



## Standard Operating Procedure for the Turbidimetric Determination of Lead in Paint Extracts

# SCIENCE





## **Standard Operating Procedure for the Turbidimetric Determination of Lead in Paint Extracts**

Prepared by

William Studabaker, Kristen Sorrell, Michelle McCombs,  
Cynthia Salmons, and William Gutknecht

Environmental and Industrial Sciences Division  
RTI International  
Research Triangle Park, NC 27709

### **Disclaimer**

The information in this document has been funded wholly or in part by the U.S. Environmental Protection Agency (EPA) under EPA Contract No. EP-D-05-065 to Alion Science and Technology, Inc., and RTI Subcontract No. SUB1174861RB. It has been subjected to the Agency's peer and administrative review. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

## **Acknowledgments**

This document was prepared under the direction of the Work Assignment Contracting Officer's Representative, Ms. Sharon L. Harper, National Environmental Research Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC.

Special acknowledgment is given to Dr. Hunter Daughtrey, Alion Science and Technology, Inc., for his support of this effort and careful review of this document.



## Table of Contents

|             |   |          |
|-------------|---|----------|
|             | List of Appendixes.....                                   | vi       |
| <b>1.0</b>  | <b>PRINCIPLE AND APPLICABILITY .....</b>                  | <b>1</b> |
| <b>2.0</b>  | <b>SUMMARY OF METHOD .....</b>                            | <b>1</b> |
| 2.1         | Sample Preparation and Turbidimetric Measurement .....    | 1        |
| 2.2         | Method Performance.....                                   | 2        |
| <b>3.0</b>  | <b>DEFINITIONS, ACRONYMS, AND ABBREVIATIONS .....</b>     | <b>3</b> |
| <b>4.0</b>  | <b>HEALTH AND SAFETY.....</b>                             | <b>3</b> |
| <b>5.0</b>  | <b>EQUIPMENT, SUPPLIES, AND REAGENTS .....</b>            | <b>4</b> |
| 5.1         | Test Samples .....  | 4        |
| 5.2         | Apparatus .....   | 4        |
| 5.3         | Materials and Supplies .....                              | 4        |
| 5.4         | Reagents .....  | 5        |
| 5.4.1       | List of Reagents .....                                    | 5        |
| 5.4.2       | Preparation of Reagents and Standards.....                | 5        |
| <b>6.0</b>  | <b>SAMPLE COLLECTION, PRESERVATION, AND STORAGE .....</b> | <b>6</b> |
| <b>7.0</b>  | <b>QUALITY CONTROL.....</b>                               | <b>6</b> |
| 7.1         | Reagent Blank.....  | 6        |
| 7.2         | Calibration Check .....                                   | 6        |
| 7.3         | Duplicates.....   | 6        |
| <b>8.0</b>  | <b>INSTRUMENT CALIBRATION AND STANDARDIZATION .....</b>   | <b>7</b> |
| <b>9.0</b>  | <b>PROCEDURE .....</b>                                    | <b>7</b> |
| 9.1         | Turbidimeter Performance Check .....                      | 7        |
| 9.2         | Sample Analysis.....                                      | 7        |
| 9.3         | Calculations.....   | 8        |
| <b>10.0</b> | <b>DATA AND RECORDS MANAGEMENT .....</b>                  | <b>8</b> |
| <b>11.0</b> | <b>WASTE MANAGEMENT .....</b>                             | <b>9</b> |
| <b>12.0</b> | <b>REFERENCES.....</b>                                    | <b>9</b> |

## **List of Appendixes**

|             |   |     |
|-------------|---|-----|
| Appendix 1. | Results of Rotor/Stator-Turbidimetric Analysis of Six NIST RM 8680 Samples, Five ELPAT Samples and NIST SRM 2581 .....                          | A-1 |
| Appendix 2. | Results for Rotor/Stator-Turbidimetric Method on Lead-in-Paint Diagnostic Materials ...   | A-2 |
|             | Summary of ORD 2008 Lead-in-Paint Diagnostic Paint Materials .....  | A-3 |
| Appendix 3. | Results of Comparison of Real-World Paint Samples Analyzed for Lead Using the Rotor/Stator-Turbidimetric Method and Method 3051a/ ICP-OES ..... | A-5 |

## 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.<sup>1</sup> 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),<sup>2</sup> as equal to or greater than 0.5% Pb by weight or 1.0 mg Pb/cm<sup>2</sup>. 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)<sup>3</sup> 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 Federal regulated level.<sup>3</sup> 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 the 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.<sup>4</sup> 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.

In response to this need for solubilization, a new method has been developed that simultaneously grinds a paint sample and quantitatively extracts the Pb.<sup>4</sup> This procedure is presented in the Standard Operating Procedure (SOP), "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."<sup>5</sup>

As noted above, a variety of methods are available for measurement of Pb once it is in solution. To satisfy the goals of the RRP rule, the method needs to be accurate, quick, inexpensive, and relatively easy to perform. In response to this need, research has been performed in an effort to develop a system wherein the amount of Pb equal to 1 mg Pb/cm<sup>2</sup> would be totally bound up (effectively 100%) by complexation or precipitation, and, then, any small amount of Pb over this amount would be detected by a color-forming reaction. However, no system could be identified that would quantitatively react with an amount of Pb equal to exactly 1 mg/cm<sup>2</sup>; an excess of the complexing or precipitating agent was found necessary to react with the Pb equal to 1 mg Pb/cm<sup>2</sup>. During the course of this effort, however, it was determined that the turbidity associated with a precipitation reaction could be used to determine the concentration of the Pb. Subsequently, a procedure has been developed based on the turbidimetric measurement of the product of the dissolved Pb and potassium molybdate.<sup>6</sup>

This method has been applied successfully in the laboratory with a large number of real-world paint samples removed from various substrates and is expected to perform as well in the field. It is a general use SOP and intended to be used by trained nontechnical workers.

