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Environmental Technology Verification Program

Environmental and Sustainable Technology Evaluations

Report

ANDALYZE
LEAD-IN-PAINT TEST KIT
QUALITATIVE SPOT TEST KIT FOR LEAD IN PAINT

Prepared by
Battelle

Battelle
The Business of Innovation

for

EPA U.S. Environmental Protection Agency

ETV ETV ETV

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Qualitative Spot Test Kit for Lead in Paint

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Notice

Funding for this verification test was provided under Contract No. EP-W-09-024, Work Assignments 4-16, 0-06, and 1-06, Office of Pollution Prevention, and Toxics, US EPA. The U.S. Environmental Protection Agency, through its Office of Research and Development, managed the research described herein. It has been subjected to the Agency's peer and administrative review and has been approved for publication. Any opinions expressed in this report are those of the author(s) and do not necessarily reflect the views of the Agency, therefore, no official endorsement should be inferred. Any mention of trade names or commercial products does not constitute endorsement or recommendation for use.

Foreword

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the Nation's land, air, and water resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, EPA's research program is providing data and technical support for solving environmental problems today and building a science knowledge base necessary to manage our ecological resources wisely, understand how pollutants affect our health, and prevent or reduce environmental risks in the future.

The National Risk Management Research Laboratory (NRMRL) is the Agency's center for investigation of technological and management approaches for preventing and reducing risks from pollution that threaten human health and the environment. The focus of the Laboratory's research program is on methods and their cost-effectiveness for prevention and control of pollution to air, land, water, and subsurface resources; protection of water quality in public water systems; remediation of contaminated sites, sediments and groundwater; prevention and control of indoor air pollution; and restoration of ecosystems. NRMRL collaborates with both public and private sector partners to foster technologies that reduce the cost of compliance and to anticipate emerging problems. NRMRL's research provides solutions to environmental problems by: developing and promoting technologies that protect and improve the environment; advancing scientific and engineering information to support regulatory and policy decisions; and providing the technical support and information transfer to ensure implementation of environmental regulations and strategies at the national, state, and community levels.

This publication has been produced as part of the Laboratory's strategic long-term research plan. It is published and made available by EPA's Office of Research and Development to assist the user community and to link researchers with their clients.

Sally Gutierrez, Director
National Risk Management Research Laboratory

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List of Abbreviations and Acronyms

AMS	Advanced Monitoring Systems
ASTM	American Society for Testing and Materials
CCV	continuing calibration verification
COC	chain of custody
CRM	certified reference material
DOT	Department of Transportation
EPA	U.S. Environmental Protection Agency
ESTE	Environmental and Sustainable Technology Evaluations
ETV	Environmental Technology Verification
ICP-AES	inductively coupled plasma-atomic emission spectrometry
LCS	laboratory control spike
mg/cm^2	milligrams per centimeter squared
µL	microliter
mL	milliliter
mm	millimeter
MSDS	material safety data sheets
NLLAP	National Lead Laboratory Accreditation Program
PE	performance evaluation
PEM	performance evaluation material
ppb	parts per billion
PT	performance test
QA	quality assurance
QC	quality control
QCS	quality control sample
QMP	quality management plan
RRP	Renovation, Repair, and Painting
SOP	standard operating procedure
TSA	technical systems audit

Chapter 1
Background

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by 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 testing organizations; with stakeholder groups consisting of buyers, vendor organizations, and permitters; and 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 according to rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

This verification test was conducted under the U.S. EPA ETV program. Testing was performed by Battelle, which served as the verification organization under the Environmental and Sustainable Technology Evaluations (ESTE) arm of ETV. Battelle evaluated the performance of qualitative spot test kits for lead in paint.

This verification test was developed with the support of a stakeholder technical panel. A voluntary stakeholder technical panel consisting of individuals from the American Industrial Hygiene Association (Kenn White), U.S. Department of Housing and Urban Development (Warren Friedman), National Institute for Occupational Safety and Health (Kevin Ashley), U.S. Army Center for Health Promotion and Preventative Medicine (Al Liabastre), National Center for Healthy Housing (David Jacobs), National Association of Homebuilders (Matt Watkins), the U.S. Consumer Product Safety Commission (Joanna Matheson), the Center for Disease Control and Prevention (Larry Franklin), and U.S. EPA (Paul Carroll and Moira Lataille) was formed for this verification test. Participants on this panel were reviewed and approved by EPA. This panel gave input during the entire ETV process, including providing guidance and input on the development of the performance evaluation materials used in this test, on the development of the test design and test/QA plan, and comments on this report.

Chapter 2
Technology Description

This report provides results for the verification testing of the Lead-in-Paint Test Kit for lead-based paint by ANDalyze. The following is a description of the Lead-in-Paint Test Kit, based on information provided by the vendor. The information provided below was not verified in this test.

The ANDalyze Lead-in-Paint Test Kit utilizes a sensor/fluorimeter platform to quantitatively detect lead in paint. The test is based on a sensing technology which uses DNA to identify lead. Research done at the University of Illinois, Urbana Champaign used combinatorial biology to identify a particular DNA sequence that specifically binds to lead ion (Pb^{2+}) and catalyzes the cleavage of another DNA sequence. These special DNA sequences capable of performing catalysis are called DNAzymes (DNA enzymes). ANDalyze has converted this patented technology into a test kit for lead. The DNA sequence specific for Pb^{2+} is linked to fluorophores/quencher pair as depicted in Figure 2-1. Two strands of DNA, an enzyme strand (shown in green) linked to a quencher and a substrate strand (shown in black) linked to a fluorophore are held together by DNA hybridization. The fluorescence of the fluorophore is quenched due to its close proximity to the quencher. In the presence of lead, the DNAzyme catalyzes the cleavage of the substrate strand which releases the cleaved fragment containing the fluorophore into solution thereby enhancing the fluorescence. The increased level of fluorescence upon reaction with lead can be measured using a fluorimeter. The rate of this increase is proportional to the lead concentration.

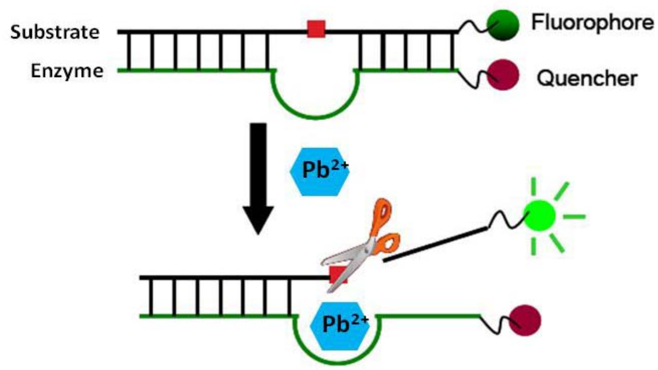

Figure 2-1: Schematic representation of DNAzyme based lead sensing

The Lead-in-Paint Test Kit consists of two parts: the extraction kit (shown in Figure 2-2, left) and the testing kit (shown in Figure 2-2, right). The extraction kit includes a razor blade, ruler, drill bit, plastic tissue grinder, plastic transfer pipette, weighing paper and a bottle of 25% nitric acid. A drill required for drilling the paint sample from surfaces may be purchased from ANDalyze if the user does not already own one. The testing kit includes a portable fluorimeter instrument and the following consumables: dried sensor in a plastic housing, syringe, glass tube, 30 milliliter (mL) plastic tube with test buffer, and 10 microliter (µL) fixed volume pipette with tips. A lead paint standard for calibrating the instrument is also supplied with the kit.

Figure 2-2. ANDalyze Lead-in-Paint Test Kit Test extraction kit (left) and testing kit (right)

To extract soluble lead (as Pb^{2+}) from a dry paint surface, a 1.2 cm^2 area of paint is either drilled using a drill fitted with a ½ inch drill bit or cut using a razor blade. The selection of a drill or manual scraping device is dependent upon the type of surface that is being tested. The entire paint sample is transferred into a plastic tissue grinder to which 2 mL 25% nitric acid is added using the plastic pipette. The paint chips are then ground to a fine powder by rotating the pestle of the tissue grinder for approximately 2 – 5 minutes which results in Pb^{2+} being extracted into the acidic solution. The test is performed by first transferring and mixing 10 µL volume (using the fixed volume pipette) of the acidified Pb^{2+} extract into the 30 mL plastic tube containing 20 mL of testing buffer. This is the test solution. A glass tube is inserted into the sample chamber of the fluorimeter and sensor housing is placed on the glass tube. Using the syringe, 0.7 mL of test solution is withdrawn and pushed through the sensor housing into the glass tube. The lead reacts with the DNA-based sensor during this step. The housing is immediately removed, the lid is closed and the START button is pressed. The lead concentration in paint is displayed on the screen within 30 seconds in units of mg/cm^2.

At the time of testing, a test kit included a fluorimeter at $1500 and consumables for 50 tests at $300. Refill consumables could be purchased for further testing. Optional: A Craftsman drill could be purchased from ANDalyze at a cost of $310 if the user does not own one. The ANDalyze fluorimeter could be used for any other tests which utilize fluorescent sensing methods.

Chapter 3
Test Design and Procedures

3.1 Introduction

This verification test was conducted according to procedures specified in the *Test/QA Plan for Verification of Qualitative Spot Test Kits for Lead in Paint*.[1] Lead-based paints were commonly used in houses in both interior and exterior applications prior to 1978, when the US government banned the use of lead-based paint in residential applications. The term lead-based paint means paint or other surface coatings that contain lead at contents that equal or exceed a level of 1.0 milligrams per centimeter squared (mg/cm^2) or 0.5 percent by weight.[2] This paint still exists in many of these houses across the country. The accurate and efficient identification of lead-based paint in housing is important to the Federal government as well as private individuals living in residences containing such paints. Renovation, repair, and painting (RRP) activities may disturb painted surfaces and produce a lead exposure hazard. Such disturbances can be especially harmful to children and pregnant women as lead exposure can cause neurological and developmental problems in both children and fetuses. In fact, because of the large amount of pre-1978 housing stock, a report by the President's Task Force on Environmental Health Risks and Safety Risks to Children found that approximately 24 million US dwellings were at risk for lead-based paint hazards.[3]

There are lead-based paint test kits available to help home owners and contractors identify lead-based paint hazards before any RRP activities take place so that proper health and safety measures can be taken. However, many of these test kits have been found to have high rates of false positives (i.e., test kit indicates that lead in excess of 1.0 mg/cm^2 is present, while in fact the true lead level is below 1.0 mg/cm^2).[4] This verification test was conducted in response to the call of the Renovation, Repair, and Painting rule[2] for an EPA evaluation and recognition program for test kits that are candidates to meet the goal of a demonstrated probability (with 95% confidence) of a false negative response less than or equal to 5% of the time for paint containing lead at or above the regulated level, 1.0 mg/cm^2 and a demonstrated probability (with 95% confidence) of a false positive response less than or equal to 10% of the time for paint containing lead below the regulated level, 1.0 mg/cm^2. This test incorporated ASTM International's E1828, *Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Spot Test Kits for Lead in Paint*[5] guidelines into the test design.

The objective of this verification test was to evaluate the performance of the test kits for the detection of lead in paint. This evaluation assessed the capabilities of the lead paint spot test kits against laboratory-prepared performance evaluation material (PEM) samples and compared the lead paint test kit results with those of a standard technique, inductively coupled plasma-atomic emission spectrometry (ICP-AES). Additionally, this verification test relied on verification testing staff observations to assess other performance characteristics of the lead paint test kits. Only qualitative results (e.g., detect/non-detect of lead at specified levels) were considered for each technology.

The ANDalyze Lead-in-Paint Test Kit was verified by evaluating the following parameters:

- False positive and false negative rates
- Precision
- Sensitivity
- Modeled probability of test kit response
- Matrix effects
- Operational factors.

Verification testing of the test kit was conducted from January to June 2010. This timeframe included testing of the test kit and also completion of all ICP-AES and QC analyses. False positive and negative rates were determined by comparing test kit responses to actual lead concentrations of the PEM as determined through ICP-AES. Precision was determined by reproducibility of responses for replicate samples. Sensitivity was determined as the lowest detectable level of the test kit. The modeled probability and matrix effects were determined using logistic regression models.

Operational factors such as ease of use, operator bias, average cost, average time for kit operation, helpfulness of manuals, and sustainability metrics such as volume and type of waste generated from the use of each test kit, toxicity of the chemicals used, and energy consumption were determined based on documented observations of the testing staff and the Battelle Verification Test Coordinator. Operational factors were described qualitatively, not quantitatively; therefore, no statistical approaches were applied to the operational factors.

3.2 Test Facility

Laboratory analyses of the ANDalyze Lead-in-Paint Test Kit were conducted in Battelle laboratories in Columbus, Ohio. No field testing was conducted during this technology verification.

3.3 Test Procedures

Qualitative spot test kits for lead in paint were evaluated against a range of lead concentrations in paint on various substrates through the use of PEMs. PEMs were 3 inch by 3 inch square panels of wood (pine and poplar), metal, drywall, or plaster that were prepared by Battelle.[6] Pine and poplar were chosen for the wood panels as they are representative of woods most commonly found in homes. Table 3-1 shows the PEMs prepared for each test kit. Poplar and pine PEMs were distributed in random mixtures (e.g., two poplar and one pine or one poplar and two pine) for each set of three wood PEMs listed in Table 3-1. Each PEM was coated with either white lead (lead carbonate) or yellow lead (lead chromate) paint. The paint contained lead targeted at

Table 3-1. PEMs Testing Scheme for Each Test Kit

Lead Type	Lead Level (mg/cm^2)	Substrate	PEMs Analyzed Per Test Kit by Topcoat Color			
			White	Red-Orange	Grey-Black	Total
Control Blank	0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
White Lead (Lead Carbonate)	0.3	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	0.6	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	1.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	1.4	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	2.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	6.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
Yellow Lead (Lead Chromate)	0.3	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	0.6	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	1.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	1.4	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	2.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	6.0	Wood	3	3	3	9
		Metal	3	3	3	9
		Drywall	3	3	3	9
		Plaster	3	3	3	9
	Painted PEMs Subtotal		156	156	156	468
	Unpainted PEMs Subtotal (2 per each substrate)					8
	Total					476

[a] Actual number of PEMs used to evaluate performance at specific lead levels varied based on actual concentrations observed during analysis.

0.3, 0.6, 1.0, 1.4, 2.0, and 6.0 mg/cm^2. These lead concentrations were chosen with input from the stakeholder technical panel based on criteria provided in EPA's lead RRP rule as well as to represent potential lead levels in homes. Paint containing no lead (0.0 mg/cm^2) was also applied to each substrate and tested.

Two different layers of paint were applied over the leaded paint. One was a primer designed for adhesion to linseed oil-based paint and the second coat was a typical interior modern latex paint tinted to one of three colors: white, red-orange, or grey-black. These colors were chosen by EPA, with input from the stakeholder technical panel, based on the potential of certain colors to interfere or not with lead paint test kit operations. The topcoat paint manufacturers' recommended application thickness was used. Two coats at the recommended thickness were applied. Details on the PEM production process can be found in Appendix A.

The ANDalyze Lead-in-Paint Test Kit for lead paint was operated by a technical and non-technical operator. The technical operator was a Battelle staff member with laboratory experience. The technical operator was trained by a representative of the vendor company in the operation of its test kit. The same technical operator operated this test kit throughout testing. Because this lead paint test kit is anticipated to be used by certified remodelers, renovators, and painters, it was also evaluated by a non-technical operator. The non-technical operator was a certified renovator with little to no experience with lead analysis. The non-technical operator was also a college graduate. The non-technical operator was provided the instruction manual, demonstrational DVD, and other materials (operational tip sheet, material safety data sheets (MSDS)) typically provided by the vendor with the test kit for training. The non-technical operator viewed the materials himself to understand how to operate the test kit. The non-technical operator was also permitted to ask questions or clarifications of the vendor on the operation of the test kit. This scenario approximated the training renovators are expected to receive under the RRP rule.

Tests were performed in duplicate on each PEM by each operator, technical and non-technical (i.e., two samples were taken from each PEM by each operator). Duplicates were tested in succession by each operator on a given PEM. PEMs were analyzed blindly by each operator in that the PEMs used for analysis were marked with a non-identifying number. Test kit operators were not made aware of the paint type, lead level, or substrate of the PEM being tested. PEMs were tested in random order (i.e., PEMs were placed in plastic bins and the operators arbitrarily selected a PEM for analysis). To determine whether the substrate material affected the performance of the test kits, two unpainted PEMs of each substrate were tested using each test kit, in the same manner as all other PEMs (i.e., per the test kit instructions). Three PEMs at each lead level, substrate, and topcoat color were prepared for use in this test. In total, 468 painted PEMs were prepared for use in the verification test of each test kit.

Paint chip samples from each PEM were analyzed by a National Lead Laboratory Accreditation Program (NLLAP) recognized laboratory, Schneider Laboratories, Inc., using ICP-AES to confirm the lead level of each PEM used for testing. The paint chip samples for reference analyses were collected by Battelle according to a Battelle SOP[7], which was based on ASTM E1729.[8] The reference analyses confirmed the lead level of each PEM. Lead levels determined through the reference analysis were used for reporting and statistical analyses.

The procedures for collecting, storing, and shipping test samples are provided below.

3.3.1 Test Sample Collection, Storage, and Shipment

Chips of lead paint were taken from each PEM and sent for ICP-AES analysis at a NLLAP-recognized laboratory, Schneider Laboratories, Inc. A glass screw-top vial was labeled with the PEM identification number located on the back of the panel. The number was also recorded on the Chain of Custody (COC) form. Sampling was performed per the Battelle SOP for collection of dried paint samples for lead determination. All safety precautions and personal protective equipment were used. A one inch square, metal template was placed adjacent to the tested area. A utility knife was used to trace around the template. Tweezers and a utility knife were used to scrape and remove the paint within the one inch area, using caution to minimize introduction of the substrate into the paint sample. The topcoat and remaining paint were transferred to a glassine weighing paper with the assistance of a paintbrush. The sample was then transferred from the glassine paper into a glass vial using the paintbrush. All instruments and templates were wiped with tissue paper and the bench top was cleaned and gloves were changed between each sample to minimize contamination. The paint brush was carefully flicked and tapped over a trash can to remove any residual lead dust. All wipes and gloves were disposed of as lead waste. The vials were then collected into a zip-top bag and taped up securely for shipping. The bags and COC were then shipped together using overnight delivery to Schneider Laboratories, Inc.

Paint chip samples were stored at room temperature as received by Schneider Laboratories, Inc. and then analyzed by ICP-AES. Analytical results were reported to Battelle within 2-3 days. Sample digests were stored separately by Schneider Laboratories, Inc. at room temperature.

PEMs were stored individually in zip-top bags. The back of each PEM was labeled with an identifying number. The outside of the zip-top bag was labeled with the same number. Each PEM was wrapped in a Kimwipe and each zip-top bag was sealed when not in use. The zip-top bags containing the PEMs were housed in large plastic bins in the laboratory during testing.

3.3.2 Test Sample Analysis Procedure

At the beginning of each day of testing, prior to the analysis of any samples, the fluorimeter apparatus for the lead measurement was calibrated using a three-point calibration. First the fluorimeter was plugged into an electrical outlet and allowed to warm up for at least 5 minutes. Then standard solutions of 0.2, 1.0 and 5.0 mg/cm^2 lead were prepared as follows: 10 μL of the 0.2 mg/cm^2 standard, Stock Solution 1 as supplied with the test kit, were added to the pre-prepared buffer Tube 1 and mixed. Calibration standards for the remaining two levels were prepared similarly, with 10 μL of the 1.0 mg/cm^2 lead standard, Stock Solution 2, added to the pre-prepared buffer tube 2 and 10 μL of the 5.0 mg/cm^2 lead standard, Stock Solution 3, added to Tube 3.

The following steps were taken to prepare the fluorimeter for calibration. On the meter display panel of the fluorimeter, “Menu” was pressed. Then the operator scrolled down to “Calibration” on the screen and pressed “Select” and then “Next”. Once the fluorimeter was prepared, a clean glass test tube was placed into the receptacle of the meter. Then a green sensor housing was placed over the test tube. Using the 1 mL sterile syringe supplied with the test kit, 0.7 mL of solution from Tube 1 was transferred into the sensor housing. The syringe and housing were removed and discarded and the fluorimeter lid was quickly closed. Immediately following the closing of the lid on the fluorimeter, “OK” was pressed on the display panel. After a reading was obtained, the glass test tube was removed and discarded. This process was repeated using Tubes 2 and 3. After the final calibration solution (Tube 3) was analyzed, a R^2 value was generated by the fluorimeter. If the R^2 reading was above 0.98, the calibration was considered acceptable and “Apply” was pressed on the meter display panel. If the R^2 reading was below 0.98, the calibration process was repeated.

Once the fluorimeter was calibrated, sampling was conducted. Paint samples were obtained from the selected PEM using either the drill or scrape method, depending on the surface that was being tested.

A modified half-inch spade bit attached to an 18 volt Craftsman cordless hand drill was used on the wood and plaster PEMs. The spade bit was lightly placed on the PEM surface, and the drill was activated to a slow speed to remove the paint and try to get as little of the substrate as possible.

An X-acto® knife was used for collection on the metal and drywall PEMs. The ANDalyze Lead-in-Paint Test Kit came with a razor blade for cutting up the paint sample and scraping paint from the sample area of interest. The vendor indicated that it was not necessary to use this razor blade if the user had a suitable replacement. The ANDalyze Lead-in-Paint Test Kit instructions called for using a ruler to measure a 1.1 cm x 1.1 cm area on metal and drywall substrates for removal of a paint sample. Because of the large number of samples being generated in this study, a stainless steel template was used by both the technical and non-technical operator. The X-acto® knife was used to trace around the outside of the stainless steel template. The paint sample was then removed.

