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Environmental Technology Verification Program

Environmental and Sustainable Technology Evaluations

Test/QA Plan for Verification of Qualitative Spot Test Kits for Lead in Paint

Verification of
Qualitative Spot Test Kits for Lead in Paint

March 30, 2010

Prepared by

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ETV ESTE

Test/QA Plan for Verification of Qualitative Spot Test Kits for Lead in Paint

March 30, 2010

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(TBD)

SECTION A
PROJECT MANAGEMENT

A1 VERIFICATION TEST ORGANIZATION

The verification test will be conducted under the auspices of the U.S. Environmental Protection Agency (EPA) through the Environmental Technology Verification (ETV) Program. It will be performed by Battelle, which is serving as the verification organization under the Environmental and Sustainable Technology Evaluations (ESTE) arm of ETV.

The day to day operations of this verification test will be coordinated and supervised by Battelle, with the participation of the vendors who will be having the performance of their technologies for detecting lead in paint verified. Testing will be conducted at Battelle in Columbus, Ohio. Each vendor will provide Battelle with their respective technology and will train the Battelle staff in their technology use. Battelle technical staff as well as non-technical operators will operate the technologies during verification testing.

The organization chart in Figure 1 identifies the responsibilities of the organizations and individuals associated with the verification test. Roles and responsibilities are defined further below. Quality Assurance (QA) oversight will be provided by the Battelle Quality Manager and also by the EPA Quality Manager, at her discretion.

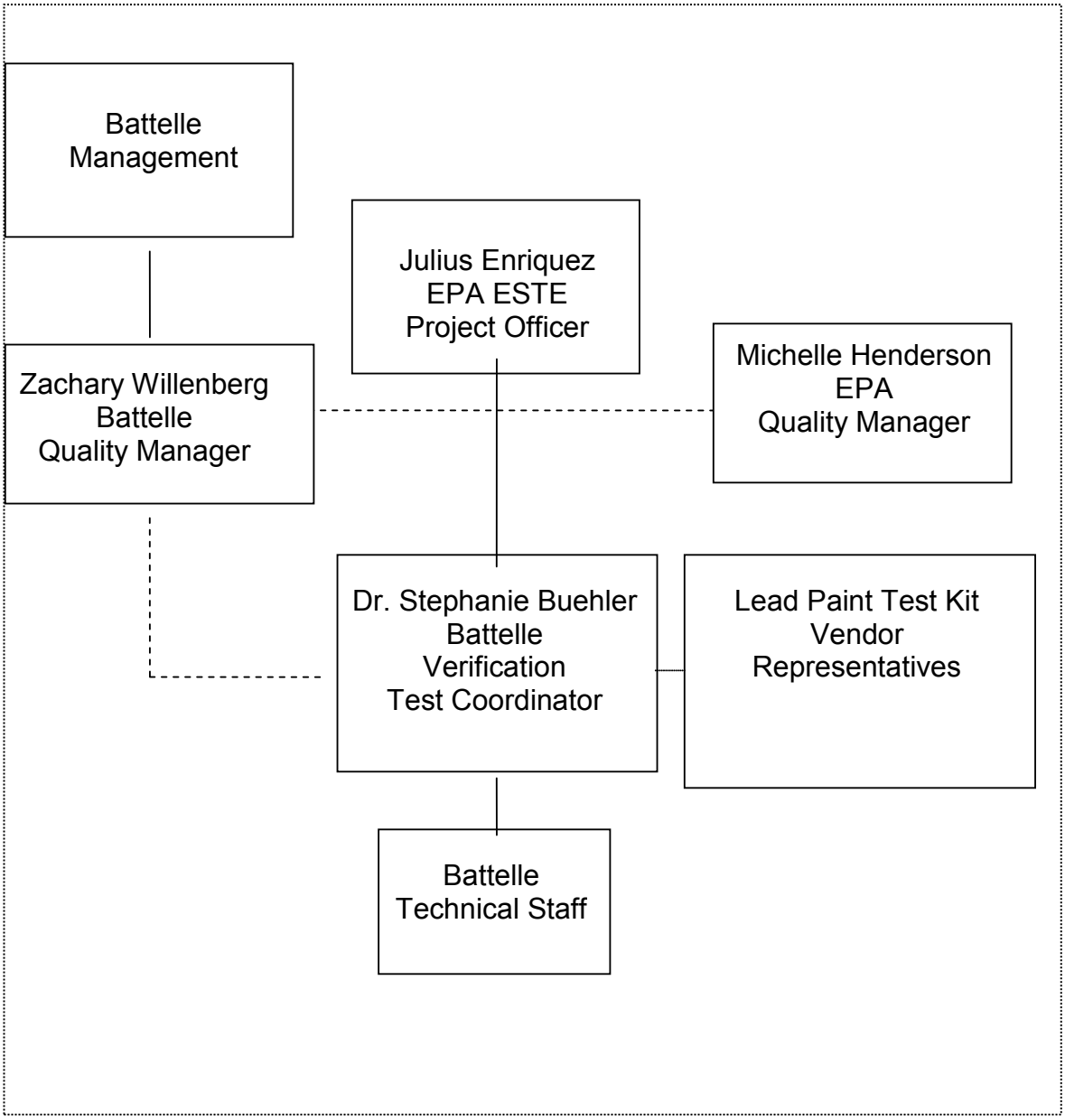

Figure 1. Organization Chart for the Verification Test

A1.1 Battelle

Dr. Stephanie Buehler is Battelle's Verification Test Coordinator for this test. In this role, Dr. Buehler will have overall responsibility for ensuring that the technical, schedule, and cost goals established for the verification test are met. Specifically, Dr. Buehler will:

- Prepare the draft test/QA plan, verification reports, and verification statements.
- Establish a budget for the verification test and manage staff to ensure the budget is not exceeded.
- Revise the draft test/QA plan, verification reports, and verification statements in response to reviewers' comments.
- Assemble a team of qualified technical staff to conduct the verification test.
- Direct the team in performing the verification test in accordance with this test/QA plan.
- Hold a kick-off meeting approximately one week prior to the start of the verification test to review the critical logistical, technical, and administrative aspects of the verification test. Responsibility for each aspect of the verification test will be confirmed.
- Serve as the primary point of contact for vendor representatives.
- Ensure that confidentiality of sensitive vendor information is maintained.
- Assist vendors as needed during verification testing.
- Become familiar with the operation and maintenance of the technologies through instruction by the vendors, if needed.
- Review and approve internal QA reviews and assessment reports.
- Review independent QA document reviews and assessment reports by EPA quality manager.
- Respond to any issues raised in assessment reports, audits, or from test staff observations, and institute corrective action as necessary.
- Coordinate distribution of the final test/QA plan, verification reports, and verification statements.

Technical staff from Battelle will support Dr. Buehler in planning and conducting the verification test. The responsibilities of the technical staff will be to:

- Assist in planning for the test, and making arrangements for the receipt of and training on the technologies.
- Attend the verification test kick-off meeting.
- Assist vendor staff as needed during technology receipt and training.
- Conduct verification testing using the vendor's technology and per the final test/QA plan.
- Conduct reference testing.
- Perform statistical calculations specified in this test/QA plan on the technology data as needed.
- Provide results of statistical calculations and associated discussion for the verification reports as needed.
- Support Dr. Buehler in responding to any issues raised in assessment reports and audits related to statistics and data reduction as needed.

Mr. Zachary Willenberg is Battelle's Quality Manager. Mr. Willenberg will:

- Review and approve the draft and final test/QA plan.
- Attend the verification test kick-off meeting.
- Conduct a technical systems audit at least once during the verification test, or designate other QA staff to conduct the audit.
- Audit at least 10% of the verification data or designate other QA staff to conduct the data audit.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.
- Request that Battelle's Verification Test Coordinator issue a stop work order if audits indicate that data quality is being compromised.
- Provide a summary of the QA/QC activities and results for the verification reports.
- Review and approve the draft and final verification reports and verification statements.

A1.2 Technology Vendors

The responsibilities of the technology vendors are as follows:

- Review and provide comments on the draft test/QA plan.
- Accept (by signature of a company representative) the final test/QA plan prior to test initiation.
- Provide adequate units of their technology for evaluation during the verification test.
- Provide all other equipment/supplies/reagents/consumables needed to operate their technology for the duration of the verification test.
- Supply training on the use of the technology, and provide written consent and instructions for test staff to carry out verification testing, including written instructions for routine operation of their technology.
- Provide maintenance and repair support for their technology, on-site if necessary, throughout the duration of the verification test.
- Review and provide comments on the draft verification report and statement for their respective technology.

A1.3 EPA

EPA's responsibilities are based on the requirements stated in the "Environmental Technology Verification Program Quality Management Plan" (EPA QMP).[1] The roles of specific EPA staff are as follows:

Ms. Michelle Henderson is EPA's Quality Manager for the verification test. Ms. Henderson will:

- Review the draft test/QA plan.
- Approve the final test/QA plan.
- Perform at her option one external technical systems audit during the verification test.
- Notify the EPA ESTE Project Officer of the need for a stop work order if the external audit indicates that data quality is being compromised.
- Prepare and distribute an assessment report summarizing results of the external audit.
- Review draft verification reports and verification statements.
- Approve final verification reports and statements.

Mr. Julius Enriquez is EPA's ESTE Project Officer. Mr. Enriquez will:

- Review the draft test/QA plan.

- Approve the final test/QA plan.
- Review independent QA document reviews and assessment reports by EPA quality manager.
- Review Battelle QA reviews and assessment reports and initiate corrective actions.
- Review the draft verification reports and verification statements.
- Oversee the EPA review process for the test/QA plan, verification reports, and verification statements.
- Approve final verification reports and statements.
- Coordinate the submission of verification reports and verification statements for signature by laboratory director and posting on the ETV website.

A1.4 Subcontract Laboratory

Any laboratory providing reference measurements will follow the requirements of the reference methods as well as the QC requirements as stated in this test/QA plan. A subcontract laboratory will provide reference measurements for the paint chip samples from each PEM. The responsibilities of this laboratory will include:

- Proper receipt and handling of sample material.
- Accurate measurement of the target analyte(s) or target parameter(s).
- Submission of data and any supporting documents to Battelle.
- Participation in audit by Battelle Quality Manager and/or EPA's Quality Manager, if requested.
- Submission of QC limits/criteria used by the laboratory for inclusion in this document.

A2 BACKGROUND

The ETV Program conducts third-party performance testing of commercially available technologies. The purpose of ETV is to provide objective and quality assured performance data on environmental technologies, so that users, developers, regulators, and consultants can make informed decisions about purchasing and applying these technologies. Stakeholder committees of buyers and users of such technologies provide input on technology verifications.

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. 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[2].

A3 VERIFICATION TEST DESCRIPTION AND SCHEDULE

A3.1 Summary of Technology Category

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 enacted. However, many of these test kits have been found to have high rates of false positives[3]. The Renovation, Repair, and Painting rule[4] calls for an EPA evaluation and recognition program for test kits that are candidates to meet the goal of a 5% false negative rate and 10% false positive rate. As stated in the Preamble to the rule, the test kit performance must be validated by a laboratory independent of the kit manufacturer, using ASTM International's E1828, *Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Spot Test Kits for Lead in Paint*[5] or an equivalent validation method. EPA will then only recognize those kits that have been verified through this process. ETV will coordinate the testing and supply the data that will be used in the recognition process. This plan incorporates ASTM Method E1828 guidelines[5].

A3.2 Verification Test Schedule

Table 1 shows the planned schedule of testing and data analysis/reporting activities to be conducted in this verification test. The planned dates for conducting verification tests of lead paint test kits are December 2009 - April 2010 at Battelle's laboratories in Columbus, Ohio. A final verification test schedule with specific test dates will be provided to participating vendors once those details are known. It will be necessary for participating vendors to provide their technologies to Battelle by the specified date so testing staff may become familiar with operating the kits before testing begins. Vendor staff will provide training in operating the technologies either in person or by teleconference. The period of operation for verification testing will be approximately four to six weeks. The test procedures are described in Section B of this test/QA plan.

