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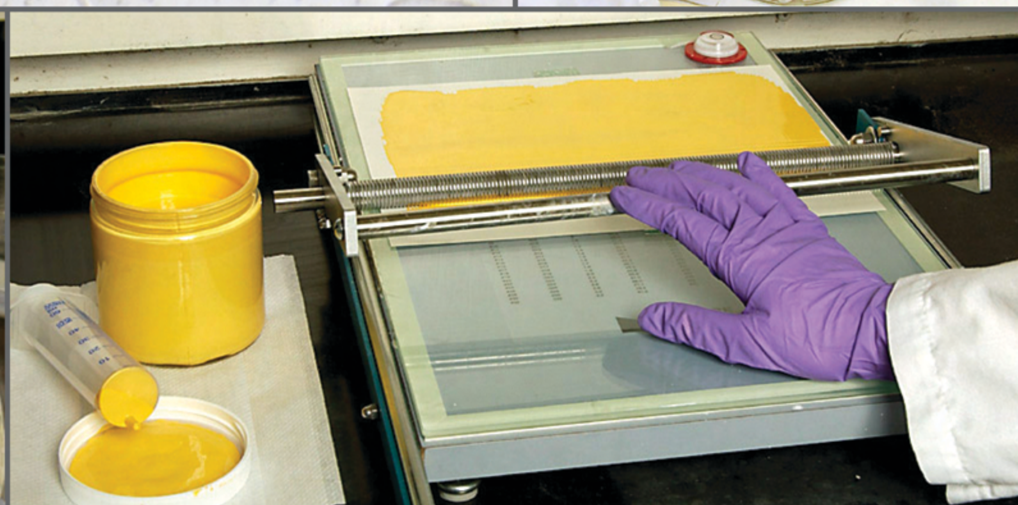

Standard Operating Procedure for the Preparation of Lead-Containing Paint Films and Lead-in-Paint Diagnostic Test Materials

SCIENCE

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Standard Operating Procedure for the Preparation of Lead-Containing Paint Films and Lead-in-Paint Diagnostic Test Materials

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1.0 PRINCIPLE AND APPLICABILITY

Exposure to lead (Pb) may adversely impact children's brains, nervous systems, and many organs. An estimated 310,000 U.S. children ages 1 to 5 have elevated blood leads. In the United States, the major exposure pathway for children to Pb is from deteriorated Pb-based paint (LBP), Pb-contaminated house dust, and residential soil. Approximately 40% of all U.S. housing units (about 38 million homes) have some LBP.[1] The Federal regulated Pb standard has been defined by the U.S. Department of Housing and Urban Development (Title X of the Housing and Community Development Act, 1992[2)] as equal to or greater than 0.5% Pb by weight or 1.0 mg Pb/cm^2. Homes built before 1978 are the most likely to contain LBP. Each year, more than 10 million renovation activities occur in homes, child-care facilities and schools potentially containing LBP. To reduce the exposures to Pb hazards during renovation, the U.S. Environmental Protection Agency (EPA) promulgated the "Lead: Renovation, Repair, and Painting Program; Final Rule" (RRP)[3] in April 2008. The rule requires the use of inexpensive test kits. However, no currently available commercial test kit can meet the performance requirements of no more than 5% false negative results at levels greater than the Federal regulated level and no more than 10% false positive results at levels less than the Federal regulated level.[3] Additional goals for the test kit procedures are that they should be inexpensive, take less than an hour per sample, and be easy to perform.

The simple, commercially produced test kits currently available for home testing for Pb in paint are very sensitive but do not provide quantification of Pb to meet the specifications in the RRP. As noted in Gutknecht et al., there are several field techniques already available for direct (in situ) quantitative analysis of Pb in painted surfaces, including field-portable, X-ray fluorescence and portable laser microprobe spectrometry.[4] The instrumentation for these methods is relatively expensive and requires extensive training. Additionally, there are numerous less expensive field methods available for quantitatively measuring Pb in solution. These include electrochemical reduction/oxidation (anodic stripping voltammetry), complexation (colorimetry), precipitation (gravimetry), or turbidimetry. However, to apply these methods, paint first must be removed quantitatively from the surface and Pb quantitatively solubilized from the paint. Grinding may be needed to facilitate solubilization.

There is a need for Pb-in-paint testing materials that will challenge current and new paint sampling methods, Pb extraction methods, and Pb measurement methods. This standard operation procedure (SOP) describes procedures for making challenging stand-alone, leaded paint films, as well as Pb-in-paint diagnostic materials that simulate old paints on a variety of substrates. Users of this SOP should evaluate the durability of the paint materials produced under their specific conditions of use. This is a general use SOP and is intended to be used by trained technical workers.

2.0 SUMMARY OF THE METHOD

This SOP describes the preparation of stand-alone, leaded paint films prepared according to old paint recipes. Further, this SOP describes the use of these paint films for the preparation of simulated old paints on a variety of substrates. Substrates used included wood, steel, masonry, and plaster. The leaded paint varied in pigment, color, and the presence of ions that have a potential for interfering with the Pb measurement performed with different methods, including various types of test kits.

2.1 Stand-Alone Paint Films

2.1.1 Preparation

The process of making stand-alone paint films starts with mixing the ingredients of the material, which for old, white paint recipes, consists of white Pb, zinc oxide, raw linseed oil, boiled linseed oil, and mineral spirits. The recipe may be varied by using a different Pb pigment, for example, lead chromate (yellow in color), a colorant such as red or black iron oxide, and metals that have the potential for interfering with Pb measurement techniques, including old or new test kits. Once the ingredients are selected, they are mixed in a ball mill. The homogenized mixture then is spread across a special piece of release paper laid on a flat glass surface using a wire-wound bar to control the thickness of the paint layer. The paint then is allowed to dry. Once dry, it is removed from the release paper for chemical analysis to determine areal (mg/cm^2) and mass (%) Pb concentrations.

2.1.2 Performance

Stand-alone paint films were prepared and analyzed for uniformity of thickness and variability in areal concentration (mg Pb/cm^2) and mass concentration (% Pb).[5] The paint films were marked off in 3 x 3-cm areas across a paint film, and the film was laid on a sheet of steel. An electronic meter then was used to measure the thickness in micrometers of the paint at various locations on a film. Appendix 1 presents thickness information for a number of paint films prepared using the old paint recipes. The thickness usually varied less than 5% across a paint film that is approximately 15 x 24 cm. Appendix 1 also presents Pb concentration data for a variety of paint films. The areal concentration (mg Pb/cm^2) usually varied by less than 10%.

2.2 Method Diagnostic Test Materials

2.2.1 Preparation

The stand-alone, leaded paint films are cut into appropriately sized pieces and then attached to a variety of substrates, including wood, metal, masonry, and plaster, using commercially available, oil-based primer. A second leaded paint film may be laid (attached with primer) over the first to make a more challenging test material. These paint films then can be overcoated with one or more layers of commercially available oil-based paint, followed by one or more layers of commercially available water-based paint to simulate what one might expect for old dwelling paint. As noted above, these diagnostic test materials can be made more challenging by adding colorants such as red or black iron oxide. Also, metals that might interfere with a particular type of measurement method can be added during the preparation of the stand-alone paint films. Test samples having different combinations of these materials can be prepared to vary the analytical challenge. Once the substrates are coated with the desired paint films and overcoats, they are baked in a laboratory oven to harden the paint to simulate some degree of aging.

2.2.2 Performance

In a project performed for the EPA, 31 different combinations of substrate, leaded paint films, and overcoats were prepared.[5] A table is presented in Appendix 2 that shows the results of analysis of representative samples or “coupons” of these materials. The variability of areal Pb concentration (mg Pb/cm^2) is generally less than 10%. Specification sheets were prepared for each of the 31 types of diagnostic test materials, which were analyzed for 15 elements in addition to Pb. Appendix 3 is an example of a specification sheet for a complex test material that combined a layer of paint with Pb chromate; a layer of paint with white Pb, black iron oxide, aluminum oxide, barium oxide, and magnesium oxide added; two overcoats of oil-based paint; and four overcoats of water-based paints.

3.0 DEFINITIONS, ACRONYMS, AND ABBREVIATIONS

A number of acronyms and abbreviations are used in this SOP. These acronyms and their meanings are as follows.

- EPA – U.S. Environmental Protection Agency
- ICP-OES – inductively coupled plasma-optical emission spectrometry
- LBP – lead-based paint
- Pb – elemental or ionic lead
- RRP – Lead; Renovation, Repair, and Painting Program; Final Rule
- RTI – RTI International
- SOP – standard operating procedure

4.0 HEALTH AND SAFETY WARNINGS

4.1 Safety with Paint Ingredients, Including Powdered Lead Compounds and Solvents

Weighing, mixing, and pouring of paint ingredients must be done in an approved chemical fume hood. While mixing ingredients, the technician is to wear all specified personal safety equipment including a lab coat/apron, dust mask, gloves, and safety glasses. Spilled materials are to be wiped up using wetted paper towels and the towels placed into labeled, resealable bags. It's very important to completely close the lid of the ball mill jar after filling it with the paint ingredients and before removing it from the hood.

Note: Powdered white Pb (lead carbonate) and lead chromate are extremely hazardous.[6,7,8]

4.2 Safety with Ball Mill

A cover (see Figure 1) should be made for the ball mill to protect the user from entanglement with the rollers. Place the ball mill jar securely and evenly on the rollers and close the cover before turning on the power to the mill. The rotation speed control dial position should be set before turning on power.