In both sampling methods, after removal from the PEM, the paint sample was cut up into small pieces using the X-acto® knife and then placed into the labeled grinding tube. Once the paint sample was removed and placed into the grinding tube, 2 mL of the 25% nitric acid solution was added to the grinding tube using a 3 mL plastic transfer pipette. Per tips for successful operation of the kit provided by the vendor, the paint sample was allowed to sit in the acid for 5 minutes. This softened up the paint sample and allowed for easier grinding. After 5 minutes in the acid, the pestle was screwed onto the grinding tube and the pestle was rotated, with an up and down motion, or the pestle was held and the tube was rotated. The ANDalyze Lead-in-Paint Test Kit instructions indicated that the paint sample should be ground until it turns into a powder, up to seven minutes. It was determined during testing that 3 minutes was needed to grind each of the samples for this test.

Because nearly 1000 samples were needed to be ground by each operator using the pestle and grinding tube, there was concern that the operators might acquire a repetitive motion disorder over the course of testing. To alleviate this concern, additional Battelle laboratory technicians were brought in to perform the grinding step, in conjunction with the efforts of the operators, for both the technical and non-technical operator. The technical and non-technical operator each calibrated the fluorimeter, removed the paint sample from the PEM, added acid to the pestle, and analyzed the resulting extract (see below for details). However, for approximately half of the samples analyzed on a given day, one to two additional Battelle laboratory technicians helped conduct the grinding step for a given sample. It was noted on the data collection sheets when a person other than the operator conducted the grinding. All grinding was performed for three minutes.

After the grinding of the sample was complete, 10 µL of the ground paint/acid mixture was removed using a pipette and placed into a plastic tube containing 20 mL of the testing buffer. The tube was capped and shaken to mix.

Next, the fluorimeter was prepared for analyzing the sample extract. A clean glass test tube was placed into the receptacle of the fluorimeter. Then a green sensor housing was placed over the test tube. Using a sterile 1 mL syringe supplied with the test kit, 0.7 mL of the lead test solution was transferred into the test tube. The syringe and housing were removed and discarded and the fluorimeter lid was quickly closed. "Start" was pressed on the fluorimeter display panel and the sample was analyzed. The value displayed on the fluorimeter screen provided the concentration (mg/cm^2) of lead in the sample.

After each sample the pestle and grinding tube had to be cleaned. After emptying the pestle and grinding tube of any lead solution, both components were cleaned with a brush and tap water. Then 2 mL of the cleaning solution was placed into the grinding tube. Pestles were then placed into tube. The pestle and grinding tube were allowed to sit for at least 30 minutes. Then the pestle was removed and the cleaning solution was poured out. The brush was used to remove any remaining debris, and the pestle and grinding tube were rinsed and completely dried before the next use.

Chapter 4
Quality Assurance/Quality Control

QA/QC procedures were performed according to the quality management plan (QMP) for the Battelle ETV Advanced Monitoring Systems (AMS) Center[9], except where differences were noted for ESTE per the EPA ETV Program QMP[10], and the test/QA plan for this verification test.[1] Test procedures were as stated in the test/QA plan; however a deviation to the test/QA plan was made during the ICP-AES analyses. For some sample runs, continuous calibration verification (CCV) samples were run once every 20 instead of 10 samples. This deviation is described below. This change was assessed to have no impact on the quality of the results as described below. QA/QC procedures and results are described below. Additional information on QA/QC outcomes for the PEMs is provided in Appendix A.

4.1 Quality Control Samples

Steps were taken to maintain the quality of data collected during this verification test. This included analyzing specific quality control samples for the reference method (ICP-AES) and the test kit.

4.1.1 ICP-AES Blank Sample Results

Various blank samples were analyzed for the ICP-AES analyses. Method blank samples were analyzed in each set of 10-20 paint samples to ensure that no sources of contamination were present. An initial calibration blank was analyzed at the beginning of each run and used for initial calibration and zeroing the instrument. A continuing calibration blank was analyzed after each CCV to verify blank response and freedom from carryover. No blank samples failed during the analyses.

4.1.2 ICP-AES Matrix Spike Samples and Calibration Verification Standards

Initial calibration standards were run at the beginning of each set of analyses. The acceptance criterion for the calibration coefficient of the calibration standards was ≥ 0.998. If this criterion was not met, the analysis was stopped and recalibration was performed before samples were analyzed. A 500 parts per billion (ppb) CCV standard was analyzed at the beginning of each run (following the initial calibration), at the end of each run, and every 10-20 samples. CCV recoveries ranged from 96% to 108%. Per the test/QA plan, CCV sample frequency was once every 10 samples. For most of the sample sets CCVs were performed with this frequency.

However, for later sample sets CCVs were run once every 20 samples. CCV samples were used to verify instrument performance. CCV samples were run every 10 samples as a preventative measure so that large amounts of samples do not need to be re-run if a CCV sample fails. In the course of this study, one CCV sample failed. All samples from the last passing CCV of that sample set were re-analyzed.

A matrix spike sample and laboratory control sample (LCS), as well as duplicates of these samples, were also analyzed. Duplicate samples were run once every 10-20 samples. Acceptable recoveries for matrix spike samples were between 80-120%. Acceptable recoveries for LCS samples were between 80-120%. Duplicate samples had acceptance criteria of ±25% relative percent difference (RPD).

All matrix spike samples were performed as post-digestion spikes as there was insufficient sample volume to perform a pre-digestion spike. Matrix spike recoveries ranged from 86% to 207%. Six matrix spike samples failed, with recoveries above the specified acceptance criteria. In these instances, the lead concentration in the sample was well above the spike level. Matrix spike results indicated that matrix interferences were not observed. Duplicate samples were within the specified RPD.

LCS samples were analyzed once every 10-20 samples. LCS recoveries ranged from 17% to 225%. Schneider Laboratories, Inc. noted that LCS failures on one sample set were attributed to improper spiking technique. Training on spiking procedures was immediately implemented by Schneider Laboratories for all analysts spiking samples. All LCS failures occurred prior to a revision to the Schneider Laboratories, Inc. SOP[11] for analyzing paint samples written specifically for this verification test. In the original version of the SOP, LCS samples were prepared by spiking a known amount of lead onto a certified reference material (CRM). This practice was changed because there were over-recovery issues. This was because the spike was not >3x the background lead concentration because of the high lead concentrations in the actual CRM samples. In the revised SOP, the LCS was prepared by spiking a piece of lead-free latex paint. There were no LCS failures after that. In addition, a QC check sample containing only the CRM, which had a known concentration of lead weighed out to a particular amount, was analyzed with each sample set throughout the verification test. These QC check samples all passed acceptance criteria.

4.1.3 Test Kit Quality Controls and Blank PEMs

As indicated in Section 3.3.2, quality control measures were built into the test procedures through the calibration of the fluorimeter. All fluorimeter calibrations had to obtain an R^2 value of 0.98 or higher to pass calibration. All calibrations obtained for this test were above this R^2 value. Painted PEMs containing no lead as well as each of the PEM substrates containing no paint were also run as part of the verification test. All samples of PEM substrates containing no paint returned negative results from the test kit (i.e., no lead was present). All of painted PEMs containing no lead returned negative results.

4.2 Audits

Three types of audits were performed during the verification test: a performance evaluation (PE) audit of the reference method measurements made in this verification test, a technical systems audit (TSA) of the verification test performance, and a data quality audit. Audit procedures are described below.

4.2.1 Performance Evaluation Audits

A PE audit was conducted to assess the quality of the reference method measurements made in this verification test. The reference method PE audit was performed by supplying an independent, NIST-traceable lead paint standard (Reference Material 8680, panel CB3), to the reference laboratory. The PE audit samples were analyzed in the same manner as all other samples and the analytical results for the PE audit samples were compared with the nominal concentration. The target criterion for this PE audit was in agreement with the analytical result within 20% of the nominal concentration. The specified acceptable concentration range for the NIST standard panel was 1.13 – 1.75 mg/cm^2 (1.44 ±0.31 mg/cm^2). The PE samples taken from this standard panel were 1.38, 1.38, 1.19, and 1.31 mg/cm^2. The PE audit result met the target criterion. This audit was performed once at the start of the test.

4.2.2 Technical Systems Audit

The Battelle Quality Manager performed one TSA during this verification test to ensure that the verification test was being performed according to the Battelle AMS Center and ETV Program QMPs, the test/QA plan, any published reference methods, and standard operating procedures. In the TSA, the Battelle Quality Manager reviewed the reference methods used, compared actual test procedures with those specified or referenced in the test/QA plan, and reviewed data acquisition and handling procedures. Also in the TSA, the Battelle Quality Manager observed testing, observed reference method sample preparation and analysis, inspected documentation, and reviewed technology-specific record books. He also checked standard certifications and technology data acquisition procedures and conferred with the technical staff. A TSA report was prepared. There were no findings. The records concerning the TSA are permanently stored with the Battelle Quality Manager.

The EPA ETV Quality Manager also performed a TSA of both the reference laboratory and the testing conducted at Battelle Columbus, OH facilities. No findings were reported in the TSA of the reference laboratory, Schneider Laboratories, Inc. In the TSA of the lead paint test kit evaluations at Battelle’s Columbus, OH facilities, the EPA ETV Quality Manger cited two findings. These findings were related to ease of use observations and were immediately and adequately addressed and did not affect the quality of the test.

4.2.3 Audit of Data Quality

Records generated in the verification test received a one-over-one review (i.e., review by a Battelle technical staff who did not generate the records) before these records were used to calculate, evaluate, or report verification results. A Battelle technical staff member involved in the verification test reviewed the data. Datasheets generated by the operators during testing were reviewed for completeness and errors. The person performing the review added his/her initials and the date to a hard copy of the record being reviewed. At least 10% of the data acquired during the verification test, including the ICP-AES results, were audited by Battelle. At least 25% of the ICP-AES data acquired during the verification test were audited by EPA. Battelle's Quality Manager traced the data from the initial acquisition, through reduction and statistical analysis, to final reporting to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked. Minor transcription errors were identified and corrected before the results were used for the calculations described in Chapter 5. Battelle's and EPA's Quality Managers also reviewed the PEM ICP-AES results thoroughly to ensure that all data quality indicators as stated in the test/QA plan were followed and that reported results matched the data generated on the instrument. Findings were cited by the EPA Quality Manager. Appropriate corrective actions were taken. Significant QA/QC concerns identified during EPA's audit are discussed in Section 4.1.

Chapter 5
Statistical Methods

The statistical methods used to evaluate the performance factors listed in Section 3.1 are presented in this chapter. The ANDalyze Lead-in-Paint Test Kit was evaluated for qualitative results (i.e., positive/negative responses to samples). All data analyses were based on these qualitative results. QC samples and unpainted PEM substrates were not included in any of these analyses. Results are provided in Chapter 6.

5.1 False Positive and False Negative Rates

A false positive response was defined as a positive result when regulated lead-based paint was not present. The test/QA plan[1] defined false positive rates as being based on target lead levels at and below 0.6 mg/cm^2 with confirmed values not to exceed 0.8 mg/cm^2. Because confirmed lead levels of particular PEMs did not sometimes match target concentrations for those PEMs, false positive rates were assessed on panels with confirmed lead levels at 0.8 mg/cm^2 and lower. Consistent with the EPA's April 22, 2008 RRP rule[2], panels with an ICP-AES confirmed lead level between 0.8 and 1.0 mg/cm^2 were not used in the false positive analysis.

A false negative response was defined as a negative response when regulated lead-based paint was present. The test/QA plan defined false negative rates as being based on target lead levels at and above 1.4 mg/cm^2 with confirmed values not to exceed 1.2 mg/cm^2. Because confirmed lead levels of particular PEMs did not sometimes match target concentrations for those PEMs, false negative rates were assessed on panels with confirmed lead levels at 1.2 mg/cm^2 and higher. Consistent with the EPA's April 22, 2008 RRP rule, panels with an ICP-AES confirmed lead level between 1.0 and 1.2 mg/cm^2 were not used in the false negative analysis.

Based on stakeholder technical panel input, the EPA lead paint action level of 1.0 mg/cm^2 lead was included for analysis as part of the verification test. Though evaluations of test kit performance based on this level is not in the EPA RRP rule, false positive and negative rates, in addition to those stated above, were also calculated for each test kit based on 1.0 mg/cm^2 lead. Thus, false positive rates were assessed on PEMs with confirmed lead levels at 1.0 mg/cm^2 and lower and false negative rates were assessed on PEMs with confirmed lead levels at 1.0 mg/cm^2 and higher. For panels that measure 1.0 mg/cm^2, positive results were considered "correct" and negative results were considered false negative. If the confirmed lead concentration of the PEM was greater than 1.0 mg/cm^2 (e.g., 1.1 mg/cm^2), then negative results were considered false negatives. If the confirmed lead concentration of the PEM was less than 1.0 mg/cm^2 (e.g., 0.9 mg/cm^2), then positive results were considered false positives.

False positive and negative rates were calculated as shown in Equations 1 and 2, respectively:

$$\textit{False Positive Rate} = \frac{\#\ of\ \ positive\ results}{total\ \#\ of\ \ PEMs\ with\ lead\ level\ below\ 0.8\ (or\ 1.0)\ mg/cm^2} \quad (1)$$

$$\textit{False Negative Rate} = \frac{\#\ of\ \ negative\ results}{total\ \#\ of\ \ PEMs\ with\ lead\ level\ above\ 1.2\ (or\ 1.0)\ mg/cm^2} \quad (2)$$

5.2 Precision

Precision was measured by the reproducibility of responses for replicate samples within a group of PEMs. Precision results were reported as the percentage of consistent responses from all replicate sets for those paint types (see Equation 3). Responses were considered inconsistent if 25% or more of the replicates differed from the response of the other samples in the same group of PEMs.

$$\text{Precision (\% consistent results)} = \frac{\text{\# of consistent responses of replicate sets}}{\text{total number of replicate sets}} \times 100 \quad (3)$$

5.3 Sensitivity

The sensitivity or lowest detectable lead level for each test kit was identified based on the detection results across all PEM lead levels. The lowest PEM lead level with consistent (>75%) positive or “detect” responses was considered the lowest detectable level. The identified lowest detectable lead level was reported and discussed.

5.4 Modeled Probability of Test Kit Response

Logistic regression models were used to determine the probabilities of positive or negative responses of the test kit at the 95% confidence level, as a function of lead concentration and other covariates, such as substrate type, lead paint type, operator type, and topcoat color. An evaluation of the bivariate relationship between the response variable and each candidate explanatory variable was performed by fitting single covariate logistic models to assess the predictive ability of each of the PEM parameters. Using the results from these bivariate analyses, a parsimonious multivariate model was developed including a set of explanatory variables which were most predictive of the probability of the test kit response variable. The potential logistic regression model took the form below:

$$\text{logit}(\Pr(Y_i = 1)) = X_i \beta \qquad (4)$$

where Y_i is the outcome of the test kit, X_i is a vector of explanatory variables associated with Y_i and β represent a vector of unknown parameters which was estimated with the model. Test results that indicated that lead was present were represented with Y=1; negative results were represented with Y=0. Candidate independent variables associated with the response variable were lead level (continuous), operator type (categorical), lead type (categorical), substrate type (categorical), and topcoat color (categorical). Interactions between categorical predictor variables were also assessed. Categorical covariates were modeled using indicator variables.

SAS's PROC LOGISTIC was used to evaluate the association between each explanatory variable and the probability of a positive test kit result. Then multivariable models were fit using a backward selection process whereby all explanatory variables were included in the initial model. In a multi-step backwards elimination process, the variable with the weakest association (highest Type III p-value) was eliminated from the model until all of the variables that remained had Type III p-values less than 0.05. The list of variables that remained formed the basis for evaluating interactions. Measured lead level was retained as an explanatory variable in all multivariable models. Two-way interactions were tested between all pairs of categorical explanatory variables that had p-values below 0.05. Interactions were retained in the multivariable models if their p-values were smaller than 0.05.

5.4.2 Accounting for Measurement Error – SIMEX Background and Intuition

Categorical covariates in this experiment were measured without error, but the lead level measurements were subject to some measurement error due both to variability inherent in the measurement (ICP-AES) process and possibly due to spatial heterogeneity in lead concentrations in paint on the PEMs themselves. The experimental design did not include multiple ICP-AES analyses per PEM so there is no direct estimate of the variability in measurements for these data. To account for the uncertainty associated with that error, the final multivariable model for each test kit was subjected to a simulation and extrapolation (SIMEX) analysis.[12-16]

A detailed description of SIMEX is beyond the scope of this report, but in short, it is a robust method of accounting for measurement error. The method requires either replicate measures of the quantity that is measured with error, or a characterization of the variability in the measurements. It then estimates what the regression model coefficients would be in the absence of measurement error. The technique estimates standard errors for the regression model coefficients using the bootstrap technique. SIMEX analyses were carried out in Stata version 11.1 using the programs described in Hardin et al (2003c).[15]

The premise of the analysis is that one of the independent variables, namely lead concentration, has been measured with error. In the logistic regression models considered here, lead concentration is the only continuous independent variable; all of the other covariates are categorical. Thus, lead concentration may be considered the 'x' in a simple linear regression. The observed variability in 'x' is comprised of two components, actual variation in lead concentration and measurement error. If we were able to remove the measurement error then we would observe less variability in that independent variable.

There are two important points of intuition that will inform expectations about what is seen in the SIMEX results. First, the data along the x-axis of a scatterplot would “tighten up” if measurement error were removed. “Tightening up” the independent variable in a regression analysis will result in a steeper slope or a regression coefficient with a larger magnitude. This is a fundamental consequence of any technique that adjusts for measurement error in the independent variable in a regression analysis. In the lead paint analysis, steeper logistic regression curves will result from the SIMEX analysis than would result from a non-SIMEX analysis where lead levels were considered to be fixed and known.

Second, when the statistical analyses acknowledge and account for the measurement error, then the regression output prediction intervals may be wider than those for a non-SIMEX analysis where ‘x’ is considered to be fixed and known. For any given predicted value of the outcome variable, the prediction interval will most likely be wider, or at least not narrower. But for a fixed value of ‘x’, (such as 0.8 or 1.2 mg/cm^2) whether the SIMEX prediction intervals are wider or narrower than the non-SIMEX intervals depends on how much the slopes of the SIMEX and non-SIMEX regression line differ. For typical logistic regression models, prediction intervals are very narrow at the extreme low and high asymptotic ends of the x-axis, and only appreciably wide in the region where the probability of the outcome is not near zero and not near one. So if the SIMEX analysis has only a moderate impact on the slope then wider prediction intervals might be observed at 0.8 and 1.2 mg/cm^2. But if the slope changes dramatically, then 0.8 or 1.2 mg/cm^2 might now be in the part of the prediction curve that is near zero or one and the SIMEX prediction interval might be dramatically more narrow than a non-SIMEX interval.

Thus, the prediction curves for every SIMEX analysis are expected to be steeper than, or at least not less steep than, a non-SIMEX analysis. However, the assessment of test kit performance is based on the upper and lower bounds of prediction intervals at 0.8 and 1.2 .mg/cm^2, respectively.

5.4.3 SIMEX Input and Analysis

During pre-production of the PEMs, replicate paint chip samples were analyzed from selected metal PEMs that served as reference panels (see Appendix A). Three metal panels were prepared for the pre-production homogeneity testing. Four paint chip samples, one from each quadrant of the PEM, were taken and analyzed via ICP-AES for their lead levels. Data are available on the coefficients of variation for these metal PEMs for both white and yellow lead. These data are shown below in Table 5.1. Though these data did not come from actual PEMs used during the lead paint test kit verification test, this information was used as a surrogate measure of homogeneity variability on the PEMs.

For each PEM in the study, nine random pseudo-replicates were generated from a normal distribution with a mean equal to the confirmed lead concentration for that panel, and a standard deviation computed from the metal reference PEM data in Table 5-1 and indexed by the panel’s lead type and target lead level. The nine measurements were used as inputs to the Stata SIMEX algorithm as if they were true replicate measurements.

Table 5-1. Results from Final Homogeneity Testing for each Set of ETV PEMs

Lead Type	Target Lead Level	Mean Levels ICP (mg/cm^2)	CoV* ICP
White Lead	0.3	0.30	13.3
	0.6	0.65	7.1
	1.0	0.99	3.9
	1.4	1.56	7.2
	2.0	1.85	5.6
	6.0	5.97	14.2
Yellow Lead	0.3	0.30	9.6
	0.6	0.62	4.1
	1.0	1.07	11.0
	1.4	1.42	4.1
	2.0	1.92	10.1
	6.0	6.88	5.2

* Coefficient of Variation (Standard Deviation/Mean x 100)

There are two user-specified parameters for the Stata SIMEX algorithm: 1) the number of replicate measurements for the covariate measured with error, and 2) the number of bootstrap samples used to estimate standard errors on regression parameters. In testing not detailed here, the sensitivity of the SIMEX algorithm to different settings of these parameters was investigated. It was determined that the qualitative results were not sensitive to the values used in the analysis. The values used were nine pseudo-replicates per PEM and 199 bootstrap samples, respectively.

The predicted regression curves and associated prediction intervals were generated in the interval 0.0 to 6.0 mg/cm^2 using Stata. The relevant prediction bounds (the upper bound at 0.8 mg/cm^2 and lower bound at 1.2 mg/cm^2) were assessed and the predicted false positive and false negative rates based on these prediction bounds were determined.

5.4.4 Goodness of Fit

To assess whether the logistic regression models fit the data well, standardized Pearson residuals were computed for every observation and those with an absolute value greater than two were flagged and plotted versus lead level. Standardized Pearson residuals greater than two are associated with observations that are not well fit by the model. In the logistic regression context observations that are not well fit might be those with high lead levels where the kit results were negative or very low lead levels where the kit results were positive. In the absence of categorical variables the standardized Pearson residuals should be normally distributed, so we would expect approximately 5% of the observations to have residuals with absolute value greater than two. In this case there are categorical covariates so the residuals are not strictly expected to be distributed normally but the proportion of observations with large residuals is still informative. That proportion is reported in Section 6.4.