Subsequent to the verification test, a separate verification report will be drafted for each participating technology. These reports will be reviewed by the respective vendor and by peer reviewers, and submitted to EPA for final signature and subsequent publication. Technologies and associated equipment (but not consumables) will be returned to the vendors at the completion of report writing, unless other arrangements have been made with Battelle.

Table 1. Planned Verification Test Schedule

Dates	Testing Activities	Data Analysis and Reporting
December 2009	Training of verification testing staff on technology use	
December 2009 – April 2010	Conduct verification testing	Review and compile test data and records as they become available. Review and summarize verification testing staff observations.
February - May 2010		Prepare report templates and complete common sections of reports. Evaluate and analyze data generated during testing
May – July 2010		Complete draft reports and submit for vendor, EPA, and peer reviews.
July – September 2010		Revise draft reports and submit final reports for EPA approval.

A3.3 Test Site

Testing will be conducted in Battelle laboratories in Columbus, Ohio. There will be no field testing, i.e., testing at an offsite location outside of the laboratory, such as a house,

conducted during this technology verification. EPA is considering the possibility of a future verification test involving real-world field environments.

A3.4 Health and Safety

Battelle will conduct all verification testing and reference lead paint spot test kit measurements following the safety and health protocols in place for the laboratory and facilities. This includes maintaining a safe work environment and a current awareness of handling potentially toxic chemicals. Exposure to potentially toxic chemicals will be minimized, personal protective equipment will be worn, and safe laboratory practices will be followed, as necessary. Health and Safety will be reviewed with Battelle's Safety Officer once the specific technologies to participate in the test are known.

A4 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

In performing the verification test, Battelle will follow the technical and QA procedures specified in this test/QA plan and will comply with the data quality requirements in the EPA-approved QMP[6] for the Advanced Monitoring Systems (AMS) Center, except where differences are noted for ESTE per the EPA ETV Program QMP.[1] The objective of this verification test is to evaluate the performance of test kits for the detection of lead in paint. This evaluation will assess the capabilities of the lead paint spot test kits against laboratory prepared performance evaluation material (PEM) samples, and will include a comparison between the lead paint test kit results and those of a standard technique as described in Section B4. Additionally, this verification test will rely upon 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) will be considered for each technology. Below is a discussion of the quality objectives and the criteria for measurement data that have been established to assure that the test objectives are met.

A4.1 Quality Objectives

Data quality objectives indicate the minimum data quality required to meet the lead paint spot test kits verification objectives. Data quality objectives for this verification test include those related to the reference method performance and those related to the test kit performance.

Data quality objectives for the reference method (see Section B4) are presented in terms of data quality indicator (DQI) criteria for the critical measurements associated with the reference method and are listed in Table 2 and discussed in Section A4.2. The quality of the reference measurements will be monitored using QC samples and procedures, as described in the testing laboratory's procedures or the method. These requirements are further discussed in Section B. Method blanks, positive control samples, and negative control samples are expected to be included as QC samples for each technology. Method blank samples will include performance evaluation materials (PEMs) with 0.0 mg/cm^2 lead paint as well as each PEM substrate (wood, metal, drywall, and plaster) with no paint. Positive and negative control samples, if provided with a test kit, will be analyzed according to the kit's instructions.

The EPA Quality Manager will perform a technical systems audit (TSA) of the subcontract laboratory conducting reference analyses. The Battelle Quality Manager or his designee will perform a TSA of the actual evaluation of the test kits at least once during this verification test and will audit at least 10% of the verification data acquired, including the data packages received from the subcontract reference laboratory. The EPA Quality Manager also may conduct an independent TSA of the verification test, at her discretion.

A4.2 Criteria for Measurement Data

Reference measurements will be conducted by an NLLAP-accredited laboratory using inductively coupled plasma-atomic emission spectrometry (ICP-AES) on paint chip samples from each PEM used in the verification test. Table 2 presents the minimum DQIs and criteria for the reference method critical measurements. These DQIs and criteria are based on NLLAP guidelines and are consist with the selected NLLAP-accredited laboratory's criteria and procedures. The method detection limit for the reference analyses will be determined based on the criteria provided by the subcontract laboratory selected to perform the reference analyses. The reference method measurement quality will be assured by adherence to these DQI criteria. For batches of less than 20 samples, at least one sample will be analyzed for each applicable DQI. Recommendations for appropriate positive and negative controls and their critical measurements for the lead paint spot test kits will be provided by each vendor, as appropriate.

A5 SPECIAL TRAINING/CERTIFICATION

Documentation of training related to technology testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective Battelle location. The Battelle Quality Manager may verify the presence of appropriate training records prior to the start of testing. The technology vendor will be required to train Battelle technical staff in the operation of his/her technology prior to the start of testing. Battelle will document this training with a consent form, signed by the vendor, which states which specific Battelle staff have been trained and determined by the vendor to be competent in operation of the vendor's technology.

Table 2. DQIs and Criteria for Critical Measurements for Reference Method

DQI	Method of Assessment	Frequency	Minimum Acceptance Criteria	Corrective Action
Precision	Replicate (duplicate) analyses of test sample extract	One per 20 samples or batch (min. 5% frequency)	Within ±25% relative percent difference (RPD)	Flag data; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples
Bias and Accuracy of Instrument	Instrument calibration/performance verification using matrix-matched reference standard materials of the same matrix as the samples	Verified daily or prior to analyzing samples	Per most stringent instrument, laboratory, or method guidelines	Recalibrate instrument
Bias and Accuracy of Sample Measurements	Independent Calibration Verification (ICV) – lead standard at concentration in the range of lead levels tested	Once per day after calibration	Within ±10% of known value	Recalibrate instrument
	Initial Calibration Blank (ICB) - contains no lead and is used for initial calibration and zeroing instrument response. The ICB must be matrix matched to acid content present in sample digestates.	Once per run at the beginning of the run	Absolute value not more than 20% of the regulatory limit or the level of concern	Prepare new calibration curves

DQI	Method of Assessment	Frequency	Minimum Acceptance Criteria	Corrective Action
	Continuing Calibration Verification (CCV) – independent reference standards or the same standards used to prepare the instrument calibration curve	At the beginning and end of a sample run, as well as every 12 hours, or according to instrument manufacturer's recommendations, or according to instrument Performance Characteristic Sheet, or at a predetermined SOP frequency (once every 10 samples), whichever is more frequent	Within ±20% of known value	Establish new calibration curve and reanalyze samples; sample analysis shall not continue or be restarted until a new calibration curve is established and verified
	Interference Check Sample (ICS) - A standard solution (or set of solutions) used to verify accurate analyte response in the presence of possible interferences from other analytes present in samples. The ICS must be matrix matched to the reagent content present in sample digestates.	At the beginning and end of each run or twice every 12 hours	Within 20% of known value	Apply correction factors to sample results as appropriate
	Continuing Calibration Blank (CCB) - A standard solution which has no lead and is used to verify blank response and freedom from carryover	After each ICS and CCV	Absolute value not more than 20% of the regulatory limit or level of concern	Flag data; attempt to determine source of contamination; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples
	Laboratory Control Sample (LCS) – same matrix as test samples with lead concentration near the level of concern or regulatory level; wherever possible shall not require extensive pretreatment dilution or concentration prior to	1 per 10-20 samples or batch (minimum 5%)	Within ±20% of known value	Flag data; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples

DQI	Method of Assessment	Frequency	Minimum Acceptance Criteria	Corrective Action
	analysis; shall be either NIST Standard Reference Materials or commercially available certified reference materials			
	Matrix Spike Sample – prepared using split sample (before any digestion when possible); lead level spiked shall be enough to result in final lead concentration of the prepared sample of 5x the sample's observed lead concentration, or 5x the method detection limit, whichever is greater	1 per 20 samples or batch (minimum 5%)	Within ±25% of calculated value	Flag data; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples
	Duplicate Samples – of test sample extract only	1 per 20 samples or batch (minimum 5%)	Within ±25% of RPD	Flag data; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples
	Method Blank – mixture of all reagents used for digestion but without the matrix; is carried through all steps of the analysis starting with digestion	1 per 20 samples or batch (minimum 5%)	Absolute value not more than 20% of the regulatory limit or level of concern	Flag data; attempt to determine source of contamination; reanalyze QC samples; if these QC samples are out of range, then repeat entire analysis including recalibration and all QC samples

A6 DOCUMENTATION AND RECORDS

The records for this verification test include the test/QA plan, the protocols, laboratory record books (LRB), data collection forms, electronic files (both raw data and spreadsheets), and the final verification report and statement. All of these records will be maintained in the Verification Test Coordinator's office or at the testing locations during the test and will be transferred to permanent storage at Battelle's Records Management Office at the conclusion of the verification test. All Battelle LRBs are stored indefinitely, either by the Verification Test Coordinator or Battelle's Records Management Office. The EPA ESTE project officer or appropriate EPA ETV management will be notified before disposal of any files. The results from

the reference measurements made by the subcontractor laboratory will be submitted to Battelle after making the measurement and obtaining the results of the analyses. Table 3 provides further details the data recording practices and responsibilities. QA documents generated over the course of this verification test include audit and assessment reports and will be maintained by the Battelle Quality Manager. Copies of audit and assessment reports will be downloaded into the ETV web database so that EPA may access it if needed.

All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed, and dated by the person making the correction. In all cases, strict confidentiality of the raw data from each vendor's technology, and separation of data from different technologies, will be maintained throughout the verification test. Separate files (including manual records, printouts, and/or electronic data files) will be kept for each instrument.

Table 3. Summary of Data Recording Process

Data to Be Recorded	Where Recorded	How Often Recorded	By Whom	Disposition of Data
Dates, time, and details of test events	LRBs or data recording forms	Start/End of test procedure, and at each change of test parameter	Battelle	Used to organize and check test results; manually incorporated in data spreadsheets as necessary
Sample (PEMs) used (IDs, dates, etc.)	LRBs or data recording forms	When each PEM is used, throughout test duration	Battelle	Incorporated into verification report as necessary
Test kit procedures and sample results	Data sheets and LRB	Throughout test duration	Battelle	Manually incorporated into data spreadsheets for statistical analysis and comparisons
Reference method sample preparation	LRB	Throughout sample preparation	Battelle or subcontract laboratory	Used to demonstrate validity of samples submitted for reference measurements
Reference method procedures, calibrations, QA, etc.	LRB or data recording forms	Throughout sampling and analysis processes	NLLAP-accredited laboratory	Retained as documentation of reference method performance

SECTION B
MEASUREMENT AND DATA ACQUISITION

B1 EXPERIMENTAL DESIGN

This verification test will specifically address verification of spot test kits for the detection of lead in paint. This test follows procedures described in ASTM E1828[5]. The lead paint test kits will be tested only in a laboratory under controlled conditions; no field testing will take place during this verification test. This will allow comparison of the technology results to a reference method using a specified set of performance evaluation materials (PEMs). PEMs will be 3 inch by 3 inch squares of wood, metal, plaster, or drywall coated with paint of various colors containing a range of lead concentrations. PEM samples will be analyzed in at least duplicate by the test kits and also analyzed by the reference method (ICP-AES). The lead paint test kits participating in this test will be evaluated based on qualitative results, indicating only the presence or absence of lead in the paint at specified concentrations (see Section B1.1). Some test kits may provide quantitative results. In the instance where quantitative measures are used in determining the results for a particular technology, a qualitative result will be reported (i.e., presence or absence of the contaminant of interest) as with the other technologies, and the quantitative measure used to determine that result will also be reported for that sample but will not be used in any other data analyses as described in Section B1.2. The performance of the lead paint test kits will be verified based on sensitivity, precision, false positive/negative rates, matrix effects, and operational factors. These parameters are discussed in detail in Section B1.1 and B1.2.