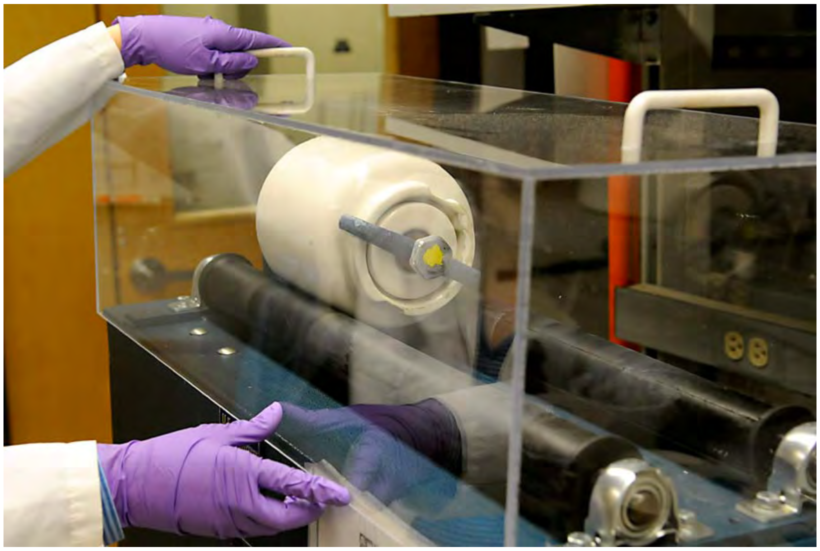

Figure 1. Ball mill being covered with safety shield to prevent injury from rollers.

4.3 Proper Disposal of Waste

Proper procedures for disposing of leaded and unleaded paint and paint cleanup solutions and materials should be followed. Proper procedures for disposing of flammable materials (mineral spirits) also should be followed. See Section 11.0 for more information on waste management.

5.0 EQUIPMENT, SUPPLIES, AND REAGENTS

5.1 Paint Mixing

5.1.1 Paint Ingredients

5.1.1.1 Basic Components

5.1.1.1.1 Boiled linseed oil, VWR Catalog No. EM-LX0305-3, or equivalent

5.1.1.1.2 Lead (III) chromate,[9] VWR Catalog No. AA14125-22, or equivalent

5.1.1.1.3 Mineral spirits, Klean-Strip Odorless,[10] or equivalent

5.1.1.1.4 Raw linseed oil, VWR Catalog No. IC96012205, or equivalent

5.1.1.1.5 Lead carbonate, basic[11] (white Pb), Aqua Solutions (Deer Park, TX), Catalog No. L3200-500G, or equivalent

5.1.1.1.6 Zinc oxide, VWR Catalog No. AA44263-A1, or equivalent

5.1.1.2 Potential Test Kit Interferences

5.1.1.2.1 Aluminum oxide, VWR Catalog No. EM-AX0612-1, or equivalent

5.1.1.2.2 Barium carbonate, VWR Catalog No. BDH0218-500G, or equivalent

5.1.1.2.3 Magnesium carbonate, VWR Catalog No. JT2436-1, or equivalent

5.1.1.3 Example Colorants

5.1.1.3.1 Black iron oxide, VWR Catalog No. 100202-020, or equivalent

5.1.1.3.2 Red iron oxide, Hoover Color Corporation,[12] Syn-Ox HR- 1201 RED, or equivalent

5.1.2 Equipment and Supplies for Mixing Paint Ingredients

5.1.2.1 Balance, up to 400 g capacity, such as Fisher Scientific, Model S-400, or equivalent

5.1.2.2 Cylindrical alumina grinding media, U.S. Stoneware,[13] 1/2 x 1/2 in (1.27 x 1.27 cm), 94 pieces/lb, "for rapid dispersion of liquid based systems," or equivalent

5.1.2.3 Disposable beakers, 250 mL capacity, VWR Catalog No. 25384-154 or 400 mL capacity, VWR Catalog No. 25384-156, or equivalent

5.1.2.4 Disposable spatulas, VWR Catalog No. 80081-188, or equivalent

5.1.2.5 Glass jar, 16 oz (500 mL) capacity, VWR Catalog No. 15900-064, for storing paint between casts, or equivalent

5.1.2.6 One-liter Grinding mill jar, U.S. Stoneware,[13] Size 0, or equivalent

5.1.2.7 Unitized ball mill, U.S. Stoneware,[13] Model 764AVM, holds two jars, or equivalent

5.1.3 Equipment and Supplies for Casting Paint Films

5.1.3.1 Coating thickness gauge, Positector 6000 by Defelsko,[14] or equivalent

5.1.3.2 Drawdown machine, Gardco,[15] Item No. DP-1230A, or equivalent

5.1.3.3 Drawdown rod, Gardco,[15] Wire Diameter 0.090 in (#90 [0.229 cm]), Item No. AP-1/2 x 90, or equivalent

5.1.3.4 Permanent marker, such as a Sharpie, or equivalent for labeling jars of paint and paint films

5.1.3.5 Release paper by Leneta, Gardco,[15] Item No. PC-RP-1K, or equivalent

5.1.3.6 Steel plate, needed under paint film to measure thickness, Gardco,[15] Catalog No. PP-02-06x12 (6 x 12 x 0.032 in [15.2 x 30.5 x 0.081 cm]), or equivalent

5.1.3.7 Syringe, Luer-slip plastic disposable, 50 mL capacity, VWR Catalog No. 660640764, for spreading paint onto release paper, or equivalent

5.1.3.8 Tape for securing release paper to drawdown machine

5.1.4 Supplies and Equipment for Preparing Test Kit Diagnostic Materials

5.1.4.1 Supplies

5.1.4.1.1 Fired bricks, either standard size or pavers from local brick company

5.1.4.1.2 Three-inch (7.6-cm) disposable paint rollers for application of primer and overcoats, Shur-Line[16] 3"/75-mm Trim Roller, or equivalent

5.1.4.1.3 Disposable plates (coated paper or plastic) to serve as trays for loading a roller with paint

5.1.4.1.4 Oil-based paint for applying overcoats, Sherwin Williams[17] ProMar 200, or equivalent

5.1.4.1.5 Paint for coating metal to prevent formation of rust, Rust- Oleum[18] Professional Primer (Gray), or equivalent

5.1.4.1.6 Pine or oak boards from local lumberyard

5.1.4.1.7 Plaster for making moulds, USG Hobby Plaster, Plaster of Paris, United States Gypsum Company,[19] Chicago, IL, Plaster.com, or equivalent

5.1.4.1.8 Primer paint for attaching paint films to substrates, BEHR[20] Premium Plus Oil-Base, or equivalent

5.1.4.1.9 Smooth metal rod 1/4 to 3/8 in (0.64 to 0.95 cm) in diameter and about 6 in (15.2 cm) long for smoothing out the paint pieces after their attachment to the substrate. The paint roller handle (without the roller) will serve this purpose as well.

5.1.4.1.10 Steel or iron plating or sheeting from local hardware supplier, typically 1/32 to 1/16 in (0.079 to 0.16 cm) thick

5.1.4.1.11 Tray with flat bottom and sides at least 3/8 in (0.95 cm) high for casting plaster

5.1.4.1.12 Vented oven that can be set to 150 °C ± 5 °C for baking paints on substrates, VWR Catalog No. 16000-212, or equivalent

5.1.4.1.13 Water-based paint for applying overcoats, Glidden[21] Evermore Semi-gloss, or equivalent

5.1.4.2 Equipment

5.1.4.2.1 Hacksaw or metal shears to cut metal substrate pieces

5.1.4.2.2 Hand-held or power saw to cut wood substrate pieces

5.1.4.2.3 Ruler and fine tip marker to draw out dimensions on substrates and paint films

5.1.4.2.4 Straightedge and pizza cutter to cut cast paint film into desired dimensions, or specially made guide for cutting paint film (shown below in Figure 7)

5.1.4.2.5 Table saw or miter saw to cut plaster substrate pieces

5.1.4.2.6 Water-cooled masonry saw to cut masonry substrate pieces

5.2 Cleanup

5.2.1 Acetone, for cleaning glass surface of drawdown machine, VWR Catalog No. BDH 2002-1GLP, or equivalent

5.2.2 Brass bristle brush for cleaning drawdown rods, VWR 101413-026, or equivalent

5.2.3 Two-liter graduated cylinder, VWR Catalog No. 24780-369, or equivalent for soaking drawdown rods

5.2.4 Mineral spirits, Klean-Strip Odorless,[10] or equivalent

5.2.5 Paper towels

5.2.6 Razor blade for scraping dried paint residue from drawdown machine

5.2.7 Scrub brush for cleaning ball mill jar, VWR 17170-005, or equivalent

5.3 Safety Equipment

Safety equipment is required for protection from harm from various paint ingredients. Useful items include the following.

5.3.1 Dust mask, VWR Catalog No. 56219-004, or equivalent

5.3.2 Eyewash bottle, VWR Catalog No. AA39980-KT, or equivalent

5.3.3 Gloves, VWR Catalog No. 40101-348, or equivalent

5.3.4 Laboratory apron, VWR Catalog No. 10845-016, or equivalent

5.3.5 Safety glasses, VWR Catalog No. 89032-944, or equivalent

Note: Paint ingredients must be handled in fume hood to avoid inhaling dust particles and solvent fumes.