5.5 Matrix Effects

The covariate-adjusted logistic regression model described in Section 5.4 was used to assess the significance of PEM parameters and the interactions among them on the performance of the test kits. PEM parameters were included in the model as explanatory variables associated with the Y_i response variable.

Comparison of the observed values of the response variable to predicted values obtained from models with and without the predictor variable in question was the guiding principle in the logistic regression model. The likelihood function is defined as

$$L(\beta) = \prod_{i=1}^{n} \pi(Y_i).[1 - \pi(Y_i)] \tag{5}$$

where $\pi(Y_i)$ is the conditional probability of Y_i =1 and $[1 - \pi(Y_i)]$ is the conditional probability of Y_{il} =0 given the vector of explanatory variables (X). For purposes of assessing the significance of a group of p predictor variables (where p can be 1 or more), we computed the likelihood ratio test statistic, G, as follows:

$$G = -2 \log_e [\text{likelihood without the p variables / likelihood with the p variables}] \tag{6}$$

Under the null hypothesis, this test statistic followed a chi-square distribution with p degrees of freedom. If the test statistic was greater than the 95th percentile of the chi-square distribution, then the group of variables, taken together, were statistically significant.

5.6 Operational Factors

There were no statistical calculations applicable to operational factors. Operational factors were determined qualitatively based on assessments from the Operator (both technical and non-technical) and the Battelle Verification Test Coordinator. Operational factors such as ease of use, operator bias, average cost, average time for kit operation, and helpfulness of manuals, were determined. Sustainability metrics such as volume and type of waste generated from the use of each test kit, toxicity of the chemicals used, and energy consumption are discussed. This discussion is based on how much waste was generated and what the waste was composed of, information from the vendor on how the waste should be properly handled, a summary of the pertinent MSDS information, when available, and noting whether the test kit used batteries, a power supply, or no energy source was needed. Information on how many tests each kit could perform as well as the shelf life of the test kit and chemicals used as part of the test kit was also reported.

Chapter 6
Test Results

The results for the ANDalyze Lead-in-Paint Test Kit are presented below for each of the performance parameters. The interpretation of results for this test kit relied on the use of a fluorimeter. A specific numerical response was provided by the fluorimeter, indicating the actual lead concentration, in mg/cm^2, of the test sample. All responses that indicated the presence of lead at or above 1.0 mg/cm^2 were considered positive for the purposes of the statistical analyses presented in this section. All responses that indicated that lead was present below the 1.0 mg/cm^2 threshold were considered negative for the purposes of this report. Only the qualitative results (i.e., positive or negative) were used in conducting the statistical analyses presented here.

In this report each PEM is associated with three definitions of lead levels:

- Target lead level - the expected concentration of each PEM as outlined in Table 3-1. These target lead levels were 0, 0.3, 0.6, 1.0, 1.4, 2.0, or 6.0 mg/cm^2.
- Confirmed lead level - the concentration as measured by the reference laboratory using ICP-AES analysis.
- Closest target lead level - the target level that is closest to the confirmed level. If a panel has a target lead level of 1.4 mg/cm^2 and a confirmed lead level of 1.9 mg/cm^2 then the closest target level is 2.0 mg/cm^2.

Under ideal circumstances the confirmed lead level would equal the target lead level, but this was sometimes not the case. Analyses where lead level was a categorical variable (i.e., consistency, precision, and sensitivity analyses) characterized the panels by their closest target lead level. Analyses where lead level was a continuous variable (i.e., the false positive/negative and logistic regression analyses) characterized the panels by their confirmed lead level. Each analysis described clearly which level was used to characterize the lead level.

6.1 False Positive and False Negative Rates

Observed false positive and negative rates were calculated based on confirmed lead levels as measured though ICP-AES analysis. For example, if the PEM was confirmed to have a lead level of 1.4 mg/cm^2, and the test kit returned a negative result, this would be considered a false negative. Table 3-1 details the target lead levels for the PEMs and the number of PEMs that were anticipated at each lead level. Because of variations in PEM production, the confirmed lead level of a particular PEM did not always match the target lead level. Table 6-1 compares the number of PEMs at the confirmed and target lead levels used for the false positive and negative analyses. The data are divided into three categories: those panels eligible for false positive analysis (lead levels up to and including 0.8 mg/cm^2), those excluded from false positive and false negative analyses (lead levels between 0.8 and 1.2 mg/cm^2) and those eligible for false negative analysis (lead levels 1.2 mg/cm^2 and above). If the confirmed lead levels had been equal to the target lead levels, all of the numbers would lie along the shaded diagonal. Because the confirmed levels sometimes differed significantly from the target levels, (i.e., the target lead level was at 0.6 mg/cm^2 but confirmed near 1.4 mg/cm^2) some panels appear in the off-diagonal table entries and were therefore included in portions of the analysis other than those for which they had been targeted.

Table 6-1. The number of panels in each false positive and false negative analysis category

		Confirmed Lead Levels			
		Eligible for False Positive Analysis	Excluded from Analysis	Eligible for False Negative Analysis	Total
Target Lead Levels	Eligible for False Positive Analysis	146	22	11	179
	Excluded from Analysis	7	43	22	72
	Eligible for False Negative Analysis	1	17	197	215
	Total	154	82	230	466

Tables 6-2 and 6-3 list the observed false positive and false negative rates for the ANDalyze Lead-in-Paint Test Kit under two sets of conditions:

- Table 6-2 shows the observed false positive results for panels with confirmed lead levels ≤ 0.8 mg/cm^2 and observed false negative results for panels with confirmed lead levels ≥ 1.2 mg/cm^2, per the RRP ruling[2].
- Table 6-3 shows observed false positive results for panels with confirmed lead levels < 1 mg/cm^2 and observed false negative results for panels with confirmed lead levels ≥ 1 mg/cm^2.

Results for both the technical and non-technical operator are presented. Results are presented as overall rates (i.e., false positive and negative results across all applicable PEMs combined) and also false positive and negative rates based on lead paint type (i.e., white or yellow lead), substrate (i.e., drywall, metal, plaster, or wood), and topcoat paint color (i.e., grey red or white).

The observed overall false positive rate for the technical and non-technical operators, based on confirmed lead levels of ≤0.8 mg/cm^2 (see Table 6-2) was 4-5%. Observed false positive rates across both operators based on PEM characteristics ranged from 0% for metal PEMs and PEMs with a red topcoat to 8% for yellow lead PEMs and PEMs with a grey topcoat. The observed false positive rates across different PEM factors were similar to the overall rates and were similar between the two operators. Observed false positive rates were 10% or lower in all cases.

Observed false negative rates for the ANDalyze Lead-in-Paint Test Kit were slightly higher than the observed false positive rates and were close to two times higher than the desired RRP rule[4] of a 5% or lower false negative rate.   Observed false negative rates for the technical operator were 9% overall. Observed false negative rates for substrate and topcoat color were similar to the overall rates found for each operator. Observed false negative rates for the non-technical operator were 12% overall with comparable observed false negative rates on the various PEM sub-factors except for metal PEMs. The observed false negative rate for the non-technical operator on metal PEMs was 22%.

The observed false positive rates for both the technical and non-technical operator using 1.0 mg/cm^2 as the deciding concentration (see Table 6-3) were slightly higher than those found using RRP rule concentration limits (PEMs with confirmed lead levels $\leq$0.8 mg/cm^2) (see Table 6-2), with overall observed false positive rates of 7% and 6%, respectively. The observed false positive rate for white lead panels was twice as high as that for yellow lead panels for the technical operator. The observed false negative rates were also higher overall for both operators than those found on PEMs with confirmed lead levels $\geq$1.2 mg/cm^2, 14% for the technical operator and 19% for the non-technical operator. Observed false positive rates for the substrates and topcoat colors were similar to the overall rate for the technical operator when panels were divided based on 1.0 mg/cm^2. As with Table 6-2, analysis of metal PEMs by the non-technical operator resulted in a higher observed false negative rate.

Table 6-2. ANDalyze Lead-in-Paint Test Kit false positive results for panels with confirmed lead levels ≤ 0.8 mg/cm² and false negative results for panels with confirmed lead levels ≥ 1.2 mg/cm²

	ANDalyze Lead-in-Paint Test Kit			
	False Positives[i]		False Negatives[ii]	
	Technical Operator	Non-technical Operator	Technical Operator	Non-technical Operator
Overall	15 / 308 = 5%	12 / 308 = 4%	41 / 462 = 9%	54 / 462 = 12%
None	0 / 70 = 0%	0 / 70 = 0%	NA	NA
White	4 / 120 = 3%	7 / 120 = 6%	23 / 232 = 10%	21 / 232 = 9%
Yellow	9 / 118 = 8%	5 / 118 = 4%	18 / 230 = 8%	33 / 230 = 14%
Drywall	4 / 76 = 5%	5 / 76 = 7%	13 / 124 = 10%	17 / 124 = 14%
Metal	0 / 94 = 0%	2 / 94 = 2%	5 / 90 = 6%	20 / 90 = 22%
Plaster	3 / 54 = 6%	1 / 54 = 2%	11 / 138 = 8%	3 / 138 = 2%
Wood	6 / 84 = 7%	4 / 84 = 5%	12 / 110 = 11%	14 / 110 = 13%
Grey	5 / 102 = 5%	8 / 102 = 8%	10 / 158 = 6%	16 / 158 = 10%
Red	3 / 104 = 3%	0 / 104 = 0%	17 / 140 = 12%	21 / 140 = 15%
White	5 / 102 = 5%	4 / 102 = 4%	14 / 164 = 9%	17 / 164 = 10%

[i]False positives on PEMs with confirmed lead levels ≤ 0.8 mg/cm²

[ii]False negatives on PEMs with confirmed lead levels ≥ 1.2 mg/ cm²
NA: If the paint did not contain lead then a false negative is not possible, those entries are 'NA' (not applicable).

Table 6-3. ANDalyze Lead-in-Paint Test Kit false positive results for panels with confirmed lead levels < 1 mg/cm^2 and false negative results for panels with confirmed lead levels ≥ 1 mg/cm^2

	ANDalyze Lead-in-Paint Test Kit			
	False Positives[i]		False Negatives[ii]	
	Technical Operator	Non-technical Operator	Technical Operator	Non-technical Operator
Overall	29 / 398 = 7%	25 / 398 = 6%	77 / 536 = 14%	100 / 536 = 19%
None	0 / 70 = 0%	0 / 70 = 0%	NA	NA
White	19 / 172 = 11%	11 / 172 = 6%	39 / 262 = 15%	39 / 262 = 15%
Yellow	10 / 156 = 6%	14 / 156 = 9%	38 / 274 = 14%	61 / 274 = 22%
Drywall	7 / 94 = 7%	9 / 94 = 10%	26 / 144 = 18%	33 / 144 = 23%
Metal	5 / 126 = 4%	3 / 126 = 2%	11 / 110 = 10%	33 / 110 = 30%
Plaster	6 / 68 = 9%	4 / 68 = 6%	24 / 164 = 15%	17 / 164 = 10%
Wood	11 / 110 = 10%	9 / 110 = 8%	16 / 118 = 14%	17 / 118 = 14%
Grey	8 / 134 = 6%	14 / 134 = 10%	21 / 178 = 12%	28 / 178 = 16%
Red	13 / 140 = 9%	4 / 140 = 3%	31 / 172 = 18%	42 / 172 = 24%
White	8 / 124 = 6%	7 / 124 = 6%	25 / 186 = 13%	30 / 186 = 16%

[i]False positives on PEMs with confirmed lead levels < 1.0 mg/cm^2

[ii]False negatives on PEMs with confirmed lead levels ≥ 1.0 mg/ cm^2

NA: If the paint did not contain lead then a false negative is not possible, those entries are 'NA' (not applicable).

Note that the observed false positive and negative rates presented in this section provide a general representation of the ability of the ANDalyze Lead-in-Paint Test Kit to correctly identify regulated lead paint when it is present or absent. The results presented in Table 6-2 provide rates based on the cut-off concentration (0.8 or 1.2 mg/cm^2) as well as all levels evaluated below or above those concentrations. To evaluate test kit performance based on the RRP rule, lead paint test kits should have a demonstrated probability (with 95% confidence) of a negative response at or above the regulated lead level ≤5% of the time. Test kits should also have a demonstrated probability (with 95% confidence) of a positive response below the regulated lead level ≤10% of the time. Because the RRP rule also indicated that test kit performance would not be based on lead levels between 0.8 and 1.2 mg/cm^2, the false positive and negative probabilities assessed in this report were then based around the excluded concentrations (of 0.8 and 1.2 mg/cm^2). False positive and negative rates associated with these criteria are discussed in Section 6.4.

6.2 Precision

To compute precision, it is first necessary to compute the number of replicate sets with consistent responses. Replicate sets are defined in the test/QA plan[1] to be groups of panels with similar lead levels. The target lead levels in this experiment were 0, 0.3, 0.6, 1.0, 1.4, 2, and 6 mg/cm^2 but the lead levels that were achieved, as confirmed by ICP-AES, sometimes varied from those target levels. To assemble replicate sets that represented the target lead levels, the panels were assigned to the replicate set that was nearest their confirmed lead level. In other words, if a particular panel was targeted for 0.3 mg/cm^2 but was measured to have 0.9 mg/cm^2 then it was assigned to the replicate set nearest 0.9 mg/cm^2, which is the set labeled 1.0 mg/cm^2. Table 6-4 shows the thresholds that defined the replicate set bins as well as the range of measured levels that fell in each bin.

Table 6-4. Actual lead levels and their replicate set labels

Replicate Set Bin Label (mg/cm^2) (Closest Target Lead Level)	Bin Thresholds (mg/cm^2)	Confirmed Lead Levels In This Bin (mg/cm^2)
0	Targeted to have zero lead	0.000 - 0.032
0.3	0 ≤ Confirmed Lead Level < 0.45	0.051 - 0.448
0.6	0.45 ≤ Confirmed Lead Level < 0.8	0.451 - 0.795
1	0.8 ≤ Confirmed Lead Level < 1.2	0.804 - 1.198
1.4	1.2 ≤Confirmed Lead Level < 1.7	1.218- 1.694
2	1.7 ≤ Confirmed Lead Level < 4	1.714 - 3.914
6	4 ≤ Confirmed Lead Level	4.280 - 15.23

Table 6-5 shows the number of panels in which confirmed lead levels fell nearest their target level and the number of panels whose confirmed levels fell closer to a level other than their target level. The shaded values along the diagonal of the table are the panels in which measured levels fell closer to their target than to any of the other targets. If all of the panels had measured levels that were equal to their target levels, then all of the numbers would lie along the diagonal of Table 6-5. The numbers off the diagonal represent panels with confirmed lead levels closer to some other target value. Note, for example, that of the 72 panels that were targeted to have 1.0 mg/cm^2 of lead, 43 achieved that level, one fell closer to 0.3 mg/cm^2, six fell closer to 0.6 mg/cm^2 than 1.0 mg/cm^2, 16 fell closer to 1.4 mg/cm^2, and six fell closer to 2.0 mg/cm^2 than to any other target level. In the consistency analysis described below, each panel was grouped into sets labeled with the target level that its measured level fell closest to, rather than by its target lead level.

Table 6-5. The number of panels at each target level and the number in each replicate set bin

Replicate Set Bin
(Target level that is closest to the panel's actual measured lead level)

Target Lead Level (mg/cm^2)	0	0.3	0.6	1	1.4	2	6	Total
0	35	-	-	-	-	-	-	35
0.3	-	62	7	3	-	-	-	72
0.6	-	5	37	19	9	2	-	72
1.0	-	1	6	43	16	6	-	72
1.4	-	-	1	16	46	8	1	72
2.0	-	-	-	1	5	62	3	71
6.0	-	-	-	-	-	-	72	72
Total	35	68	51	82	76	78	76	466

Table 6-6 lists consistency results for the ANDalyze Lead-in-Paint Test Kit by operator type, lead type, substrate, and lead level. Each table entry lists the number of test results with those characteristics (N) as well as the proportion of the results that were positive for lead (Pos). Table entries where the proportion is below 25% or above 75% are 'consistent', meaning that more than three-quarters of the results were the same (negative or positive). Table entries where the proportion of positive results ranges from 25% to 75% are considered to be 'inconsistent'. Inconsistent entries are shaded in the tables. Overall consistency results across all substrates for white and yellow lead panels for each operator type are also provided in the last row of Table 6-6. Results across both operators and lead paint types are provided in the last column of the table.

Overall inconsistencies for the non-technical operator were found at the 1.4 mg/cm^2 lead level for white lead PEMs (see the last row of Table 6-6). This was also true for drywall, metal, and wood substrates, with consistencies ranging from 50-75% at the 1.4 mg/cm^2 lead level for white lead PEMs. Overall inconsistencies were also found at the 1.0 mg/cm^2 lead level for the yellow lead PEMs. However, the lead level of inconsistencies on yellow-lead PEMs varied across substrates, with only the 0.0 and 0.3 mg/cm^2 lead levels not showing any inconsistencies regardless of substrate type. Overall inconsistencies for all PEMs for the non-technical operator were found at the 1.0 and 1.4 mg/cm^2 lead level.

Inconsistencies for the technical operator overall and across all white and yellow lead PEMs were at the 1.0 mg/cm^2 lead level. Inconsistencies were also found at 0.6 mg/cm^2 on yellow lead PEMs and 1.4 mg/cm^2 on white lead PEMs for some substrates. Across both operators and white and yellow lead PEMs, the test kit was inconsistent at 0.6 mg/cm^2 for drywall (28% consistent) and 1.4 mg/cm^2 for drywall, metal, and wood (74%, 72%, and 70% consistent, respectively) Across both operators and all substrates and lead paint type, the ANDalyze Lead-in-Paint Test Kit was inconsistent at only 1.0 mg/cm^2.

The consistency results provided in Table 6-6 were used to calculate precision. Precision was estimated for panels with no lead, white lead, and yellow lead and broken out by type of operator and then aggregated across both types of operators. For any column in Table 6-6, the precision is simply the proportion of consistent (unshaded) table entries in the rows for the four different substrates. The 'All' rows are not counted in the precision calculation because those table entries are summaries of the entries for the four substrates. Thus, precision was calculated as:

$$Precision(\%\ consistent\ results) = \frac{\#\ of\ unshaded\ table\ entries\ in\ the\ drywall, metal, plaster, and\ wood\ sections}{total\ entries\ in\ those\ sections} \quad (7)$$

Table 6-7 lists the results of the precision calculations for the ANDalyze Lead-in-Paint Test Kit. Higher proportions of consistent results indicate more consistency and higher precision.

The ANDalyze Lead-in-Paint Test Kit was precise on PEMs (100%) that contained no lead. The precision of the non-technical operator was higher than that of the technical operator on white lead PEMs (85% vs. 73%), while the results were reversed for the yellow lead PEMs, with the technical operator having a precision of 81% while the non-technical operator only had a precision of 66%. The overall precision across both operators was similar (79% and 73%) for both lead paint types.

Table 6-6. ANDalyze Lead-in-Paint Test Kit consistency results by operator type, lead type, substrate, and lead level

ANDalyze Lead-in-Paint Test Kit

	NON-TECHNICAL								TECHNICAL								TOTAL	
Lead Type	None		White		Yellow		Total		None		White		Yellow		Total		Total	
	N	Pos	N	Pos	N	Pos	N	Pos	N	Pos	N	Pos	N	Pos	N	Pos	N	Pos
DRYWALL																		
0	18	0%			2	0%	20	0%	18	0%			2	0%	20	0%	40	0%
0.3			16	0%	18	0%	34	0%			16	0%	18	0%	34	0%	68	0%
0.6			12	17%	10	30%	22	23%			12	0%	10	40%	22	18%	44	20%
1			18	0%	20	40%	38	20%			18	33%	20	20%	38	27%	76	23%
1.4			24	75%	18	67%	42	71%			24	75%	18	78%	42	76%	84	74%
2			22	100%	22	91%	44	95%			22	91%	22	95%	44	93%	88	94%
6			18	100%	20	85%	38	92%			18	100%	20	100%	38	100%	76	96%
METAL																		
0	18	0%			2	0%	20	0%	18	0%			2	0%	20	0%	40	0%
0.3			16	13%	18	0%	34	6%			16	0%	18	0%	34	0%	68	3%
0.6			22	0%	18	0%	40	0%			22	0%	18	0%	40	0%	80	0%
1			28	18%	24	13%	52	15%			28	36%	24	38%	52	37%	104	26%
1.4			10	50%	16	69%	26	59%			10	80%	16	88%	26	84%	52	72%
2			14	93%	12	67%	26	80%			14	93%	12	100%	26	96%	52	88%
6			20	100%	18	72%	38	86%			20	100%	18	100%	38	100%	76	93%
PLASTER																		
0	16	0%	2	0%			18	0%	16	0%	2	0%			18	0%	36	0%
0.3			16	0%	6	0%	22	0%			16	0%	6	0%	22	0%	44	0%
0.6			6	17%	8	0%	14	8%			6	0%	8	38%	14	19%	28	14%
1			16	50%	24	29%	40	40%			16	50%	24	33%	40	42%	80	41%
1.4			16	94%	30	93%	46	94%			16	75%	30	87%	46	81%	92	87%
2			34	100%	16	100%	50	100%			34	94%	16	100%	50	97%	100	99%
6			20	100%	22	100%	42	100%			20	100%	22	95%	42	98%	84	99%
WOOD																		
0	18	0%	4	0%			22	0%	18	0%	4	0%			22	0%	44	0%
0.3			16	0%	20	0%	36	0%			16	13%	20	0%	36	6%	72	3%
0.6			10	20%	16	13%	26	16%			10	20%	16	13%	26	16%	52	16%
1			20	15%	14	50%	34	33%			20	25%	14	29%	34	27%	68	30%
1.4			16	56%	22	73%	38	64%			16	75%	22	77%	38	76%	76	70%
2			20	95%	16	100%	36	98%			20	90%	16	100%	36	95%	72	96%
6			18	100%	18	100%	36	100%			18	100%	18	94%	36	97%	72	99%
ALL																		
0	70	0%					80	0%	70	0%					80	0%	160	0%
0.3			64	3%	62	0%	126	2%			64	3%	62	0%	126	2%	252	2%
0.6			50	10%	52	10%	102	10%			50	4%	52	17%	102	12%	204	10%
1			82	20%	82	30%	164	26%			82	35%	82	30%	164	34%	328	30%
1.4			66	71%	86	78%	152	74%			66	76%	86	83%	152	79%	304	77%
2			90	98%	66	91%	156	95%			90	92%	66	98%	156	95%	312	95%
6			76	100%	78	90%	154	95%			76	100%	78	97%	154	99%	304	98%

N = number of test results in each bin of the table
POS = Proportion of those N test results that were 'Positive' for the presence of lead.
Lead levels in the left-most column represent the target level closest to the measured level of lead in the panel.
Shaded cells represent 'inconsistent' results. i.e., % positive is between 25% and 75%

Table 6-7. ANDalyze Lead-in-Paint Test Kit precision results by lead type and operator type

	No Lead	White Lead	Yellow Lead[i]
Non-technical	4/4 = 100%	22/26 = 85%	17/26 = 66%
Technical	4/4 = 100%	19/26 = 73%	21/26 = 81%
All	8/8 = 100%	41/52 = 79%	38/52 = 73%

[i] Results were consistently negative across all lead levels for this test kit on yellow lead paint panels, even those samples containing detectable levels of lead.

