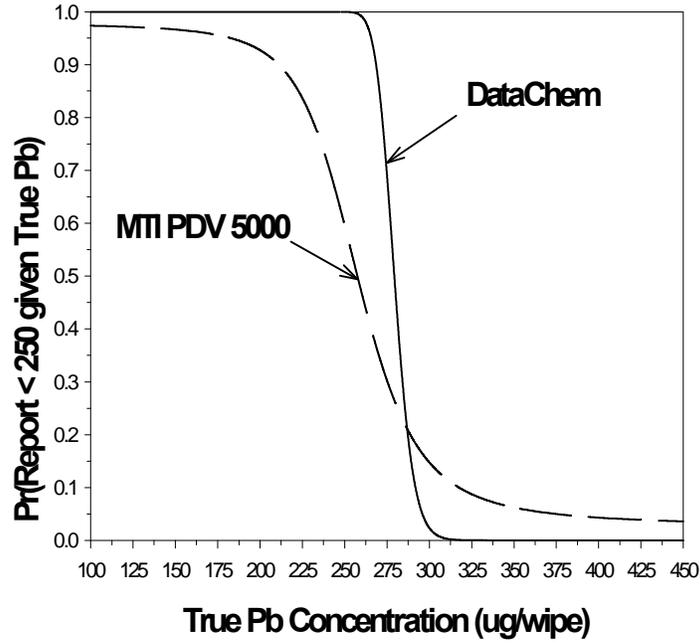


Figure 9. Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 250 µg/wipe.

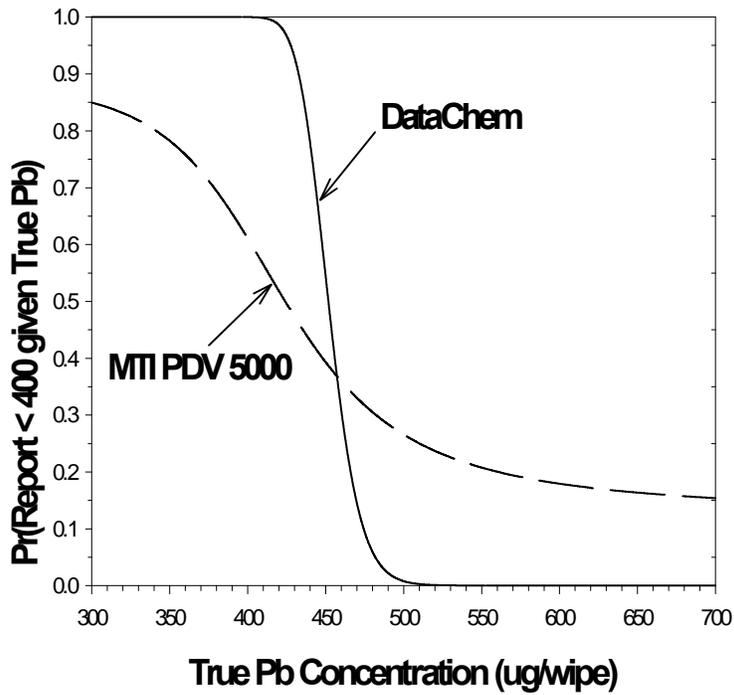


Figure 10. Comparison of the false negative probabilities for MTI PDV 5000 and DataChem at a target concentration level of 400 µg/wipe.

**Table 10.** Summary of the Linear Regression and Recovery Data for the PDV 5000 Response versus the Estimated Concentrations

Evaluation Parameter	40 µg/wipe		250 µg/wipe		400 µg/wipe	
	UC	ELPAT <sup>a</sup>	UC	ELPAT	UC	ELPAT
slope	0.997	0.626	1.3583	0.356	1.047	6.356
intercept	-10.700	18.655	-99.780	123.757	-43.847	-2284.1
correlation coefficient	0.388	0.0317	0.431	0.130	0.267	0.372
average % recovery	73%	103%	96%	88%	94%	83%
SD of % recovery	14%	17%	16%	29%	21%	21%
Reported concentration at CL	29 µg/wipe	44 µg/wipe	240 µg/wipe	213 µg/wipe	375 µg/wipe	258 µg/wipe

<sup>a</sup> Excludes four ELPAT samples at 29.8 µg/wipe because MTI report two of the four samples at non-detects.

### Completeness

Completeness is defined as the percentage of measurements that are judged to be usable (i.e., the result was not rejected). Results were reported by the technology for all 160 dust wipe samples. However, two results for UC samples were reported as non-detects for sample concentrations of 40 and 250 µg/wipe and were excluded from the data analysis. Therefore, completeness was 99% and within the acceptable completeness rate of 95% or greater.