6.0 QUALITY CONTROL AND QUALITY ASSURANCE

6.1 Quality control activities to be practiced during the performance of this method include those that follow.

6.1.1 Use only beakers, scoops, etc., dedicated to particular components when weighing paint ingredients.

6.1.2 Use only reagent-grade ingredients for components, except mineral spirits.

6.1.3 Jars and grinding media must be cleaned thoroughly with mineral spirits between each batch of paint.

6.1.4 Discard syringes after use.

6.1.5 Clean and dry drawdown rods thoroughly between each cast.

6.1.6 Use only clean glass jars to store paint between mixing and each cast.

6.2 The quality objectives for the leaded paint films are (1) a variability of less than ±10% in thickness (µm) as measured with an electronic thickness gauge and (2) a variability of less than 10% in Pb concentration (mg/cm^2) as measured by sampling and analysis using EPA Microwave Method 3051A,[22] EPA "Standard Operating Procedure for the Grinding and Extraction of Lead in Paint Using Nitric Acid and a Rotor/Stator System Powered by a High-Speed Motor,"[23] or an equivalent method for extracting Pb from the paint, and measurement by inductively coupled plasma-optical emission spectrometry(ICP-OES).[24]

6.3 The quality objective for the Pb method diagnostic test materials is a variability of less than 10% in Pb concentration (mg/cm^2) as measured by sampling and analysis using EPA Method 3051A,[22] EPA "Standard Operating Procedure for the Grinding and Extraction of Lead in Paint Using Nitric Acid and a Rotor/Stator System Powered by a High-Speed Motor,"[23] or an equivalent method for extracting Pb from the paint, and measurement by ICP-OES.[24]

7.0 PROCEDURE FOR PREPARATION OF STAND-ALONE PAINT FILM

7.1 Selection of Components

The preparation of a paint films starts with selection of the components. The categories will be

- Pb pigment such as white Pb[11] or lead chromate,[9]
- filler such as zinc oxide or titanium dioxide,

- binder such as linseed oil,
- optional—colorants such as red iron oxide, and
- optional—potential measurement interferences such as barium carbonate.

The selection will be based on the intended use of the paint film. For example, one might select lead chromate as the pigment because it is more difficult to dissolve than white Pb and red iron oxide to serve as a potential interference with rhodizonate-based test kits that give a pink color on reaction with Pb.[25, 26] The relative amounts of the components selected are based on literature sources[27, 28] and experience. A typical old paint recipe includes, by weight,

- 37% white Pb,
- 37% zinc oxide,
- 17% linseed oil (half raw, half boiled), and
- 9% mineral/petroleum spirits.

Following this basic recipe, the relative amounts of Pb pigment and zinc oxide can be varied to achieve the desired Pb-in-paint film concentration (i.e., mg Pb/cm^2). A large number of paint films were produced and analyzed, as illustrated in Appendixes 1 and 4. From these results, a relationship between the areal Pb concentration (mg Pb/cm^2) in the final paint film and the percent white Pb in the "wet" mix was determined.[5] This relationship is shown in Figure 2.

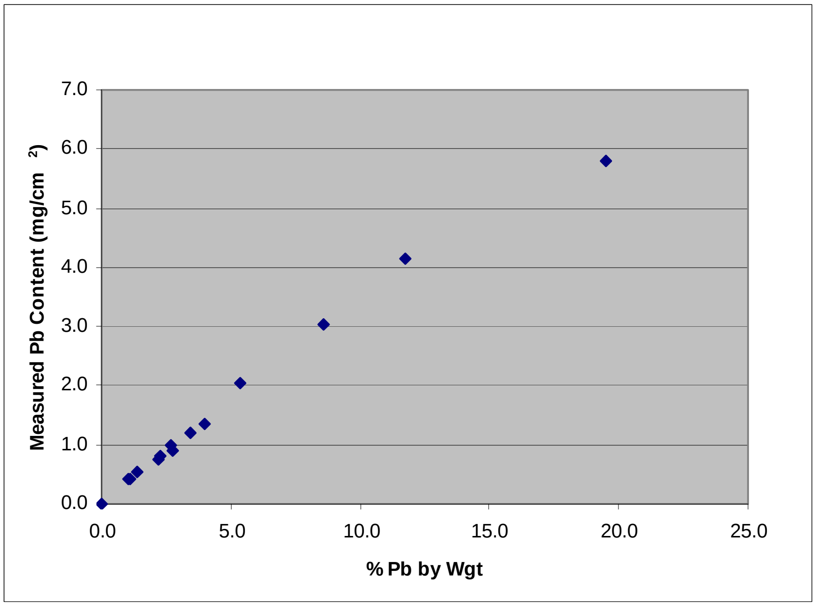

Figure 2. Plot of paint film Pb concentrations (mg Pb/cm^2) versus Pb concentration (% Pb) in "wet" paint mixture (assuming white Pb = 80% Pb by weight).

To calculate, the amount of white Pb needed, first select the Pb-in-paint film concentration (mg/cm^2) desired. From the plot in Figure 2, determine the required percent Pb in the wet mix. The amount of white Pb to be used is then calculated as

White Pb in wet paint mix (g) = (% Pb in wet paint mix/100) x 1.25 x total weight of wet paint mix (g),

where 1.25 is the weight ratio of white Pb to Pb.

Note: Recipes for amounts of each ingredient to use to achieve specific concentrations are listed specifically in Appendix 4 in the batch/recipe table.

7.2 Mixing Paint Ingredients in Ball Mill

The paint ingredients are mixed in the following manner.

7.2.1 Use safety equipment, including dust mask, safety glasses, gloves, and lab coat.

7.2.2 Place the containers of the ingredients (Pb pigment, zinc oxide, raw linseed oil, white linseed oil, and mineral spirits) in a laboratory fume hood.

7.2.3 Fill the ball mill jar approximately half full with grinding media, as shown in Figure 3, and place the ball mill jar with grinding media in the fume hood.

Figure 3. Ball mill jar with grinding media along with principal ingredients of paint.

7.2.4 Place a clean 250-mL beaker on the top loading balance and tare the balance.

7.2.4.1 Carefully use a spatula dedicated to the Pb pigment to place the desired weight of the Pb pigment into the beaker while still on the balance.

7.2.4.2 Very carefully transfer the Pb pigment into the ball mill over the grinding media.

7.2.4.3 Repeat this procedure with the zinc oxide using a clean beaker and spatula dedicated to the zinc oxide.

7.2.4.4 Next, use a clean beaker and weigh out the required amount of raw linseed oil and transfer this material to the ball mill jar.

7.2.4.5 Using another clean beaker, weigh out the required amount of boiled linseed oil and transfer this material to the ball mill jar.

7.2.4.6 Finally, use another clean beaker and weigh out the required amount of mineral spirits and transfer this material to the ball mill jar.

7.2.5 When all ingredients are in the jar, securely lock the lid into place.

7.2.6 Remove the closed jar from the fume hood and place it on its side on the rollers of the ball mill (see Figure 1). Two jars can be placed on the ball mill at the same time, if desired.

7.2.6.1 Cover the ball mill rollers and jars with an approved safety cover.

7.2.6.2 The dial should be set to approximately 69 rpm (dial position 20). Turn on the ball mill using the power switch located on the cord.

7.2.6.3 Allow the jars to turn at this speed, with no interruption, for 96 h.

7.3 Varying Recipes (adding metals and pigments)

7.3.1 Lead chromate or some pigment other than white Pb may be used. If lead chromate is to be added, it is added as a replacement for the white Pb, not in addition to it. It has been determined that the mineral spirits need to be increased by 10 g when preparing a 810g mixture to ensure even flow of the paint during the casting process when using lead chromate. Using lead chromate will lead to a yellow paint film. Several recipes for lead chromate paint films are listed in Appendix 4 in the batch/recipe table.

7.3.2 Chemicals that have the potential for interfering with the measurement may be added (aluminum oxide, barium carbonate, magnesium carbonate). When using these compounds, the weight of zinc oxide used is reduced by an amount equal to the weight of the added chemical. Illustrative data are presented in Appendix 4.

7.3.3 Pigmentation can be achieved by adding materials like red iron oxide (Fe_2O_3), black iron oxide (FeO), red Pb (lead tetraoxide, Pb_3O_4, or $2PbO.PbO_2$), or chrome oxide green (Cr_2O_3) with the white Pb. When using these compounds, the weight of zinc oxide used is reduced by an amount equal to the weight of the added chemical. Illustrative data are presented in Appendix 4.

7.4 Casting Paint into Films

A paint film is cast as follows.

7.4.1 When 96 h have elapsed, turn off the ball mill and remove the protective cover.

7.4.2 Remove the jar from rollers and carefully unlock the lid.

7.4.3 Using caution, pour the paint into a glass jar leaving the grinding media in the ball mill jar. Cap the glass jar. Be sure that this jar is clearly labeled with the batch number and other important information, such as date of preparation and nominal Pb concentration. To help with cleanup, it is recommended to wipe up spills on the side of the jar, etc., immediately. More specifics on cleanup are presented in Section 7.7.

7.4.4 Set up and level a drawdown machine in a fume hood; allow 8 to 12 in (20 to 30 cm) of open space on each side of the machine.

7.4.4.1 Place a sheet of release paper, positioned in “landscape,” on the machine’s flat glass surface and secure with small pieces of tape at the top and bottom, taking care that the paper is as flat as possible.