6.3 Sensitivity

Sensitivity was calculated using the bottom six rows in Table 6-6. These rows aggregate results across all four substrates. For the white lead and yellow lead columns in these tables, the sensitivity is the lowest lead level ≥ 1 mg/cm^2 that is consistently detected with positive results (unshaded and > 75%). Ideally the kit would give consistently negative results for lead levels < 1 mg/cm^2 and consistently positive results for levels ≥ 1 mg/cm^2 so the optimal sensitivity results would be 1 across every row of Table 6-8.

Table 6-8. ANDalyze Lead-in-Paint Test Kit sensitivity results – lowest lead level for which the kit gave consistent positive results (mg/cm^2)

	Non-technical Operator			Technical Operator			All
Lead Type	White	Yellow	Total	White	Yellow	Total	Total
Sensitivity	2.0	1.4	2.0	1.4	1.4	1.4	1.4

Across all lead paint types and operators, the ANDalyze Lead-in-Paint Test Kit generated consistent positive results at 1.4 mg/cm^2 lead. When sensitivity is evaluated by operator type, consistently positive results were found at 1.4 mg/cm^2 on white and yellow as overall for the technical operator. Consistently positive responses were found at the 2.0 mg/cm^2 lead level for the non-technical operator on white lead PEMs and the 1.4 mg/cm^2 lead level for yellow lead PEMs. The overall sensitivity as determined through evaluations performed by the non-technical operator to be at the 2.0 mg/cm^2 lead level. This is higher than the sensitivity determined by evaluations from the technical operator.

Note that the ANDalyze Lead-in-Paint Test Kit is quantitative in nature. That is, the test kit provides the measured lead level in mg/cm^2 of the sample being evaluated. As such, the ANDalyze Lead-in-Paint Test Kit can provide results lower than 1.0 mg/cm^2, and in fact did. Table 6-9 presents the average concentration and standard deviation of the samples as indicated by the ANDalyze Lead-in-Paint Test Kit for each of the replicate bin sets. As shown in Table 6-9, the average values indicated by the ANDalyze Lead-in-Paint Test Kit are close to those of the replicate bin sets. However, as the standard deviation indicates, the range of concentrations given for samples within a particular replicate set are sometimes outside of the bin thresholds (see Table 6-4). Also, the concentrations given for PEMs in the 6.0 mg/cm^2 replicate set by the ANDalyze Lead-in-Paint Test Kit were sometimes small in comparison to the actual confirmed lead levels in this bin. Confirmed lead levels went up to 15 mg/cm^2, while the ANDalyze Lead-in-Paint Test Kit readings never went higher than 10 mg/cm^2. Concentrations indicated by the test kit were up to 7.5 times lower than confirmed lead levels for a PEM. This is likely because of the limited upper range of the calibration curve used for the fluorimeter.

Table 6-9. Average and standard deviation of ANDalyze Lead-in-Paint Test Kit results compared to the concentration of the replicate sets

Replicate Set Bin Label (mg/cm^2) (Closest Target Lead Level)	Average ANDalyze Lead-in-Paint Measured Lead Level (mg/cm^2)	ANDalyze Lead-in-Paint Measured Lead Level Standard Deviation (mg/cm^2)
0	BL[i]	0
0.3	0.089	0.25
0.6	0.49	0.69
1	0.86	0.29
1.4	1.46	0.79
2	2.17	0.87
6	3.49	2.07

[i] Below limit on fluorimeter; indicative of no lead present

6.4 Modeled Probability of Test Kit Response

Table 6-10 lists the explanatory variables which had significant ($p<0.05$) univariate associations with the probability of obtaining a positive test kit result. All potential explanatory variables except for lead type and operator type showed a statistically significant univariate association with the probability of a positive response. Lead level, substrate type, and topcoat color were significant in the multivariable model after backward selection. Table 6-11 lists the parameter estimates for the multivariable logistic regression models from the Stata SIMEX program. There were no statistically significant interactions between categorical covariates.

Table 6-12 lists the modeled probability of a positive test result for the ANDalyze Lead-in-Paint Test Kit when the lead level is 0.8 mg/cm^2 (PREDICTION) along with the upper bound of a 95% prediction interval (UPPER). That upper bound can be considered to be a worst-case estimate of the false positive probability when the true lead level is 0.8 mg/cm^2 (FALSE POS RATE). Ideally the numbers in the UPPER/FALSE POS RATE column would be ≤ 10%. Note that the FALSE POS RATE in Table 6-12 is higher than those in Tables 6-2 and 6-3. In those earlier tables the rates considered panels at a variety of comparatively low lead levels so some cases should have been easier for the kit to obtain the correct answer. In Table 6-12, the false positive rate is evaluated only at 0.8 mg/cm^2 so the rate does not benefit from the comparatively lower lead concentrations. Evaluating at only this level also ensures that a test kit can adequately perform at concentrations of lead paint closest to the current regulatory level.

Based on the upper prediction bound estimates shown in Table 6-12, the ANDalyze Lead-in-Paint Test Kit did not meet the false positive criteria for any scenario at the 0.8 mg/cm^2 lead level. The lowest upper bound prediction expected to be achieved at this lead level is 19.6% for a metal substrate with a red topcoat, and this rate is approximately two times the ideal rate.

Table 6-13 lists the modeled probability of a positive test result for the ANDalyze Lead-in-Paint Test Kit when the lead level is 1.2 mg/cm^2 (PREDICTION) along with the lower bound of a 95% prediction interval (LOWER). The difference between the lower bound and 100% can be considered to be a worst-case estimate of the false negative probability when the true lead level is 1.2 mg/cm^2 (FALSE NEG RATE). Ideally, for the purposes of the RRP rule, the numbers in the FALSE NEG RATE column would be ≤ 5%. Based on the lower bound estimates shown in Table 6-13, the ANDalyze Lead-in-Paint Test Kit did not meet the false negative criterion (<5%) at the 1.2 mg/cm^2 lead level.

Note that the FALSE NEG RATE in Table 6-12 is higher than those in Tables 6-2 and 6-3. In the earlier tables, the false negative rates considered panels at a variety of comparatively high lead levels so some cases should have been easier for the kit to obtain the correct answer. In Table 6-12, the false negative rate is evaluated only at 1.2 mg/cm^2 so the rate does not benefit from the comparatively higher lead concentrations. Evaluating at only this level also ensures that a test kit can adequately perform at concentrations of lead paint closest to the current regulatory level.

Table 6-10. ANDalyze Lead-in-Paint Test Kit univariate associations between probability of positive response and explanatory variables

Explanatory Variable	Significant Univariate Association?	Included in Multivariable Model?
Lead Level	Yes (p-value < 0.0001)	Yes
Lead Type	No (p-value = 0.9960)	No
Operator Type	No (p-value = 0.2116)	No
Substrate Type	Yes (p-value < 0.0001)	Yes
Topcoat Color	Yes (p-value = 0.0168)	Yes

Table 6-11. ANDalyze Lead-in-Paint Test Kit multivariable Stata SIMEX logistic regression parameter estimates

```
Simulation extrapolation                          No. of obs         =      1868
                                                  Bootstraps reps    =       199

Residual df  =      1861                          Wald F(6,1861)     =     15.86
                                                  Prob > F           =    0.0000

Variance Function: V(u) = u(1-u)                     [Bernoulli]
Link Function    : g(u) = log(u/(1-u))               [Logit]

------------------------------------------------------------------------------
             |              Bootstrap
      result |      Coef.   Std. Err.      t    P>|t|     [95% Conf. Interval]
-------------+----------------------------------------------------------------
Substrate    : (wood is the reference level)
     drywall |  -.1469332   .1650832    -0.89   0.374     -.470701    .1768345
       metal |  -.4716426   .1601065    -2.95   0.003    -.7856498   -.1576354
     plaster |   .4060918    .161856     2.51   0.012     .0886534    .7235301
Topcoat      : (white is the reference level)
        grey |   .0398478   .1474259     0.27   0.787    -.2492896    .3289852
         red |  -.3201983   .1320513    -2.42   0.015    -.5791826    -.061214

  lead level |   2.198884   .3316751     6.63   0.000      1.54839    2.849378
    constant |  -2.657648   .3875847    -6.86   0.000    -3.417795   -1.897502
------------------------------------------------------------------------------
```

Table 6-12. ANDalyze Lead-in-Paint Test Kit modeled probability of positive test results and upper 95% prediction bound when lead level = 0.8 mg/cm^2

TOPCOAT	SUBSTRATE	LEAD LEVEL	PREDICTION	UPPER (FALSE POS RATE)
GREY	DRYWALL	0.8	26.8%	32.4%
	METAL	0.8	20.9%	25.3%
	PLASTER	0.8	38.9%	45.1%
	WOOD	0.8	29.8%	35.3%
RED	DRYWALL	0.8	20.3%	25.4%
	METAL	0.8	15.6%	19.6%
	PLASTER	0.8	30.7%	37.1%
	WOOD	0.8	22.8%	27.9%
WHITE	DRYWALL	0.8	26.0%	31.6%
	METAL	0.8	20.3%	24.7%
	PLASTER	0.8	37.9%	45.2%
	WOOD	0.8	28.9%	35.2%

Table 6-13. ANDalyze Lead-in-Paint Test Kit modeled probability of positive test results, lower 95% prediction bound, and corresponding conservative estimate of the false negative rate when lead level = 1.2 mg/cm^2

TOPCOAT	SUBSTRATE	LEAD LEVEL	PREDICTION	LOWER	FALSE NEG RATE
GREY	DRYWALL	1.2	46.9%	40.6%	59.4%
	METAL	1.2	38.9%	32.8%	67.2%
	PLASTER	1.2	60.5%	55.0%	45.0%
	WOOD	1.2	50.5%	44.1%	55.9%
RED	DRYWALL	1.2	38.1%	32.4%	67.6%
	METAL	1.2	30.8%	25.5%	74.5%
	PLASTER	1.2	51.7%	46.0%	54.0%
	WOOD	1.2	41.6%	35.8%	64.2%
WHITE	DRYWALL	1.2	45.9%	40.4%	59.6%
	METAL	1.2	38.0%	32.6%	67.4%
	PLASTER	1.2	59.6%	53.8%	46.2%
	WOOD	1.2	49.5%	43.2%	56.8%

As another means of reporting the results for the ANDalyze Lead-in-Paint Test Kit, modeled probability curves were also plotted based on the results of the regression analysis. To better understand the information being provided in these probability curves, a brief explanation is presented here. Figure 6-1 shows that for the perfect or ideal test kit, the probability of a positive test result would be a step function. The probability of a positive result would be zero below 1.0 mg/cm^2 and 100% at or above 1.0 mg/cm^2. Under the RRP rule, a test kit must yield a demonstrated probability (with 95% confidence) of no more than 10% false positives at lead concentrations below 0.8 mg/cm^2 and a demonstrated probability (with 95% confidence) of no more than 5% false negatives at concentrations above 1.2 mg/cm^2. Figure 6-1 also shows a performance curve for a hypothetical test kit that achieves those rates. The upper bound of the 90% prediction interval is at 10% at 0.8 mg/cm^2 and the lower bound of the prediction interval is at 95% at 1.2 mg/cm^2.

One way to think of the test kit performance guidelines is in terms of regions of the probability plots. Figure 6-2 demonstrates this concept. For the kit to be within limits set up by the RRP rule, the probability curve must trace a path through the white region in the figure and must not stray into the shaded regions. If the curve crosses the shaded region at the left side of the graph then there are lead levels < 0.8 mg/cm^2 where the false positive rate is > 10%. If the curve crosses the shaded region at the right side of the graph then there are lead levels > 1.2 mg/cm^2 where the false negative rate is > 5%. Either type of intersection between the curve and the shaded region indicates that the kit does not meet the performance levels stipulated in the RRP rule.

Note that results for the region between 0.8 and 1.2 mg/cm^2 were not discussed in this report. This is consistent with the RRP rule stipulation that lead concentrations between 0.8 and 1.2 mg/cm^2 were not to be considered for the evaluation of the performance of lead paint test kits.

Figures 6-3 through 6-5 show the predicted probability of obtaining a positive test result using the ANDalyze Lead-in-Paint Test Kit over the full range of explanatory variables along with the bounds of a 90% prediction interval. Note that the upper and lower bounds of the 90% prediction interval may also be considered to be upper and lower 95% prediction bounds for one-sided inference. In every instance the upper end of the probability curves (above 1.2 mg/cm^2) pass through the shaded regions of the plot until above 2.0 mg/cm^2. The low ends of the curves (below 0.8 mg/cm^2) cross out of the shaded regions well below 0.8 mg/cm^2.

In every instance, both the upper end of the probability curves (above 1.2 mg/cm^2) and the lower end (below 0.8 mg/cm^2) pass through the shaded regions of the plot, indicating that the kit's false positive and false negative performance do not meet the RRP rule requirements.

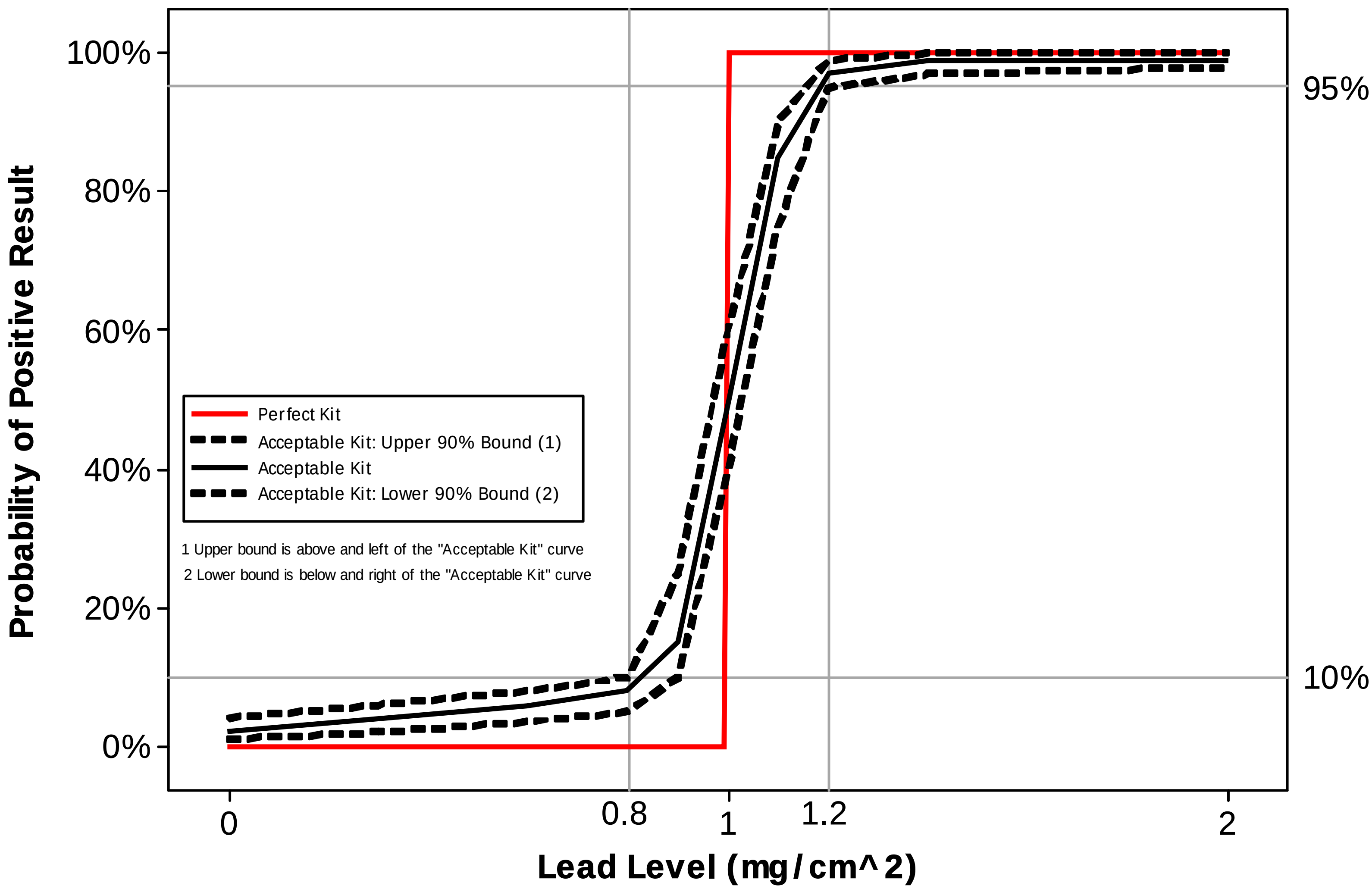

Figure 6-1. Probability curves that represent test kit results that are both perfect (red line) and within RRP rule criteria (black solid line).

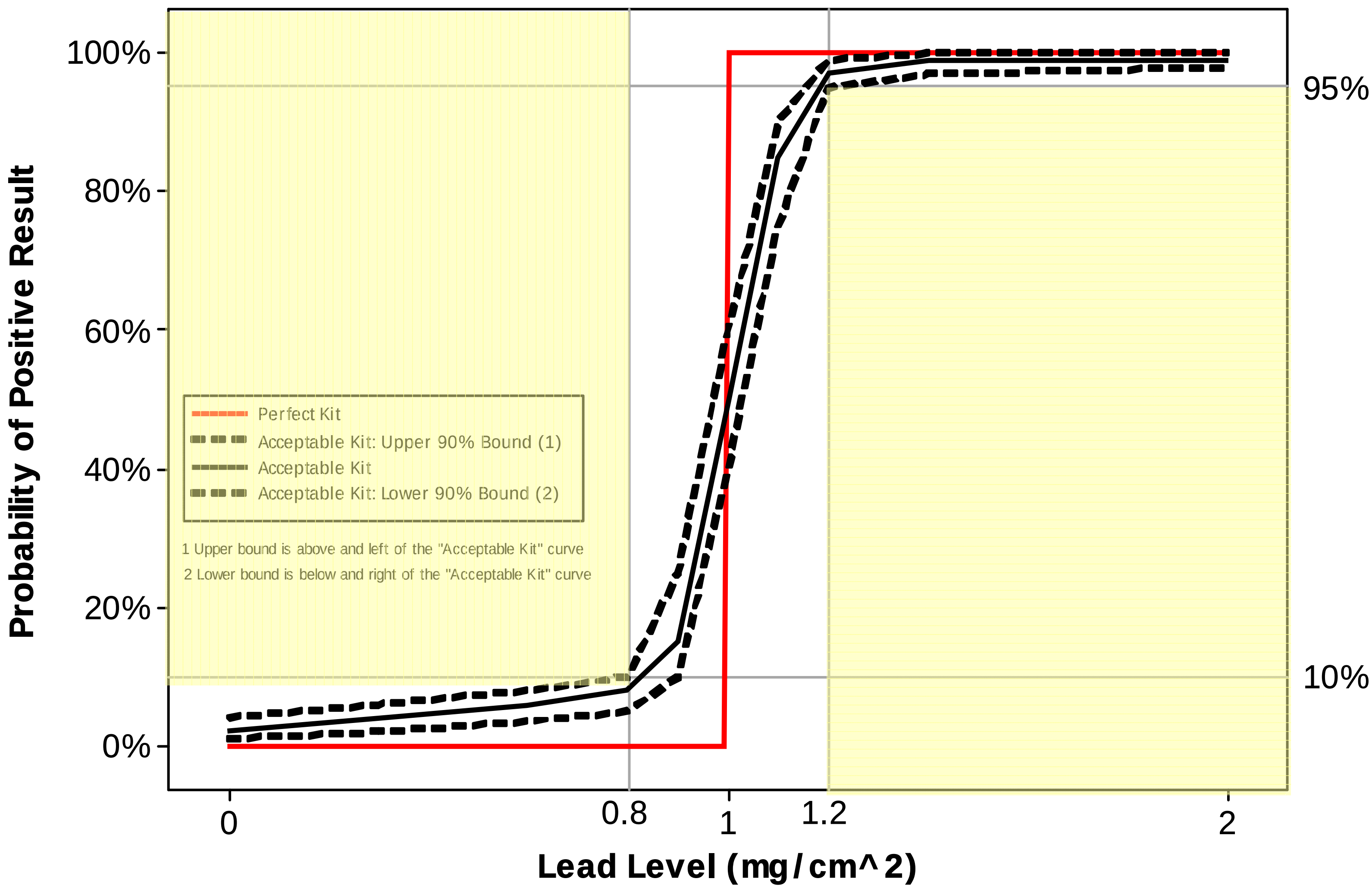

Figure 6-2. Probability curves with shaded region to denote performance results that meet RRP rule false positive and negative criteria. Test kits with curves that fall within the shaded region and avoid the white region meet the RRP rule.