The analyses will be performed according to the vendor's recommended procedures as described in the user's instructions or manual, which should be consistent with training provided to Battelle staff. Similarly, calibration and maintenance of the technologies will be performed as specified by the vendor. Results from the technologies being verified will be recorded manually by the operator on appropriate data sheets or captured in an electronic data system and then transferred manually or electronically for further data workup. Qualitative characteristics of each technology such as ease of use will be assessed through observations made by the Test Coordinator and operators throughout the verification test. The results from each technology will be reported individually. No direct comparison will be made between technologies, but each

technology will undergo similar testing so it is convenient for end users to evaluate the ETV testing results.

B1.1 Test Procedures

Qualitative spot test kits for lead in paint will be evaluated against a range of lead concentrations in paint on various substrates through the use of PEMs. PEMs are 3 inch by 3 inch square panels of wood (pine and poplar), metal, drywall, or plaster that will be prepared by Battelle[7]. Table 4 shows the PEMs to be tested for each test kit. Each PEM will be coated with the same thickness of either white lead or lead chromate paint. The paint will contain lead at 0.4, 0.6, 1.0, 1.4, 2.0, and 6.0 mg/cm^2. These lead concentrations were chosen based on guidelines provided in EPA's lead RRP rule[4] as well as to represent potential lead levels in homes.

After production of the 6.0 mg/cm^2 PEMs, reference analyses results indicated that actual lead levels for these PEMs were outside of the anticipated 6.0 mg/cm^2 target level. For lead chromate PEMs at 6.0 mg/cm^2, 18 reference panels dispersed throughout the PEMs during production[7] indicated that actual concentrations ranged from 4.8 - 6.2 mg/cm^2 with a mean of 5.2 mg/cm^2 and a CoV of 9.6%. The 18 reference panels coated during the production of 6.0 mg/cm^2 PEMs using white lead indicated that actual concentrations ranged from 5.6 to 18.4 mg/cm^2. Four white lead reference PEMs were considered to be "outliers" (18.4, 12.8, 11.3, and 11.1 mg/cm^2). Excluding these panels results in an average concentration of 8.0 mg/cm^2 with a CoV of 12% for this level. Though both the lead chromate and white lead PEMs are outside of the expected 6.0 mg/cm^2 concentration, it was determined that these PEMs were acceptable for use. The purpose of the 6.0 mg/cm^2 lead level is to evaluate the test kit's response at a level well above the action level of 1.0 mg/cm^2. Both the lead chromate and white lead PEMs satisfy this need and will be used in this verification test. Some modifications will be made, however, for the use of the white lead PEMs at this level. Because there were four reference panels with quite high lead levels, PEMs produced within the range of these four reference panels, and thus thought to have the same lead levels, will be distributed evenly amongst all participating test kits, to the extent possible. When analyzing the results from this lead level, consideration will be given to conducting analyses with all data for this lead level as well as without data from PEMs with >10 mg/cm^2.

Paint containing no lead (0.0 mg/cm^2) will also be applied to each substrate and tested. Two different layers of paint will be applied over the leaded paint. One will be a primer

designed for adhesion to linseed oil-based paint and the second coat will be a typical interior modern latex paint tinted to one of three colors: white, red-orange, or grey-black. These colors were chosen by EPA based on the potential of certain colors to interfere or not with lead paint test kit operations. The top-coat paint manufacturers' recommended application thickness will be used. Two coats at the recommended thickness will be applied. Each substrate will be tested without paint, in the same manner as all other PEMs (i.e., per the test kit instructions), to determine if the substrate material itself is causing any effects on the performance of the test kits. Two unpainted PEMs of each substrate will be evaluated using each test kit.

Each spot test kit for lead paint will be operated by a technical operator. This operator will be a Battelle staff member with laboratory experience. The technical operator will be trained by the vendor in the operation of the vendor's test kit. The same technical operator will operate a given test kit throughout the course of testing. Multiple technical operators may operate different lead paint test kits. Because these test kits are anticipated to be used by certified remodelers, renovators and painters, the test kits will also be evaluated by a non-technical operator depending on the operational and potential safety issues surrounding each test kit. Because this verification test will involve the evaluation of lead-based paint, any disturbance of that paint could pose a potential health hazard. If a test kit's operation does involve disturbing the paint on the PEM and thus pose a health risk to the operator, then it may not be feasible to evaluate that test kit using a non-technical operator. In such an instance, any technical operator from Battelle would have to undergo specific health and safety training to operate the test kit and all appropriate health and safety practices would have to be followed during testing. If a non-technical operator is used, the non-technical operator will be a certified renovator with little to no experience with lead. The non-technical operator will be trained in the use of the lead paint test kit by a Battelle staff person who has experience operating test kits in general, but not by the technical operator who was trained by the vendor. This scenario will approximate the training renovators are expected to receive under the RRP rule[3].

Table 4. 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	0.4	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
Lead Chromate	0.4	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

Tests will be performed in at least duplicate on each PEM by each operator, technical and non-technical, depending on available space and test kit operation requirements. Replicates will be tested in succession by each operator on a given PEM. PEMs will be analyzed blindly by each operator in that the PEMs used for analysis will be marked with a non-identifying number. Test kit operators will not be made aware of the paint type, lead level, or substrate of the PEM being tested. PEMs will be tested in no particular order.

Paint chip samples from each PEM will be analyzed by a National Lead Laboratory Accreditation Program (NLLAP) accredited laboratory using ICP-AES to confirm the lead level of each PEM used for testing. The paint chip samples for reference analyses will be collected by Battelle according to ASTM E1729[8]. The reference analyses will confirm the lead level of each PEM to ensure an accurate understanding of each test kit's performance. Lead levels determined through the reference analysis will be used for reporting and statistical analyses.

The technologies will be evaluated for the following parameters:

B1.1.1 False Positive and False Negative Rates

A false positive response will be defined as a positive result when regulated lead-based paint is not present. For this test, false positive rates will be assessed on panels with target lead levels at 0.6 mg/cm^2 and lower. Per the guidelines set forth in EPA's April 22, 2008 RRP rule[4], panels with an ICP-AES confirmed lead level greater than 0.8 mg/cm^2 will not be used in the false positive analysis.

A false negative response will be defined as a negative response when regulated lead-based paint is present. For this test, false negative rates will be assessed on panels with target lead levels at 1.4 mg/cm^2 and higher. Per the guidelines set forth in EPA's April 22, 2008 RRP rule[4], panels with an ICP-AES confirmed lead level less than 1.2 mg/cm^2 will not be used in the false negative analysis

False positive and negative rates will be grouped by paint type (lead chromate vs. white lead). Results will also be grouped across paint types by PEM substrate and by color. Results will also be examined by operator type (i.e., technical vs. non-technical).

Based on stakeholder 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 within the guidelines of the EPA RRP rule[4], false positive and negative

rates, in addition to those stated above, will also be calculated for each test kit based on 1.0 mg/cm^2 lead. Thus, false positive rates will be assessed on panels with lead levels at 1.0 mg/cm^2 or lower and false negative rates will be assessed on panels with lead levels at 1.0 mg/cm^2 or higher. For panels that measure 1.0 mg/cm^2, positive results will be considered "correct" and negative results will be considered false negative. If the lead concentration of the PEM is actually greater than 1.0 mg/cm^2 (e.g., 1.1 mg/cm^2), then negative results will be considered false negatives. If the lead concentration of the PEM is actually less than 1.0 mg/cm^2 (e.g., 0.9 mg/cm^2), then positive results will be considered false positives.

B1.1.2 Precision

Precision will be measured by the reproducibility of responses for replicate samples within a group of PEMs. Groups of PEMs to be evaluated for precision will include lead concentrations and substrate material at a specific lead concentration.

B1.1.3 Sensitivity

The sensitivity or lowest detectable lead level will be identified based on the detection results across all PEM lead levels.

B1.1.4 Modeled Probability of Test Kit Response

Logistic regression models will be 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.

B1.1.5 Matrix Effects

Covariate adjusted logistic regression models will be used to determine whether any of the PEMs parameters (color, substrate, etc) affects the performance of the test kit. Type III Statistics and comparison of Likelihoods from logistics regression models will be used to determine the statistical significance of these factors.

B1.1.9 Operational Factors

Operational factors such as ease of use, operator bias, and helpfulness of manuals, will be evaluated based on Operator and Verification Test Coordinator observations. 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 will also be evaluated. These metrics will be discussed

by detailing how much waste is generated and what the waste is composed of, providing information on how the waste should be properly handled, presenting a summary of the pertinent MSDS information, when available, and noting whether the test kit used batteries, a power supply, or no energy source is needed. Information on how many tests each kit can perform as well as the shelf life of the test kit and chemicals used as part of the test kit will also be reported.

B1.2 Statistical Analysis

The statistical methods and calculations used for evaluating quantitative performance parameters are described in the following sections. ICP-AES reference analyses will confirm the lead level of each PEM to ensure an accurate understanding of each test kit's performance. Lead levels determined through the reference analysis will be used for reporting and statistical analyses.

B1.2.1 False Positive and False Negative Rates

A false positive response will be defined as a detect from the lead paint test kit when evaluated on PEMs with target lead levels at and below 0.6 mg/cm^2 (i.e., 0, 0.4, and 0.6 mg/cm^2 levels). Per the guidelines set forth in EPA's April 22, 2008 RRP rule[4], panels with an ICP-AES confirmed lead level greater than 0.8 mg/cm^2 will not be used in the false positive analysis. A false negative response will be defined as a non-detect from the lead paint test kit when evaluated on PEMs with target lead levels at and above 1.4 mg/cm^2 (i.e., 1.4, 2.0, 6.0 mg/cm^2). Per the guidelines set forth in EPA's April 22, 2008 RRP rule[4], panels with an ICP-AES confirmed lead level less than 1.2 mg/cm^2 will not be used in the false negative analysis

False positive and negative rates will be grouped by paint type (lead chromate vs. white lead). Results will also be grouped across paint types by PEM substrate and by color. Results will also be examined by operator type (i.e., technical vs. non-technical, where applicable).

Based on stakeholder 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 within the guidelines of the EPA RRP rule[4], false positive and negative rates, in addition to those stated above, will also be calculated for each test kit based on 1.0 mg/cm^2 lead. Thus, false positive rates will be assessed on panels with lead levels at 1.0 mg/cm^2 and lower and false negative rates will be assessed on panels with lead levels at 1.0 mg/cm^2 and higher.

False positive and negative rates will be evaluated as the number of positive or negative results, respectively, out of the total number of PEM samples evaluated without or with regulated lead-based paint, per the concentration levels stated above.

$$False\ Positive\ Rate = \frac{\#\ of\ \ positive\ results}{total\ \#\ of\ \ PEMs\ without\ regulated\ lead\ -\ based\ \ paint} \quad (1)$$

$$False\ Negative\ Rate = \frac{\#\ of\ \ negative\ results}{total\ \#\ of\ \ PEMs\ with\ regulated\ lead\ -\ based\ \ paint} \quad (2)$$

B1.2.2 Precision

Precision will be measured by the reproducibility of responses for replicate samples within a group of PEMs. Groups of PEMs to be evaluated for precision will include individual lead concentration levels (e.g., all PEMs at 0.4 mg/cm^2) and substrate material at a specific lead concentration (e.g., all metal PEMs at 1.4 mg/cm^2). Responses will be considered inconsistent if 25% or more of the replicates differ from the response of the other samples in the same group of PEMs. An overall precision for each test kit will be assessed by paint type by calculating the overall number of consistent responses for all the sample sets of either white lead or lead chromate-painted PEMs. The results will be reported as the percentage of consistent responses out of all replicate sets for those paint types (see Equation 3).