7.4.4.2 Lock the drawdown bar into place on the appropriate mechanism on the drawdown machine and leave in the “open” position.

7.4.5 Using a syringe, draw up about 12 mL of paint from the glass jar. Spread this paint across the release paper, approximately 1 in (2.5 cm) from the top. Paint will begin to spread slightly on its own.

7.4.5.1 Put drawdown bar into the lowered position so that it touches the top of the paint and steadily draw it down towards the bottom of the machine, spreading the paint evenly down the release paper, as shown in Figure 4. The drawdown should take about 3 s. Do not be concerned if a bit of the paint spills over onto the glass surface of the machine.

7.4.5.2 When reaching the bottom of the paper, pick up drawdown bar and remove it from the machine, and place it in a 2-L graduated cylinder filled with mineral spirits.

7.4.5.3 Do not move the machine or paper at all, as this will create an uneven paint distribution.

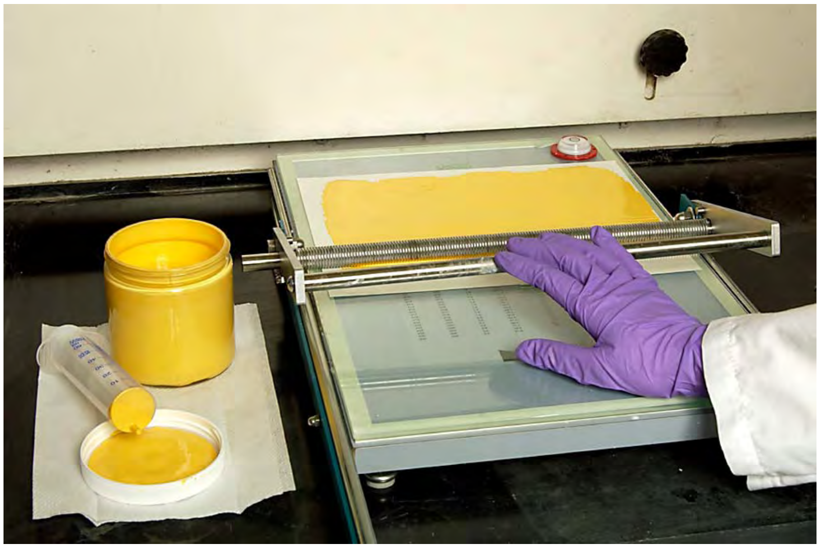

Figure 4. Using the wire-wound drawdown bar to cast a lead chromate paint film on release paper.

7.4.6 Label the paint film by writing with a permanent marker on the unused part of the release paper or by placing a separate sheet of paper beside the drawdown machine, clearly indicating the batch and film number. Do not attempt to write on the paint.

7.4.7 Allow paint to cure for at least 2 days before attempting to remove it from the paper.

7.5 Retrieval of Paint Film from Release Paper

The dry paint film is removed from the release paper as follows.

7.5.1 After a minimum of 48 h, remove the release paper from the machine by pulling up the tape.

7.5.2 Carefully peel the paint film off of the release paper and place it on a clean sheet of release paper, labeled with the batch and film number. If desired, this is an appropriate time to label the paint film itself using a permanent marker

7.5.3 Place the film on the release paper in a folder, label the folder, and store it in a clean, dry place.

7.6 Testing for Variability of Film Thickness

A quality control parameter is the variability in area thickness of the cast paint film measured in micrometers using an electronic thickness meter. To measure the variability of a cast paint film, perform the following steps.

7.6.1 Make a grid on the paint film (squares approximately 3 x 3 cm) using a ruler and an ink pen. Or, if markings on the film are undesirable, use a gridded template.

7.6.2 Place the paint film on top of a steel plate.

7.6.3 Place the thickness gauge straight down, perpendicular to the steel plate, on the film and allow the button on the bottom to be compressed. Do this in each square on the grid (see Figure 5).

7.6.4 The gauge will beep when it takes a measurement. Take three measurements before recording to give the gauge time to come to a final value.

Figure 5. Marked paint film being measured for thickness.

7.6.5 Measurements are displayed in micrometers and should be recorded appropriately in a laboratory notebook.

7.7 Cleaning

It is extremely important to thoroughly clean jars, grinding media, drawdown bars and machines, etc., between each batch and each film casting to ensure accuracy in film preparation.

7.7.1 Ball Mill Jar and Grinding Media

After emptying the paint into a glass jar and setting it aside, the grinding media will still be in the ball mill jar. To clean the jar and media, do the following.

7.7.1.1 Fill the ball mill jar with mineral spirits until it just covers the grinding media, which will be approximately half full.

7.7.1.2 Secure the lid on the jar and roll on the ball mill for 15 min, being sure to use the safety cover.

7.7.1.3 After 15 min, pour the mineral spirits and paint residue into a disposable, 400-mL beaker and discard this waste into an appropriate flammable materials waste container.

7.7.1.4 Again, fill the jar half-way with mineral spirits and roll on the ball mill for another 15 min.

7.7.1.5 Pour the mineral spirits and paint residue into the disposable beaker and discard this waste into an appropriate flammable materials waste container.

7.7.1.6 By either pouring or using a gloved hand, remove the grinding media from the ball mill jar and place it in a quart container, cover the media with mineral spirits, and cap the container. Before reusing, remove and allow to dry.

7.7.1.7 Fill the ball mill jar with soapy water and scrub the inside with a scrub brush. The lid of the jar should also be cleaned using soapy water. Discard the wash water down the drain.

7.7.1.8 After washing, the jar and lid should be allowed to air dry before the next use.

7.7.2 Drawdown Rod and Drawdown Machine

7.7.2.1 Immediately after drawing down paint with the drawdown rod, place the rod into a 2-L graduated cylinder that contains mineral spirits.

7.7.2.1.1 Allow it to soak for approximately 10 min.

7.7.2.1.2 Working in the hood, remove the rod from the mineral spirits and use a brass brush to scrub between the wire wound around the rod. Do this over paper towels to capture drips.

7.7.2.1.3 After scrubbing with the brush, use gloved hands to wash the rod with soapy water to clean off the mineral spirits.

7.7.2.1.4 Place the rod on paper towels and allow it to air dry.

7.7.2.2 After removing the release paper holding the film, use a razor blade to scrape off any dried paint that is on the glass surface of the drawdown machine.

7.7.2.2.1 Using a paper towel dipped in acetone, wipe the glass surface to remove any remaining paint residue. Do this until surface appears completely clear. If any tape is left on the surface, remove this as well. Be sure that the glass surface is clean and dry before placing another piece of release paper on it.

8.0 PROCEDURE FOR PREPARATION OF LEAD METHOD DIAGNOSTIC TEST MATERIALS

8.1 Selection of Components

The components of the Pb method diagnostic test materials include a substrate, one or more leaded paint films, and one or more overcoats of oil-based and/or water-based, nonleaded paint. The leaded paints may vary in composition as described in Section 7.1.

8.1.1 Selection of Substrates

Four common substrates found in old dwellings are (1) wood, (2) metal, (3) masonry, and (4) plaster. Figure 6 shows examples of each of these types of substrates.

Figure 6. Photograph of metal, wood, plaster, and masonry substrate materials ready for preparation of diagnostic test materials.

8.1.1.1 Wood substrates for the method diagnostic materials can be prepared by cutting typical dwelling wood types like pine and oak into the desired dimensions.

8.1.1.2 Typically, the metal plate or sheeting found in old houses will be made of iron.

8.1.1.2.1 Iron plate or sheeting can be acquired from various sources, such as hardware supply stores.

8.1.1.2.2 The metal plate can be cut to the desired dimensions using a hacksaw or metal cutting shears.

8.1.1.2.3 It has been found that the steel can rust if it is not stainless. To prevent this, paint both sides of the metal with a light coat of anti-rust coating such as Rust-Oleum Professional Primer.[18]

8.1.1.3 Masonry substrate is made from bricks. The brick is cut into the desired dimensions using a water-cooled masonry saw equipped with a diamond-tipped saw blade.

8.1.1.4 The plaster is a greater challenge. To make the plaster, purchase the type of plaster mix made for making castings.

8.1.1.4.1 Mix up the plaster with water according to the instructions on the container.

8.1.1.4.2 Pour the wet plaster into a tray with a flat bottom and sides that are at least 3/8 in (0.95 cm) high.

8.1.1.4.3 Quickly shake the tray to smooth out the surface of the plaster and/or smooth it with a small trowel because it begins setting quickly.

8.1.1.4.4 Allow the plaster to dry for 24 h and then gently remove the slab of plaster from the tray.

8.1.1.4.5 Using a table saw or skill saw, cut the plaster slab into pieces of the desired dimensions.

8.1.2 Selection of Leaded Paint Films

The leaded paint films are chosen to challenge the Pb testing method at various levels of complexity. Variables are the desired type of Pb pigment, the concentration of the Pb, the absence or presence of colorants, and the absence or presence of potentially interfering agents, such as certain trace metals.

8.2 Cutting Paint Films into Desired Dimensions

8.2.1 Once the type of leaded paint film is chosen, the films cast as described above are cut to the desired dimensions. This may be done using a ruler and a fine-tipped marking pen to first draw out the dimensions of the desired paint pieces across a cast film. The paint may be cut with a pizza cutter (or a scalpel or hobby knife), using a straightedge as a guide.