## 2.0 SUMMARY OF METHOD

### 2.1 Sample Preparation and Turbidimetric Measurement

This procedure assumes that established procedures are used for sample collection<sup>7</sup> and extraction.<sup>5</sup> Paint sample extracts are to be prepared in 25% (v/v) nitric acid and free of particulate matter. If necessary, samples are filtered. A reagent solution is prepared in a turbidimeter vial using 1M ammonium acetate and solid potassium molybdate.<sup>6</sup> An aliquot of the sample extract is added to this

reagent solution and the resultant mixture is allowed to react for 5 min. The turbidity of this mixture is then read on a portable turbidimeter and the result is converted to a Pb concentration (in mg Pb/cm<sup>2</sup>) using a standard curve generated by the user.

## 2.2 Method Performance

A series of tests was carried out to determine the performance characteristics of the turbidimetric method, and the results of these tests follow.

- **Range.** Using the method, the linear calibration curve range is 0.03 to 0.9 mg/mL Pb<sup>++</sup> in paint extracts. For a paint sample removed from 1 cm<sup>2</sup> of surface and dissolved in 3 mL of 25% (v/v) nitric acid (2 mL extraction + 1 mL diluent), this corresponds to 0.09 to 2.7 mg/cm<sup>2</sup> Pb on the original surface.
- **Sensitivity.** The Hach turbidimeter<sup>8</sup> used for this method provides a readout from 1 to 1000 NTU (nephelometric turbidity units). For a typical calibration, this corresponds to a sensitivity of 0.0009 mg Pb<sup>++</sup>/mL extract per NTU, or 0.0027 mg Pb/cm<sup>2</sup> per NTU.
- **Minimum Detection Limit (MDL).** The MDL for Pb in paint extracts is based on testing of laboratory blanks and is estimated at 0.001 mg/mL. This is equivalent to 0.003 mg/cm<sup>2</sup> in the original paint sample. It is calculated as the mean (in NTU) of 10 to 20 replicates  $\pm$  3 standard deviations, multiplied by the sensitivity.
- **Interferences.** The following cations were tested at a concentration of 10 mg/mL in 25% (v/v) nitric acid: Ba<sup>+2</sup>, Zn<sup>+2</sup>, Fe<sup>+3</sup>, Cu<sup>+2</sup>, Co<sup>+2</sup>, Mg<sup>+2</sup>, Ca<sup>+2</sup>. None gave a signal above background (1 NTU).
- **Precision and Bias.** In a laboratory evaluation of the method, extracts of 14 samples collected from six different substrates, with concentrations near the Federal regulated level of 1 mg/cm<sup>2</sup>, were analyzed using the subject method and reference method Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES). The accuracy (as mean difference from value determined by ICP-OES) was -2.8%. The precision of 14 standards at the Federal regulated level (1.67 mM Pb<sup>++</sup>) tested at the same time was 1.4%.

The method was evaluated using a series of paint reference materials, synthetic diagnostic paint materials, and real-world paints from the RTI repository of paint for the Environmental Lead Proficiency Analytical Testing (ELPAT) program.<sup>9</sup> Included were six samples of certified National Institute of Standards and Technology (NIST) Reference Material (RM) 8680 Paint on Fiberboard<sup>10</sup> (actually collected by RTI for EPA/NIST in the 1990s). There was no statistical difference at the 95% confidence level between the results for the NIST RM 8680 samples analyzed using the new rotor/stator grinding and extraction procedures with turbidity measurement and samples prepared by EPA nitric acid/microwave digestion Method 3051a<sup>11</sup> with ICP-OES measurement, as shown in Appendix 1. These values also did not differ statistically from the expected values for these reference materials. As also noted in Appendix 1, there was no difference between the results with the ELPAT samples analyzed using the new rotor/stator grinding and extraction procedure with turbidity measurement and the expected values determined by multilaboratory consensus.

To provide diagnostic performance information on materials of known composition, 31 different synthetic paint diagnostic materials were developed and used that incorporated different Pb compounds, multiple layers of leaded and unleaded paint, various substrates, and chemical and color interferences. The resulting values for paint samples of different weights, shown in Appendix 2, were compared to the final concentrations listed in the 2008 EPA Office of Research and Development (ORD) Specification Sheets for the Lead-in-Paint Diagnostic Test Materials<sup>12</sup> that were generated from EPA Method 3051a extraction and ICP-OES quantitation. The rotor/stator-turbidity data either showed no statistical difference or agreed to better than  $\pm$ 20% for all but one diagnostic material (type H). The details for the paint materials are shown in the summary table in Appendix 2.

Finally, the method was further evaluated with a series of real-world paints collected from various sources. As shown in Appendix 3, except for Hospital Sample "Window 2," the methods results are statistically equivalent. The high uncertainties for these results reflect the real-world variation in Pb

concentration from point to point within any source, such as a painted door or wall. Other studies<sup>13,14</sup> have reported similar variabilities.