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

B1.2.3 Sensitivity

The sensitivity or lowest detectable lead level for each test kit will be identified based on the detection results across all PEM lead levels. The lowest PEM lead level with consistent positive or "detect" responses will be considered the lowest detectable level. The identified lowest detectable lead level will be reported and discussed.

B1.2.4 Modeled Probability of Test Kit Response

Logistic regression models will be 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. An evaluation of the bivariate relationship between

the response variable and each individual candidate explanatory variable will be 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 will be developed including a set of explanatory variables which are most predictive of the probability of the test kit response variable. The potential logistic regression model will take the form as below:

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

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 will be estimated with the model. Candidate independent variables associated with the response variable are operator type, lead type, lead level, substrate type, and topcoat color. Interactions between these predictor variables will also be assessed. Each level of the covariates can also be included using indicator variables. SAS procedures GENMOD or LOGISTIC will be used to fit the logistic model

B1.2.5 Matrix Effect

The covariate-adjusted logistic regression model described in section B1.2.4 will be used to assess the significance of PEM parameters and the interactions among them on the performance of the test kits. PEM parameters are included in the model as explanatory variables associated with the Y_{ijkl} 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 will be the guiding principle in logistic regression model. The likelihood function is defined as

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

where $\pi(Y_{ijkl})$ is the conditional probability of $Y_{ijkl}=1$ and $[1-\pi(Y_{ijkl})]$ is the conditional probability of $Y_{ijkl}=0$ 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 compute the likelihood ratio test statistic, G, as follows:

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

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

B1.2.6 Operational Factors

There are no statistical calculations applicable to operational factors. Operational factors such as ease of use, operator bias, average cost, average time for kit operation, and helpfulness of manuals, will be determined qualitatively based on Operator (both technical and non-technical) and Verification Test Coordinator observations. The non-technical operator will not receive any vendor support on the operation of the test kit throughout the test. Descriptions of observations made throughout testing will be reported and discussed. 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 will be discussed. This discussion will be based on how much waste is generated and what the waste is composed of, information 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 is needed. Information on how many tests each kit can perform as well as the shelf life of the test kit and chemicals used as part of the test kit will also be reported.

B1.3 Reporting

The data obtained in the verification test will be compiled separately for each vendor's technology, and the data evaluations will be applied to each technology's data set without reference to any other. At no time will data from different vendor's technology be intercompared or ranked. Following completion of the data evaluations, a draft verification report and verification statement will be prepared for each vendor's technology, stating the verification test procedures and documenting the performance observed. Each report will briefly describe the ETV Program and the procedures used in verification testing. The results of the verification test will then be stated, without comparison to any other technology tested, or comment on the acceptability of the technology's performance. Each draft verification report will be submitted

for review by the respective technology vendor and by EPA and other peer reviewers. Comments on the draft report will be addressed in revisions of the report. The peer review comments and responses will be tabulated to document the peer review process. The reporting and review process will be conducted according to the requirements of the ETV QMP[1]. All final verification reports and statements will be made 508 compliant and will be posted on the ETV website (www.epa.gov/etv).

B2 SAMPLING REQUIREMENTS

B2.1 Sample Collection, Storage and Shipment

PEM samples will be produced and stored prior to the beginning of the verification test and in accordance with the *Revised Plan For Development And Production Of Performance Evaluation Materials For Testing Of Test Kits For Lead In Paint under the Environmental Technology Verification Program*[7]. The film thickness, homogeneity, and lead levels of the paint applied to the PEMs will be verified prior to full-scale PEMs production via spray or draw down application on quality-controlled metal panels[7]. This process will test the paint formulation and application to ensure that the desired lead level can be achieved during full PEM production. PEMs will be measured for film thickness using a Positector 6000 coating thickness gauge. Subsequently, paint chip samples corresponding to the locations of the film thickness measurements will be obtained following ASTM E1729, *Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination*.[8] These samples will be shipped to the NLLAP-accredited laboratory for analysis. This process will not verify the lead levels of individual PEMs used for verification testing and will take place prior to the production of PEMs for this verification test. Details on this process can be found in the PEMs development plan[7]. The PEMs development plan, along with a summary of the homogeneity and lead level analyses, can be found in Appendix A.

As part of the verification test, the lead level of paint from each PEM used in the verification test will be verified through ICP-AES analysis by an independent NLLAP-accredited laboratory. Paint chip samples from unused portions of each PEM will be collected by Battelle using guidelines set forth in ASTM E1729 *Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination*.[8] Paint chips will be collected into small glass vials

according to a procedure to be prepared by Battelle (see Appendix B). This procedure will detail paint chip sampling guidelines for each substrate to ensure consistent paint chip collection throughout the verification test. These paint chips will be supplied to the subcontract laboratory for analysis. ICP-AES reference analyses will confirm the lead level of each PEM to ensure an accurate understanding of each test kit's performance. Lead levels determined through the reference analysis will be used for reporting and statistical analyses. The lead concentrations expected across a batch of PEMs will be evaluated prior to their use in the ETV test through the use of reference PEMs during the production phase[7]. This process should help ensure that large deviations in concentrations do not exist across a particular lead level.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Sample custody will be documented for the shipping and analysis of paint chip samples to the subcontract laboratory using standard chain-of-custody (COC) forms provided by Battelle or supplied by the laboratory, as appropriate. Samples transferred within Battelle may be documented in bound sample login LRBs. Each COC form will summarize the analyses requested. The COC forms will track sample release from Battelle to the NLLAP laboratory. Each COC form will be signed by the person relinquishing the samples once that person has verified that the COC form is accurate. The original sample COC forms will accompany the samples; the shipper will keep a copy. Any discrepancies will be noted on the form and the sample receiver will immediately contact the Verification Test Coordinator to report missing, broken, or otherwise compromised samples. Copies of all COC forms will be delivered to the Verification Test Coordinator, and maintained with the test records.

B4 LABORATORY REFERENCE METHOD

Paint chips from an unused portion of each PEM will be analyzed by ICP-AES by an independent NLLAP-accredited laboratory. Paint chip samples will be collected by Battelle using guidelines set forth in ASTM E1729[8] and supplied to the subcontract laboratory for analysis. Because PEMs will be assumed to be homogenous, based on pre-production testing[7], the specific place of paint chip collection will not matter, as any spot on the PEM should be representative of the entire panel. However, to the extent practicable, based on space needed for individual test kit operation on each PEM, varying places on each PEM will be selected for

collection of paint ships for reference analyses. The NLLAP-accredited laboratory will use ASTM 1645[9] or equivalent for paint digestion and EPA method 6010B[10] or equivalent, along with their own laboratory SOPs for ICP-AES analysis. The subcontract laboratory will be responsible for providing calibrated instrumentation, performing all method QA/QC, and providing calibration records for any instrumentation used. ICP-AES reference analyses will confirm the lead level of each PEM to ensure an accurate understanding of each test kit's performance. Lead levels confirmed through the reference analysis shall have a percent error of less than ±15 percent of expected values. Reference measurements outside this range will be used in place of expected lead concentrations for reporting and statistical analyses.

B5 QUALITY CONTROL

Steps will be taken to maintain the quality of data collected during this verification test. When confirmation analyses of the lead levels of the PEMs are performed, QC measures as noted in the subcontract laboratory's operating procedures or the reference method and provided in Table 2 will be followed. The QC measures for the reference method will at least include the analysis of a method blank sample. Method blank samples will be analyzed to ensure that no sources of contamination are present. If the analysis of a method blank sample indicates a concentration above the minimum acceptance criteria provided in Table 2, contamination will be suspected. Any contamination source(s) will be corrected, and proper blank readings will be achieved, before proceeding with the analyses. A matrix spike sample as well as calibration verification standards will also be analyzed. Average acceptable recoveries for these samples are between 75-125%. Initial calibration standards will be run at the beginning of each set of analyses or at least once daily. The calibration coefficient must be at least 0.995. If this criteria is not met, the analysis will be stopped and recalibration will be performed. A continuing calibration verification will be run once every 10 samples. Duplicate samples will be run once every 10-20 samples.

If quality control samples as provided with each lead paint test kit (e.g., positive/negative controls), then they will also be run per the vendor's instructions. Painted PEMs containing no lead as well as each of the PEMs substrates containing no paint will also be run as part of the verification test.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

The instruments used for the reference analyses will be tested and inspected as per the standard operating procedures or instrument manuals of the subcontract laboratory or per the standard methods being used to make each measurement. Any discovered deficiencies with a particular instrument will be resolved per the protocol of the laboratory in a timely manner. When technical staff operate and maintain technologies undergoing testing, those activities will follow directions provided by the technology vendor. Any maintenance required on components of the lead paint test kits will be the responsibility of the vendor.

B7 CALIBRATION/VERIFICATION OF TEST PROCEDURES

The ICP instrument used for the reference analyses will be calibrated per the standard reference method or the standard operating procedures of the analysis laboratory. A calibration will be run at the beginning of each set of analyses or at least once daily. The calibration coefficient must be at least 0.995 or higher. An independent calibration verification (ICV) standard will be run once a day after calibration and a continuous calibration verification (CCV) standard will be run at the beginning and end of each sample run. The ICV and CCV must be within ±10% and ±20%, respectively, of known values. If these evaluation criteria are not met, analysis must be stopped and recalibration performed. If the recalibration fails, the standards must be re-made and/or the equipment must be evaluated. If any component of a lead paint test kit requires calibration, the vendor will provide Battelle technical staff with instructions on how to properly maintain such components.

B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

PEMs used for verification testing will be made prior to the initiation of this test by Battelle in accordance with the Revised Plan For Development And Production Of Performance Evaluation Materials For Testing Of Test Kits For Lead In Paint under the Environmental Technology Verification Program[7]. The film thickness, homogeneity, and lead levels of the paint applied to the PEMs will be verified prior to full-scale PEMs production[7]. This process will test the paint formulation and application to ensure that the desired lead level is being

achieved during full PEM production. PEMs will be measured for film thickness using a Positector 6000 coating thickness gauge. Subsequently, paint chip samples corresponding to the locations of the film thickness measurements will be obtained following ASTM E1729.[8] These samples will be shipped to the NLLAP-accredited laboratory for analysis. This process will not verify the lead levels of individual PEMs used for verification testing and will take place prior to the production of PEMs for this verification test. Details on this process can be found in the PEMs development plan[7].

B9 NON-DIRECT MEASUREMENTS

No non-direct measurements will be used during this verification test.

B10 DATA MANAGEMENT

Various types of data will be acquired and recorded electronically or manually by Battelle during the verification test. Table 2 summarizes the types of data to be recorded. All data and observations for the operation of the test kits will be documented by Battelle technical staff on data sheets or in laboratory record books. Results from the subcontract laboratory reference instruments will be compiled by the subcontractor's staff in electronic format and submitted to Battelle upon obtaining the results.

Records received by or generated by any technical staff during the verification test will be reviewed by a Battelle staff member within two weeks of generation or receipt, before the records are used to calculate, evaluate, or report verification results. If a Battelle staff member generated the record, this review will be performed by a Battelle technical staff member involved in the verification test, but not the staff member who originally generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, any calculations performed by technical staff will be spot-checked by Battelle QA and/or technical staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this test/QA plan. The data obtained from this verification test will be compiled and reported independently for each technology. Results for technologies from different vendors will not be compared with each other.

Among the QA activities conducted by Battelle QA staff will be an audit of data quality. This audit will consist of a review by the Battelle Quality Manager (or his designee) of at least 10% of the test data. During the course of any such audit, the Battelle Quality Manager will inform the technical staff of any findings and any need for immediate corrective action. If serious data quality problems exist, the Battelle Quality Manager will request that Battelle's Verification Test Coordinator issue a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken.