8.2.2 If large numbers of films are to be cut, a guide system may be built. One such system is shown in Figure 7. It is composed of two pieces of plastic constructed with slots. One piece has the slots separated by the desired length of the film piece and the other has the slots separated by the desired width of the film piece.

8.2.2.1 The film is laid on a cutting board and one of the pieces is laid over the film. A pizza cutter is run through the slots.

8.2.2.2 Then, the second piece of plastic is laid down, with the slots perpendicular to the slots of the first cut. Again, the pizza cutter is run through the slots.

8.2.2.3 The final result is several pieces of paint cut to the desired size. If the paint pieces are 5 x 7 cm, then about nine pieces of paint are cut from one cast paint film.

8.3 Assembly of the Test Pieces

A test piece consists of the substrate with one or more layers of leaded paint film and overcoats. The assembly is as follows.

8.3.1 Place the wood, metal, or masonry on a flat surface. [Plaster is discussed in Section 8.3.2.]

8.3.1.1 Pour a small amount of well-mixed primer onto a disposable plate and then use the roller to apply a thin layer of oil-based paint primer on the substrate.

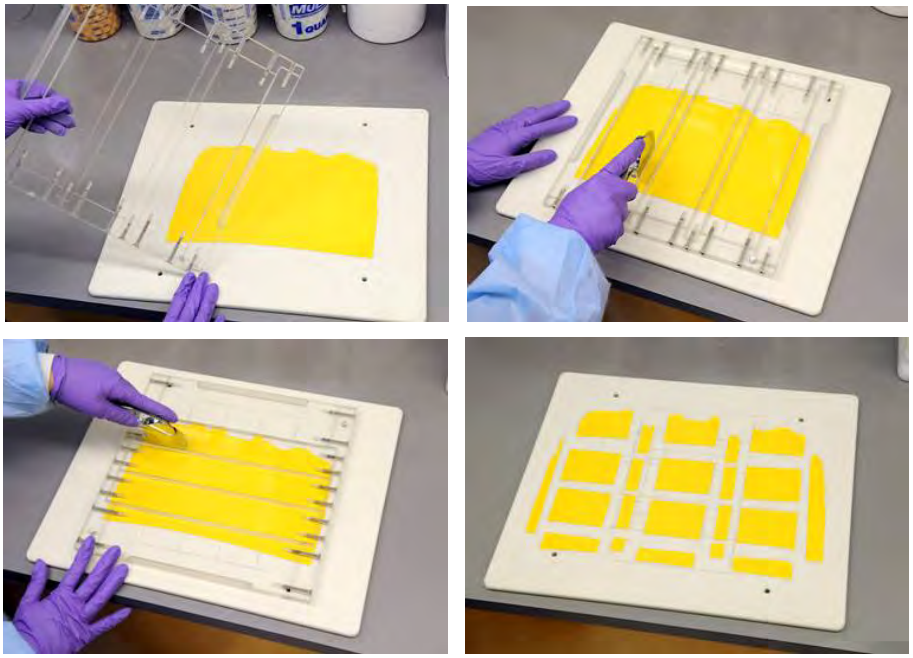

Figure 7. Paint film cutting apparatus and process: (1) Paint film is laid on cutting board; (2) the first template is placed in guide holes to make vertical cuts, leaving 2-cm-wide strips for film analysis; (3) the second template is placed in guide holes to make horizontal cuts; (4) the process leaves six to nine, 5 x 7-cm film pieces for preparation of coupons and 2-cm pieces for analysis.

8.3.1.2 Carefully lay a piece of the leaded paint on the coated substrate, taking care to center the paint piece as shown in Figure 8.

Figure 8. Placing leaded paint film on wood substrate coated with primer to serve as adhesive.

8.3.1.3 Using a small metal rod, very carefully and gently roll/push it across the paint piece to push the paint piece into the partially dry primer and smooth out any wrinkles as shown in Figure 9.

8.3.1.4 After drying for 24 h, perform any additional smoothing needed.

Figure 9. Smoothing out paint film after allowing time for the primer to partially dry.

8.3.1.5 Allow the primer to dry for a total of 2 days.

8.3.2 In the case of plaster substrate, the first layer of primer will soak quickly into the plaster and not be a suitable adhesive for the paint piece. In this case, apply a coating of primer on the plaster and allow it to dry for 2 days. Then apply another coating of the primer for attachment of the paint piece.

8.3.2.1 If a second leaded paint film is to be attached, coat the paint piece on the substrate with a thin layer of primer and carefully place the second paint film over the first paint film.

8.3.2.2 Using a small metal rod, very carefully and gently roll/push it across the paint piece to push the paint piece into the partially dry primer and smooth out any wrinkles.

8.3.2.3 After drying for 24 h, perform any additional smoothing needed.

8.3.2.4 Allow the primer to dry for a total of 2 days.

8.3.3 If overcoating the paint piece with oil-based paint, pour a small amount of well-mixed, nonleaded, oil-based paint onto a disposable plate and use a roller to overcoat the paint film as shown in Figure 10. Make every effort to make a smooth and uniform coating.

8.3.3.1 Allow the oil-based coating to dry for 2 days.

8.3.3.2 Repeat the previous two steps for each coating of oil-based paint desired.

8.3.3.3 If overcoating the paint piece or the oil-based overcoats with water-based paint, pour a small amount of well-mixed, nonleaded, water-based paint onto a disposable plate and use a roller to overcoat the previously applied paint film. Make every effort to make a smooth and uniform coating.

8.3.3.4 Allow the water-based coating to dry for 1 day.

8.3.3.5 Repeat the previous two steps for each coating of water-based paint desired.

8.3.4 With the test piece fully assembled, place the piece in a vented oven for 24 h at 40 °C and then 48 h at 150 ± 5 °C to harden the paint as an approximation of the hardening that occurs with paints over many years of aging.

8.3.5 The plaster substrate may crack when samples are taken for analysis. To minimize the potential for cracking and breaking, a thin piece of wood is glued to the back of the piece after baking.

Figure 10. Applying overcoat.

Figure 11 shows completed test pieces or coupons and bare substrates.

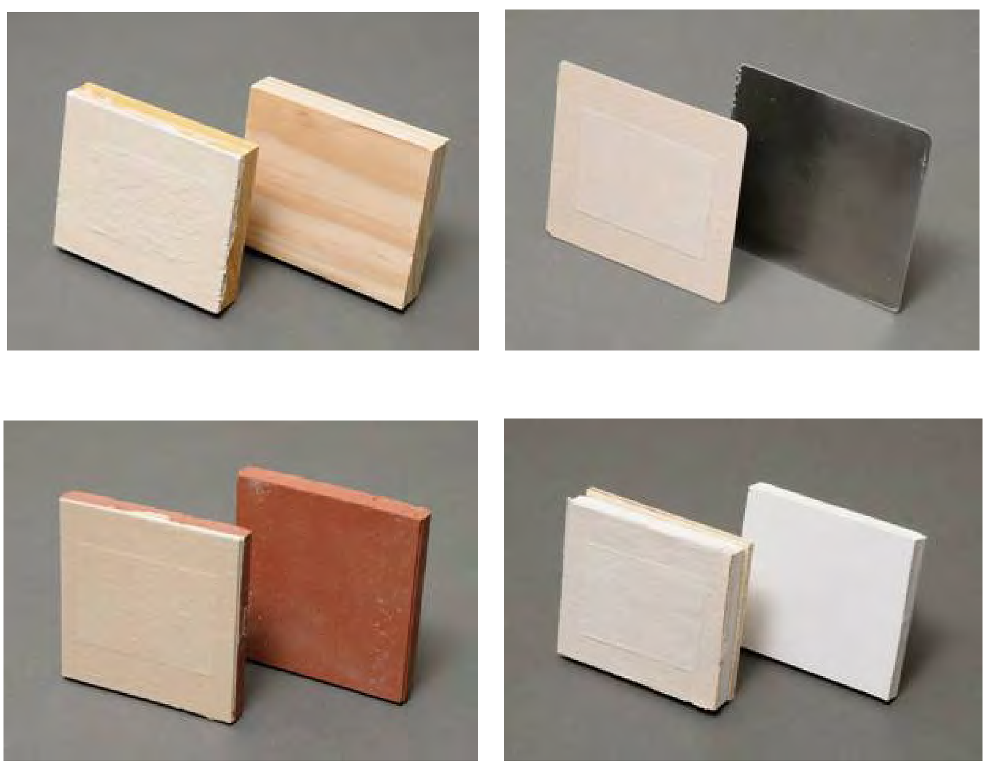

Figure 11. Photographs of overcoated leaded paint films on wood, metal, masonry, and plaster substrate pieces, along with bare substrate pieces. Note backing board on finished plaster piece to minimize potential for breaking.

8.4 Example of Preparation of Test Piece

In one EPA project, 31 different combinations of substrate, type of leaded paint films, and type and number of overcoats were prepared.[5] The following describes the preparation of one of the more complex combinations.

8.4.1 Place a 10 x 8 x 2-cm piece of pine wood on a flat surface.

8.4.1.1 Using a small roller, coat the top of the piece with a thin layer of white primer.

8.4.1.2 Carefully lay down a 7 x 5-cm piece of paint film prepared with lead chromate at 0.4 mg Pb/cm^2.

8.4.1.3 Smooth the paint film out as needed with a smooth metal rod (repeat after 24 h).

8.4.1.4 Allow this combination to dry for 2 days.

8.4.2 Using a small roller, coat the paint film and the primed wood with a thin layer of white primer.

8.4.2.1 Carefully lay down a 7 x 5-cm piece of paint film prepared with white Pb at 0.8 mg Pb/cm^2 and doped with aluminum oxide, barium carbonate, and magnesium carbonate (as potential measurement interferences), and black iron oxide (as a colorant).