### Sample Throughput

Sample throughput is representative of the estimated amount of time required to prepare and analyze the sample and perform the data analysis. Two analysts (one expert and one novice analyst) each operated their own instrument, with the expert running odd-numbered samples and the novice analyzing the even-numbered samples. The analysts completed the analysis of 160 samples over the course of three days. The first day was spent setting up, training the novice, and running approximately 32 samples. On the second day (a 14-hour day), 130 samples were analyzed. The data was checked and transposed onto the results sheets on the third day. The MTI team spent a total of about 18 hours analyzing the samples.

### Ease of Use

Two operators were used for the test because of the number of samples and the working conditions, but the technology can be operated by a single person. Users unfamiliar with the technology may need approximately one-half day of additional training to operate the instrument. No particular level of

educational training is required for the operator. During the test, one analyst that operated the PDV 5000 was an expert and the other was a novice.

### Cost Assessment

The purpose of this economic analysis is to estimate the range of costs for analysis of lead in dust wipe samples using the PDV 5000 and a conventional analytical laboratory method. The analysis was based on the results and experience gained from this verification test, costs provided by MTI, 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 PDV 5000 instrument and by the laboratory.

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

**Table 11.** Estimated analytical costs for lead dust wipe samples

<b>Analysis method:</b> PDV 5000	<b>Analysis method:</b> EPA SW846 6010b
<b>Analyst/manufacturer:</b> MTI	<b>NLLAP Laboratory:</b> DataChem
<b>Sample throughput:</b> 80 samples/day	<b>Actual turnaround:</b> 18 working days
<b>Cost category</b> <b>Cost (\$)</b>	<b>Cost category</b> <b>Cost (\$)</b>
<b>Sample shipment</b> 0	<b>Sample shipment</b>
	Labor                                      100–200
	Overnight shipping                      50–150
<b>Labor</b>	<b>Labor</b>
Rate                                      50–100/h per analyst	Rate                                      30 per sample
<b>Equipment</b>	<b>Equipment</b> Included <sup>a</sup>
Mobilization/demobilization      0–150	
Instrument purchase price          \$7,500	
Reagents/supplies                      \$99 per 10 samples	
<b>Waste Disposal</b> 250	<b>Waste Disposal</b> Included

<sup>a</sup> “Included” indicates that the cost is included in the labor rate.

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.

***PDV 5000 Costs***

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

**Labor**

Labor costs included on-site labor to perform the analyses. The cost of the on-site labor was estimated at a rate of \$50–100/h, depending on the required expertise level of the analyst. This cost element included the labor involved during the entire analytical process, comprising sample preparation, sample management, analysis, and reporting. If the user would have to travel to the site, the cost of mobilization and demobilization, travel, and per diem expenses should also be considered. However, in a typical application where the PDV 5000 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.

- *Instrument purchase.* The instrument can be purchased for \$7,500. This price includes the handheld analyzer with Voltscan software, patented three- electrode device, battery and charger, AC adapter, and carrying case. Leasing of the instrument is available; terms are dependent on the intended application for use. Training is provided upon instrument purchase for a nominal fee. The fee varies based on the number of trainees and the scope of the application.
- *Reagents and supplies.* The dust sample preparation kit can be purchased for \$99 for 10 tests.

***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 reference laboratory. Tasks included packing the shipping coolers, completing the chain-of-custody documentation, and completing the shipping forms. The estimate to complete this task ranged from 2 to 4 h, at \$50 per hour.

- *Overnight shipping.* The overnight express shipping service cost was estimated to be \$50 - 100 for two boxes of samples.

### **Labor, Equipment, and Waste Disposal**

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

### **Cost Assessment Summary**

An overall cost estimate for use of the PDV 5000 instrument 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 PDV 5000 instrument:

- During the test, the PDV 5000 was run using electrical power, but it does come with a rechargeable nickel metal hydride battery.
- The MTI analysts were ready for the first set of samples within 1 h of arriving on site.
- Tests with the PDV 5000 generated a 5-gal bucket full of vials containing 2M hydrochloric acid waste, which cost approximately \$250 to dispose by a commercial vendor. The waste contained a small amount of mercury (parts per billion level) from the electrode.
- Two analysts analyzed the samples using two

instruments. One analyst was an expert (the developer of the technology) and analyzed the odd-numbered samples. The other analyst, a novice who was operating the instrument for the first time, analyzed the even-numbered samples. It is possible that the experience level of the novice versus the expert operator impacted the consistency of the data. (Of the 14 ELPAT sample results that were outside of the acceptable range, 10 of these were even-numbered samples that were presumably analyzed by the novice.) This trend was not further investigated, as the vendor was informed prior to the test that ORNL would make no distinction between operators and/or instruments in the performance of the PDV 5000.