SECTION C
ASSESSMENT AND OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

Every effort will be made in this verification test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of this test/QA plan is to establish mechanisms necessary to ensure this. Internal quality control measures described in this test/QA plan, which is peer reviewed by a panel of outside experts, implemented by the technical staff and monitored by the Verification Test Coordinator, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the Verification Test Coordinator. Technical staff have the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the Verification Test Coordinator, who will work with the Battelle Quality Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of any EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this verification test.

C1.1 Performance Evaluation Audits

A Performance Evaluation (PE) audit will be conducted to assess the quality of the reference method measurements made in this verification test. The reference method PE audit will be performed by supplying an independent, NIST-traceable lead paint standard, to the subcontract laboratory. The PE audit samples will be analyzed in the same manner as all other samples and the analytical results for the PE audit samples will be compared to the nominal concentration. The target criterion for this PE audit is agreement of the analytical result within 20% of the nominal concentration. If the PE audit result does not meet the target criterion, the PE audit will be repeated. If the outlying results persist, the source of error will be investigated and corrective action taken as necessary until successful PE audit results are obtained. This audit will be performed once at the start of the test, and will be the responsibility of the Verification Test Coordinator or designee.

C1.2 Technical Systems Audits

The Battelle Quality Manager or designee will perform a TSA of the actual evaluation of the test kits at least once during this verification test. The purpose of this audit is to ensure that the verification test is being performed in accordance with the AMS Center and ETV Program QMPs[1,6], this test/QA plan, any published reference methods, and any Standard Operating Procedures (SOPs) used. In the TSA, the Battelle Quality Manager, or a designee, may review the reference methods used, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures In the TSA, the Battelle Quality Manager will observe testing in progress, observe the reference method sample preparation and analysis (when available), inspect documentation, and review technology-specific record books. He or she will also check standard certifications and technology data acquisition procedures, and may confer with technical staff. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The EPA Quality Manager will receive a copy of Battelle's TSA report. An initial, draft TSA report, not including verification of corrective actions, will be submitted to EPA within 2 weeks of completion of the audit. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the verification test. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

C1.3 Data Quality Audits

The Battelle Quality Manager or his designee will audit at least 10% of the verification data acquired in the verification test, including any data packages received from the subcontract reference laboratory. Data packages will conform to ADQ guidelines provided by EPA. The Battelle Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked. An initial, draft TSA report, not including verification of corrective actions, will be submitted to EPA within 2 weeks of completion of the audit. Data packages received from the subcontract laboratory will be briefly reviewed by Battelle for completeness and then provided to EPA within three days of receipt of the data package. The packages will

not have gone through any data quality audits at this time, as these audits will be ongoing at the time of submission of the data to EPA.

C1.4 QA/QC Reporting

Each assessment and audit will be documented in accordance with the AMS Center and ETV Program QMP.[1,6] The results of the technical systems and data quality audit will be submitted to EPA. Initial, draft reports, not including verification of corrective actions, will be submitted to EPA within 2 weeks of completion of the audit. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problems
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others.

C2 REPORTS TO MANAGEMENT

The Battelle Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Battelle Quality Manager will notify the Battelle Management to authorize a stop work order. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle Quality Manager will ensure that follow-up corrective action has been taken. The test/QA plan and final report are reviewed by EPA QA Manager and the EPA ESTE Project Officer. Upon final review and approval, both documents will then be posted on the ETV website (www.epa.gov/etv).

SECTION D
DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The key data review requirements for the verification test are stated in Section B10 of this test/QA plan. In general, the data review requirements specify that the data generated during this test will be reviewed by a Battelle technical staff member within two weeks of data generation. The reviewer will be familiar with the technical aspects of the verification test, but will not be the person who generated the data. This process will serve both as the data review and the data verification, and will ensure that data have been recorded, transmitted, and processed properly. Furthermore, this process will ensure that the lead paint test kit data and the reference method data are collected under appropriate testing conditions and that the reference method data meet the reference method specifications.

The data validation requirements for this test involve a data quality assessment relative to the DQIs and audit acceptance criteria specified for this test. The DQIs listed in Section A 4.2 and B5 will be used to validate the data quality. The QA audits described within Section C of this document, including the performance evaluation audit and data quality audit, are designed to validate the data quality.

D2 VALIDATION AND VERIFICATION METHODS

Data verification is conducted as part of the data review, as described in Section B10 for this test/QA plan. A visual inspection of handwritten data will be conducted to ensure that all entries were properly recorded or transcribed, and that any erroneous entries were properly noted (i.e., single line through the entry with an error code and the initials of the recorder and date of entry). Data manually incorporated into spreadsheets for use in calculations will be checked against handwritten data to ensure that transcription errors have not occurred. All calculations used to transform the data will be reviewed to ensure the accuracy and the appropriateness of the calculations. Calculations performed manually will be reviewed and repeated using a handheld calculator or commercial software (e.g., Excel). Calculations performed using standard commercial office software (e.g., Excel) will be reviewed by inspecting the equations used in calculations and verifying selected calculations by handheld calculator. Calculations performed

using specialized commercial software (i.e., for analytical instrumentation) will be reviewed by inspection and, when feasible, verified by handheld calculator, or standard commercial software.

To ensure that the data generated from this test meet the goals of the test, a number of data validation procedures will be performed. Section C of this test/QA plan provides a description of the validation safeguards employed for this verification test. Data validation and verification efforts include the completion of QC activities and the performance of TSA audits as described in Section C. The data from this test will be evaluated relative to the measurement DQIs described in Section A7.1 and B5 of this test/QA plan. Data failing to meet these criteria will be flagged in the data set and not used for evaluation of the technologies, unless these deviations are accompanied by descriptions of their potential impacts on the data quality.

A data quality audit will be conducted by the Battelle Quality Manager to ensure that data review, verification, and validation procedures were completed, and to assure the overall data quality.

D3 RECONCILIATION WITH USER REQUIREMENTS

This purpose of this verification test is to evaluate the performance of qualitative spot test kits for lead based paint. To meet the requirements of the user community, the data obtained in this verification test should include thorough documentation of the performance of each lead paint test kit during the verification test. The data review, verification, and validation procedures described in previous sections will assure that data meet these requirements, are accurately presented in the verification reports generated from this test, and that data not meeting these requirements are appropriately flagged and discussed in the verification reports. Additionally, all data generated using the reference method, which are used to evaluate technology results during the verification test, should meet the QA requirements of any applicable standard operating procedures or instrumentation instruction manuals.

The data from this verification test will be compiled into an ETV verification report. The report will be submitted to EPA in Word and Adobe pdf format and subsequently posted on the ETV website. This test/QA plan and the resulting ETV verification report(s) will be subjected to review by the lead paint test kit vendors, Battelle staff, EPA, and expert peer reviewers. The reviews of this test/QA plan will assure that this verification test and the resulting reports meet the needs of potential users of the qualitative spot test kits for lead based paint.

SECTION E
REFERENCES

E1 REFERENCES

1. "Environmental Technology Verification Program Quality Management Plan", December 2002, EPA/600/R-03/021.

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Appendix A

REVISED PLAN FOR DEVELOPMENT AND PRODUCTION OF PERFORMANCE EVALUATION MATERIALS FOR TESTING OF TEST KITS FOR LEAD IN PAINT UNDER THE ENVIRONMENTAL TECHNOLOGY VERIFICATION PROGRAM

July 2009

1. Overview of Problem

The accurate and efficient identification of lead-based paint in housing is important to the Federal government and to private individuals living in residences containing such paints, especially when renovation, repair or painting work is planned. Renovation, repair and painting (RRP) may disturb painted surfaces and produce a lead exposure hazard. According to a recent report by the President's Task Force on Environmental Health Risks and Safety Risks to Children, approximately 24 million U.S. dwellings were at risk for lead-based paint hazards in 1999. 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 milligram per centimeter squared or 0.5 percent by weight."

The Preamble for the proposed EPA rule for RRP activities mentions the development of an improved test kit for paint that has a false negative rate of no more than 5 percent and a false positive rate of no more than 10 percent vis-à-vis the federal standards for lead-based paint. The Preamble also refers to an EPA evaluation and recognition program for test kits, initially for kits that are candidates to meet the goal of a 5 percent false negative rate, and then for kits that are candidates to meet the joint goals of a 5 percent false negative rate and a 10 percent false positive rate. As stated in the Preamble, test kit performance would have to be "validated by a laboratory independent of the kit manufacturer, using ASTM International's E1828, Standard Practice for Evaluating the Performance Characteristics of Qualitative Chemical Test Kits for Lead in Paint (Ref. 50) or an equivalent validation method. The instruction for use of any particular kit would have to conform to the results of the validation, and the certified renovator must follow the manufacturer's instructions when using the kit."

This effort includes the development and production of Performance Evaluation Materials (PEMs) for use in the evaluation of the performance characteristics of qualitative test kits for lead in paint. These PEMs will be used by independent laboratories for the verification of existing and new test kits under the Environmental Technology Verification (ETV) program. This effort includes the development and production of as many as 6,600 PEMs measuring approximately 3 inches by 3 inches. Note that estimates of the number of test panels needed assume that multiple tests can be conducted on each panel.

2. Study Objectives

The EPA requires a sufficient number of PEMs to evaluate up to 14 different test kits. Although a formal power study was not conducted, it is assumed that this number of panels should be sufficient to thoroughly test the kits, given that multiple tests can be conducted on each panel. How many tests can be conducted on each panel will depend on the specific requirements of each test kit.

In comparison to the NISTIR 6398 study "Spot Test Kits for Detecting Lead in Household Paint: A Laboratory Evaluation", this effort will include many of the same variables. Table 1 highlights the differences and similarities to both studies. Neither the NISTIR 6398 study nor the current PEM production effort described here claims to produce Certified Reference Materials (CRM's) as defined by ISO Guides 31, 34 and 35. Meeting that certification requires storage and transportation stability studies which can take up to 36 months. These PEMs are expected to be utilized within 18 months of production. The PEMs produced for this study will

each be labeled and individually wrapped and sealed to minimize surface abrasions during storage and transport.

Table 1: NISTIR 6398 vs. PEM Production

Category	NISTIR 6398 (2000)	PEM Production (2008)	Comments
Certified Reference Materials per ISO Guides 31, 34 & 35	no	no	See note below
Lead Types	Lead Carbonate & Lead Chromate	Lead Carbonate & Lead Chromate	
Lead Levels (mg/cm^2)	10 levels (0 up to 3.5) depending on lead type and Coefficient of Variation results after production	Seven levels (0, 0.3, 0.6, 1.0, 1.4, 2.0, 6.0)	
Substrates	Metal - Mylar and release paper then glue applied to wood and plaster	Apply directly to wood, metal, drywall and plaster	Application directly to the substrates closely simulates real-world scenario
Application methods	Drawdown and roller	Spray for zero lead panels and topcoats; Drawdown for all others	Drawdown allows for direct contact between leaded paint and substrate and better consistency of film thickness
Leaded Paint Type	Linseed Oil/ Lead paste added to alkyd paint	Linseed Oil Based	Historically accurate linseed oil paint formulation
Fineness of Lead Dispersion Test	None	ASTM D1210	Ensure lead pigment agglomerates are adequately pulverized and sufficiently dispersed through paint
Topcoats	2 = Alkyd and latex	2 = Primer and latex	
Verification Study	Originally 37 different paints were produced and applied, once analyzed, results from only 19 total paints were actually included in the test	Verification of lead level applied with a given film thickness on a small number of panels prior to full production	
Homogeneity Analysis	After panel production was completed a Coefficient of Variation was determined using ICP analysis and a variation of less than 20% was accepted	Independent homogeneity study planned prior to and throughout full PEM production using ICP analysis	

3. Study Design Options

The EPA specified four main design factors of interest – paint speciation, substrate, paint lead level, and overcoat color. These are discussed below along with topcoat color, number of

topcoats, and panel size. Table 2 presents a simple design that would yield 468 panels per test kit for the ETV evaluation. Across the nine kits originally planned for this adds up to 4,212 panels and across 14 test kits this adds up to 6,552 panels. This number of PEMs should allow for sufficient replicates of each design factor of interest for the ETV tests.