8.4.2.2 Smooth the paint film out as needed with a smooth metal rod (repeat after 24 h).

8.4.2.3 Allow this combination to dry for 2 days.

8.4.3 Using a small roller, coat this combination with a uniform layer of oil-based white paint.

8.4.3.1 Allow this combination to dry for 2 days.

8.4.3.2 Using a small roller, coat this combination with a second, uniform layer of oil-based white paint.

8.4.3.3 Allow this combination to dry for 2 days.

8.4.4 Using a small roller, coat this combination with a uniform layer of water-based white paint.

8.4.4.1 Allow this combination to dry for 1 day.

8.4.4.2 Using a small roller, coat this combination with a second, uniform layer of water-based white paint.

8.4.4.3 Allow this combination to dry for 1 day.

8.4.4.4 Using a small roller, coat this combination with a third, uniform layer of water-based white paint.

8.4.4.5 Allow this combination to dry for 1 day.

8.4.4.6 Using a small roller, coat this combination with a fourth, uniform layer of water-based white paint.

8.4.4.7 Allow this combination to dry for 1 day.

8.4.5 Bake this piece for one day at 40 °C in a vented oven to remove volatiles.

8.4.5.1 Bake this piece for 2 days at 150 ± 5 °C in a vented oven to harden the paint such that it better represents old, hard paints found in old dwellings.

This combination presents numerous challenges, including

- lead chromate, which is a fairly insoluble form of Pb;
- two layers of leaded paint;

- the presence of potential measurement interferences, Al, Ba, and Mg plus Fe from the colorant;
- the second layer of leaded paint being black, which has the potential for interfering with visualization of a Pb-caused color change; and
- multiple layers of oil-based and water-based over the leaded-paint layers, which add the challenge of relatively thick, multilayered paint.

9.0 DATA PROCESSING

9.1 Variability in Paint Film Thickness

The variability in paint film thickness is measured using an electronic thickness gauge. The average and standard deviation are calculated using either software such as Excel or an advanced calculator. The variability or uncertainty in the value of thickness (µm) is not to exceed ±10%.

9.2 Variability in Lead Concentration in Prepared Paint Films

The average value and the variability in the Pb concentration are determined for each film based on the analysis of aliquots taken from that film. The average and standard deviation are calculated using either software such as Excel or an advanced calculator. The variability or uncertainty in the value of Pb concentration (mg/cm^2) thickness is not to exceed ±10%.

9.3 Variability in Lead Concentration in Method Diagnostic Test Materials

The average value and the variability in the Pb concentration are determined for each film based on the analysis of aliquots taken from the method diagnostic test materials. The average and standard deviation are calculated using either software such as Excel or an advanced calculator. The variability or uncertainty in the value of Pb concentration (mg/cm^2) should not exceed ±10%.

10.0 DATA AND RECORDS MANAGEMENT

Keeping complete records will help ensure accuracy in making paint films. Activities to be performed include those that follow.

10.1 Maintain all records in a bound notebook or on a form prepared specifically for recording information pertinent to this SOP. The forms shall be maintained in a binder.

10.2 Each paint batch and each paint film are to be given an identifying name or number that is recorded.

10.3 Each block or piece of method diagnostic test material is to be given an identifying name or number that is recorded.

11.0 WASTE MANAGEMENT

There are several forms of waste generated during the operation of this SOP. Each is dealt with as follows.

11.1 Mineral Spirits

Leftover mineral spirits used for cleaning should be contained in labeled, flammable waste containers until they can be disposed of by hazardous waste personnel.

11.2 Leaded Paint

Any excess Pb-containing paint that is not cast into a film should be kept in labeled, closed containers until it can be disposed of by hazardous waste personnel. Any unused pieces of leaded paint film should be kept in labeled, closed containers until they can be disposed of by hazardous waste personnel.

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APPENDIX 1

Results of Analysis of Multiple Samples Taken from Cast Paint Films Needed To Prepare All Planned Types of Coupons

Batch	Nominal Pb Conc.[a] (mg/cm^2) and Film Description	No.[b] Films Prepared	Color	Overall Thickness[c] (µm)	Pb Conc. (mg/cm^2)	Pb Conc. by wt (%)	n =
19	Blank	8	White	101.9 ± 9.5 (9.4%)	Blank	Blank	8
39	0.4 White Pb	11	White	107.2 ± 7.3 (6.8%)	0.391 ± 0.027 (6.97%)	1.21 ± 0.02 (1.3%)	12
35	0.8 White Pb	24	White	102.5 ± 6.2 (6.1%)	0.808 ± 0.051 (6.25%)	2.56 ± 0.06 (2.3%)	12
41	1.0 White Pb	8	White	100.7 ± 5.1 (5.1%)	1.02 ± 0.05 (4.7%)	3.07 ± 0.03 (0.9%)	8
29	1.2 White Pb	6	White	100.9 ± 4.0 (3.9%)	1.31 ± 0.06 (4.4%)	4.07 ± 0.05 (1.1%)	8
45	2.0 White Pb	6	White	100.8 ± 7.3 (7.3%)	1.98 ± 0.12 (6.2%)	6.16 ± 0.11 (1.7%)	8
46	0.4 Lead Chromate	16	White	99.7 ± 6.7 (6.8%)	0.378 ± 0.028 (7.42%)	1.24 ± 0.03 (2.7%)	20
37	0.8 Lead Chromate	19	White	98.6 ± 6.1 (6.2%)	0.763 ± 0.056 (7.39%)	2.41 ± 0.02 (0.8%)	12
49	0.4 White Pb with Al, Ba, Mg	15	White	103.2 ± 6.0 (5.8%)	0.358 ± 0.027 (7.64%)	1.21 ± 0.02 (1.4%)	12
50	0.8 White Pb with Al, Ba, Mg	11	White	102.4 ± 6.8 (6.6%)	0.784 ± 0.096 (12.3%)	2.60 ± 0.05 (1.8%)	12
53	0.4 White Pb with Black Iron Oxide	8	Black	88.0 ± 6.0 (6.8%)	0.406 ± 0.050 (12.25%)	1.20 ± 0.02 (1.3%)	8
54	0.8 White Pb with Black Iron Oxide	8	Black	89.5 ± 6.9 (7.7%)	0.765 ± 0.045 (5.82%)	2.55 ± 0.04 (1.5%)	8
55	0.4 White Pb with Red Iron Oxide	6	Red	101.5 ± 6.2 (6.1%)	0.364 ± 0.012 (3.39%)	1.24 ± 0.02 (1.4%)	8
56	0.8 White Pb with Red Iron Oxide	8	Red	99.2 ± 6.5 (6.6%)	0.734 ± 0.026 (3.53%)	2.45 ± 0.04 (1.5%)	8
51	0.4 White Pb with Al, Ba, Mg, Black Iron Oxide	18	Black	90.2 ± 6.9 (7.7%)	0.358 ± 0.033 (9.35%)	1.19 ± 0.01 (0.6%)	12
52	0.8 White Pb with Al, Ba, Mg, Black Iron Oxide	12	Black	88.5 ± 6.4 (7.3%)	0.747 ± 0.046 (6.18%)	2.66 ± 0.04 (1.3%)	12

[a]Conc. = concentration
[b]No. = number
[c]Thickness tested on all films.

APPENDIX 2

Summary of Results of Analysis of Each Type of Coupon Prepared

Type[a]	No. Coupons Made	n =	Average Pb (mg/cm^2)	Average Pb by wt (%)
A	18	6	Blank	Blank
B	18	6	0.341 ± 0.014 (4.11%)	0.359 ± 0.023 (6.41%)
C	18	6	0.759 ± 0.017 (2.24%)	1.018 ± 0.063 (6.19%)
D	18	6	0.955 ± 0.050 (5.24%)	1.141 ± 0.078 (6.84%)
E	18	6	1.167 ± 0.069 (5.91%)	1.369 ± 0.140 (10.2%)
F	18	6	1.917 ± 0.136 (7.09%)	1.973 ± 0.080 (4.05%)
G	18	6	0.744 ± 0.051 (6.85%)	1.021 ± 0.100 (9.78%)
H	18	6	1.196 ± 0.040 (3.34%)	0.980 ± 0.059 (6.02%)
I	18	6	0.741 ± 0.034 (4.59%)	0.851 ± 0.053 (6.23%)
J	18	6	1.115 ± 0.079 (7.09%)	0.946 ± 0.113 (12.0%)
K	18	12	0.716 ± 0.067 (9.36%)	1.555 ± 0.101 (6.50%)
L	18	6	1.147 ± 0.052 (4.53%)	1.162 ± 0.048 (4.13%)
M	18	6	0.701 ± 0.034 (4.85%)	1.463 ± 0.065 (4.41%)
N	18	6	1.164 ± 0.115 (9.88%)	1.355 ± 0.027 (1.99%)
O	18	7	0.821 ± 0.114 (13.9%)	0.421 ± 0.093 (22.1%)
P	18	7	1.322 ± 0.056 (4.24%)	0.660 ± 0.098 (14.9%)
Q	18	6	0.658 ± 0.038 (5.78%)	0.617 ± 0.088 (14.3%)
R	18	6	1.143 ± 0.085 (7.44%)	0.858 ± 0.123 (14.3%)
S	18	6	0.675 ± 0.031 (4.59%)	0.908 ± 0.143 (15.7%)
T	18	6	1.08 ± 0.07 (6.54%)	0.829 ± 0.069 (8.32%)
U	18	6	0.699 ± 0.044 (6.29%)	0.898 ± 0.059 (6.57%)
V	18	6	1.134 ± 0.098 (8.64%)	0.853 ± 0.054 (6.33%)
W	16	8	0.734 ± 0.045(6.13%)	0.494 ± 0.062 (12.6%)
X	16	6	1.078 ± 0.061 (5.66%)	0.646 ± 0.066 (10.22%)
Y	18	6	1.061 ± 0.084 (7.92%)	0.666 ± 0.063 (9.46%)
Z	16	5	0.717 ± 0.072 (10.0%)	0.523 ± 0.033 (6.31%)
AA	16	6	1.062 ± 0.090 (8.47%)	0.825 ± 0.080 (9.70%)
AB	18	6	1.018 ± 0.044 (4.32%)	0.776 ± 0.023 (2.96%)
AC	16	6	0.696 ± 0.064 (9.20%)	0.387 ± 0.054 (14.0%)
AD	16	6	1.03 ± 0.10 (9.71%)	0.644 ± 0.050 (7.76%)
AE	18	6	0.945 ± 0.112 (11.9%)	0.641 ± 0.073 (11.4%)