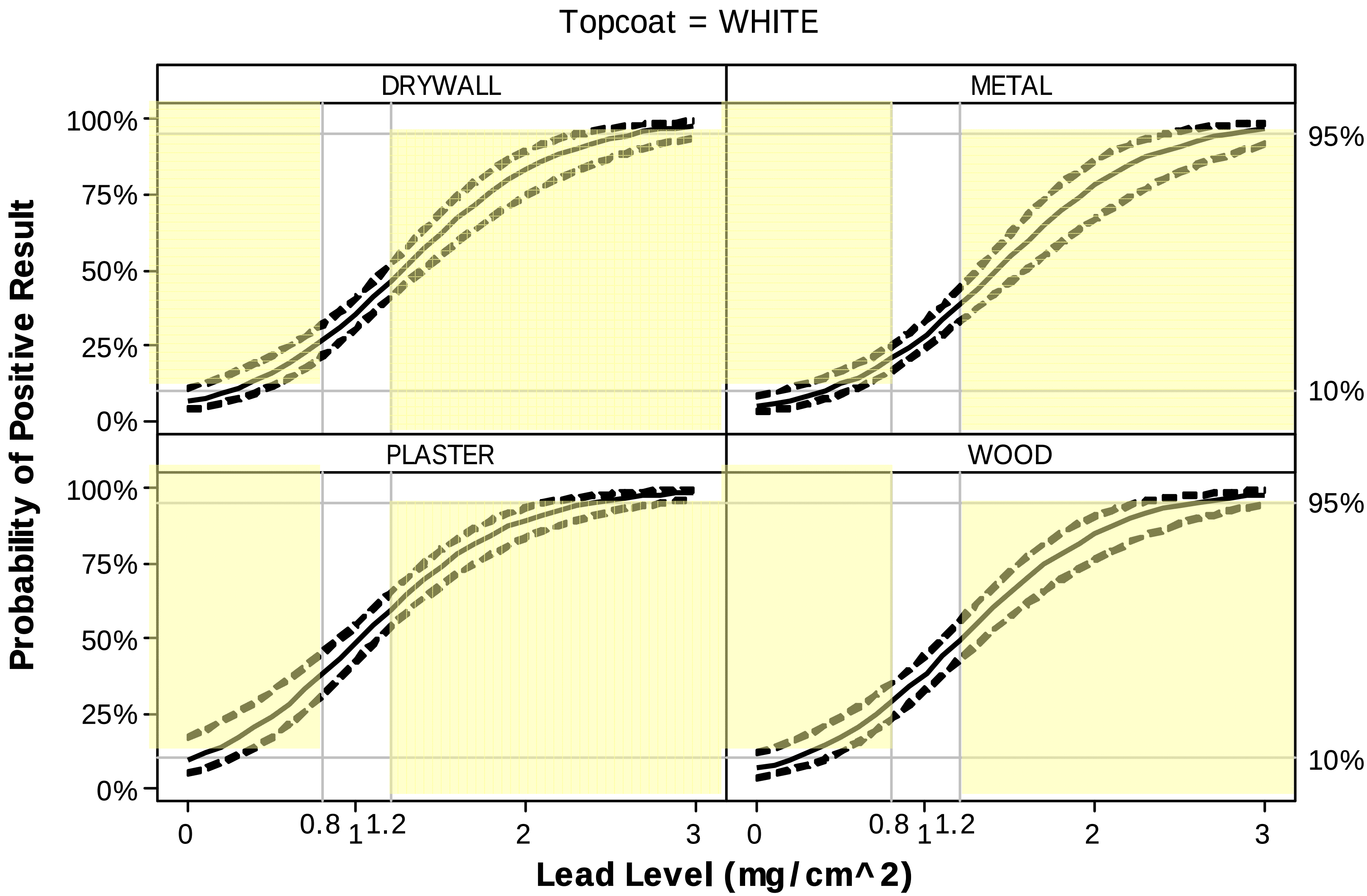

Figure 6-3. ANDalyze Lead-in-Paint Test Kit predicted probability of positive test result (solid lines) with 90% prediction interval (dotted lines) for a white paint topcoat on various substrates.

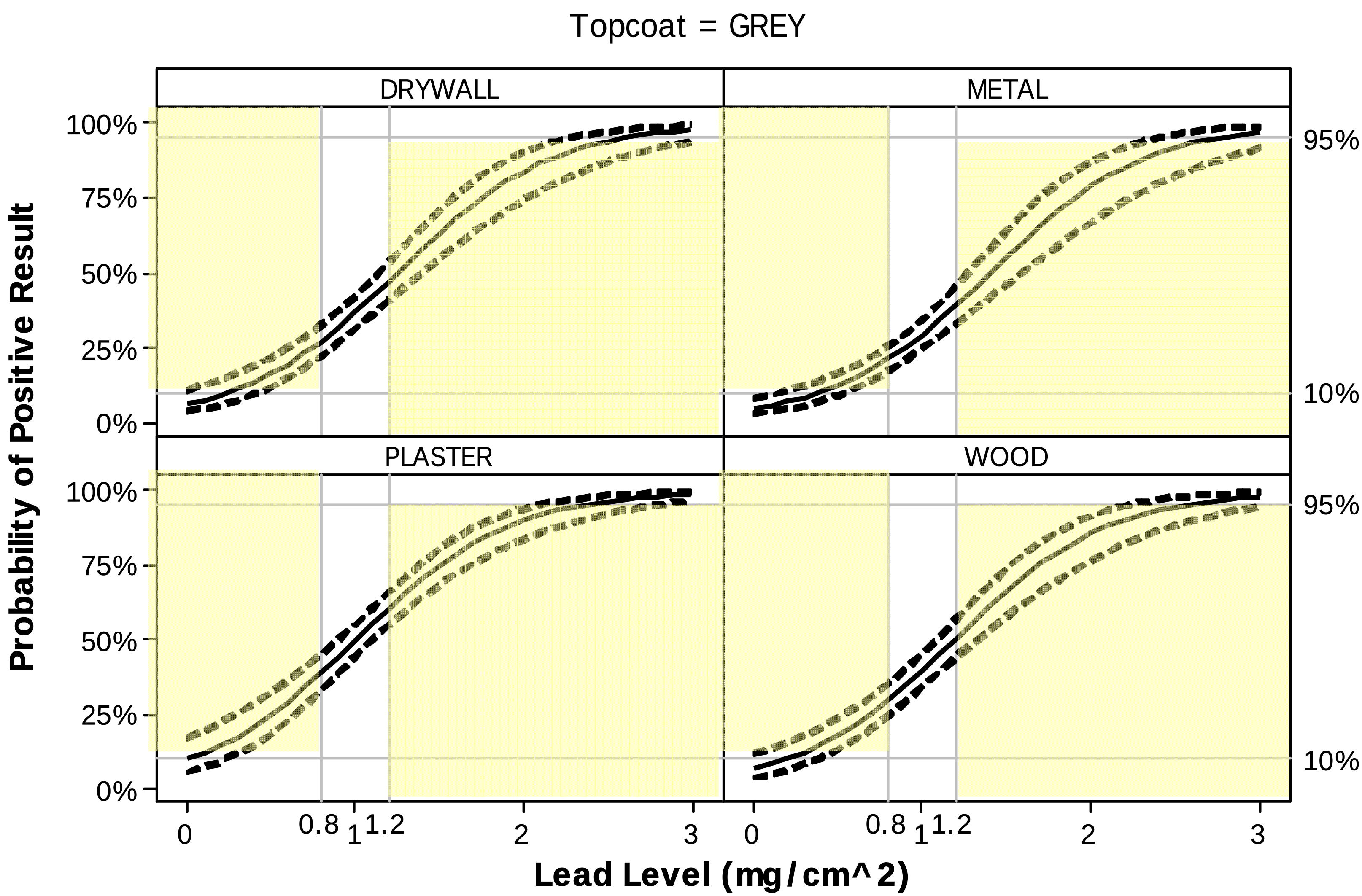

Figure 6-4. ANDalyze Lead-in-Paint Test Kit predicted probability of positive test result (solid lines) with 90% prediction interval (dotted lines) for a grey paint topcoat on various substrates.

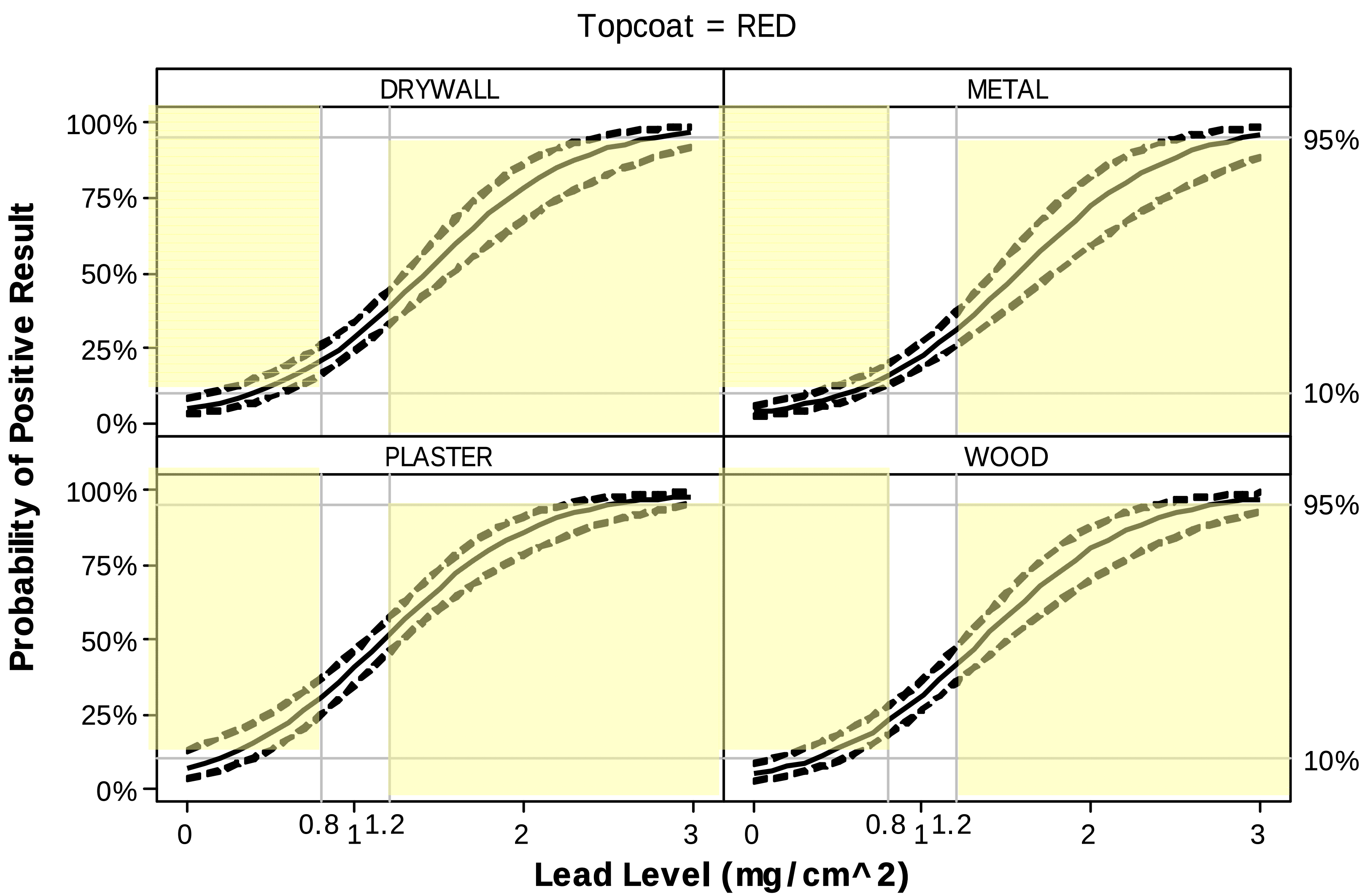

Figure 6-5. ANDalyze Lead-in-Paint Test Kit predicted probability of positive test result (solid lines) with 90% prediction interval (dotted lines) for a red paint topcoat on various substrates.

Based on the modeled probabilities shown in Figures 6-3 through 6-5, threshold values for false positive and negative rates were established for the ANDalyze Lead-in-Paint Test Kit. For the false positive rate, this threshold value is the lead level, with 95% confidence, below which the ANDalyze Lead-in-Paint Test Kit would yield fewer than 10% false positive results. For the false negative rate, this threshold value is the lead level, with 95% confidence, above which the ANDalyze Lead-in-Paint Test Kit would yield fewer than 5% false negative results. These threshold values are then the lead levels where the ANDalyze Lead-in-Paint Test Kit is predicted to meet the false positive and negative criteria set forth in the RRP rule.

Table 6-14 presents the false positive and negative threshold values for the ANDalyze Lead-in-Paint Test Kit. Threshold lead levels are provided for each topcoat and substrate combination shown in Tables 6-12 and 6-13.

Table 6-14. ANDalyze Lead-in-Paint Test Kit false positive and negative threshold values (95% confidence) based on the modeled probability of test results

TOPCOAT	SUBSTRATE	FALSE POSITIVE THRESHOLD (mg/cm^2)	FALSE NEGATIVE THRESHOLD (mg/cm^2)
GREY	DRYWALL	NA	3.13
	METAL	0.18	3.35
	PLASTER	NA	2.79
	WOOD	NA	3.05
RED	DRYWALL	0.16	3.33
	METAL	0.37	3.55
	PLASTER	NA	2.99
	WOOD	0.09	3.25
WHITE	DRYWALL	NA	3.13
	METAL	0.18	3.34
	PLASTER	NA	2.79
	WOOD	NA	3.05
OVERALL		0.20	3.15

NA in the FALSE POSITIVE THRESHOLD column means that the false positive rate was > 10% for all lead levels.
NA in the FALSE NEGATIVE THRESHOLD column means that the false negative rate was > 5% for all lead levels.

Table 6-14 indicates that overall, across all factors, the false positive threshold for the ANDalyze Lead-in-Paint Test Kit is 0.20 mg/cm^2. That is, this test kit is predicted, with 95% confidence, to not yield fewer than 10% false positive results until lead levels reach 0.20 mg/cm^2 or lower. The overall false negative threshold for the ANDalyze Lead-in-Paint Test Kit is 3.12 mg/cm^2. False positive and negative thresholds for individual combinations of factors were similar to the overall false positive and negative thresholds across all factors of significance. A false positive threshold could not be established for the plaster substrate with either a grey or white topcoat. The red topcoat plaster had a false positive threshold of 0.0 mg/cm^2.

Standardized Pearson residuals were calculated to assess goodness of fit of the logistic regression models. For the ANDalyze Lead-in-Paint Test Kit model, 95.1% of the residuals had absolute values smaller than two.

6.5 Matrix Effect

The matrix effects for the ANDalyze Lead-in-Paint Test Kit were evaluated with results in Table 6-11. The variables that were retained in the multivariable logistic regression model each add significant explanatory power to their respective models. Those variables are significantly associated with the probability of obtaining a positive test result from the kits tested in this study.

For the ANDalyze Lead-in-Paint Test Kit, Table 6-11 indicates that after controlling for the significant covariates, the likelihood of a positive test result is positively and significantly associated with: higher lead levels, plaster, drywall, and metal substrates, and grey and white topcoats.

6.6 Operational Factors

A tip sheet was also provided with the instructions, containing helpful hints in the test kit operation. The technical operator found the ANDalyze Lead-in-Paint Test Kit instructions to be clear, informative, and easy to follow. The non-technical operator received no training from the vendor and relied solely on the test kit instructions, tip sheet, and instructional DVD for his understanding of the operation of the test kit. He did not believe that the kit was easy to follow based solely on the supporting information.

Both the technical and non-technical operator stated that a significant amount of training and possibly previous experience or laboratory knowledge would be needed to successfully operate this test kit. The calibration step and number of complicated steps (including multiple pipetting steps) and pieces of equipment needed for the operation of this test kit could make it difficult for an average contractor or renovator to feel comfortable with the test kit and to conduct successful evaluations without extensive training. It was the opinion of the non-technical operator that the kit would be too complicated for the typical lead removal contractor. As an example, the grinding step was established as 3 minute per sample for this verification test. The ANDalyze Lead-in-Paint Test Kit instructions indicate that 10 minutes or more could be needed to grind a single paint sample. The completeness of grinding is subjective and is left to the operator to determine. The cleaning of the grinding tubes was a time-consuming step as each tube had to soak, filled with cleaning solution, for at least 30 minutes after each use. The use of the drill and drill bit was not complicated, but care must be taken to operate the drill as slowly as possible so as not to remove much, if any, of the substrate.

The ANDalyze Lead-in-Paint Test Kit, as supplied for this verification test, included a drill, modified half-inch drill bit, fluorimeter, razor blades, ruler, 15 mL grinding tubes and pestles, nitric acid, plastic pipettes, calibration solution and buffers, sensor housings, 30 mL plastic tubes pre-filled with testing buffer, 10 x 76 millimeter (mm) glass test tubes, 1 mL syringes, 10 µL mini-pipette and disposable tips, and grinder cleaning solution. The test kit instructions indicated that the kit should be stored at room temperature and out of direct sunlight. The housing sensors are sensitive to light and needed to be stored out of direct light until their use. Expiration dates were not supplied with any of the reagents. When new supplies were provided for verification testing, mixing of the new and old reagents was discouraged. The grinding tubes began to crack and wear down after repeated use and had to be thrown away in some instances. The point of their disposal was determined by the operator.

The test kit instructions indicated that appropriate safety precautions should be taken when using this test kit, such as wearing the necessary protective equipment and using caution when handling the drill to prevent injury. The extraction solution is 25% nitric acid and the kit instructions note that protective equipment should be used when handling the acid. Both the technical and non-technical operators followed general laboratory safety procedures and wore a lab coat, protective eyewear, and gloves at all times. A MSDS sheet was provided with the test kit for the acid solution.

All reagents came prepared and ready to use. The solutions used for different steps were easily identifiable within the kit. Storage conditions of the reagents were not marked on the containers, although the ANDalyze Lead-in-Paint Test Kit instruction manual did indicate storage requirements and a temperature range for test kit operation.

The waste generated for this test kit includes both liquid and solid waste. Solid waste included pipette tips, glass test tubes, housing sensors, plastic tubes, and disposable plastic pipettes. Liquid waste included approximately 2 mL of ground paint in nitric acid; 20 mL of testing buffer, 3 mL of calibration solutions, and 2-3 mL of cleaning solution. The ANDalyze Lead-in-Paint Test Kit tip sheet provided some waste disposal guidelines. It indicated that any used glass test tubes should be disposed of into a sturdy container, such as a cardboard box, and labeled as "Broken Glass". Any uncontaminated nitric acid solution should be neutralized with testing buffer or baking soda and flushed down the sink. If a positive result is obtained with the test kit, the sample would be assumed to contain lead. As such, any lead-containing waste, such as the paint extract, would be considered lead waste. As such, the ANDalyze Lead-in-Paint Test Kit instructions indicated that EPA and Department of Transportation (DOT) regulations pertaining to disposal of lead waste should be followed and non-positive tests (indicating that no lead at the regulated level is present) would be considered non-lead waste and disposed of with normal waste procedures. (Note: Because regulations for the disposal of wastes generated from the use of lead test kits may vary from state to state, EPA recommends that test kit users contact their state government agency for proper waste disposal requirements.) After preparation, the calibration tubes contain 60, 300, and 1500 ppb lead waste. The lead levels in these solutions are very small and the vendor recommends that they be disposed in the drain by flushing with enough water to dilute the lead to < 15 ppb (EPA limit for lead in drinking water). The vendor recommends diluting each buffer tube with at least 100X the volume of water. That is, use 100 mL of tap water to dilute the 1 mL buffer containing lead. Similarly, the buffer solutions containing the diluted lead paint extract from the sample should contain minimal levels of lead and could be flushed down the drain with copious amounts of water.

Interpretation of the fluorimeter output for the ANDalyze Lead-in-Paint Test Kit was easy to determine. Outputs were clear and well labeled. However, it was not clear how rugged the fluorimeter itself would be for taking into the field if paint samples were to be evaluated onsite.

The ANDalyze Lead-in-Paint Test Kit was viewed by both the technical and non-technical operators to be complicated and difficult to use. Operation of the test kit took approximately 18 minutes by both the technical and non-technical operator, not including the grinder washing procedure. A normal power supply was needed for the operation of the fluorimeter. As of the writing of this report, the cost of the fluorimeter is $1500 and the cost for the drill is $310. The cost for all necessary consumables to conduct 50 tests is $300.

Chapter 7
Performance Summary

The observed overall false positive rate for the ANDalyze Lead-in-Paint Test Kit on PEMs with confirmed lead levels of ≤ 0.8 mg/cm^2 was 4-5% for both the technical and non-technical operator. Observed false positive rates across both operators based on PEM characteristics ranged from 0% for metal PEMs and PEMs with a red topcoat to 8% for PEMs with a grey topcoat. The observed false positive rates across different PEM factors (e.g., substrate type, topcoat color, lead paint type) were similar to the overall rates and were similar between the two operators. Observed false positive rates were 10% or lower in all cases.