3.1 Paint types

A typical interior paint formulation containing either white lead (lead carbonate) or lead chromate will be prepared. The binder composition will be linseed oil based. The preparation of linseed oil-based paint PEMs is substantially longer due to the increased drying time required for this type of chemistry.

3.2 Substrates

We will use wood (both poplar and pine), metal (zinc phosphated cold-rolled steel), drywall, and plaster (limestone base) applied over drywall. The two types of wood are proposed in order to better represent potential substrates across the range of pre-1978 housing. These represent typical substrates to which leaded paint was originally applied.

3.3 Lead Levels

This plan proposes producing panels at seven lead levels: 0.0 mg/cm^2, 0.3 mg/cm^2, 0.6 mg/cm^2, 1.0 mg/cm^2, 1.4 mg/cm^2, 2.0 mg/cm^2, and 6.0 mg/cm^2. These levels represent two levels containing lead below the current lead based paint standard of 1.0 mg/cm^2, one level at the standard, two levels slightly above the standard, and additional higher level (6.0 mg/cm^2) to investigate differences in performance at significantly higher lead levels as well as represent potential lead levels on exterior surfaces, which are higher than those found on interior walls. The level of the current standard, 1.0 mg/cm^2, is included to allow for the evaluation results to provide a smoother performance evaluation curve. In addition, blank samples with no lead paint applied will also be produced for testing and quality control purposes.

3.4 Paint Colors

Three colors of topcoats will be applied randomly across the panels. The following paint colors will be used: white, reddish-orange, and grayish-black. These colors were chosen based on potential for certain colors to cause interferences with the performance of some kits.

3.5 Layers of paint on top of lead paint (Topcoats)

Two different layers of paint will be applied over the leaded paint. One will be a primer designed for adhesion to linseed oil-based paint and the second coat will be a typical interior modern latex paint tinted to one of three colors. The thickness of each topcoat is not a design variable. The manufacturers' recommended application thickness will be used. Two coats at the recommended thickness will be applied.

3.6 Panel Size

3 inch by 3 inch PEMs will be produced. The assumption is that multiple tests (up to four replicates) can be conducted per panel.

Table 2: PEMs Produced for ETV Test

Lead Type	Lead Level	Substrate	# Samples Produced Per Test Kit by Topcoat Color				9 Test Kits	14 Test Kits
			White	Red-Orange	Grey-Black	Total		
Control Blank	0	Wood-Poplar	2	1	2	5	45	70
		Wood-Pine	1	2	1	4	36	56
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
White Lead	0.3	Wood-Poplar	2	1	2	5	45	70
		Wood-Pine	1	2	1	4	36	56
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
	0.6	Wood-Poplar	1	2	1	4	36	56
		Wood-Pine	2	1	2	5	45	70
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
	1.0	Wood-Poplar	2	1	2	5	45	70
		Wood-Pine	1	2	1	4	36	56
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
	1.4	Wood-Poplar	1	2	1	4	36	56
		Wood-Pine	2	1	2	5	45	70
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
	2.0	Wood-Poplar	2	1	2	5	45	70
		Wood-Pine	1	2	1	4	36	56
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
	6.0	Wood-Poplar	1	2	1	4	36	56
		Wood-Pine	2	1	2	5	45	70
		Metal	3	3	3	9	81	126
		Drywall	3	3	3	9	81	126
		Plaster	3	3	3	9	81	126
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)			27	243	378
		Wood-Pine				27	243	378
		Metal				54	486	756
		Drywall				54	486	756
		Plaster				54	486	756
	Subtotal - Per Test Kit		156	156	156	468	4,212	6,552

4. Production Plan

4.1 Materials

Nine different paint formulations will be 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 will be designed to consistently achieve the lead levels required when applied at typical wet film builds. The paint samples will be 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. Battelle staff members completing this task have many years of direct industry experience in commercial and laboratory paint production. The paint formulations are shown below on Tables 3 and 4. Since the molecular compositions of the two lead pigments are different, the formulations have accounted for this by adjusting the load levels. Additional formulation changes will be investigated and implemented as necessary during production of the paints for the ETV evaluation.

The lead pigment fineness of dispersion will be monitored throughout the grinding process and once a Hegman grind of 5-6 has been achieved (ASTM D1210), the paint will be let-down with additional turpentine and Japan drier.

Table 3: Lead Carbonate Paint Formulations

0% Zero Lead Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	59.67	1491.75
Pb CO_3	American Elements	0.00	0.00
TiO2	DuPont	24.86	621.56
Linseed Oil	Recochem Inc.	5.97	149.18
Boiled Linseed Oil	Recochem Inc.	0.60	14.92
Turpentine	Recochem Inc.	8.70	217.55
Japan Drier	Barr	0.20	5.04
		100.00	2500.00

0.3% Lead Carbonate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	59.08	1477.08
Pb CO_3	American Elements	5.91	147.71
TiO2	DuPont	19.69	492.36
Linseed Oil	Recochem Inc.	5.91	147.71
Boiled Linseed Oil	Recochem Inc.	0.59	14.77
Turpentine	Recochem Inc.	8.62	215.41
Japan Drier	Barr	0.20	4.97
silica (TS 100)	Degussa	1.00	25.00
		101.00	2525.00

1.0% Lead Carbonate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	55.53	1388.14
Pb CO_3	American Elements	11.57	289.20
TiO2	DuPont	18.51	462.71
Linseed Oil	Recochem Inc.	5.55	138.81
Boiled Linseed Oil	Recochem Inc.	0.56	13.88
Turpentine	Recochem Inc.	8.10	202.44
Japan Drier	Barr	0.19	4.82
silica (TS 100)	Degussa	1.50	37.50
		101.50	2537.50

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

2.0% Lead Carbonate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	48.16	1204.04
Pb CO_3	American Elements	22.73	568.31
TiO2	DuPont	15.41	385.29
Linseed Oil	Recochem Inc.	5.28	132.00
Boiled Linseed Oil	Recochem Inc.	0.53	13.20
Turpentine	Recochem Inc.	7.70	192.50
Japan Drier	Barr	0.19	4.67
silica (TS 100)	Degussa	1.50	37.50
		101.50	2537.50

6.0% Lead Carbonate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	16.73	418.16
Pb CO_3	American Elements	64.49	1612.23
TiO2	DuPont	5.58	139.39
Linseed Oil	Recochem Inc.	5.09	127.34
Boiled Linseed Oil	Recochem Inc.	0.51	12.73
Turpentine	Recochem Inc.	7.43	185.70
Japan Drier	Barr	0.18	4.45
silica (TS 100)	Degussa	2.00	50.00
		102.00	2550.00

Table 4: Lead Chromate Paint Formulations

0.3% Lead Chromate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	60.03	1500.74
$PbCrO_4$	American Elements	4.40	110.05
TiO2	DuPont	20.01	500.25
Linseed Oil	Recochem Inc.	6.00	150.07
Boiled Linseed Oil	Recochem Inc.	0.60	15.01
Turpentine	Recochem Inc.	8.75	218.86
Japan Drier	Barr	0.20	5.01
silica (TS 100)	Degussa	0.70	17.50
		100.70	2517.50

1.0% Lead Chromate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	55.53	1388.14
$PbCrO_4$	American Elements	11.57	289.20
TiO2	DuPont	18.51	462.71
Linseed Oil	Recochem Inc.	5.55	138.81
Boiled Linseed Oil	Recochem Inc.	0.56	13.88
Turpentine	Recochem Inc.	8.10	202.44
Japan Drier	Barr	0.19	4.82
silica (TS 100)	Degussa	1.00	25.00
		101.00	2525.00

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

2.0% Lead Chromate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	42.32	1058.12
$PbCrO_4$	American Elements	27.83	695.72
TiO2	DuPont	14.81	370.34
Linseed Oil	Recochem Inc.	5.80	145.00
Boiled Linseed Oil	Recochem Inc.	0.58	14.50
Turpentine	Recochem Inc.	8.46	211.46
Japan Drier	Barr	0.19	4.85
silica (TS 100)	Degussa	1.00	25.00
		101.00	2525.00

6.0% Lead Chromate Paint Formulation			
Materials	**Supplier**	**% by wt.**	**Gram wt**
ZnO	The Carry Co.	5.99	149.69
$PbCrO_4$	American Elements	77.84	1946.00
TiO2	DuPont	2.00	49.90
Linseed Oil	Recochem Inc.	5.47	136.75
Boiled Linseed Oil	Recochem Inc.	0.55	13.68
Turpentine	Recochem Inc.	7.98	199.43
Japan Drier	Barr	0.18	4.54
silica (TS 100)	Degussa	1.00	25.00
		101.00	2525.00

The applied lead levels will be verified prior to full-scale PEM production via drawdown application on quality-controlled metal panels. Dried paint chip samples will be obtained and evaluated by an independent NLLAP-accredited laboratory using ICP-AES or equivalent analysis.

Quadrant #1		Quadrant #2	
Film Thickness Test Area	ICP Lead Level Test Area	Film Thickness Test Area	ICP Lead Level Test Area
Quadrant #3		**Quadrant #4**	
Film Thickness Test Area	ICP Lead Level Test Area	Film Thickness Test Area	ICP Lead Level Test Area

This lead level verification study will test the formulation and application to ensure that the desired lead level can be achieved during full PEM production. The test map in Figure 1 describes how each panel will be divided and where each test will be performed.

Figure 2. PEM Lead Level Verification Step Test Map

One panel of each film thickness will be prepared using the previously manufactured paints. After drying, the panel will be measured once in each quadrant for film thickness using a Positector 6000 coating thickness gauge. Subsequently, paint chip samples corresponding to the locations of the film thickness measurements will be obtained following ASTM E1729, "Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination." These samples will be shipped to the NLLAP-accredited laboratory for analysis. The laboratory will prepare the paint chip samples for analysis following ASTM 1656 and analyze the samples according to the ASTM 1613 method, "Standard Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques." Results will be provided electronically along with relevant QA/QC data on calibration samples, analytical duplicates, and analytical spikes.

Since the thickness of the paint film will determine the final lead level per unit area, the purpose of this verification step is to identify the approximate film thickness that will result in the targeted lead level for each paint. The ICP lead level measurements will be used to determine the film thickness that led to the desired lead level. If average lead levels of the nearest measurements differ from the target by more than 10 percent for target lead levels of 1.0 mg/cm^2 and higher and 15 percent for target lead levels <1.0 mg/cm^2, corresponding adjustments will be made to the targeted film thickness measurements prior to the homogeneity testing to follow.

Once the desired film thicknesses are determined from the verification step, panels will be coated for a Homogeneity Study consisting of producing three additional panels at each of the six lead levels for both types of lead pigments, obtaining four paint chip samples from each panel (one from each quadrant), and conducting ICP-AES analysis on those paint samples to determine precise lead measurements. The results from this process will confirm the ability to apply consistent and accurate lead levels across each panel. Prior to paint chip sampling, film thickness measurements also will be obtained to verify the desired film thickness for each leaded paint. Table 5 provides an example of the data that will be collected for the Homogeneity study.

Table 5: Lead Level Verification/Homogeneity Study for ETV Panels

Lead Type	Lead Level	Replicate	Film Thickness Measurements - 1 per Quadrant				ICP Testing - 1 per Quadrant			
			Q-1	Q-2	Q-3	Q-4	Q-1	Q-2	Q-3	Q-4
none	0	1								
		2								
		3								
White Lead	0.3	1								
		2								
		3								
	0.6	1								
		2								
		3								
	1.0	1								
		2								
		3								
	1.4	1								
		2								
		3								
	2.0	1								
		2								
		3								
	6.0	1								
		2								
		3								
Lead Chromate	0.3, 0.6, 1.0, 1.4, 2.0, 6.0	1	Same testing scheme as the White Lead paints							
		2								
		3								

The ICP lead level measurements obtained will be analyzed to establish accuracy of the lead levels in reference to target lead levels as well as the variability across and within panels. Variability will be measured using the Coefficient of Variation (i.e., the standard deviation divided by the mean). A Coefficient of Variation (CoV) less than 15 percent will be deemed acceptable.