[a]See pages A-3 and A-4 for details of types of coupons (Summary of ORD 2008 Lead-in-Paint Diagnostic Paint Materials[5]).

Summary of ORD 2008 Lead-in-Paint Diagnostic Paint Materials[5]

Type	Diagnosis	Substrate	No. Films and Pb Compound[a]	White Overlayers No. and Type[b]	Chemical Interferences Al, Ba, Mg[c]	Chemical Interference Fe[c,d]	Pb Film Colors[d]	Areal Pb[e] (mg/cm^2)
A	Sensitivity, accuracy, and precision	Wood	1 non-Pb	1O 2L			White	<0.0004
B	Sensitivity, accuracy, and precision	Wood	1w	1O 2L			White	0.341 ± 0.014 (4.11%)
C	Sensitivity, accuracy, and precision	Wood	1w	1O 2L			White	0.759 ± 0.017 (2.24%)
D	Sensitivity, accuracy, and precision	Wood	1w	1O 2L			White	0.955 ± 0.050 (5.24%)
E	Sensitivity, accuracy, and precision	Wood	1w	1O 2L			White	1.167 ± 0.069 (5.91%)
F	Sensitivity, accuracy, and precision	Wood	1w	1O 2L			White	1.917 ± 0.136 (7.09%)
G	Accuracy and precision (A & P) with form of Pb	Wood	1c	1O 2L			Yellow	0.744 ± 0.051 (6.85%)
H	A & P with form of Pb	Wood	2c	2O 2L			Yellow	1.196 ± 0.040 (3.34%)
I	A & P with multiple layers	Wood	1w	2O 4L			White	0.741 ± 0.034 (4.59%)
J	A & P with multiple layers	Wood	2w	3O 4L			White	1.115 ± 0.079 (7.09%)
K	A & P with substrate effects	Steel	1w	1O 2L			White	0.716 ± 0.067 (9.36%)
L	A & P with substrate effects	Steel	2w	2O 2L			White	1.147 ± 0.052 (4.53%)
M	A & P with substrate effects	Masonry	1w	1O 2L			White	0.701 ± 0.034 (4.85%)
N	A & P with substrate effects	Masonry	2w	2O 2L			White	1.164 ± 0.115 (9.88%)
O	A & P with substrate effects	Plaster	1w	1O 2L			White	0.821 ± 0.114 (13.89%)
P	A & P with substrate effects	Plaster	2w	2O 2L			White	1.322 ± 0.056 (4.24%)
Q	A & P with chemical interferences	Wood	1w	1O 2L	Y		White	0.658 ± 0.038 (5.78%)
R	A & P with chemical interferences	Wood	2w	2O 2L	Y		White	1.143 ± 0.085 (7.44%)
S	A & P with color interferences	Wood	1w	1O 2L		Y	Red	0.675 ± 0.031 (4.59%)
T	A & P with color interferences	Wood	2w	2O 2L		Y	Red	1.08 ± 0.07 (6.54%)
U	A & P with color interferences	Wood	1w	1O 2L		Y	Black	0.699 ± 0.044 (6.29%)
V	A & P with color interferences	Wood	2w	2O 2L		Y	Black	1.134 ± 0.098 (8.64%)

Summary of ORD 2008 Lead-in-Paint Diagnostic Paint Materials[5] (cont'd.)

Type	Diagnosis	Substrate	No. Films and Pb Compound[a]	White Overlayers No. and Type[b]	Chemical Interferences Al, Ba, Mg[c]	Chemical Interference Fe[c,d]	Pb Film Colors[d]	Areal Pb[e] (mg/cm^2)
W	A & P with all potential interferences	Steel	1c 1w	3O 4L	Y	Y	Yellow, Black	0.734 ± 0.045 (6.13%)
X	A & P with all potential interferences	Steel	1c 1w	3O 4L	Y	Y	Yellow, Black	1.078 ± 0.061 (5.66%)
Y	A & P with all potential interferences	Steel	1c 1w	3O 4L	Y	Y	Yellow, Black	1.061 ± 0.084 (7.92%)
Z	A & P with all potential interferences	Masonry	1c 1w	3O 4L	Y	Y	Yellow, Black	0.717 ± 0.072 (10.04%)
AA	A & P with all potential interferences	Masonry	1c 1w	3O 4L	Y	Y	Yellow, Black	1.062 ± 0.090 (8.47%)
AB	A & P with all potential interferences	Masonry	1c 1w	3O 4L	Y	Y	Yellow, Black	1.018 ± 0.044 (4.32%)
AC	A & P with all potential interferences	Plaster	1c 1w	3O 4L	Y	Y	Yellow, Black	0.696 ± 0.064 (9.20%)
AD	A & P with all potential interferences	Plaster	1c 1w	3O 4L	Y	Y	Yellow, Black	1.03 ± 0.10 (9.71%)
AE	A & P with all potential interferences	Plaster	1c 1w	3O 4L	Y	Y	Yellow, Black	0.945 ± 0.112 (11.9%)

[a]Pb compounds: 1w = 1 white Pb film, 2w = 2 white Pb films; 1c = 1 lead chromate film, 2c = lead chromate films
[b]Overlayers: 1O = 1 oil-based paint overcoat; 2L = 2 water-base paint overcoats
[c]Chemical interferences: Elements Al, Ba, Mg added to paint film; red or black iron oxide added for color.
[d]Colors and sources: Red iron oxide added to paint film for red color; black iron oxide added to paint film for black color; yellow from lead chromate.
[e]Mean ± standard deviation for n=6, except K (n=12), Z (n=5), and O and P (n=7). Paint extracted using EPA 3051a and analyzed by ICP-OES.

APPENDIX 3

2008 EPA/ORD SPECIFICATION SHEET

Lead-in-Paint Diagnostic Test Material: Type AA

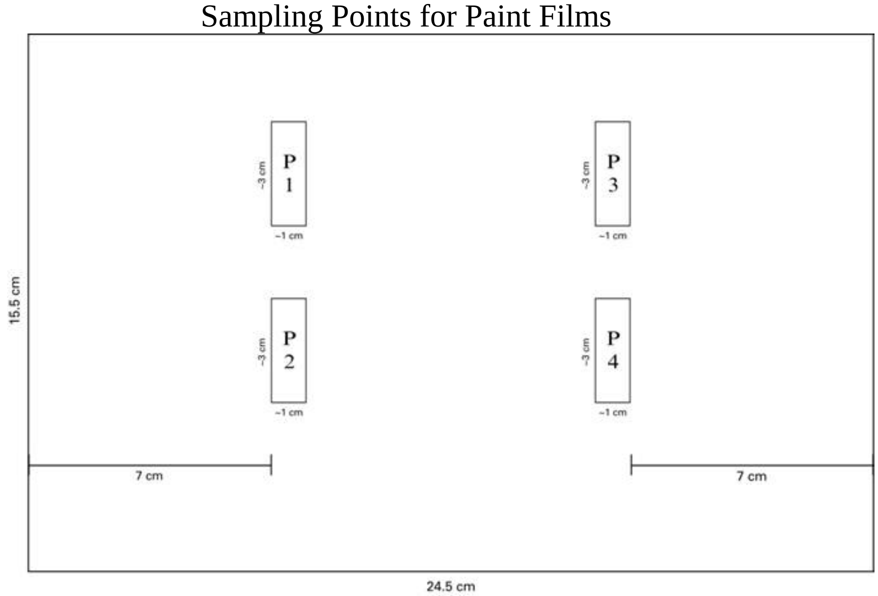
- Substrate Masonry
- Source of Material — Date of Preparation:

  Synthetic- 10-2-08

  Layers-
  Primer Paint
  Lead Chromate Paint Film (0.4 mg/cm^2)
  Oil-based Paint
  White Lead Paint Film with Al, Ba, Mg and Black Iron Oxide (0.8 mg/cm^2)
  2 Oil-based Paint Overcoats
  4 Water-based Paint Overcoats
- Target Concentration 1.2 mg/cm^2
- Verification Analysis