### 3.0 DEFINITIONS, ACRONYMS, AND ABBREVIATIONS

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

- DI – deionized water
- ELPAT – Environmental Lead Proficiency Analytical Testing Program
- EPA – U.S. Environmental Protection Agency
- ICP-OES – inductively coupled plasma-optical emission spectrometry
- ID – identification [number]
- LBP – lead-based paint
- MDL – minimum detection limit
- NIST – National Institute of Standards and Technology
- NTU – nephelometric turbidity units
- ORD – Office of Research and Development
- Pb – elemental or ionic lead
- RTI – RTI International
- RM – NIST Reference Material
- RRP – Lead; Renovation, Repair, and Painting Program; Final Rule
- R/S-T – rotor/stator-turbidity
- SOP – standard operating procedure
- SRM – NIST Standard Reference Material
- v/v – volume-to-volume ratio

### 4.0 HEALTH AND SAFETY

The part of this procedure requiring the greatest care is working with nitric acid ( $\text{HNO}_3$ ). Nitric acid is a strong, corrosive, oxidizing agent that requires protection of the eyes, skin, and clothing. The diluted acid (25%, v/v) used with the method is less harmful than concentrated nitric acid, but still requires full protection, especially of the eyes. Items to be worn during use of this reagent include

- safety goggles (or safety glasses with side shields),
- acid-resistant rubber gloves, and
- a protective garment, such as a laboratory apron. Nitric acid spilled on clothing will destroy the fabric and result in a hole; contact with the skin underneath will result in a chemical burn.

It is also essential that an eye wash bottle be available during performance of this method. This is a bottle with a spout that covers the eye. If acid or any other corrosive gets into the eye, the water in this bottle is squirted onto the eye to wash out the harmful material. Eye washing should be performed immediately after exposure with the eye wash bottle or with large amounts of water from another source if available. Medical help should be sought immediately after washing. If nitric acid is spilled onto the skin, wash immediately with large amounts of water. Medical attention is not required unless the burn appears to be

significant. Even after washing and drying, the nitric acid may leave the skin slightly brown in color. This will heal and fade with time.

## **5.0 EQUIPMENT, SUPPLIES, AND REAGENTS**

### **5.1 Test Samples**

Each test sample shall consist of paint extract, prepared by collection of paint from nominally 1 cm<sup>2</sup> of painted surface, followed by extraction with 2.0 mL of 25% (v/v) nitric acid, according to the 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."<sup>5</sup>

### **5.2 Apparatus**

**5.2.1** Analytical balance, minimum 100-g capacity, with accuracy to 0.1 mg

**5.2.2** Dry box or glove bag

**5.2.3** Hach 2100P turbidimeter,<sup>15</sup> or equivalent (provided with reference standards and reusable sample cells)

**5.2.4** Laboratory timer, VWR 33501-418, or equivalent

**5.2.5** Stirring motor, VWR 12620-974, or equivalent

**5.2.6** Stirring bar, VWR 58947-142, or equivalent

### **5.3 Materials and Supplies**

**5.3.1** AutoVial 5, 0.45-μm PTFE, Whatman AV115NPUORG, or equivalent

**5.3.2** Blank labels or labeling tape

**5.3.3** Two bottles, 1 L, Wheaton W216853, or equivalent

**5.3.4** Five bottles, 1 oz, Wheaton W216848, or equivalent

**5.3.5** Caps, black phenolic for turbidimeter vials, 22 to 400 size, VWR 16199-602, or equivalent

**5.3.6** Desiccant packets, VWR 89024-382, or equivalent

**5.3.7** Disposable pipette, 5 mL, MarketLab ML9086, or equivalent

**5.3.8** Eye wash bottle, VWR 45000-803, or equivalent

**5.3.9** Graduated cylinder, 1 L, VWR 89000-276, or equivalent

**5.3.10** Graduated pipet, 5 mL, Kimble 37025-5, or equivalent

**5.3.11** Laboratory marker

**5.3.12** Nalgene wide-mouth jar, 250 mL, for storage of vials of dry molybdate, VWR36319-627, or equivalent

**5.3.13** One wide-mouth, 5-L, polyethylene carboy for acid waste and rinse water waste, VWR 80094-464, or equivalent

**5.3.14** Paper towels

**5.3.15** Parafilm, VWR 82024-546, or equivalent

**5.3.16** Pipette, Eppendorf style, 1000 μL, with disposable tips, or equivalent

**5.3.17** Repipetter dispenser, 20 mL capacity to attach to 1-L bottle, VWR 53527-744, or equivalent

**5.3.18** Sample cell (turbidimeter vial), 1" round glass, 10 mL, Hach 2434706, or equivalent

**5.3.19** Vials, screw cap, ~8 mL, Wheaton 225144, or equivalent

**5.3.20** Vials, shell, with cap, 1 dram, Kimble 60965D 1, or equivalent

**5.3.21** 6 Volumetric flasks, 25 mL, VWR 89090-818, or equivalent

**5.3.22** 1 Volumetric flask, 100 mL, VWR 89090-606, or equivalent

## 5.4 Reagents

### 5.4.1 List of Reagents

**5.4.1.1** Ammonium acetate, crystalline, Mallinckrodt 3272-04, or equivalent

**5.4.1.2** Lead ICP-OES standard solution, 10,000 µg/mL, Aldrich 35,6336, or equivalent

**5.4.1.3** Lead nitrate, Baker 2322-04, or equivalent

**5.4.1.4** Nitric acid, 50% (v/v), VWR 3335-1, or equivalent

**5.4.1.5** Potassium molybdate, Alfa Aesar 22898, or equivalent

**5.4.1.6** Water, deionized (DI) or distilled

### 5.4.2 Preparation of Reagents and Standards

**5.4.2.1 25% (v/v) Nitric Acid.** Add 500 mL DI water to a 1-L graduated cylinder, then slowly fill to the 1-L mark with 50% (v/v) nitric acid. Add a stirring bar, cover the cylinder with parafilm, and stir for 30 min. Transfer the solution to a suitable storage bottle and label.