4.2 Facilities

Battelle laboratories include a walk-in spray booth capable of this type of production as well as air handling equipment and monitors to ensure staff safety.

4.3 Painting Process

Some substrates, such as wood and drywall will be cut down into 3" by 3" panel dimensions. The plaster panels will be prepared by applying plaster to pre-cut drywall panels via hand trowel application. All drywall and drywall/plaster panels will require edge treatment to minimize dusting of the interior gypsum. This treatment will be performed by spray applying a latex primer to the edge surfaces only to encapsulate the inner core. All substrate preparations will take place prior to the application of any leaded paints.

The linseed oil-based paints will be applied via drawdown bar directly to the surface of the substrates. The drawdown method, although somewhat less efficient for applying coatings to a large number of panels, will allow more precise film builds to be achieved as the application device will be set to the previously determined target film build level. The drawdown paint

application method will be utilized for both the white lead and lead chromate paints at the three specified lead levels. The substrates will be lined up in sets of either two or four, depending on the substrate type (2 drywall, 2 plaster. 4 metal or 4 wood panels next to each other). The paint will be applied across the two or four PEMs in the set at the same time. The order of application for each set will be assigned randomly using a random number generator in MS Excel. For example, the order of application could be drywall, metal, wood, wood, plaster, metal, etc. The PEMs will be placed on a horizontal board to dry overnight. Using the same randomization process, a metal reference panel will be coated in the same fashion (same drawdown bar, same paint formulation). Reference panels will be inserted as one of the four metal panels in a set, with the location within the set randomly determined. Within Table 6, which provides an example drawdown scenario for 56 panels targeted for the same lead paint application and same topcoat color, three reference panels are inserted into the application process to verify the lead levels applied to surrounding panels. The first and second reference panels are randomly placed among the first eight and second eight sets, respectively, while the third reference panel is randomly placed among the last seven sets of PEMs. The position of the reference panel within the set of metal panels is also randomly determined.

Table 6. Illustration of Reference Panel Location within Set of 56 Production Panels

Drawdown Positions				
#1	#2	#3	#4	Order
drywall	drywall			1
wood	wood	wood	wood	2
drywall	drywall			3
plaster	plaster			4
plaster	plaster			5
drywall	drywall			6
metal	metal	REF	metal	7
plaster	plaster			8
plaster	plaster			9
metal	REF	metal	metal	10
wood	wood	wood	wood	11
metal	metal	metal		12
drywall	drywall			13
drywall	drywall			14
wood	wood	wood	wood	15
plaster	plaster			16
metal	metal			17
plaster	plaster			18
REF	metal	metal	metal	19
plaster	plaster			20
drywall	drywall			21
wood	wood			22
drywall	drywall			23

The reference PEMs will be tested for film thickness during application and for lead level verification by ICP analysis after the paint has dried. This test procedure will check that the application process is resulting in appropriate lead levels. Metal panels will serve as the reference panels since the metal panels yield the most accurate measurements of film thickness

and lead levels. Previous work found that the metal panels provided the best film thickness measurements and also provided a better surface for extracting a complete paint chip sample for ICP analysis. Despite the use of metal reference panels, the lead levels and paint thickness on the reference panels will be representative of the coatings applied to all wood, drywall, plaster, and metal substrate panels that will be randomly scattered throughout each area.

Once reference panel results have undergone review and are determined to have met all target specifications, the PEMs will then be mounted onto 4 foot by 8 foot drying racks for the application of topcoat layers and conditioned in constant temperature and humidity rooms to ensure consistent curing for all samples. All substrates will be attached to the drying racks for painting using double stick tape. Following application of the topcoat layers, the PEMs will be removed for packaging and shipping. Packaging will involve attaching a label to each panel, covering the panel with a chem-wipe for protection, inserting the panel in a plastic, sealable bag, and attaching a matching label to the exterior of the bag. Separate boxes will be prepared containing full sets of PEMs.

5. Quality Assurance/Quality Control

Lead levels will be verified prior to full panel production by an independent NLLAP-accredited laboratory. This detailed testing of initial batches of samples during the PEMs verification step will establish that specified lead levels can be achieved. As part of the verification step, ICP analysis will also be conducted in multiple locations on initial batches of panels to confirm lead levels and homogeneity across panels. During production application of the leaded paints, a subset of reference panels for each set of PEMs (unique lead level, lead pigment combinations) will be tested to monitor the paint application process and ensure that targeted lead levels are achieved with acceptable variability. As noted above, the average lead level across the reference panels should be within 10% of the targeted value for panels at 1.0 mg/cm^2 and higher and within 15% for panels below 1.0 mg/cm^2. The tolerance for an acceptable level of variability in film thickness and lead level is <15% Coefficient of Variation (CoV). One reference panel will be inserted into the production process for every 18 to 20 production panels.

The laboratory will be required to provide QA/QC data from each batch of paint samples analyzed on calibration samples, analytical duplicates, and analytical spikes. Percent recovery for analytical spike samples must be within 20 percent of the actual value. The relative percent difference for analytical duplicates (i.e., the absolute value of the difference between the two samples divided by their average) must be less than 20 percent. If any QC samples are found to be outside of these tolerance limits, the laboratory will be asked to reanalyze the affected batch of samples.

6. Environmental Safety and Health Issues

Battelle has a safety and health plan related to producing lead-based paint and PEMs coated with these paints. The plan was approved by appropriate environmental safety and health personnel. Environmental monitoring during paint mixing and spraying activities determined that lead exposure levels were below OSHA standards. During development of the PEMs for the ETV

evaluation, Battelle staff will continue to comply with the safety and health plan. Some of the components of the safety plan include:

- All staff (and any visitors) will need hazard communication training on lead.
- Baseline and post-work blood-lead levels will be obtained on those that will be conducting the paint mixing and spray painting.
- Respirators will be used during leaded paint production and the spray application operation by staff that have undergone the required physical, training and fit test
- The interior of the spray booth will be covered with plastic or other material that can be easily removed and disposed as hazardous waste.
- The area in front of the booth will be set up as a change out area where the coveralls, etc, can be removed without spreading lead outside of the area.

Warning signs at the paint booth door and restricting access will be posted.

Summary of Homogeneity and Lead Levels Analyses for PEMs Development

Table A-1. Results from Final Homogeneity Testing for each set of ETV PEMs

Lead Type	Target Lead Level	Mean Levels		CoV*	
		ICP (mg/cm^2)	FT (mils)	ICP	FT
No Lead	0.0	0.00	0.73	22.9	12.3
White Lead	0.3	0.30	0.79	13.3	6.1
	0.6	0.65	0.95	7.1	5.7
	1.0	1.00	1.18	7.0	4.3
	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	2.7	11.0	6.0
	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)

Table A-2. 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	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
Lead Chromate (Yellow Lead)	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	1.41	11.0	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

Appendix B

Standard Operating Procedure (SOP)
For Collection of Dried Paint Samples for Lead Determination

I. Scope

This Standard Operating Procedure (SOP) describes the manner to which dried paint samples should be collected for the determination of lead on substrates such as metal, wood, plaster and drywall.

II. Purpose

The purpose of this SOP is to create a reproducible sampling technique that minimizes variability between samples and to accurately represent a lead level for a specific substrate.

III. References

A. ASTM E 1729-05 Standard Practice for Field Collection of Dried Paint Samples for Subsequent Lead Determination.

B. SOP AMA I-006-02 Standard Operating Procedure for the Safe Handling of Lead, Lead-Spiked Paint, and Lead-Spiked Samples

C. OSHA Standards for Lead-1910.1025

D. Battelle Safety and Industrial Hygiene Program Plan (#ESHQ-SIH-PP-005)

E. ETV ESTE Test/QA Plan For The Verification Of Qualitative Spot Test Kits For Lead In Paint

IV. Definitions

A. Paint collection tray-any clean, dry, lead-free container for use in catching paint scrapings.

B. PPE-Personal protective equipment

C. Lead worker-Training for any person physically handling lead powders or lead-containing materials such as paints.

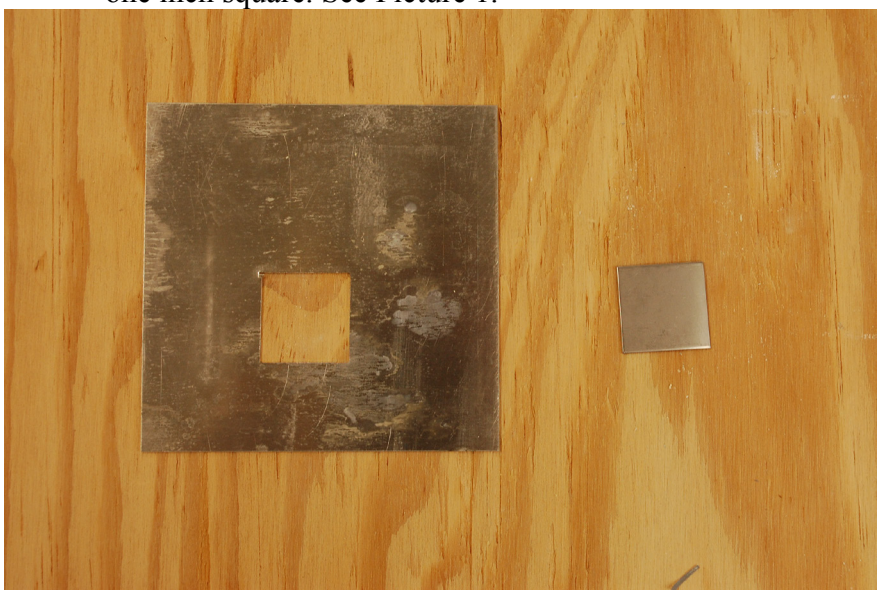
V. Procedures

A. Equipment and Materials
   i. PPE
      1. Disposable Coveralls/Scrubs
      2. Disposable Gloves
      3. Disposable Shoe Covers
      4. Safety Glasses with Side Shields

ii. Equipment

1. glass vials and lids
2. labels
3. tape
4. Sampling template – one inch square reusable aluminum or steel template with accurately known dimensions
5. disposable wipes-for cleaning off sampling equipment
6. sharp-edged knives/blades/chisels for cutting and scraping
7. tweezers
8. Paint collection tray
9. small brushes-for brushing off powder to weighing paper for transfer

B. Lead workers with full PPE in a dedicated lead hazard area with lead disposal will be used to minimize exposure to other persons or areas.
C. Label vial for collection using the number on the back of the panel
D. Place the 1 inch square template as close to the tested area as possible, without including any disturbed areas.
   i. There are two types of templates, a one inch square and a punch-out of one inch square. See Picture 1.