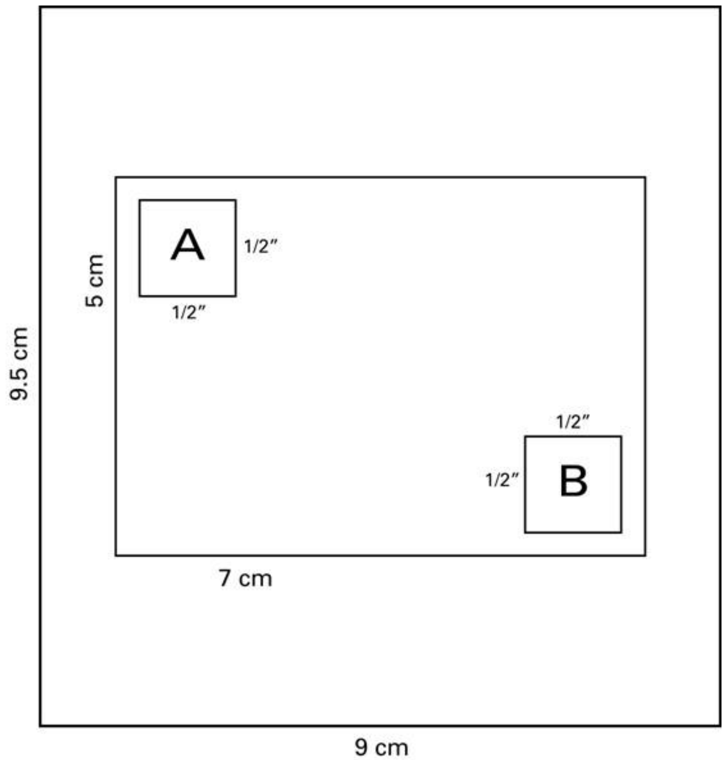
	Procedure	Results (mean ± 1 Std. Dev.) n=12	
		Pb (mg/cm^2)	Pb (%)
Preliminary-	Paint Film. Four aliquots (each 2.98cm^2) from each of 3 paint films digested by EPA Method 3051 and analyzed by ICP-AES for Pb.	1.12±0.04	3.90±0.03

Sampling Points for Paint Films



Final-	Paint Film on Masonry Substrate.	Pb (mg/cm^2)	Pb (%)
	Two aliquots paint,	n=6	
	each 1.61 cm^2, from each of 3	1.06±0.09	0.825±0.080
	masonry blocks collected with		
	½"square chisel, digested by EPA Method 3051		
	and analyzed by ICP-AES for Pb.		

Sampling Points for Masonry Substrate-based Coupon



Masonry Substrate: 9 cm x 9.5 cm
Paint Film: 5 cm x 7 cm

- References:

1. Binstock, D.A., D.L. Hardison, P.M. Grohse, and W.F. Gutknecht. Standard Operating Procedure for Lead in Paint by Hotplate- or Microwave-based Acid Digestion and Atomic Absorption or Inductively Coupled Plasma Emission Spectrometry, EPA 600/8-91/213. U.S. EPA RTP, NC 1991.

2. U.S. EPA SW-846 Method 3051. Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils. 1994.

Table of Multielement Values[a]
(Mean ± Standard Deviation)

Type AA

Element	µg/g	mg/cm²
Al	4590 ± 330	0.593 ± 0.043
Ba	1350 ± 23	0.174 ± 0.003
Be	<2	<0.0003
Ca	3.66 ± 0.07%	4.73 ± 0.09
Cd	2.09 ± 0.07	0.00027 ± 0.000009
Cr	1480 ± 19	0.191 ± 0.002
Cu	<4	<0.0005
Fe	1.42 ± 0.04%	1.83 ± 0.05
Mg	4500 ± 203	0.581 ± 0.026
Mn	116 ± 1	0.015 ± 0.00009
Mo	<2	<0.0003
Ni	<8	<0.001
Pb	8070 ± 260	1.04 ± 0.03
Sr	28.8 ± 0.1	0.004 ± 0.00002
V	3580 ± 68	0.462 ± 0.009
Zn	19.8 ± 0.8%	25.6 ± 1.0

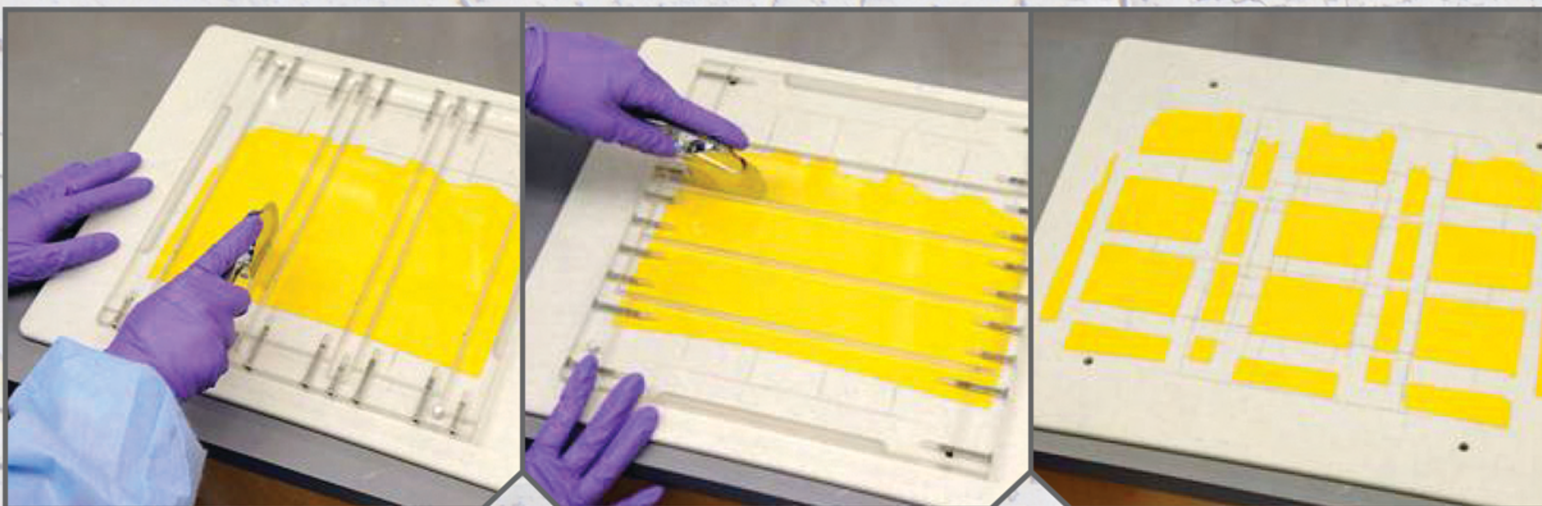
[a]n = two aliquots paint, each 1.61 cm², from two different blocks, collected with 1/2-in (1.27 cm) square chisel, digested by EPA Method 3051, and analyzed by ICP-OES.

APPENDIX 4

Summary of Recipes Used To Make Paint Films for Preparation of 31 Types of Diagnostic Test Kit Materials

Batch	Nominal Pb Conc. (mg/cm^2) and Film Description	Total wt (g)	Ingredient Weights (g)										
			White Pb	Lead Chromate	ZnO	Boiled Linseed Oil	Raw Linseed Oil	Mineral Spirits	Al_2O_3	$BaCO_3$	$MgCO_3$	Black Iron Oxide	Red Iron Oxide
19	Blank	810	0	—	600	70	70	70	—	—	—	—	—
39	0.4 White Pb	810	10.5	—	589.5	70	70	70	—	—	—	—	—
35	0.8 White Pb	810	22	—	578	70	70	70	—	—	—	—	—
41	1.0 White Pb	810	27	—	573	70	70	70	—	—	—	—	—
29	1.2 White Pb	810	35	—	565	70	70	70	—	—	—	—	—
45	2.0 White Pb	810	54	—	546	70	70	70	—	—	—	—	—
46	0.4 Lead Chromate	820	—	14	586	70	70	80	—	—	—	—	—
37	0.8 Lead Chromate	820	—	26	574	70	70	80	—	—	—	—	—
49	0.4 White Pb with Al, Ba, Mg	810	10.5	—	431	70	70	70	65.2	37.6	55.8	—	—
50	0.8 White Pb with Al, Ba, Mg	810	22	—	419.4	70	70	70	65.2	37.6	55.8	—	—
53	0.4 White Pb with Black Iron Oxide	810	10.5	—	524.5	70	70	70	—	—	—	65	—
54	0.8 White Pb with Black Iron Oxide	810	22	—	513	70	70	70	—	—	—	65	—
55	0.4 White Pb with Red Iron Oxide	810	10.5	—	468	70	70	70	—	—	—	—	121.5
56	0.8 White Pb with Red Iron Oxide	810	22	—	456.5	70	70	70	—	—	—	—	121.5
51	0.4 White Pb with Al, Ba, Mg, Black Iron Oxide	810	10.5	—	365.9	70	70	70	65.2	37.6	55.8	65	—
52	0.8 White Pb with Al, Ba, Mg, Black Iron Oxide	810	22	—	354.4	70	70	70	65.2	37.6	55.8	65	—

Method: Ball mill 69 rpm, 1-L jar, roll for 96 h.



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