**5.4.2.2 1 M Ammonium Acetate.** Weigh 77.1 g (1 mol) ammonium acetate into a 600-mL beaker. Add approximately 300 mL DI water and stir until the acetate is dissolved. Transfer to a 1-L graduated cylinder, rinse the beaker with DI water, and add the rinse to the graduated cylinder. Fill to the 1-L mark with DI water, add a stirbar, cover the cylinder with parafilm, and stir for 30 min. Transfer the solution to a suitable storage bottle and label.

**5.4.2.3 Lead Nitrate Primary Standard.** Weigh 0.676 g (2.04 mmol) dry, reagent-grade lead nitrate using a weighing boat or paper. Transfer to a 100-mL volumetric flask and fill part way with 25% (v/v) nitric acid. Rinse the weighing boat or paper into the flask with 5 to 10 mL 25% (v/v) nitric acid, and swirl until all the solids have dissolved. Fill to the mark with 25% (v/v) nitric acid, stopper, and mix by inverting three times. Primary standard contains 4.23 g/L Pb.

**5.4.2.4 Lead Nitrate Calibration Solutions.** Label 5, 25-mL volumetric flasks as 0, 0.5, 1.0, 1.5, and 2.0 mg/cm<sup>2</sup>. Add 0, 1.25, 2.5, 3.75, and 5 mL of the primary standard solution, respectively, to the labeled flasks. Fill each flask to the mark with 25% (v/v) nitric acid, stopper, and mix by inverting 3 times. Transfer each solution to a labeled 1-oz bottle.

Example calculation:

$$\frac{4.23 \text{ g/L Pb} \times 2.5 \text{ mL}}{25 \text{ mL}} = 0.423 \text{ g/L Pb}$$

$$0.423 \text{ g/L Pb} \times \frac{0.003 \text{ L extract}}{1.27 \text{ cm}^2 \text{ paint}} = 0.00099 \text{ g/cm}^2 \text{ Pb.}$$

**5.4.2.5 Lead Nitrate Calibration Check Sample.** Label a 25-mL flask as "Check Sample." Add 1.06 mL of Pb ICP-OES standard solution (Section 5.4.1.2). Fill to the mark with 25% (v/v) nitric acid.

*Note:* This check sample is equivalent to an areal, Pb-in-paint concentration of 1 mg Pb/cm<sup>2</sup>, assuming the paint sample is taken with a 1/2-in (1.27-cm<sup>2</sup>) drill according to EPA “Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit with a Variable-Speed Portable Electric Drill or Using a Wood Chisel with or without a Heat Gun.”<sup>7</sup> This value is determined as follows:

$$(1.06 \text{ mL}) \times (10 \text{ mg/mL}) = 10.6 \text{ mg}$$

$$(10.6 \text{ mg}) / (25 \text{ mL}) = 0.424 \text{ mg/mL}$$

$$(0.424 \text{ mg/mL}) \times (3.00 \text{ mL [volume of solution used as the calibration check sample; see Section 9.2.1]}) = 1.27 \text{ mg.}$$

See Section 9.3.2 for adjustment of calculation if not collecting 1.27 cm<sup>2</sup>.

**5.4.2.6 Potassium Molybdate.** Inside a dry box or a glove bag that has been purged thoroughly with dry air or nitrogen, weigh 0.357 g ± 0.010 g potassium molybdate into a shell vial for each sample and calibration check solution to be tested and cap the vial immediately.

*Note:* Potassium molybdate is water-sensitive and exposure to moisture can affect test performance. The reagent bottle should be opened only in a dry environment.

*Note:* It can be helpful to prepare a large number of reagent vials at one time. Vials should be stored in a dessicator containing fresh Dri-Rite or desiccant packets.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

This SOP assumes the paint samples are collected according to EPA “Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit with a Variable-Speed Portable Electric Drill or Using a Wood Chisel with or without a Heat Gun”<sup>7</sup> and are ground and extracted according to the 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.”<sup>5</sup>

## 7.0 QUALITY CONTROL

### 7.1 Reagent Blank

A sample of 25% (v/v) nitric acid is to be analyzed every 20 samples or each day that the analysis is performed, whichever is more frequent. A result above the limit of detection indicates contamination of the equipment. The source of the contamination needs to be identified and corrected.

### 7.2 Calibration Check

An aliquot of the calibration check sample is to be analyzed prior to beginning analysis on each day that samples are to be analyzed, then every 20 samples or on completion of daily sample analysis, whichever is more frequent. An incorrect result (calculated concentration differs from actual by more than 5%) may indicate contamination, instrument problem, or deterioration of reagent, calibration solutions, or check solution. Corrective action may include servicing or recalibration of the turbidimeter (see instrument manual), preparation of fresh calibration and/or check solutions, or replacement of the reagents.

### 7.3 Duplicates

Duplicate aliquots of paint extract should be analyzed every 20 samples. The sample selected for testing should have a Pb concentration of at least 0.07 mg/mL (0.16 mg/cm<sup>2</sup>). The difference between the two analysis results should be no more than 10% of the average concentration of the two. If the difference is greater, corrective action is to be taken including review of all original data and calculations and possible analysis of a second duplicate sample.

## 8.0 INSTRUMENT CALIBRATION AND STANDARDIZATION

The 2100P turbidimeter (or equivalent) shall be maintained, calibrated, and operated as described in the turbidimeter manufacturer's manual, and checked according to the procedure described below (Sections 9.1 and 9.2).

## 9.0 PROCEDURE

### 9.1 Turbidimeter Performance Check

**9.1.1** Before beginning the procedure, the user should be thoroughly familiar with the operation of the Hach 2100P turbidimeter, or equivalent.