Observed false negative rates for the technical operator were 9% overall. The observed false negative rates for substrate and topcoat color were similar to the overall rates found for each operator. Observed false negative rates for the non-technical operator were 12% overall with comparable observed false negative rates on the various PEM sub-factors except for metal PEMs. The observed false negative rate for the non-technical operator on metal PEMs was 22%.

Overall observed false positive rates on PEMs with confirmed lead levels <1.0 mg/cm^2 for both the technical and non-technical operator were slightly higher than those found on PEMs with confirmed lead levels ≤ 0.8 mg/cm^2, with overall observed false positive rates of 7% and 6%, respectively. The observed false negative rates were also higher overall for both operators than those found on PEMs with confirmed lead levels ≥ 1.2 mg/cm^2, 14% for the technical operator and 19% for the non-technical operator.

The ANDalyze Lead-in-Paint Test Kit produced consistent responses (either positive or negative) across all substrates and paint types at all lead levels except one; the ANDalyze Lead-in-Paint Test Kit results were inconsistent at 1.0 mg/cm^2.

Results from the ANDalyze Lead-in-Paint Test Kit indicated 100% precision on PEMs that contained no lead. The precision observed when the kit was operated by the non-technical operator was higher than that of the technical operator on white lead PEMs (85% vs. 73%), while the results were reversed for the yellow lead PEMs, with the technical operator having a precision of 81% while the non-technical operator had a precision of 66%. The overall precision across both operators was similar (79% and 73%) for both lead paint types.

Across all lead paint types and operators, the lowest lead level for which the ANDalyze Lead-in-Paint Test Kit generated consistent positive results was 1.4 mg/cm^2 lead. When sensitivity was evaluated by operator type, consistently positive results were found at 1.4 mg/cm^2 on white and yellow as overall for the technical operator. Consistently positive responses were found at the 2.0 mg/cm^2 lead level for the non-technical operator on white lead PEMs and the 1.4 mg/cm^2

lead level for yellow lead PEMs. The overall sensitivity as determined through evaluations performed by the non-technical operator to be at the 2.0 mg/cm^2 lead level. This is a higher than the sensitivity determined by evaluations from the technical operator.

Under the RRP rule[4], a test kit must yield a demonstrated probability (with 95% confidence) of no more than 10% false positives at lead concentrations below 0.8 mg/cm^2 and a demonstrated probability (with 95% confidence) of no more than 5% false negatives at concentrations above 1.2 mg/cm^2 to meet the rule criteria. Based on the upper bound estimates of the modeled probability of the ANDalyze Lead-in-Paint Test Kit, the technology did not meet the false positive criterion at the 0.8 mg/cm^2 lead level. The lowest false positive rate expected to be achieved at this lead level is 19.6% for a metal substrate with a red topcoat, and this rate is approximately two times the 10% false positive rate specified in the RRP rule. All false negative rates obtained using the ANDalyze Lead-in-Paint Test Kit results were above the 5% criterion established by the RRP rule. False negative rates were predicted to range from 45.0 to 74.5%.

Based on the modeled probabilities, the overall false positive threshold value (i.e., the lead level, with 95% confidence, below which the test kit would yield fewer than 10% false positive results) for the ANDalyze Lead-in-Paint Test Kit is 0.20 mg/cm^2. Across all factors of significance, the overall false negative threshold (the lead level, with 95% confidence, above which the test kit would yield fewer than 5% false negative results) for the ANDalyze Lead-in-Paint Test Kit is 3.12 mg/cm^2.

After controlling for the significant covariates, the likelihood of a positive test result is positively and significantly associated with: higher lead levels, drywall, metal and plaster substrates, and a grey and white topcoat. It is not significantly and positively associated with red topcoats or wood substrates.

The technical operator found the ANDalyze Lead-in-Paint Test Kit instructions to be clear, informative, and easy to follow. The non-technical operator, however, did not. A tip sheet was also provided with the instructions, containing helpful hints in the test kit operation. Both the technical and non-technical operator stated that a significant amount of training and possibly previous experience or laboratory knowledge would be needed to successfully operate this test kit.

All reagents came prepared and ready to use. The solutions used for different steps were easily identifiable within the kit. Storage conditions of the reagents were not marked on the containers, although the ANDalyze Lead-in-Paint Test Kit instruction manual did indicate storage requirements and a temperature range for test kit operation.

The ANDalyze Lead-in-Paint Test Kit, as supplied for this verification test, included, a drill, modified half-inch drill bit, fluorimeter, razor blades, ruler, 15 mL grinding tubes and pestles, nitric acid, plastic pipettes, calibration solution and buffers, sensor housings, 30 mL plastic tubes pre-filled with testing buffer, 10 x 76 mm glass test tubes, 1 mL syringes, 10 μL mini-pipette and disposable tips, and grinder cleaning solution.

The waste generated for this test kit included both liquid and solid waste. Solid waste included pipette tips, glass test tubes, housing sensors, plastic tubes, and disposable plastic pipettes. Liquid waste included approximately 2 mL of ground paint in nitric acid; 20 mL of testing buffer, 3 mL of calibration solutions, and 2-3 mL of cleaning solution. The ANDalyze Lead-in-Paint Test Kit tip sheet provided some waste disposal guidelines. It indicated that any used glass test tubes should be disposed of into a sturdy container, such as a cardboard box, and labeled as “Broken Glass”. Any uncontaminated nitric acid solution should be neutralized with testing buffer or baking soda and flushed down the sink. If a positive result is obtained with the test kit, the sample would be assumed to contain lead. As such, any lead-containing waste, such as the paint extract, calibration standards and tubes, and syringes, would be considered lead waste. As such, the ANDalyze Lead-in-Paint Test Kit instructions indicated that EPA and DOT regulations pertaining to disposal of lead waste should be followed and non-positive tests (indicating that no lead at the regulated level is present) would be considered non-lead waste and disposed of with normal waste procedures. (Note: Because regulations for the disposal of wastes generated from the use of lead test kits may vary from state to state, EPA recommends that test kit users contact their state government agency for proper waste disposal requirements.)

Operation of the test kit took approximately 18 minutes by both the technical and non-technical operator, not including the grinder washing procedure. A normal power supply was needed for the operation of the fluorimeter. At the time of the writing of this report, the cost of the fluorimeter is $1500 and the cost for the drill is $310. The cost for all necessary consumables to conduct 50 tests is $300.

Chapter 8
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Appendix A

Performance Evaluation Materials
Summary Information

List of Abbreviations and Acronyms

AMS	Advanced Monitoring Systems
ASTM	American Society for Testing and Materials
CCV	continuing calibration verification
CoV	coefficient of variation
CRM	certified reference material
DI	deionized
EPA	U.S. Environmental Protection Agency
ESTE	Environmental and Sustainable Technology Evaluations
ETV	Environmental Technology Verification
FT	film thickness
HVLP	high volume/low pressure
ICP-AES	inductively coupled plasma-atomic emission spectrometry
ICS	interference check sample
ICV	initial calibration verification
LCS	laboratory control spike
μg/L	micrograms per liter
μL	microliters
mg/cm^2	milligrams per centimeter squared
mg/kg	milligrams per kilogram
mL	milliliter
MSDS	material safety data sheets
NLLAP	National Lead Laboratory Accreditation Program
PEM	performance evaluation material
ppb	parts per billion
QA	quality assurance
QC	quality control
QMP	quality management plan
RH	relative humidity
RPD	relative percent difference
SOP	standard operating procedure

Section A1
Preparation of
Performance Evaluation Materials

Executive Summary

Battelle prepared a batch of performance evaluation materials (PEMs) for use in an Environmental Technology Verification (ETV) program evaluation of the performance of lead paint test kits. These PEMs encompass two lead types (white lead [lead carbonate] and yellow lead [lead chromate]), four separate substrates (metal, wood, drywall, and plaster), and six lead levels within each lead type (0.3, 0.6, 1.0, 1.4, 2.0, and 6.0 mg/cm^2). The goal of the production was to produce panels at a specified lead level with minimal variability across and within panels. The study design called for a verification and homogeneity study involving inductively coupled plasma (ICP) testing of the painted metal panels to determine applied lead levels. Initial application procedures included spray application for paints at 2.0 and 6.0 mg/cm^2, but testing indicated that spray application yielded high variability in lead levels. As a result, the Battelle team, in consultation with U.S. Environmental Protection Agency (EPA), decided to apply all lead paint layers via drawdown bar, which enables more precision in the thickness of the paint layer applied. Later in the development process, continued high variability measurements led to the team's decision to include silica in the formulations of each lead paint to thicken the paint and allow for a more even coating.

Verification and homogeneity testing was conducted for all 12 lead paints as well as the one no-lead control paint. Verification testing determined the formulation and drawdown bar best suited to yield a particular lead level. Homogeneity test results were assessed for proximity to target lead levels, lead level range, and variability within and between panels. All paints passed verification and homogeneity testing.

After completing the verification and homogeneity testing, base paint layers were applied for all 12 sets of lead paints (two lead types by six lead levels) and the no-lead paint. Paint chips were sampled and analyzed from the metal reference panels within each set of PEMs. The metal reference panel measurements met target specifications for all sets of PEMs. All nine sets of 468 panels *each* were appropriately labeled and packaged. All reference PEM concentrations and homogeneity results were reviewed and approved by EPA prior to full-scale production of a set.

Study Design

The initial study design specified production of the ETV PEMs using six lead levels (0.3, 0.6, 1.0, 1.4, 2.0, and 6.0 mg/cm^2), two lead types (white and yellow lead), four substrates (wood, metal, drywall, and plaster), and three topcoat colors (white, red-orange, and grey-black), as specified in Table A-1. For the wood substrates, both poplar and pine wood panels were produced, segregated, and uniquely labeled to be consistent with the design in Table A-1.

The final design specified production of 624 panels for each of seven test kits for a total of 4,368 panels. Late in the development process, the planned evaluation design changed so that only 468 panels were required to test each of nine test kits for a total of 4,212 PEMs needed for the ETV test.

Table A-1: PEMs Produced for ETV Evaluation

Lead Type	Lead Level (mg/cm^2)	Substrate	# Samples Produced Per Test Kit by Topcoat Color: White	Red-Orange	Grey-Black	Total	7 Test Kits
Control Blank	0	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
White Lead (Lead Carbonate)	0.3	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
	0.6	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
	1.0	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
	1.4	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
	2.0	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
	6.0	Wood-Poplar	2	2	2	6	42
		Wood-Pine	2	2	2	6	42
		Metal	4	4	4	12	84
		Drywall	4	4	4	12	84
		Plaster	4	4	4	12	84
Yellow Lead (Lead Chromate)	0.3, 0.6, 1.0, 1.4, 2.0, 6.0	Wood-Poplar	2 panels per cell for Wood substrates, 4 panels per cell for other substrates (same design as White Lead panels)			36	252
		Wood-Pine				36	252
		Metal				72	504
		Drywall				72	504
		Plaster				72	504
	Subtotal - Per Test Kit		208	208	208	624	4,368

The original design plan called for a target lead concentration of 0.3 mg/cm^2 for a set of PEMs. During the writing of the ETV test/quality assurance plan, preliminary ICP results indicated that the target level for this set of PEMs might be closer to 0.4 mg/cm^2. The preliminary results were used in the ETV test plan.

Substrate Preparation

The ETV PEMs included four different substrate types – metal, wood, drywall, and plaster; although two types of wood (pine and poplar) were utilized. The following bulleted lists describe the steps taken to prepare each of the types of substrates.

Metal

- Iron Phosphate Steel panels 0.032″ x 3″ x 3″ were placed in an isopropyl alcohol bath and carefully wiped and dried before being placed in plastic bags prior to coating.
- The solvent wipe step was performed to ensure that residue oils/fingerprints from the manufacturing processes were removed.

Wood

- Wood (pine and poplar) was purchased in 4″ widths, planed, and cut into 3″ x 3″ panels.
- PEMs were placed into constant temperature and humidity conditioning rooms prior to coating application to ensure uniform water content through each panel prior to coating. [Note that plaster and drywall panels are less sensitive to water absorption prior to coating.]

Drywall

- 4″ x 8″ x 3/8″ gypsum drywall sheets were cut into 3″ x 3″ panels.

Plaster

- Two joint compound materials were evaluated for ease of application and smoothness to ensure the best surface for coating. USG Joint Compound provided the smoothest surface and was used to coat panels at about 1/32″ thickness.
- A 3″ x 4′ strip of 3/8″ thick gypsum drywall was placed into jig, then plaster joint compound was smoothed over top surface to a precise 1/32″ thickness. Plastered drywall strips were then cut down into 3″ x 3″ panels.

Sealer Application to Drywall and Plaster PEMs

- Stacks of drywall and plaster PEMs were sealed on cut edges with no lead latex primer/sealer to eliminate dusting.

All panels were then placed in constant temperature and humidity conditioning rooms prior to coating application.

Spray Application Facilities and Equipment

Battelle’s laboratory includes a walk-in spray booth capable of this type of production as well as air handling equipment and monitors to ensure the safety of Battelle staff. Although the 0.3 and 6.0 mg/cm^2 white lead and no-lead paints were applied via spray application, all other application of lead paint layers was performed using drawdown bars in a laboratory setting. All topcoats were applied by spray application in the spray booth. Details on the equipment used in these processes are listed below.

Spray Booth

- 10′ x 10′ x 7.5′ double door spray booth
- Compressed air supply for spray equipment
- Spray equipment consists of a high volume/low pressure (HVLP) gravity fed DeVilbiss spray gun
- Plastic sheeting covering walls and floor to minimize clean-up time

Conditioning Rooms

- Constant temperature (75°Farenheit)) and humidity (50% relative humidity [RH])) rooms for substrate conditioning (the variability in temperature and RH is not tracked in those rooms)
- Substrates were conditioned both before and after coating application. Wood substrates were conditioned a minimum of two weeks prior to coating. All substrates were conditioned a minimum of 48 hours after coating and before bagging and wrapping.
- Plastic covering was placed on the floor to minimize clean-up time after transporting drying racks from the coating application lab into the conditioning rooms.

Environmental Health and Safety

Battelle developed a health and safety plan related to producing lead-based paint and PEMs coated with these paints. The plan was approved internally by appropriate environmental safety and health personnel. Environmental monitoring during paint mixing and spraying activities determined that lead exposure levels for workers were below Occupational Safety and Health Administration standards. Some of the components of the safety plan included:

- All staff and any visitors were required to have documented hazard communication training on lead.
- Baseline and post-work blood-lead levels were obtained for those Battelle staff that conducted the paint mixing and spray painting.
- Respirators were used during leaded paint production
- Spray application operations staff were required to have a physical, appropriate training, and to pass a respirator fit test.
- The interior of the spray booth was covered with plastic or other material that could be easily removed and was then disposed of as hazardous waste.
- The area in front of the booth was set up as a change-out area where personal protective equipment, such as coveralls, etc., could be removed without spreading lead outside of the area.
- Warning signs restricting access were posted at the paint booth door.

Preparation of Linseed Oil Based Leaded Paints

To formulate historically accurate lead-based paints to apply to PEMs, Battelle consulted Bennett's *The Chemical Formulary – A Collection of Valuable, Timely, Practical Commercial Formulae and Recipes for Making Thousands of Products in Many Fields of Industry, Volume VI.*[1] The Chemical Formulary had been printed with revisions every year until at least 1998. Sample formulations from this reference are listed below in Table A-2. Since the paints produced for the ETV verification of lead test kits were being applied to metal, drywall, plaster and wood, Battelle used a combination of formulations from *Chapter Thirteen – Paint, Varnish, Lacquer and Other Coatings* to ensure adhesion to all substrates. Battelle reviewed the various relevant historical formulations and developed formulations to apply to the PEMs that would work best for application to the four substrates being used, i.e. would provide the best adhesion to the variety of substrates required while achieving desired target lead levels.

Table A-2: Paint Formulations from The Chemical Formulary

Floor Painting and Finishing (p. 281) (for raw wood)	**Plaster, Primer (p. 332)**	**Exterior House Paint Pigments White (p. 328)**
Soft Paste White Lead, 100 lb. Raw Linseed Oil, 3 gal. Turpentine, 2 gal. Liquid Drier, 1 pt.	White Lead, Semi-Paste, 100 lb. Interior Varnish, 4 gal. Linseed Oil, Kettle Bodied, 2 gal. Turpentine, ¾ gal.	35% Leaded Oxide, 45 lb. White Lead, 18 lb. Titanium Dioxide, 15 lb. Inert, 22 lb. (Battelle used Zinc Oxide)

In preparing the lead-based paints for the PEMs, Battelle used a combination of raw and boiled linseed oil to ensure realistic drying time and good adhesion to the variety of substrates. A variety of other formulas in the reference also mix these two resins.

A similar formulation was also found in Charles Uebele's *Paint Making and Color Grinding: A Practical Treatise for Paint Manufacturers and Factory Managers*[2]. The excerpt below explains the difference in formulation requirements based on the substrate to which the paint will be applied.

> *"CHAPTER XXV - DIPPING PAINTS.*
> *Dipping Paints for Wood or Metal require to be made specially for either surface, as that intended for wood will not always serve the purpose for metal. The paint for wood requires to contain a pigment that acts as a filler, while tin or smooth sheet iron or steel does not necessarily need it, in fact, it is best without it for certain metallic surfaces. The function of a dipping paint is, first of all, to economize in labor, to cover uniformly any article immersed in it, and to dip freely without leaving fringes of paint at the edges and dry equally all over the surface thus coated.*
>
> *Metal Preservative Red may be made by grinding a base of 40 pounds bright red oxide of 95 per cent, purity, 8 pounds red lead, 2 pounds zinc chromate, 25 pounds floated silex or silica in 25 pounds raw linseed oil thinning same with 5 gallons raw linseed oil, 1 gallon hard gum japan and Y% gallon turps. This will produce 12 gallons of paint weighing a trifle over ll pounds per gallon. By substituting a long stock of hard gum varnish for part of the 5 gallons raw oil a hard drying product will be the result."*

In support of achieving consistent application of the lead-based paints in terms of film thickness and lead level, Battelle investigated additions of various elements to mitigate settling and improve application. Silicon dioxide was selected for this purpose because it was present in pre-1978 leaded paints, is used for thickening and anti-settling properties in modern paint formulations, and achieved the most consistent results. Battelle established the historical precedent for including silica in paints in a technical report submitted to EPA on February 19, 2009[3].

The primer and topcoats applied to the PEMs on top of the lead-based paints (or base paint for the no-lead panels) all contain some form of Diatomaceous Silica, as well. The primer and three topcoats applied are listed below.

- Sherwin Williams brand PreRite Bonding Primer
- Sherwin Williams Classic 99 Interior Satin Latex color Extra White
- Sherwin Williams Classic 99 Interior Satin Latex color 7047 Software (Grey)
- Sherwin Williams Accents Interior Satin Latex color 6867 Fireworks (red-orange)

Section 2 of the primer Material Safety Data Sheet (MSDS) specifies that the primer contains 9% quartz. Quartz is referred to as "Crystalline Silica" in Section 11 of the MSDS. The MSDSs for the three topcoats specify Cristobalite (CAS 14464-46-1) as an ingredient, which is a synonym for silicon dioxide and also referred to as Crystalline Silica in Section 11. All panels have some level of silica in the topcoat layers.

The paint formulations used for this effort were based on historical records. Primary ingredients included zinc oxide, raw and boiled linseed oil, turpentine, Japan drier, either lead carbonate or lead chromate, and titanium dioxide (used to balance the levels of lead). Nine different paint formulations were produced as dictated by the two lead pigments (lead carbonate and lead chromate) and the six different lead levels in addition to the zero lead level control. The formulations were designed to consistently achieve the lead levels required when applied at typical wet film builds.

The paint formulations are shown in Tables A-3 and A-4 below. Since the molecular compositions of the two lead pigments are different, the formulations have accounted for these differences by adjusting the load levels. However, the formulations for the 0% lead chromate and carbonate were the same because no lead pigment was used in either.

Table A-3. White Lead (Lead Carbonate) Paint Formulations

0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0 Lead	0 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.79	59.67%	1491.75
Pb CO_3	51	1401047-267	American Elements	0.00	0.00%	0.00
TiO2	37	931407T.12	DuPont	6.16	24.86%	621.56
Linseed Oil	7.8	83734	Recochem Inc.	1.48	5.97%	149.18
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.60%	14.92
Turpentine	7.0	83304	Recochem Inc.	2.16	8.70%	217.55
Japan Drier	7.0	PJD 40	Barr	0.05	0.20%	5.04
	Total			24.8	100%	2500

Sample reduced to 60% solids, 0% of TS-100 silica added then sprayed to thickness.

0.3% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0.3 Lead	0.3 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.85	59.08%	1477.08
Pb CO_3	51	1401047-267	American Elements	1.49	5.91%	147.71
TiO2	37	931407T.12	DuPont	4.95	19.69%	492.36
Linseed Oil	7.8	83734	Recochem Inc.	1.49	5.91%	147.71
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.77
Turpentine	7	83304	Recochem Inc.	2.17	8.62%	215.41
Japan Drier	7	PJD 40	Barr	0.05	0.20%	4.97
	Total			25.1	100%	2500

Sample reduced to 60% solids, 0% of TS-100 silica added then sprayed to 3 mils wet.

0.6% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0.6 Lead	0.6 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.77	58.45%	1461.22
Pb CO_3	51	1401047-267	American Elements	1.77	7.00%	175.11
TiO2	37	931407T.12	DuPont	4.92	19.47%	486.74
Linseed Oil	7.8	83734	Recochem Inc.	1.48	5.86%	146.42
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.84
Turpentine	7	83304	Recochem Inc.	2.15	8.51%	212.70
Japan Drier	7	PJD 40	Barr	0.03	0.12%	2.97
	Total			25.3	100%	2500

Sample reduced to 70% solids, 0.7% of TS-100 silica added then drawdown with # 24 bar.