- The PDV 5000's reporting limit was 20 µg/wipe. One set of samples around 30 µg/wipe indicated that the PDV 5000 had difficulty consistently detecting lead at this concentration level. For the four samples prepared at 29.8 µg/wipe, MTI reported 24, 25, <20, and < 20 µg/wipe, indicating that these samples were near the reporting limits of the instrument. More data would need to be generated at this concentration to substantiate this conclusion.

### **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 PDV 5000 instrument 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 PDV 5000 was eighty samples per day with two analysts each operating their own instrument.

The overall performance of the PDV 5000 for the analysis of lead in dust wipe samples was characterized as biased low, but within acceptable levels of bias, having greater than acceptable levels of variability, and in good linear agreement with the average results reported by the NLLAP laboratory.

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 where required) need to be followed.

**Table 12.** Performance Summary for the PDV 5000 System

Feature/parameter		Performance summary			
		UC Samples		ELPAT Samples	
Precision : average RSD		21%		22%	
Accuracy: average % recovery		87%		93%	
Positive results on “detectable blank” samples (< 2 µg/wipe)		n/a		0 of 20 samples	
False positive results		<u>DataChem</u>	<u>PDV 5000</u>	<u>DataChem</u>	<u>PDV 5000</u>
		0 of 30	4 of 29	2 of 12	3 of 12
False negative results		<u>DataChem</u>	<u>PDV 5000</u>	<u>DataChem</u>	<u>PDV 5000</u>
		23 of 30	17 of 29	7 of 28	12 of 28
Comparison with NLLAP-recognized laboratory results (excluding < 30 µg/wipe samples)	slope	1.074		0.885	
	intercept	-14.345		15.633	
	correlation coefficient	0.999		0.988	
Overall evaluation		<ul style="list-style-type: none"> <li>- Statistically significant negative bias but within the acceptable bias range</li> <li>- Less precise than acceptable levels</li> <li>- Strong linear relationship to the NLLAP lab results</li> <li>- Few fp results</li> <li>- Higher number of fn results</li> </ul>		<ul style="list-style-type: none"> <li>- Statistically significant negative bias but within the acceptable bias range</li> <li>- Less precise than acceptable levels</li> <li>- Strong linear relationship to the NLLAP lab results</li> <li>- Few fp results</li> <li>- Higher number of fn results</li> </ul>	
Completeness		99% of 160 dust wipe samples			
Size and Weight		10 cm x 18 cm x 4 cm; 0.7 kg			
Sample throughput (2 analysts)		Average 80 samples/day 32 samples/7 hr-day (day#1); 128 samples/11-hr day (day #2); data analysis (day#3)			
Power requirements		battery operated (nickel metal hydride) or AC power			
Training requirements		One-half day instrument-specific training			
Cost		Purchase: \$7,500 Reagents/Supplies: \$99 for 10 tests			
Waste generated		5-gal bucket of vials of 2M hydrochloric acid/extracted dust wipes (Total number of samples analyzed: 160)			

## 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*. 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*. West Conshohocken, PA.
- Ashley, Kevin, Korrie Mapp, and Mark 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.
- Ashley, K., T.J. Wise, W. Mercado, and D.B. Parry. 2001. "Ultrasonic Extraction and Field-Portable Anodic Stripping Voltammetric Measurement of Lead in Dust Wipe Samples." *Journal of Hazardous Materials*. 83, p 41-50.
- 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, Ruiguang, Paul C. Schlecht, and Kevin Ashley. 2001. "Field Screening Test Methods: performance criteria and performance characteristics." *Journal of Hazardous Materials*. 83, 29-39.