**9.1.2** Turn on the turbidimeter.

**9.1.3** Invert the 100-NTU standard, which comes with the instrument, gently 5 to 10 times until the contents are uniformly dispersed. Do not shake, as this will create bubbles that interfere with the sample measurement.

**9.1.4** Measure the turbidity of the 100-NTU standard. The reported value should fall between 97 and 103 NTU. If the value falls outside that range, consult the operating manual for the 2100P turbidimeter.

### 9.2 Sample Analysis

**9.2.1** Add 1 mL of 25% (v/v) nitric acid to the sample prepared according to 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"<sup>5</sup> (Section 5.1). Cap the sample tube and shake the mixture vigorously for about 10 s.

**9.2.2** Using a disposable pipette, transfer unfiltered sample extract to the barrel of a Whatman AutoVial filter syringe.

**9.2.3** Holding the barrel over a waste container, carefully insert the plunger into the open end of the barrel, and expel a few drops of the sample into the waste container.

**9.2.4** Hold a screw-cap vial under the barrel, and filter the sample by slowly depressing the plunger. Cap the vial and set it aside.

**9.2.5** Measure 15 mL ammonium nitrate into a clean, dry sample cell. *Note:* Bottle-top dispensers (repipets) provide acceptable precision for use in this step.

**9.2.6** Add dry potassium molybdate to the sample cell. Cap and shake vigorously for 15 s.

**9.2.7** Remove the caps from the filtered extract vial and the sample cell. Using an Eppendorf pipette, add 0.75 mL of the filtered sample to the sample cell reagent. Start timer, then immediately cap the sample cell and shake it vigorously for 5 s.

**9.2.8** Turn on the 2100P turbidimeter.

**9.2.9** After the sample has incubated for exactly 5 min, measure the turbidity and record the result.

**9.2.10** Pour the contents of the vial into the waste container. Rinse the vial three times using distilled water. Place the vial upside-down on a paper towel to dry.

**9.2.11** If the value is above the highest point on the standard curve, the sample will need to be diluted and the analysis repeated.

**9.2.12** If the turbidimeter returns a value of “E,” the sample turbidity is too high, and the sample needs to be diluted and the analysis repeated. The turbidimeter will need to be powered off and then turned back on to clear the memory.

### 9.3 Calculations

As noted above, this SOP assumes that the paint samples are collected according to the EPA “Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit with a Variable-Speed Portable Electric Drill or Using a Wood Chisel with or without a Heat Gun,”<sup>7</sup> which describes sample collection using a 1/2-in (1.27-cm) drill bit with subsequent collection of a paint sample 1.27 cm<sup>2</sup> in area. It also assumes that the paint samples are ground and extracted according to the 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.”<sup>5</sup>

**9.3.1** The calibration curve includes five points, centered on the Pb concentration corresponding to the Federal regulated level of 1 mg/cm<sup>2</sup>. As noted, at 1 mg Pb/cm<sup>2</sup>, the 1.27cm<sup>2</sup> of paint collected will contain 1.27 mg of Pb. The five calibration solutions equate to 0, 0.5, 1.0, 1.5, and 2.0 mg/cm<sup>2</sup>.

**9.3.2** Calculate the slope and intercept for the linear regression of the concentration (in mg Pb/cm<sup>2</sup>) (y axis) versus turbidity (x axis) of the calibration solutions, using Excel, SigmaPlot, JMP, or other suitable software. Use the values of 0, 0.5, 1.0, 1.5, and 2.0 mg/cm<sup>2</sup> for the calibration solution concentrations.

*Note:* If the area of the paint sample collected is not equal to 1.27 cm<sup>2</sup>, then calculate the effective area concentration as

$$(0, 0.5, 1.0, 1.5, \text{ or } 2.0 \text{ mg/cm}^2) \times (1.27 \text{ cm}^2)/(\text{actual sample area [cm}^2]),$$

and calculate the slope and intercept for the linear regression of the recalculated effective concentration (in mg/cm<sup>2</sup>) (y axis) versus turbidity (x axis) of the calibration solutions.

**9.3.3** For each unknown sample, calculate the Pb concentration as

$$\text{Pb (mg/cm}^2\text{)} = \text{Turbidity (NTU)} * \text{slope} + \text{intercept.}$$

**9.3.4** For samples where the turbidity is higher than the upper end of the calibration curve, valid results may be obtained by serial dilution of the sample in 25% (v/v) nitric acid and adjusting the calculation for the dilution.

## 10.0 DATA AND RECORDS MANAGEMENT

Keeping accurate and complete records will help assure that the final results of the testing can be used to make decisions about risk and the need for Pb-in-paint treatment. Activities to be performed include those that follow.

**10.1** Maintain all records in a bound notebook or on a form (maintained in a binder) prepared specifically for recording information pertinent to this SOP.

**10.2** Paint extracts will have been assigned identification (ID) numbers or codes as required for the related SOP, “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.”<sup>5</sup> The IDs shall be maintained throughout this procedure. Turbidimeter vials should be labeled in the way that permits sample identification without interference with sample measurement.

**10.3** The time and date of performance of this procedure shall be recorded in the notebook or on the form.

**10.4** The name of the analyst shall be recorded in the notebook or on the form.

## 11.0 WASTE MANAGEMENT

**11.1** Unused nitric acid, leftover paint extracts, solutions containing nitric acid, and used analytical samples shall be collected in a 5-L, wide-mouth, polyethylene carboy that is labeled with a description of the contents. When the carboy is about three-fourths full, it should be delivered to a commercial firm that specializes in removal of hazardous waste.