1.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	1.0 Lead	1.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.40	55.53%	832.88
Pb CO_3	51	1401047-267	American Elements	3.00	11.57%	173.52
TiO2	37	931407T.12	DuPont	4.80	18.51%	277.63
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.56%	8.33
Turpentine	7	83304	Recochem Inc.	2.10	8.10%	121.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.89
	Total			25.9	100%	1500

This formulation will be used to produce 0.6% and 1.4% lead levels at different coating thickness.

1.4% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	1.4 Lead	1.4 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	13.22	50.94%	764.16
Pb CO_3	51	1401047-267	American Elements	4.21	16.22%	243.35
TiO2	37	931407T.12	DuPont	4.81	18.54%	278.03
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.24
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.54%	8.09
Turpentine	7	83304	Recochem Inc.	2.1	8.09%	121.39
Japan Drier	7	PJD 40	Barr	0.03	0.12%	1.73
	Total			26.0	100%	1500

Sample reduced to 70 % solids, 1.5% of TS-100 silica added then drawdown with # 54 bar.

2.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	2.0 Lead	2.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	12.88	48.16%	722.42
Pb CO_3	51	1401047-267	American Elements	6.08	22.73%	340.98
TiO2	37	931407T.12	DuPont	4.12	15.41%	231.17
Linseed Oil	7.8	83734	Recochem Inc.	1.41	5.28%	79.20
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.53%	7.92
Turpentine	7	83304	Recochem Inc.	2.06	7.70%	115.50
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.80
	Total			26.7	100%	1500

Sample reduced to 65% solids, 1.5% of TS-100 silica added then drawdown with #40 bar.

6.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	6.0 Lead	6.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	4.70	16.73%	250.89
Pb CO_3	51	1401047-267	American Elements	18.10	64.49%	967.34
TiO2	37	931407T.12	DuPont	1.57	5.58%	83.63
Linseed Oil	7.8	83734	Recochem Inc.	1.43	5.09%	76.40
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.51%	7.64
Turpentine	7	83304	Recochem Inc.	2.09	7.43%	111.42
Japan Drier	7	PJD 40	Barr	0.05	0.18%	2.67
	Total			28.1	100%	1500

Sample reduced to 70% solids, 1% of TS-100 silica added then sprayed to thickness.

0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0 Lead	0 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.79	59.67%	1491.75
Pb CO_3	51	1401047-267	American Elements	0.00	0.00%	0.00
TiO2	37	931407T.12	DuPont	6.16	24.86%	621.56
Linseed Oil	7.8	83734	Recochem Inc.	1.48	5.97%	149.18
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.60%	14.92
Turpentine	7.0	83304	Recochem Inc.	2.16	8.70%	217.55
Japan Drier	7.0	PJD 40	Barr	0.05	0.20%	5.04
	Total			24.8	100%	2500

Sample reduced to 60% solids, 0% of TS-100 silica added then sprayed to thickness.

0.3% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0.3 Lead	0.3 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.85	59.08%	1477.08
Pb CO_3	51	1401047-267	American Elements	1.49	5.91%	147.71
TiO2	37	931407T.12	DuPont	4.95	19.69%	492.36
Linseed Oil	7.8	83734	Recochem Inc.	1.49	5.91%	147.71
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.77
Turpentine	7	83304	Recochem Inc.	2.17	8.62%	215.41
Japan Drier	7	PJD 40	Barr	0.05	0.20%	4.97
	Total			25.1	100%	2500

Sample reduced to 60% solids, 0% of TS-100 silica added then sprayed to 3 mils wet.

0.6% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	0.6 Lead	0.6 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.77	58.45%	1461.22
Pb CO_3	51	1401047-267	American Elements	1.77	7.00%	175.11
TiO2	37	931407T.12	DuPont	4.92	19.47%	486.74
Linseed Oil	7.8	83734	Recochem Inc.	1.48	5.86%	146.42
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.84
Turpentine	7	83304	Recochem Inc.	2.15	8.51%	212.70
Japan Drier	7	PJD 40	Barr	0.03	0.12%	2.97
	Total			25.3	100%	2500

Sample reduced to 70% solids, 0.7% of TS-100 silica added then drawdown with # 24 bar.

1.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	1.0 Lead	1.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.40	55.53%	832.88
Pb CO_3	51	1401047-267	American Elements	3.00	11.57%	173.52
TiO2	37	931407T.12	DuPont	4.80	18.51%	277.63
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.56%	8.33
Turpentine	7	83304	Recochem Inc.	2.10	8.10%	121.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.89
	Total			25.9	100%	1500

This formulation will be used to produce 0.6% and 1.4% lead levels at different coating thickness.

1.4% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	1.4 Lead	1.4 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	13.22	50.94%	764.16
Pb CO_3	51	1401047-267	American Elements	4.21	16.22%	243.35
TiO2	37	931407T.12	DuPont	4.81	18.54%	278.03
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.24
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.54%	8.09
Turpentine	7	83304	Recochem Inc.	2.1	8.09%	121.39
Japan Drier	7	PJD 40	Barr	0.03	0.12%	1.73
	Total			26.0	100%	1500

Sample reduced to 70 % solids, 1.5% of TS-100 silica added then drawdown with # 54 bar.

2.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	2.0 Lead	2.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	12.88	48.16%	722.42
Pb CO_3	51	1401047-267	American Elements	6.08	22.73%	340.98
TiO2	37	931407T.12	DuPont	4.12	15.41%	231.17
Linseed Oil	7.8	83734	Recochem Inc.	1.41	5.28%	79.20
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.53%	7.92
Turpentine	7	83304	Recochem Inc.	2.06	7.70%	115.50
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.80
	Total			26.7	100%	1500

Sample reduced to 65% solids, 1.5% of TS-100 silica added then drawdown with #40 bar.

6.0% Lead Carbonate Paint Formulation

Materials	GW	Lot#	Supplier	6.0 Lead	6.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	4.70	16.73%	250.89
Pb CO_3	51	1401047-267	American Elements	18.10	64.49%	967.34
TiO2	37	931407T.12	DuPont	1.57	5.58%	83.63
Linseed Oil	7.8	83734	Recochem Inc.	1.43	5.09%	76.40
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.51%	7.64
Turpentine	7	83304	Recochem Inc.	2.09	7.43%	111.42
Japan Drier	7	PJD 40	Barr	0.05	0.18%	2.67
	Total			28.1	100%	1500

Sample reduced to 70% solids, 1% of TS-100 silica added then sprayed to thickness.

Table A-4. Yellow Lead (Lead Chromate) Paint Formulations

0.3% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	0.3 Lead	0.3 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.97	60.03%	1500.74
$PbCrO_4$	51	1401047-267	American Elements	1.10	4.40%	110.05
TiO2	37	931407T.12	DuPont	4.99	20.01%	500.25
Linseed Oil	7.8	83734	Recochem Inc.	1.50	6.00%	150.07
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.60%	15.01
Turpentine	7	83304	Recochem Inc.	2.18	8.75%	218.86
Japan Drier	7	PJD 40	Barr	0.05	0.20%	5.01
	Total			24.9	100%	2500

Sample reduced to 70 % solids, 0.7% of TS-100 silica added then drawdown with #34 bar.

0.6% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	0.6 Lead	0.6 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.65	57.52%	1437.97
$PbCrO_4$	51	1401047-267	American Elements	2.15	8.44%	211.03
TiO2	37	931407T.12	DuPont	4.88	19.16%	478.99
Linseed Oil	7.8	83734	Recochem Inc.	1.47	5.77%	144.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.72
Turpentine	7	83304	Recochem Inc.	2.14	8.40%	210.05
Japan Drier	7	PJD 40	Barr	0.03	0.12%	2.94
	Total			25.5	100%	2500

Sample reduced to 70 % solids, 1.5% of TS-100 silica added then drawdown with #24 bar.

1.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	1.0 Lead	1.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.40	55.53%	832.88
$PbCrO_4$	51	1401047-267	American Elements	3.00	11.57%	173.52
TiO2	37	931407T.12	DuPont	4.80	18.51%	277.63
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.56%	8.33
Turpentine	7	83304	Recochem Inc.	2.10	8.10%	121.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.89
	Total			25.9	100%	1500

This formulation will be used to produce 0.6% and 1.4% lead levels at different coating thickness.

Sample reduced to 70 % solids, 1% of TS-100 silica added then drawdown with #48 bar.

1.4% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	1.4 Lead	1.4 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	13.21	50.40%	1260.02
$PbCrO_4$	51	1401047-267	American Elements	5.09	19.42%	485.50
TiO2	37	931407T.12	DuPont	4.2	16.02%	400.61
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.49%	137.35
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.53%	13.35
Turpentine	7	83304	Recochem Inc.	2.1	8.01%	200.31
Japan Drier	7	PJD 40	Barr	0.03	0.11%	2.86
	Total			26.2	100%	2500

Sample reduced to 70 % solids, 1% of Aerosil 200 silica added then drawdown with #60 bar.

2.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	2.0 Lead	2.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	10.90	42.32%	1058.12
$PbCrO_4$	51	1401047-267	American Elements	7.17	27.83%	695.72
TiO2	37	931407T.12	DuPont	3.81	14.81%	370.34
Linseed Oil	7.8	83734	Recochem Inc.	1.49	5.80%	145.00
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.58%	14.50
Turpentine	7	83304	Recochem Inc.	2.18	8.46%	211.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	4.85
	Total			25.8	100%	2500

Sample reduced to 70% solids, 1.5% of TS-100 silica added then drawdown with #42 bar.

6.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	6.0 Lead	6.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	1.65	5.99%	149.69
$PbCrO_4$	51	1401047-267	American Elements	21.43	77.84%	1946.00
TiO2	37	931407T.12	DuPont	0.55	2.00%	49.90
Linseed Oil	7.8	83734	Recochem Inc.	1.51	5.47%	136.75
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.55%	13.68
Turpentine	7	83304	Recochem Inc.	2.20	7.98%	199.43
Japan Drier	7	PJD 40	Barr	0.05	0.18%	4.54
	Total			27.5	100%	2500

Sample reduced to 70% solids, 2% of TS-100 silica added then drawdown with #54 bar.

0.3% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	0.3 Lead	0.3 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.97	60.03%	1500.74
$PbCrO_4$	51	1401047-267	American Elements	1.10	4.40%	110.05
TiO2	37	931407T.12	DuPont	4.99	20.01%	500.25
Linseed Oil	7.8	83734	Recochem Inc.	1.50	6.00%	150.07
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.60%	15.01
Turpentine	7	83304	Recochem Inc.	2.18	8.75%	218.86
Japan Drier	7	PJD 40	Barr	0.05	0.20%	5.01
	Total			24.9	100%	2500

Sample reduced to 70 % solids, 0.7% of TS-100 silica added then drawdown with #34 bar.

0.6% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	0.6 Lead	0.6 Lead % by wt.	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.65	57.52%	1437.97
$PbCrO_4$	51	1401047-267	American Elements	2.15	8.44%	211.03
TiO2	37	931407T.12	DuPont	4.88	19.16%	478.99
Linseed Oil	7.8	83734	Recochem Inc.	1.47	5.77%	144.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.59%	14.72
Turpentine	7	83304	Recochem Inc.	2.14	8.40%	210.05
Japan Drier	7	PJD 40	Barr	0.03	0.12%	2.94
	Total			25.5	100%	2500

Sample reduced to 70 % solids, 1.5% of TS-100 silica added then drawdown with #24 bar.

1.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	1.0 Lead	1.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	14.40	55.53%	832.88
$PbCrO_4$	51	1401047-267	American Elements	3.00	11.57%	173.52
TiO2	37	931407T.12	DuPont	4.80	18.51%	277.63
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.55%	83.29
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.56%	8.33
Turpentine	7	83304	Recochem Inc.	2.10	8.10%	121.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	2.89
	Total			25.9	100%	1500

This formulation will be used to produce 0.6% and 1.4% lead levels at different coating thickness.

Sample reduced to 70 % solids, 1% of TS-100 silica added then drawdown with #48 bar.

1.4% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	1.4 Lead	1.4 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	13.21	50.40%	1260.02
$PbCrO_4$	51	1401047-267	American Elements	5.09	19.42%	485.50
TiO2	37	931407T.12	DuPont	4.2	16.02%	400.61
Linseed Oil	7.8	83734	Recochem Inc.	1.44	5.49%	137.35
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.14	0.53%	13.35
Turpentine	7	83304	Recochem Inc.	2.1	8.01%	200.31
Japan Drier	7	PJD 40	Barr	0.03	0.11%	2.86
	Total			26.2	100%	2500

Sample reduced to 70 % solids, 1% of Aerosil 200 silica added then drawdown with #60 bar.

2.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	2.0 Lead	2.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	10.90	42.32%	1058.12
$PbCrO_4$	51	1401047-267	American Elements	7.17	27.83%	695.72
TiO2	37	931407T.12	DuPont	3.81	14.81%	370.34
Linseed Oil	7.8	83734	Recochem Inc.	1.49	5.80%	145.00
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.58%	14.50
Turpentine	7	83304	Recochem Inc.	2.18	8.46%	211.46
Japan Drier	7	PJD 40	Barr	0.05	0.19%	4.85
	Total			25.8	100%	2500

Sample reduced to 70% solids, 1.5% of TS-100 silica added then drawdown with #42 bar.

6.0% Lead Chromate Paint Formulation

Materials	GW	Lot#	Supplier	6.0 Lead	6.0 Lead % by wt	Gram wt
ZnO	47.3	ZC-X013	The Carry Co.	1.65	5.99%	149.69
$PbCrO_4$	51	1401047-267	American Elements	21.43	77.84%	1946.00
TiO2	37	931407T.12	DuPont	0.55	2.00%	49.90
Linseed Oil	7.8	83734	Recochem Inc.	1.51	5.47%	136.75
Boiled Linseed Oil	7.7	83404	Recochem Inc.	0.15	0.55%	13.68
Turpentine	7	83304	Recochem Inc.	2.20	7.98%	199.43
Japan Drier	7	PJD 40	Barr	0.05	0.18%	4.54
	Total			27.5	100%	2500

Sample reduced to 70% solids, 2% of TS-100 silica added then drawdown with #54 bar.

Paint Formulation Procedures

The paint samples were produced using standard painting production procedures in the Battelle laboratories, including pre-mixing, media grinding of pigment and binder resin, and paint letdown with resin and solvents. This procedure has been used for paint production both in the laboratory and in commercial paint manufacturing for over 50 years. The equipment utilized in this procedure includes the following:

- Variac that controls the speed of the dispersator
- High speed dispersator using a 5” diameter blade on the end of the mixing shaft
- Ice bath and ice
- Balance
- Paint cans
- Medium paint filters
- Red Devil paint shaker

Following are the detailed steps in the paint formulation procedure:

1. Add enough turpentine to cover mixing blade.
2. Start mixer at low speed.
3. Add zinc oxide slowly for 3-5 minutes, increasing mixing speed as needed to maintain appropriate grind viscosity as visually evaluated by an operator skilled in the art.
4. Add turpentine as needed to keep the batch rolling.
5. Mix additional 10 minutes after addition of zinc oxide.
6. Add lead pigment slowly for 2-4 minutes, increasing mixing speed as needed.
7. Add turpentine as needed to keep the batch rolling.
8. Mix additional 10 minutes after addition of lead pigments.
9. Add titanium dioxide slowly for 3-5 minutes, increasing mixing speed as needed.
10. Add turpentine as needed to keep the batch rolling.
11. Mix for 60-90 minutes, or until batch viscosity decreases, determined by rolling action of the batch.
12. Check Hegman, if < 5 continue to mix, and check Hegman every 10 minutes.[4]
13. When Hegman reaches ≠ or > 5, start the let down, which includes adding all remaining liquid raw materials after the pigment and extenders have been dispersed adequately.
14. Add boiled and raw linseed oil slowly and decrease mixing speed.
15. Add turpentine to wash out linseed oil container.
16. Mix additional 10 minutes after addition of linseed oils.
17. Add Japan drier drop wise to batch.
18. Mix additional 10 minutes after addition of Japan drier.
19. Tare quart cans.
20. Filter batch with medium paint filters into tared quart cans.
21. Note net weight and log book number of batch on quart cans.
22. Yields about 1½ quarts of lead paint per batch.
23. Allow paint to set overnight.
24. Shake paint with Red Devil paint shaker for about 10 minutes take samples for % solids check.
25. Check paint solids with moisture balance and record average of three test results on formulation sheet.

26. Store paint in aluminum cans in laboratory hood until future use.

Verification and Homogeneity Studies

Various batches of paint were prepared for the initial verification tests – one targeting each lead level. Each paint was applied via drawdown or hand spraying to 3.5″ x 5″ metal panels attached to a wooden rack. For each paint type and concentration batch, panels were coated to determine proper film thickness, formulations, and drawdown bars to use, if applicable, to achieve each desired lead level. Subsequently, homogeneity panels were coated to investigate ability to achieve target lead levels and variability within and across panels. Verification and homogeneity studies were performed on metal panels only due to ease and accuracy of sample extraction, i.e., it was easiest to obtain a 1 inch square sample from the metal surface which led to the most accurate measurements of lead content in the sampled area, which was critical for verification purposes.

After drying, paint chip samples were obtained from the metal panels following ASTM E1729.[5] Laboratory analysis for lead by inductively coupled plasma-atomic emission spectrometry (ICP-AES) was planned and conducted at an independent National Lead Laboratory Accreditation Program (NLLAP)-accredited laboratory, Schneider Laboratories, Inc. ICP-AES testing was conducted on three panels for each lead level with four samples obtained from each panel, referred to as Homogeneity Panels since the primary purpose of the samples was to assess consistency of lead levels across and within panels. The paint chips were digested using EPA Method 3050B[6] and the ICP-AES analysis was conducted following EPA Method 6010C[7] as well as the Schneider Laboratories, Inc. ICP SOP.[8] The laboratory electronically reported lead level measurements along with quality control (QC) sample results. Laboratory spike and duplicate results as well as calibration verification sample results were supplied and reviewed for each batch of samples analyzed. Acceptable recoveries for spike samples ranged from 80% to 120%. Acceptable recoveries for calibration verification samples were 90-110%. Acceptable duplicate samples had a relative percent difference of 25% or less. Percent recoveries for calibration verification samples ranged from 93-110%. Recoveries for QC spike samples ranged from 92-115%. All duplicate samples had less than 25% relative percent difference. There were no QC failures or problems.

Film thickness measurements were obtained by Battelle for each paint sample taken. Results of the final batches of homogeneity samples for each set of PEMs are included in Table A-5. Results were evaluated to determine correspondence to target lead levels and level of variability as measured by the coefficient of variation (CoV), the standard deviation divided by the mean. The production plan, agreed to in advance, specified a minimum acceptability of a CoV of less than 15 percent. Following analysis, the results were forwarded to EPA with recommendations regarding ability to proceed with production or the need for additional homogeneity testing. The results shown in Table A-5 met the acceptability requirements and were thus deemed acceptable for proceeding with the production of sets of PEMs at each lead level.

Table A-5. Results from Final Homogeneity Testing on Metal Substrates for Each Set of ETV PEMs

Lead Type	Target Lead Level	Mean Levels		CoV*	
		ICP (mg/cm^2)	FT (mils)	ICP	FT**
White Lead	0.3	0.30	0.79	13.3	6.1
	0.6	0.65	0.95	7.1	5.7
	1.0	0.99	1.26	3.9	3.4
	1.4	1.56	1.72	7.2	3.5
	2.0	1.85	1.48	5.6	7.0
	6.0	5.97	1.94	14.2	8.3
Lead Chromate	0.3	0.30	1.16	9.6	4.0
	0.6	0.62	0.98	4.1	9.1
	1.0	1.07	1.50	11.0	7.4
	1.4	1.42	1.89	4.1	6.8
	2.0	1.92	1.38	10.1	2.4
	6.0	6.88	1.81	5.2	3.3

* Coefficient of Variation (Standard Deviation/Mean x 100)
** Film thickness

Production Application of Lead Paint Coatings

Based on the results from the Verification and Homogeneity Study summarized in Table A-5, production proceeded using the paint formulation and application method (spray or a particular size drawdown bar) that achieved the target lead levels. During production application, reference panels were coated along with the production panels at a rate of 18 for each set of 468 panels. For sets of PEMs that were sprayed, reference panels were placed at previously-assigned, randomly selected locations on the racks containing all the PEMs awaiting paint application. For sets of PEMs that had the lead paint applied via drawdown bar, production panels were drawdown in sets of two to three panels each for the wood, metal and drywall substrates and one at a time for the plaster substrates. At the discretion of the operator, a reference panel was prepared approximately every 10 sets or 25 panels.

Metal panels were used as the reference panels since metal panels yield the most accurate measurements of film thickness and lead levels. The reference PEMs were tested for film thickness during application and for lead level by ICP analysis after the paint had dried. This test procedure was used to check that the application process resulted in appropriate lead levels. Despite the use of the metal substrate only for the reference panels, the lead levels and paint thickness on these reference panels served as representative of the coatings applied to all wood, drywall, plaster, and metal substrate panels.