## Appendix

### MTI's PDV 5000 Results Compared with Laboratory Results

Sample Analysis Order	Source	Rep	MTI PDV 5000		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
87	ELPAT	1	<20	1.3	<20	1.3
111	ELPAT	2	<20	1.3	<20	1.3
115	ELPAT	3	<20	1.3	<20	1.3
123	ELPAT	4	<20	1.3	<20	1.3
145	ELPAT	1	<20	1.3	<20	1.3
5	ELPAT	2	<20	1.3	<20	1.3
42	ELPAT	3	<20	1.3	<20	1.3
78	ELPAT	4	<20	1.3	<20	1.3
136	ELPAT	1	<20	1.3	<20	1.3
150	ELPAT	2	<20	1.3	<20	1.3
15	ELPAT	3	<20	1.3	<20	1.3
22	ELPAT	4	<20	1.3	<20	1.3
109	ELPAT	1	<20	1.3	<20	1.3
151	ELPAT	2	<20	1.3	<20	1.3
155	ELPAT	3	<20	1.3	<20	1.3
28	ELPAT	4	<20	1.3	<20	1.3
32	ELPAT	1	<20	1.3	<20	1.3
49	ELPAT	2	<20	1.3	<20	1.3
60	ELPAT	3	<20	1.3	<20	1.3
131	ELPAT	4	<20	1.3	<20	1.3
70	ELPAT	1	<20	16.9	<20	16.9
116	ELPAT	2	<20	16.9	<20	16.9
67	ELPAT	3	<20	16.9	<20	16.9
81	ELPAT	4	<20	16.9	<20	16.9
33	ELPAT	1	<20	17.6	30	17.6
9	ELPAT	2	<20	17.6	<20	17.6
68	ELPAT	3	<20	17.6	<20	17.6
69	ELPAT	4	<20	17.6	<20	17.6
101	ELPAT	1	24	29.8	33	29.8
34	ELPAT	2	25	29.8	26	29.8
6	ELPAT	3	<20	29.8	28	29.8
24	ELPAT	4	<20	29.8	28	29.8

Sample Analysis Order	Source	Rep	MTI PDV 5000		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
104	UC LAB	1	27	38.6	33	35.4
117	UC LAB	2	32	38.9	32	35.7
133	UC LAB	3	34	43.2	31	38.5
37	UC LAB	4	27	39.5	29	36.4
47	UC LAB	1	36	44.2	32	35.1
138	UC LAB	2	23	35.9	38	40.7
76	UC LAB	3	31	39.6	37	39.4
106	UC LAB	4	32	41.8	36	41.0
50	UC LAB	1	35	37.0	37	41.0
103	UC LAB	2	20	39.3	37	38.8
26	UC LAB	3	33	43.7	33	39.3
30	UC LAB	4	27	38.5	41	44.7
112	UC LAB	1	<20	40.6	32	36.0
129	UC LAB	2	40	41.3	38	44.7
134	UC LAB	3	20	40.2	30	39.9
144	UC LAB	4	22	42.5	35	37.5
110	UC LAB	1	28	38.9	36	37.4
99	UC LAB	2	24	40.8	31	36.7
90	UC LAB	3	39	42.1	34	35.8
147	UC LAB	4	27	37.0	34	39.7
94	ELPAT	1	30	41.3	37	41.3
43	ELPAT	2	51	41.3	42	41.3
53	ELPAT	3	52	41.3	44	41.3
149	ELPAT	4	45	41.3	41	41.3
119	ELPAT	1	55	49.0	43	49.0
65	ELPAT	2	48	49.0	52	49.0
154	ELPAT	3	49	49.0	49	49.0
105	ELPAT	4	50	49.0	48	49.0
137	ELPAT	1	45	49.1	70	49.1
55	ELPAT	2	57	49.1	54	49.1
18	ELPAT	3	38	49.1	48	49.1
124	ELPAT	4	53	49.1	44	49.1
121	ELPAT	1	54	58.6	64	58.6
95	ELPAT	2	61	58.6	55	58.6
132	ELPAT	3	57	58.6	56	58.6
122	ELPAT	4	51	58.6	52	58.6