**11.2** Unused ammonium acetate solution may be disposed of by washing down the sink.

## 12.0 REFERENCES

- (1) Jacobs, D.E., R.P. Clickner, J.Y. Zhou, S.M. Viet, D.A. Marker, J.W. Rodgers, D.C. Zeldin, P. Broene, and W. Friedman. 2002. The prevalence of lead-based paint hazards in U.S. housing. *Environmental Health Perspectives*, 110(10):A599-A606.
- (2) Title X of the Housing and Community Development Act, Residential Lead-Based Paint Hazard Reduction Program Act of 1992. Public Law 102-550.
- (3) USEPA. 2008. Lead; Renovation, Repair, and Painting Program; Final Rule. *Federal Register*. April 22. (pp.21691-21769). U.S. EPA, 40 CFR Part 745, EPA-HQ-2005-0049.
- (4) Gutknecht, W.F., S.L. Harper, W. Winstead, K. Sorrell, D.A. Binstock, C.A. Salmons, C. Haas, M. McCombs, W. Studabaker, C. Wall, and C. Moore. 2009. Rapid new methods for paint collection and lead extraction. *Journal of Environmental Monitoring*, 11, 166-173.
- (5) USEPA. 2009a. 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. U.S. Environmental Protection Agency: Research Triangle Park, NC.
- (6) Kaza, S.R., and V.G. Vaidya. 1976. Determination of lead by the precipitation of lead molybdate from a homogeneous solution by the diffusion method. *Proceedings of the Indian Academy of Science*, 84A(2), 52-56.
- (7) USEPA. 2009b. Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit with a Variable-Speed Portable Electric Drill or Using a Wood Chisel with or without a Heat Gun. U.S. Environmental Protection Agency: Research Triangle Park, NC.
- (8) Hach Company, Loveland, CO. [www.hach.com](http://www.hach.com). Accessed 6/22/09.
- (9) ELPAT. 2007. Web site, Environmental Lead Proficiency Analytical Testing (ELPAT) Program, [www.aiha.org/Content/LQAP/PT/ELPAT.htm](http://www.aiha.org/Content/LQAP/PT/ELPAT.htm). American Industrial Hygiene Association: Fairfax, VA. Accessed 09/21/07.
- (10) NIST. 1997. Certificate of Analysis. Research Material 8680 Lead-Based Paint on Fiberboard. National Institute of Standards and Technology: Gaithersburg, MD.
- (11) USEPA. 2007. EPA Method 3051A, Revision 1, Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils. Washington, D.C: U.S. Environmental Protection Agency, Web site: <http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/3051a.pdf>. Accessed 01/12/09.
- (12) Gutknecht, W.F., D.A. Binstock, K. Sorrell, C.M. Haas, M. McCombs, C.A. Salmons, W. Winstead, W. Studabaker, C. Moore, and C.V. Wall. 2008. "Lead Paint Test Kit Development, Refinements, and Test Materials." National Exposure Research Laboratory, National U.S. Environmental Protection Agency: Research Triangle Park, NC 27711.
- (13) Hodson, L.L., Hardison, E.D., Gutknecht, W. F., Binstock, D. A., Messner, M. J., and Leinbach, A. A. 1997. An Intercomparison of Grinding Techniques Used for the Preparation of Lead-in-Paint Samples, EPA 600/R-95/112, U.S. Environmental Protection Agency: Research Triangle Park, NC. Available from NTIS (NTIS #PB98131881).
- (14) Harper, S.L., and W.F. Gutknecht. 2001. Sources of variability in collection and preparation of paint and lead-coating samples. *Journal of Environmental Monitoring*, 3, 3335-340.

(15) NIST. 1997. Certificate of Analysis. Standard Reference Materials 2581, Powdered Paint, Nominal 0.5% Lead. National Institute of Standards and Technology: Gaithersburg, MD.

## APPENDIX 1

### Results of Rotor/Stator-Turbidimetric Analysis of Six NIST RM 8680<sup>10</sup> Samples, Five ELPAT<sup>9</sup> Samples, and NIST Standard Reference Material 2581<sup>15</sup>

| Sample ID    | Rotor/Stator-Turbidity Results | Method 3051a <sup>11</sup> /ICP-OES Results <sup>11</sup> | Expected Pb Value     | Turbidity Pb Recovery Based on Expected Value |
|--------------|--------------------------------|---|-----------------------|---|
| NIST RM 8680 | (mg/cm <sup>2</sup> )          | (mg/cm <sup>2</sup> )                                     | (mg/cm <sup>2</sup> ) | (%)   |
| KB2          | 1.10 ± 0.04                    | 1.21 ± 0.12   | 1.25 ± 0.35           | 88.0  |
| TD5          | 1.43 ± 0.10                    | 1.48 ± 0.05   | 1.21 ± 0.38           | 118   |
| DG2          | 1.07 ± 0.06                    | 1.13 ± 0.07   | 1.14 ± 0.32           | 93.8  |
| HA3          | 1.21 ± 0.39                    | 1.29 ± 0.36   | 1.31 ± 0.34           | 92.4  |
| MD2          | 0.98 ± 0.09                    | 1.09 ± 0.09   | 1.10 ± 0.30           | 89.1  |
| JH1          | 1.56 ± 0.08                    | 1.57 ± 0.09   | 1.29 ± 0.40           | 121   |
| ELPAT        | (%)                            | (%)   | (%)                   | (%)   |
| ELPAT 51P1   | 2.52                           | —   | 2.22 ± 0.13           | 113   |
| ELPAT 51P2   | 1.58                           | —   | 1.51 ± 0.11           | 105   |
| ELPAT 39P3   | 0.52                           | —   | 0.558 ± 0.039         | 93  |
| ELPAT 40P2   | 0.47                           | —   | 0.506 ± 0.032         | 92  |
| ELPAT 51P3   | 0.47                           | —   | 0.461 ± 0.035         | 102   |
| NIST SRM     | (%)                            | (%)   | (%)                   | (%)   |
| SRM 2581     | 0.42                           | 0.43  | 0.449 ± 0.011         | 94  |
| SRM 2581     | 0.44                           | 0.44  | 0.449 ± 0.011         | 98  |