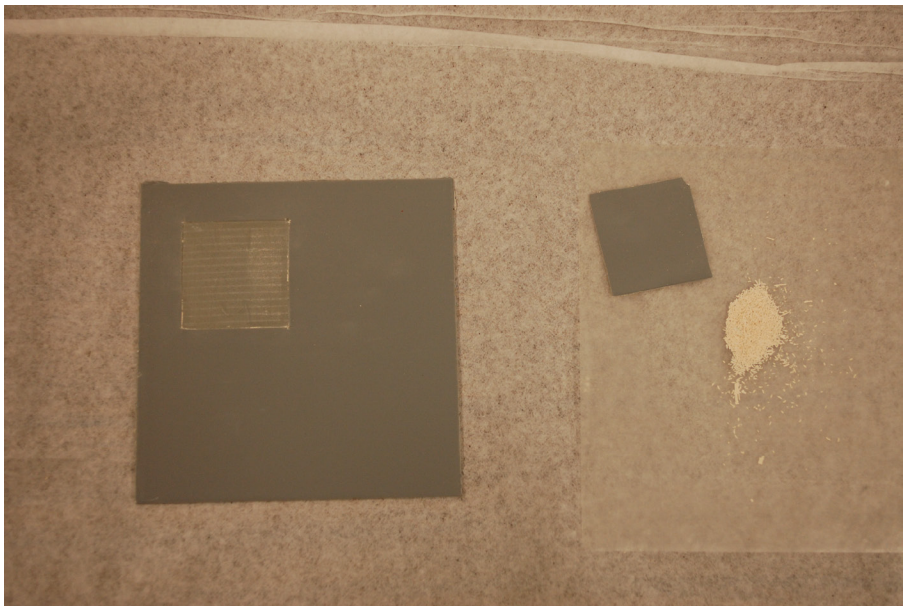


Picture 1: Templates

E. Metals
   i. While holding the template in place, score an outline of the area using a sharp edge.
   ii. Remove square template and clean the template with a wipe. Set aside

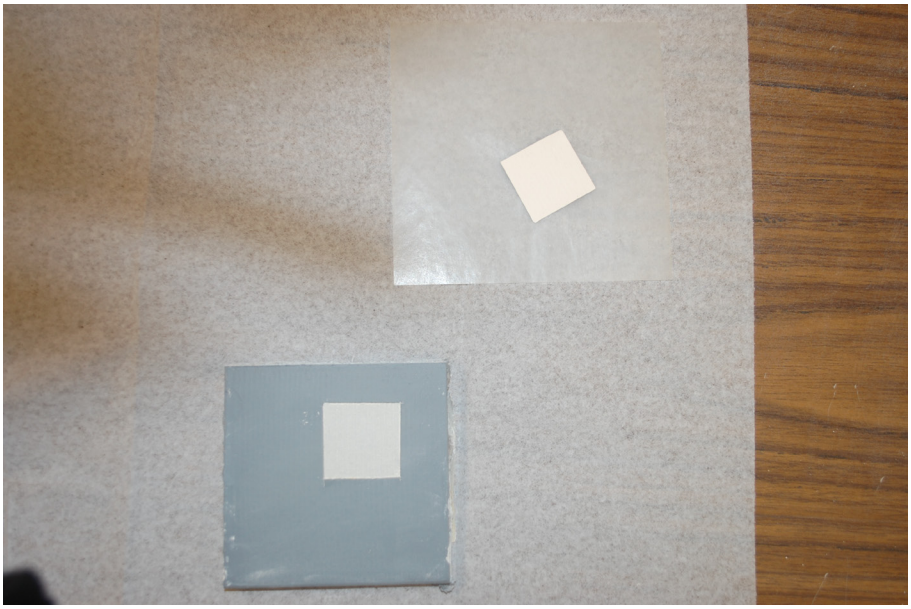
iii. Using tweezers, remove square latex topcoat and place in vial. See Picture 2.
iv. Using a blade, scrape the lead off the exposed one inch area and brush on to a collection tray. Make sure to brush all remaining lead and do not disturb adjacent area. See Picture 3.
v. Put contents of tray into the same vial, close lid tightly and label appropriately.

Picture 2: Metal with latex topcoat removed



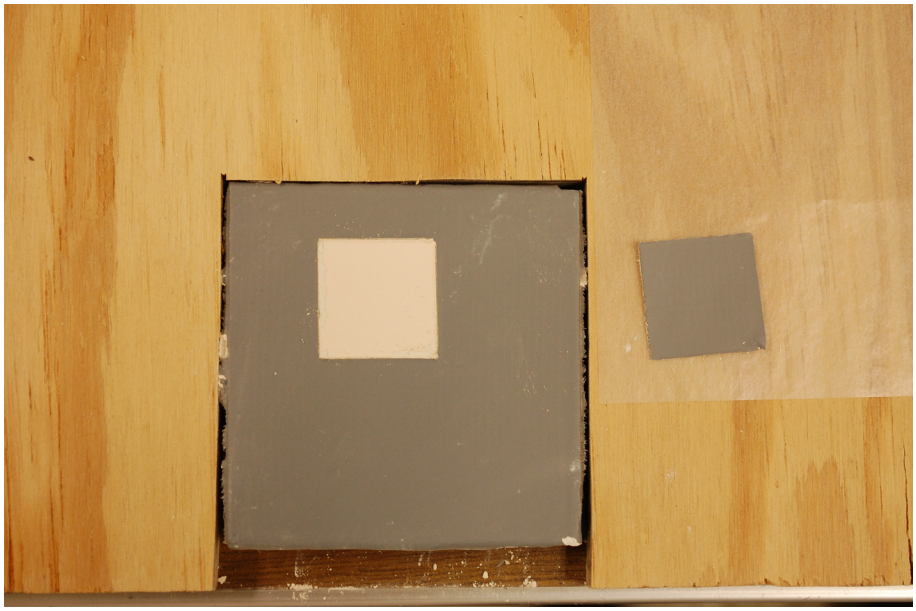
Picture 3: Metal with lead scraped and brushed on to collection tray

F. Drywall
   i. While holding the template in place, score an outline of the area using a sharp edge.
   ii. Remove template and clean template with a wipe, set aside.
   iii. Using tweezers remove latex topcoat. See Picture 4
   iv. Using a blade, scrape the lead off the exposed one inch area, minimizing any drywall paper within the area.
   v. Brush all remaining lead off the panel onto the collection tray without disturbing the adjacent area.
   vi. Place all material into a vial, close tightly and label properly.

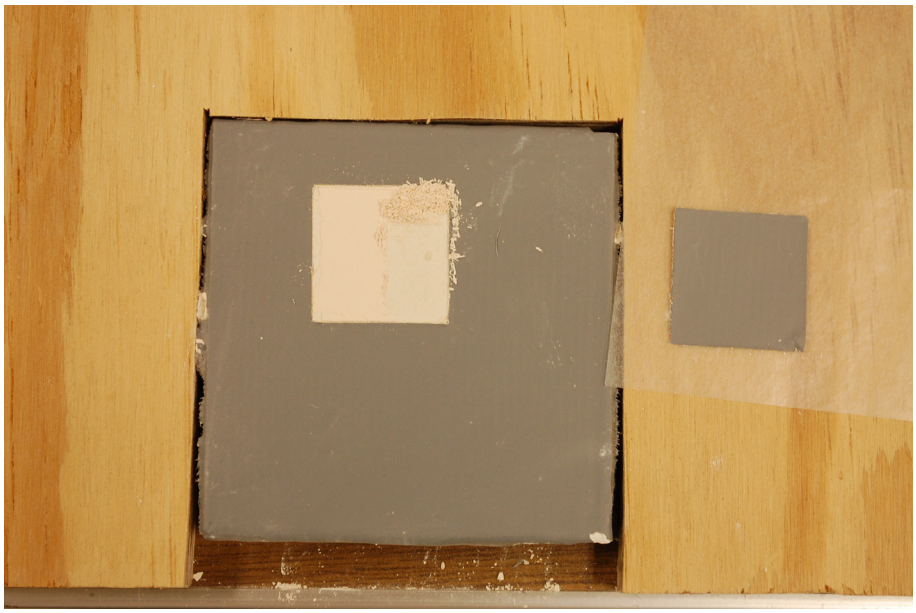


Picture 4: Drywall with latex topcoat removed

G. Plaster
   i. While holding the template in place, score an outline of the area using a sharp edge.
   ii. Remove template and clean template with a wipe, set aside.
   iii. Using tweezers remove latex topcoat. See Picture 5
   iv. Using a blade, scrape the lead off the exposed one inch area, minimizing the collection of any plaster with the sample.
   v. Brush on to a collection tray without disturbing the adjacent area. See Picture 6
   vi. Place all material into a vial, close tightly and label appropriately



Picture 5: Latex topcoat removed from plaster sample

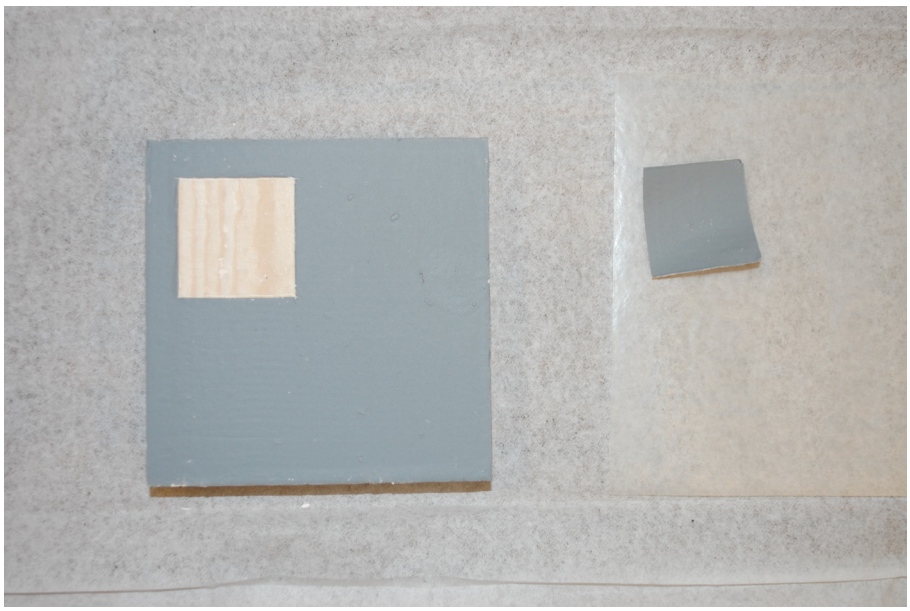


Picture 6: Scraping of lead from plaster

H. Pine and Poplar

i. While holding the template in place, score an outline of the area using a sharp edge.
ii. Remove template and clean template with a wipe, set aside.
iii. Using tweezers remove latex topcoat. See Picture 7
iv. Using a blade, scrape the lead off the exposed one inch area, minimizing the collection of any wood with the sample.

v. Brush on to a collection tray without disturbing the adjacent area. See Picture 7
vi. Place all material into a vial and close tightly



Picture 7: Latex topcoat removed from wood panel

VI. Safety

A. Project staff who work with lead or lead products must complete lead worker training. Visitors are not permitted in the lead contaminated areas without prior approvals and proof of required medical surveillance and training.
B. Staff will be on a Battelle Medical Monitoring Program, and will have had an annual physical, etc., as required for working with lead, and will be approved to work on projects using lead
C. Staff working with lead will be kept to the minimum number required to do the project.
D. Clothing or equipment will not be blown, shaken, etc. to remove dust.
E. Waste (such as filters, liners, PPE, wipes, ETC.) will be disposed of as per Battelle policies/procedures for lead contaminated items.
F. MSDS for Lead and Paint used in projects are in Room 5148
G. Battelle has strict policies that no food or beverages be present or consumed, nor any cosmetics applied within the laboratory area. Contact lenses will not be handled in or around the area
H. Any known or suspected exposure to lead outside of the engineering controls will be reported to Battelle Health Services or Safety Health/Emergency Response (SH/ER) representative as soon as possible
I. Refer to Battelle Safety and Industrial Hygiene Program Plan (ESHQ-SIH-PP-005) for additional guidance

VII. Shipping

A. Lead-containing painted samples will be analyzed further at a different location
B. Samples will be protected from possibly contaminating the shipping route by enclosing the vials in an additional plastic bag and securing the bag with tape.
C. Sample custody will be documented for the shipping and analysis of PEMs to the subcontract laboratory using standard chain-of-custody (COC) forms provided by the laboratory performing the analysis.
D. The original sample COC forms will accompany the samples; the shipper will keep a copy.
E. Copies of all COC forms will be delivered to the Verification Test Coordinator and maintained with the verification test records.