Sample Analysis Order	Source	Rep	MTI PDV 5000		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
12	ELPAT	1	48	88.0	82	88.0
54	ELPAT	2	63	88.0	83	88.0
39	ELPAT	3	89	88.0	79	88.0
10	ELPAT	4	59	88.0	100	88.0
36	ELPAT	1	112	117.0	120	117.0
89	ELPAT	2	77	117.0	120	117.0
74	ELPAT	3	133	117.0	120	117.0
114	ELPAT	4	130	117.0	110	117.0
79	ELPAT	1	130	162.3	150	162.3
85	ELPAT	2	163	162.3	160	162.3
128	ELPAT	3	185	162.3	150	162.3
7	ELPAT	4	147	162.3	160	162.3
98	ELPAT	1	78	201.6	200	201.6
44	ELPAT	2	270	201.6	190	201.6
3	ELPAT	3	255	201.6	200	201.6
141	ELPAT	4	188	201.6	220	201.6
152	ELPAT	1	278	239.0	230	239.0
107	ELPAT	2	219	239.0	250	239.0
62	ELPAT	3	199	239.0	250	239.0
108	ELPAT	4	115	239.0	230	239.0
102	UC LAB	1	273	263.3	210	244.0
25	UC LAB	2	224	244.5	250	274.4
64	UC LAB	3	308	250.0	230	252.8
16	UC LAB	4	212	240.6	230	258.9
143	UC LAB	1	179	252.3	200	241.7
84	UC LAB	2	151	226.3	240	274.9
148	UC LAB	3	222	253.4	210	244.5
130	UC LAB	4	219	225.7	210	236.2
125	UC LAB	1	240	273.8	220	244.0
139	UC LAB	2	306	263.3	220	242.3
127	UC LAB	3	183	233.5	230	260.0
31	UC LAB	4	306	246.7	170	228.5
17	UC LAB	1	219	226.8	190	242.3
91	UC LAB	2	242	241.2	210	267.2
27	UC LAB	3	231	268.9	210	236.2
66	UC LAB	4	258	245.1	250	275.5
160	UC LAB	1	253	250.0	220	262.2
126	UC LAB	2	205	258.9	210	226.3
38	UC LAB	3	<20	261.1	210	227.4
4	UC LAB	4	282	253.9	220	243.4

Sample Analysis Order	Source	Rep	MTI PDV 5000		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
51	ELPAT	1	215	256.7	290	256.7
75	ELPAT	2	276	256.7	240	256.7
88	ELPAT	3	106	256.7	230	256.7
13	ELPAT	4	258	256.7	250	256.7
23	ELPAT	1	182	260.8	220	260.8
52	ELPAT	2	169	260.8	250	260.8
72	ELPAT	3	244	260.8	210	260.8
41	ELPAT	4	291	260.8	210	260.8
45	UC LAB	1	474	436.5	320	377.8
40	UC LAB	2	436	390.0	360	395.0
1	UC LAB	3	351	383.4	350	399.4
80	UC LAB	4	145	416.6	340	385.0
146	UC LAB	1	508	370.1	350	395.5
153	UC LAB	2	258	390.0	340	382.8
8	UC LAB	3	340	363.5	370	413.8
120	UC LAB	4	311	410.5	340	374.0
48	UC LAB	1	382	391.7	370	426.5
46	UC LAB	2	448	407.7	340	378.9
100	UC LAB	3	329	370.6	370	401.1
57	UC LAB	4	438	417.7	390	423.2
73	UC LAB	1	261	369.0	330	372.9
21	UC LAB	2	444	408.8	320	362.9
58	UC LAB	3	363	395.0	330	384.5
29	UC LAB	4	476	437.6	360	411.0
63	UC LAB	1	418	407.7	340	397.2
2	UC LAB	2	330	372.9	360	393.3
113	UC LAB	3	379	418.8	390	437.6
61	UC LAB	4	378	414.9	330	375.1
59	ELPAT	1	406	408.7	360	408.7
140	ELPAT	2	405	408.7	430	408.7
92	ELPAT	3	170	408.7	410	408.7
96	ELPAT	4	274	408.7	410	408.7
11	ELPAT	1	327	418.1	440	418.1
93	ELPAT	2	382	418.1	410	418.1
77	ELPAT	3	427	418.1	430	418.1
83	ELPAT	4	358	418.1	420	418.1

Sample Analysis Order	Source	Rep	MTI PDV 5000		DataChem	
			Result	Estimated	Result	Estimated
			µg/wipe	µg/wipe	µg/wipe	µg/wipe
158	ELPAT	1	651	561.9	580	561.9
20	ELPAT	2	596	561.9	540	561.9
82	ELPAT	3	403	561.9	560	561.9
159	ELPAT	4	580	561.9	540	561.9
156	ELPAT	1	689	564.7	560	564.7
142	ELPAT	2	598	564.7	560	564.7
56	ELPAT	3	620	564.7	570	564.7
71	ELPAT	4	613	564.7	530	564.7
86	ELPAT	1	704	805.1	760	805.1
118	ELPAT	2	898	805.1	770	805.1
135	ELPAT	3	719	805.1	760	805.1
19	ELPAT	4	804	805.1	740	805.1
35	ELPAT	1	1069	1482.6	1500	1482.6
97	ELPAT	2	885	1482.6	1500	1482.6
157	ELPAT	3	1716	1482.6	1500	1482.6
14	ELPAT	4	1281	1482.6	1400	1482.6