## APPENDIX 2

### Results for Rotor/Stator-Turbidimetric Method on Lead-in-Paint Diagnostic Materials

| Type <sup>a</sup> | Rotor/Stator-Turbidity (R/S-T) Results               |   |                                   |   |                      | R/S-T Results vs Diagnostic Materials  |                                   |                                 |
|-------------------|--|---|-----------------------------------|---|----------------------|--|-----------------------------------|---------------------------------|
|                   | Larger Weight Samples (n=1) (mg Pb/cm <sup>2</sup> ) | Smaller Weight Samples (n=1) (mg Pb/cm <sup>2</sup> ) | Mean n=2 (mg Pb/cm <sup>2</sup> ) | Std. Dev. <sup>b</sup> (mg Pb/cm <sup>2</sup> ) | RSD <sup>c</sup> (%) | RS/T Mean within 95% CI <sup>d</sup> ? | 95% Confidence Intervals Overlap? | Mean Difference Less Than +20%? |
| A                 | 0.030  | 0.046   | 0.038                             | 0.011   | 29.98                | Y                                      | Y                                 | Y                               |
| B                 | 0.290  | 0.377   | 0.334                             | 0.062   | 18.50                | Y                                      | Y                                 | Y                               |
| C                 | 0.720  | 0.825   | 0.773                             | 0.074   | 9.61                 | Y                                      | Y                                 | Y                               |
| D                 | 0.830  | 0.877   | 0.854                             | 0.033   | 3.90                 | N                                      | Y                                 | Y                               |
| E                 | 1.070  | 1.044   | 1.057                             | 0.019   | 1.76                 | Y                                      | Y                                 | Y                               |
| F                 | 1.760  | 1.596   | 1.678                             | 0.116   | 6.93                 | Y                                      | Y                                 | Y                               |
| G                 | 0.600  | 0.679   | 0.640                             | 0.056   | 8.76                 | N                                      | Y                                 | Y                               |
| H                 | 0.920  | 0.933   | 0.926                             | 0.009   | 0.96                 | N                                      | N                                 | N                               |
| I                 | 0.700  | 0.672   | 0.686                             | 0.020   | 2.85                 | Y                                      | Y                                 | Y                               |
| J                 | 0.820  | 0.919   | 0.869                             | 0.070   | 8.03                 | N                                      | Y                                 | N                               |
| K                 | 0.770  | 0.672   | 0.721                             | 0.069   | 9.58                 | Y                                      | Y                                 | Y                               |
| L                 | 1.100  | 0.922   | 1.011                             | 0.126   | 12.43                | N                                      | Y                                 | Y                               |
| M                 | 0.600  | 0.592   | 0.596                             | 0.005   | 0.89                 | N                                      | N                                 | Y                               |
| N                 | 0.760  | 0.940   | 0.850                             | 0.127   | 14.94                | N                                      | Y                                 | N                               |
| O                 | 0.700  | 0.728   | 0.714                             | 0.020   | 2.76                 | Y                                      | Y                                 | Y                               |
| P                 | 1.130  | 1.096   | 1.113                             | 0.024   | 2.17                 | N                                      | N                                 | Y                               |
| Q                 | 0.680  | 0.627   | 0.654                             | 0.037   | 5.71                 | Y                                      | Y                                 | Y                               |
| R                 | 1.150  | 1.221   | 1.185                             | 0.050   | 4.22                 | Y                                      | Y                                 | Y                               |
| S                 | 0.690  | 0.648   | 0.669                             | 0.030   | 4.44                 | Y                                      | Y                                 | Y                               |
| T                 | 1.150  | 1.075   | 1.112                             | 0.053   | 4.77                 | Y                                      | Y                                 | Y                               |
| U                 | 0.740  | 0.728   | 0.734                             | 0.009   | 1.17                 | Y                                      | Y                                 | Y                               |
| V                 | 1.100  | 1.078   | 1.089                             | 0.015   | 1.40                 | Y                                      | Y                                 | Y                               |
| W                 | 0.690  | 0.745   | 0.718                             | 0.039   | 5.44                 | Y                                      | Y                                 | Y                               |
| X                 | 1.130  | 1.061   | 1.096                             | 0.049   | 4.45                 | Y                                      | Y                                 | Y                               |
| Y                 | 1.000  | 1.148   | 1.074                             | 0.105   | 9.73                 | Y                                      | Y                                 | Y                               |
| Z                 | 0.710  | 0.794   | 0.752                             | 0.059   | 7.88                 | Y                                      | Y                                 | Y                               |
| AA                | 1.160  | 1.176   | 1.168                             | 0.011   | 0.94                 | Y                                      | Y                                 | Y                               |
| AB                | 0.920  | 1.026   | 0.973                             | 0.075   | 7.73                 | Y                                      | Y                                 | Y                               |
| AC                | 0.610  | 0.658   | 0.634                             | 0.034   | 5.40                 | Y                                      | Y                                 | Y                               |
| AD                | 0.730  | 1.058   | 0.894                             | 0.232   | 25.92                | Y                                      | Y                                 | Y                               |
| AE <sup>e</sup>   | 1.000  | 0.995   | 0.998                             | 0.003   | 0.35                 | Y                                      | Y                                 | Y                               |

<sup>a</sup>See pages A-3 and A-4 for descriptions of types.