Table A-6 presents the average lead levels, CoV, minimum, and maximum of each set of 18 reference panel measurements. Most sets are very close to target lead levels, such as the 2.03 mg/cm^2 average for the 2.0 mg/cm^2 target yellow lead set, the 0.32 mg/cm^2 for the 0.3 mg/cm^2 target yellow lead set, and the 0.64 mg/cm^2 average for the 0.6 mg/cm^2 target white lead set. There also were a few sets that were a bit off target, but were sufficient to meet the verification needs. Despite the high average lead level of 9.2 mg/cm^2, the 6.0 mg/cm^2 white lead PEMs were accepted by EPA because they still met the needs of the verification for a set of PEMs at a high lead level. In the 0.6 mg/cm^2 yellow lead batch, the measured lead levels of 17 of the 18 reference panels ranged from 0.51 to 0.66 mg/cm^2, yielding a mean of 0.55 mg/cm^2, and a CoV of 7.5%. Because only one reference panel of 18 yielded a high lead level, the set of panels was accepted.

Table A-6. Reference Panel Results from Final Production for Each Set of ETV PEMs

Lead Type	Target Lead Level	Lead Levels		Range	
		Mean (mg/cm^2)	CoV	Min	Max
No Lead	0.0	0.00	8.2	0.002	0.003
White Lead (Lead Carbonate)	0.3	0.40	17.8	0.234	0.505
	0.6	0.64	13.5	0.425	0.761
	1.0	1.00	5.1	0.918	1.095
	1.4	1.48	8.0	1.322	1.748
	2.0	2.29	5.6	2.018	2.525
	6.0	9.18	31.2	5.65	18.4
Yellow Lead (Lead Chromate)	0.3	0.32	13.1	0.252	0.428
	0.6	0.57	16.6	0.511	0.920*
	1.0	1.00	7.1	0.879	1.148
	1.4	1.39	12.0	1.194	1.601
	2.0	2.03	9.4	1.483	2.314
	6.0	5.15	9.6	3.929	6.247

* Next highest measurement was 0.659

Topcoating

The linseed oil based paints were applied to the PEMs and stored in the constant temperature and humidity rooms during a four to seven day drying time. The panels were then all topcoated with Sherwin Williams brand Prep Rite bonding Primer to ensure good adhesion between the linseed oil based paint and the latex emulsion topcoat paints. The final latex paint topcoat was then applied to the PEMs. The topcoat paints are described in more detail below:

- Primer – Sherwin Williams Prep Rite bonding primer, diluted with deionized (DI) water at a ratio of 3:1 parts by volume. Spray application was done with a 50 percent overlap on the PEMs in both horizontal and vertical directions with a total wet film build of approximately 4-5 mils (a measure of dry film thickness). The PEMs then were allowed 1-2 hours to air dry before top coats were applied.
- Top coat number 1 is Sherwin Williams Classic 99 interior satin latex; color Extra White, diluted with DI water at a ratio of 3:1 parts by volume. Spray application was done with a 50 percent overlap on the PEMs in both horizontal and vertical directions, with a total wet film build of approximately 4-6 mils. Then the PEMs were allowed to air dry for three days. The PEMs were then bagged for further testing.
- Top coat number 2 is Sherwin Williams Classic 99 interior satin latex; color 7047 (software gray), diluted with DI water at a ratio of 3:1 parts by volume. Spray application was done with a 50 percent overlap on the PEMs in both horizontal and vertical

directions for a total wet film build of approximately 4-6 mils. Then the PEMs were allowed to air dry for three days. The PEMs were then bagged for further testing.

- Top coat number 3 is Sherwin Williams Color Accents interior satin latex; color 6867 (Fireworks orange red), diluted with DI water at a ratio of 3:1.5 parts by volume. Spray application was done with a 50 percent overlap on the samples in both horizontal and vertical directions for a total wet film build of approximately 4-6 mils. The PEMs were then allowed to air dry for three days. The PEM samples were then bagged for further testing.

PEM Labeling, Packing and Storage

The PEMs were stored in the constant temperature and humidity conditioning rooms prior to being packed up for transfer to the evaluation location. Each PEM was labeled on the back with an individual identification number, wrapped in a single laboratory towel to protect the front surface, and placed inside an individual zip seal bag also labeled with the identification number.

References

1. Bennett, H. The Chemical Formulary – A Collection of Valuable, Timely, Practical Commercial Formulae and Recipes for Making Thousands of Products in Many Fields of Industry, Volume VI. 1943. Chemical Publishing Co., Inc. Copyright 1943.

2. Uebele, Charles. Paint Making and Color Grinding: A Practical Treatise for Paint Manufacturers and Factory Managers, The Trade Papers Publishing Co., Ltd., 1913.

3. Battelle. Addition of Silica to Lead-based Paints Used for Production of PEMs in Support of ETV Evaluation of Lead Test Kits: References. Technical report submitted to EPA on February 19, 2009.

4. ASTM D1210-05(2010), "Standard Test Method for Fineness of Dispersion of Pigment-Vehicle Systems by Hegman-Type Gage," ASTM International.

5. ASTM E1729, "Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination," ASTM International.

6. United States Environmental Protection Agency, "Method 3050B: Acid Digestion of Sediments, Sludges, and Soils", SW846 Online, Revision 2. December 1996.

7. United States Environmental Protection Agency, "Method 6010C: Inductively Coupled Plasma-Atomic Emission Spectrometry", SW846 Online, Revision 3. February 2007.

8. Schneider Laboratories, Inc. ICP SOP Document# III-017-08-002

Section A2

Comparison of Expected vs. Actual Lead Concentrations of Performance Evaluation Materials

The following tables present a comparison of the expected vs. confirmed lead concentration for each PEM used during the testing of the lead test kits. Expected concentrations are based on lead levels defined for sets of PEMs during the PEM production process. That is, PEMs were being made at expected lead concentrations of 0, 0.3, 0.6, 1.0, 1.4, 2.0, or 6.0 mg/cm^2. These are the expected lead levels as defined in the test/quality assurance (QA) plan. Confirmed concentrations are based on ICP-AES results from individual paint chip samples taken from each PEM during testing (see Section 3.3.1in the test/QA plan).

Table A-7 presents the results by substrate and across all PEMs. Table A-8 presents the results by lead type. The average and standard deviation for the confirmed lead levels, as well as the CoV, are presented for each expected concentration level.

Table A-7. Confirmed lead level statistics for PEMs compared to expected lead level concentrations by substrate type.

		Expected PEM Lead Level (mg/cm^2)						
Substrate		**0**	**0.3**	**0.6**	**1**	**1.4**	**2**	**6**
Drywall	N	144	288	282	290	296	288	292
	Confirmed Lead Level: Average	0.00	0.34	0.83	1.15	1.48	2.52	9.04
	Confirmed Lead Level: StdDev	0.00	0.07	0.22	0.21	0.29	0.33	2.32
	CoV (%)	143.16	20.59	26.01	17.95	19.79	13.22	25.69
Metal	N	144	288	288	288	288	288	286
	Confirmed Lead Level: Average	0.00	0.31	0.56	0.85	1.26	1.91	8.18
	Confirmed Lead Level: StdDev	0.01	0.07	0.14	0.10	0.19	0.28	1.86
	CoV (%)	368.04	22.91	24.39	11.31	14.91	14.49	22.76
Plaster	N	140	290	292	296	288	288	284
	Confirmed Lead Level: Average	0.00	0.44	1.25	1.65	1.79	2.91	10.11
	Confirmed Lead Level: StdDev	0.01	0.18	0.53	0.84	0.65	0.85	3.01
	CoV (%)	258.82	40.17	42.40	50.92	36.22	29.24	29.81
Wood	N	144	288	288	275	282	284	288
	Confirmed Lead Level: Average	0.00	0.32	0.72	1.07	1.45	2.39	8.71
	Confirmed Lead Level: StdDev	0.02	0.16	0.22	0.31	0.29	0.71	1.59
	CoV (%)	470.54	48.20	30.83	28.80	20.29	29.56	18.29
All	N	572	1154	1150	1149	1154	1148	1150
	Confirmed Lead Level: Average	0.00	0.35	0.84	1.18	1.50	2.43	9.01
	Confirmed Lead Level: StdDev	0.01	0.14	0.41	0.55	0.44	0.69	2.36
	CoV (%)	451.36	38.91	48.34	46.76	29.38	28.49	26.24

CoV = Coefficient of Variation (Standard Deviation/Mean x 100)

Table A-7 indicates that overall confirmed lead levels were similar to expected concentrations. However, there are substrate types for which, comparatively, the confirmed lead levels were higher than the expected levels. Average confirmed levels for drywall and plaster PEMs were higher than expected levels, especially when compared to average confirmed lead levels from metal and wood. The PEMs used in the verification test were produced mainly using a drawdown technique (for all panels except no lead, 0.3 mg/cm^2 and 0.6 mg/cm^2 white lead). This involved applying the paint to the PEM and pulling it down with a specially designed bar. Being porous substrates, it is possible that the plaster and drywall panels absorbed some of the paint, causing more paint to be applied to the PEM to accommodate the thickness required on the PEM. This would then lead to higher lead concentrations on these substrates. The most significant potential impact of this effect can be seen on the plaster PEMs. This potential effect is based on observations during the production of the PEMs but has not been studied or confirmed.

Table A-8. Confirmed lead level statistics for PEMs compared to expected lead level concentrations by lead paint type.

		Expected PEM Lead Level (mg/cm^2)						
Lead Type		**0**	**0.3**	**0.6**	**1**	**1.4**	**2**	**6**
None	N	572						
	Confirmed Lead Level: Average	0.00						
	Confirmed Lead Level: StdDev	0.01						
	CoV (%)	451.36						
White	N		576	574	573	578	576	572
	Confirmed Lead Level: Average		0.30	0.88	1.24	1.53	2.36	8.37
	Confirmed Lead Level: StdDev		0.08	0.41	0.72	0.52	0.58	2.05
	CoV (%)		25.56	46.53	58.18	34.08	24.60	24.47
Yellow	N		578	576	576	576	572	578
	Confirmed Lead Level: Average		0.40	0.80	1.13	1.46	2.51	9.64
	Confirmed Lead Level: StdDev		0.16	0.40	0.30	0.33	0.78	2.48
	CoV (%)		40.64	49.95	26.32	22.87	31.25	25.75
All	N	572	1154	1150	1149	1154	1148	1150
	Confirmed Lead Level: Average	0.00	0.35	0.84	1.18	1.50	2.43	9.01
	Confirmed Lead Level: StdDev	0.01	0.14	0.41	0.55	0.44	0.69	2.36
	CoV (%)	451.36	38.91	48.34	46.76	29.38	28.49	26.24

CoV = Coefficient of Variation (Standard Deviation/Mean x 100)

The results in Table A-8 show that there was no significant difference in confirmed lead levels between white and yellow lead PEMs. The CoVs values were all ≤50% at all levels except 0.0 mg/cm^2. The larger CoV at this level is reflective of small changes around the zero lead level and most likely represent ICP-AES measurement variability near the detection limit, since no lead was used in preparing these PEMs. It should be noted, as discussed in Section A1 of this appendix, that the PEMs prepared at the expected lead level of 6.0 mg/cm^2 were known to be on average higher than 6.0 mg/cm^2 and that it was purposefully decided to accept the variation present at this expected lead level.

Though there were some differences between the confirmed and expected lead levels, it should be noted that when evaluated for proper responses, test kit results were compared to confirmed lead levels. That is, test kit results were always compared to the actual PEM lead levels, not the expected.

Section A3

QA/QC Results for the ICP-AES Analysis of Performance Evaluation Materials

Summary of Lead Level Confirmation ICP-AES Analysis of PEMs

All paint chip samples from the PEMs used in this verification test were analyzed using ICP-AES by Schneider Laboratories, Inc.

Sample preparation procedures followed the SOP generated by Schneider Laboratories, Inc. for this study (Schneider Laboratories, Inc., SOP Battelle Paint Samples, Doc # III-044-10-001). Information on how QC samples were spiked and final concentrations is provided in the SOP.

Three versions of this SOP (the original and two revisions) were used dated 1/20/10, 2/24/10, and 4/25/10. Approximately 27% of the PEMs were analyzed prior to the 2/24/10 revision to the SOP. In the 2/24/10 version, revisions were made to clarify that post-digestion matrix spikes and duplicates were being evaluated. Additionally, the laboratory control spike (LCS) procedures changed such that a separate LCS and a QC check sample were now being performed. Originally, in the 1/20/10 version, the LCS was prepared by spiking the QC check sample, which was a certified reference material (CRM) (as stated in Section 6.11.2 of the 1/20/10 SOP) containing a known quantity of lead. This practice was changed because there were recovery issues. The spike concentration of 1000 micrograms per liter (µg/L) was not >3x the background lead concentration because of the high lead concentrations in the actual CRM samples (4630 milligrams per kilogram [mg/kg]). Thus, as of the 2/24/10 SOP, one LCS, one QC check sample, and one QC check sample duplicate were being evaluated for every 20 samples. The LCS (Blank Paint QC) sample in the 2/24/10 SOP was defined as a piece of non-lead containing paint that was spiked with lead solution to a resulting concentration of 1000 µg/L. The QC check sample in the 2/24/10 SOP contained 10 mg of the CRM, a known lead-containing material. The QC check (CRM) was purchased to contain 4630 ± 266 mg/kg lead. To prepare the sample, 10 mg of the CRM was weighed out and diluted to 10 mL, resulting in a final concentration of 4.630 mg/L.

The 4/25/10 revision of the SOP clarified the acceptance criteria for the LCS samples, as it did not appear to be clearly defined in previous versions.

Because of the high lead concentration in the PEM samples, dilutions were made to the samples prior to initial analysis. The dilutions were prepared by spiking 10 microliters (µL) of the original digested sample into 9.990 milliliters (mL) of reagent water for a 1:1000 dilution. The samples were thoroughly mixed by inverting, and then analyzed for lead content. If the result was below the reporting limit, the sample was reanalyzed either non-diluted or at a lower dilution level. If samples were rerun at a different dilution level, this was noted in the QC summary report for that particular sample set.

The MDL for lead was 2.91 µg/L.

The reporting limit was 40 µg/L. Therefore all blank results should be <40 µg/L.

Summary of Quality Control Measures for PEMs ICP-AES Analysis

QC procedures were performed in accordance with the quality management plan (QMP) for the Battelle ETV Advanced Monitoring Systems (AMS) Center, except where differences were noted for Environmental and Sustainable Technology Evaluations (ESTE) per the EPA ETV Program QMP, and the test/QA plan for this verification test. Test procedures were conducted as stated in the test/QA plan; however a deviation to the test/QA plan was made during the ICP-AES analyses. For some sample runs, continuous calibration verification (CCV) samples were run once every 20 instead of 10 samples. This deviation is further described below. This change was assessed to have no impact on the quality of the results as described below. QC results for the analysis of paint chip samples from the PEMs are described below.

ICP-AES Blank Sample Results

Various blank samples were analyzed for the ICP-AES analyses. Method blank samples were analyzed in each set of 10-20 paint samples to ensure that no sources of contamination were present. An initial calibration blank was analyzed at the beginning of each run and used for initial calibration and zeroing the instrument. A continuing calibration blank was analyzed after each CCV to verify blank response and freedom from carryover. No blank samples failed QC during the analyses.

Calibration Verification Standards

Initial calibration standards were run at the beginning of each set of analyses. The acceptance criterion for the calibration coefficient of the calibration standards was ≥ 0.998. If this criterion was not met, the analysis was stopped and recalibration was performed before samples were analyzed. A 500 parts per billion (ppb) CCV standard was analyzed at the beginning of each run (following the initial calibration), at the end of each run, and every 10-20 samples. CCV recoveries ranged from 96% to 108%. Per the test/QA plan, CCV sample frequency was once every 10 samples. For most of the sample sets, CCVs were performed with this frequency. However, for later sample sets, CCVs were run once every 20 samples. CCV samples are used to verify instrument performance and are evaluated usually at a specified frequency as a preventative measure so that large amounts of samples do not need to be re-run if a CCV sample fails. In the course of this study, only one CCV sample failed, and it was when the CCV was being run once every 10 samples. All samples from the last passing CCV of that sample set were re-analyzed.

QC samples also included an initial calibration verification (ICV) standard and interference check sample (ICS). Both samples were 500 ppb. ICV samples were analyzed once at the beginning of each sample run and were required to have percent recoveries between 90-110% to be acceptable. ICS samples were analyzed at the beginning and end of every run and every 10-20 samples. ICS samples had to have percent recoveries between 80-120% to be acceptable. All reported ICV and ICS samples met the acceptance criteria. Recoveries for ICV samples ranged from 96% to 108%. Recoveries for ICS samples ranged from 93% to 112%.

Matrix Spike Samples/Duplicates

Matrix spike samples, as well as duplicates of these samples, were analyzed once every 10-20 samples. Acceptable recoveries for matrix spike samples were between 80-120%. Duplicate samples had acceptance criteria of ±25% relative percent difference (RPD).

All matrix spike samples were performed as post-digestion spikes as there was insufficient sample volume to perform a pre-digestion spike. Matrix spike recoveries ranged from 86% to 207%. Six matrix spike samples failed with recoveries above the specified acceptance criteria. In these instances, the lead concentration in the sample was well above the spike level. Matrix spike results indicate that matrix interferences were not observed. Duplicate samples were within the specified RPD.

LCS Samples

LCS samples were analyzed once every 10-20 samples. Acceptable recoveries for LCS samples were between 80-120%. LCS recoveries ranged from 17% to 225% . Schneider Laboratories, Inc. noted that LCS failures on one sample set were attributed to improper spiking technique. Training on spiking procedures was immediately implemented by Schneider Laboratories, Inc. for all analysts spiking samples. All LCS failures occurred prior to a revision to the Schneider Laboratories, Inc. SOP for analyzing paint samples for this verification test. In the original version of the SOP, LCS samples were prepared by spiking a known amount of lead onto a CRM. This practice was changed on 2/24/10 because there were recovery issues. The spike was not >3x the background lead concentration because of the high lead concentrations in the actual CRM samples. In the revised SOP, the LCS was prepared by spiking a piece of lead-free latex paint. There were no LCS failures after that In addition, a QC check sample containing only the CRM, which has a known concentration of lead weighed out to a particular amount, was analyzed with each sample set throughout the verification test. These QC samples all passed acceptance criteria.

Appendix B

Vendor Comments

ANDalyze, Inc. submitted the following comments on the draft report. These comments have not been reviewed by Battelle or U.S. EPA for accuracy, and do not necessarily reflect the opinions or views of U.S. EPA. Any questions regarding the comments in this section should be addressed to the vendor.

The ANDalyze test kit is unique because on performing a lead test the instrument screen displays a quantitative result (lead amount in units of mg/cm^2) instead of a positive/ negative response. We believed that this would be the best option for our product as the user can get an estimate of the amount of lead in the paint. However, the quantitative results put additional stringency on our product when false positive and false negative criteria are analyzed by ETV testing.
In the brief description that follows, **ANDalyze shows that the false negative criteria can be met for all substrates (except metal) if a minor change is made to the display of the instrument**

- For this report, ETV considered any result > 1 to be a "positive response" and any result < 1 to be "negative response". *We propose that results > 0.1 be considered positive and results < 0.1 be considered negative response.*
- ANDalyze will **change only the display** on the fluorimeter to indicate "positive" or "negative" instead of a numerical result. When the fluorimeter calculates lead concentration to be more than 0.1 mg/cm^2, the display will be "positive" and when lead concentration is calculated to be less than 0.1 mg/cm^2 the display will be "negative". Please note the proposed modification does NOT in any way change the chemical tests that were performed during the ETV program or the software code which calculates the lead amount. Therefore tests performed during ETV remain valid.
- With this proposed modification, we recalculated the predicted false positive and false negative rates. The results are pasted below (table 6-2, 6-13 were re analyzed)
- Based on the modeled probability of test response, the ANDalyze lead in paint test kits will meet the "false negative" requirement for all substrates except metal (see Re-analyzed Table 6-13 in the following page)

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Table 6-2 Re-analyzed: ANDalyze Lead-in-Paint Test Kit false positive results for panels with confirmed lead levels ≤ 0.8 mg/cm^2 and false negative results for panels with confirmed lead levels ≥ 1.2 mg/cm^2

	False Postive Rate		False Negative Rate	
	OPERATOR_TYPE		OPERATOR_TYPE	
	TECHNICAL	NON-TECHNICAL	TECHNICAL	NON-TECHNICAL
Overall	111/310=36%	89/310=29%	2/460=0%	3/460=1%
NONE	1/70=1%	0/70=0%	NA	NA
WHITE	49/122=40%	35/122=29%	1/230=0%	0/230=0%
YELLOW	61/118=52%	54/118=46%	1/230=0%	3/230=1%
DRYWALL	39/78=50%	27/78=35%	0/122=0%	1/122=1%
METAL	24/94=26%	14/94=15%	1/90=1%	2/90=2%
PLASTER	17/54=31%	18/54=33%	0/138=0%	0/138=0%
WOOD	31/84=37%	30/84=36%	1/110=1%	0/110=0%
GREY	35/104=34%	32/104=31%	0/156=0%	1/156=1%
RED	34/104=33%	27/104=26%	2/140=1%	0/140=0%
WHITE	42/102=41%	30/102=29%	0/164=0%	2/164=1%

Table 6-13 Re-analyzed: ANDalyze Lead-in-Paint Test Kit modeled probability of positive test results, lower 95% prediction bound, and corresponding conservative estimate of the false negative rate when lead level = 1.2 mg/cm^2

TOPCOAT	SUBSTRATE	LEAD_LEVEL	PREDICTION	LOWER	false negative rate
GREY	DRYWALL	1.2	98%	96%	4%
	METAL	1.2	94%	91%	9%
	PLASTER	1.2	98%	96%	4%
	WOOD	1.2	98%	96%	4%
RED	DRYWALL	1.2	98%	96%	4%
	METAL	1.2	94%	91%	9%
	PLASTER	1.2	97%	95%	5%
	WOOD	1.2	97%	96%	4%
WHITE	DRYWALL	1.2	97%	96%	4%
	METAL	1.2	94%	90%	10%
	PLASTER	1.2	97%	95%	5%
	WOOD	1.2	97%	95%	5%