<sup>b</sup>Standard deviation

<sup>c</sup>RSD = relative standard deviation

<sup>d</sup>Confidence interval

<sup>e</sup>One replacement value used for type AE.

The Lead-in-Paint Diagnostic Test Materials were processed by the newly developed procedures described in two references.<sup>5,7</sup> The samples were collected using the drill or chisel method, extracted

using the rotor/stator procedure, and quantitated for Pb by the turbidity method. The weights of the paint diagnostic material samples collected for the rotor/stator-turbidity method varied from 0.04 to 0.30 g, which reflects the variation in collection methods, substrates, and the complexity of the diagnostic materials (number of paint films, overlayers of paint, etc.).

## Summary of ORD 2008 Lead-in-Paint Diagnostic Paint Materials<sup>12</sup>

| Type | Diagnosis                                      | Substrate | No. Films and Pb Compound <sup>a</sup> | White Overlayers No. and Type <sup>b</sup> | Chemical Interferences Al, Ba, Mg <sup>c</sup> | Chemical Interference Fe <sup>c,d</sup> | Pb Film Colors <sup>d</sup> | Areal Pb <sup>e</sup> (mg/cm <sup>2</sup> ) |
|------|--|-----------|--|--|--|---|-----------------------------|---|
| 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<sup>12</sup> (cont'd.)

| Type | Diagnosis                              | Substrate | No. Films and Pb Compound <sup>a</sup> | White Overlayers No. and Type <sup>b</sup> | Chemical Interferences Al, Ba, Mg <sup>c</sup> | Chemical Interference Fe <sup>c,d</sup> | Pb Film Colors <sup>d</sup> | Areal Pb <sup>e</sup> (mg/cm <sup>2</sup> ) |
|------|--|-----------|--|--|--|---|-----------------------------|---|
| 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%)                       |

<sup>a</sup>Pb compounds: 1w = 1 white Pb film, 2w = 2 white Pb films; 1c = 1 lead chromate film, 2c = lead chromate films

<sup>b</sup>Overlayers: 1O = 1 oil-based paint overcoat; 2L = 2 water-base paint overcoats

<sup>c</sup>Chemical interferences: Elements Al, Ba, Mg added to paint film; red or black iron oxide added for color.

<sup>d</sup>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.

<sup>e</sup>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

## Results of Comparison of Real-World Paint Samples Analyzed for Lead Using the Rotor/Stator-Turbidimetric Method and Method 3051a<sup>11</sup>/ICP-OES

| Samples                               | Rotor/Stator-Turbidimetric (mg/cm <sup>2</sup> ) <sup>a</sup> | Method 3051a/ICP-OES (mg/cm <sup>2</sup> ) <sup>a</sup> |
|---------------------------------------|---|---|
| <b><i>Durham Residential Wood</i></b> |   |   |
| Ashe 1 <sup>b</sup>                   | 0.239 ± 0.056   | 0.202 ± 0.029   |
| Ashe 2a <sup>b</sup>                  | 5.49 ± 0.97   | 5.15 ± 0.69   |
| Rox 1 <sup>b</sup>                    | 4.49 ± 1.67   | 5.94 ± 0.68   |
| Rox 2 <sup>b</sup>                    | 37.6 ± 20.9   | 38.6 ± 6.8  |
| Worth 1 <sup>b</sup>                  | 18.8 ± 2.8  | 22.4 ± 1.3  |
| Worth 2 <sup>b</sup>                  | 4.34 ± 1.54   | 4.79 ± 1.32   |
| Worth 3 <sup>b</sup>                  | 0.385 ± 0.205   | 0.148 ± 0.054   |
| <b><i>Tobacco Factory</i></b>         |   |   |
| Door <sup>c</sup>                     | 1.18 ± 0.22   | 1.14 ± 0.25   |
| Brick <sup>c</sup>                    | 0.42 ± 0.32   | 0.61 ± 0.41   |
| <b><i>Residential Hospital</i></b>    |   |   |
| Metal <sup>c</sup>                    | 2.63 ± 0.48   | 2.55 ± 0.62   |
| Window 1 <sup>b</sup>                 | 19.6 ± 1.8  | 19.6 ± 1.7  |
| Window 2 <sup>b</sup>                 | 79.5 ± 3.7  | 109 ± 11  |
| Coal chute <sup>c</sup>               | 1.42 ± 0.33   | 1.25 ± 0.19   |
| <b><i>Power Station</i></b>           |   |   |
| Steel beam 1 <sup>b</sup>             | 1.86 ± 0.14   | 2.33 ± 1.03   |
| Steel beam 2 <sup>c</sup>             | 2.96 ± 0.83   | 3.23 ± 0.75   |
| Steel beam 3 <sup>c</sup>             | 0.709 ± 0.071   | 0.738 ± 0.207   |
| Steel beam 4 <sup>c</sup>             | 4.42 ± 0.46   | 5.33 ± 0.20   |
| Ceiling <sup>d</sup>                  | 12.9 ± 0.6  | 11.8 ± 3.6  |

<sup>a</sup>N = 3 to 5 for each result

<sup>b</sup>Collected with modified drill bit

<sup>c</sup>Collected with steel chisel

<sup>d</sup>Chips from ceiling







# SCIENCE



PRESORTED STANDARD  
POSTAGE & FEES PAID  
EPA  
PERMIT NO. G-35

Office of Research and Development (8101R)  
Washington, DC 20460

Official Business  
Penalty for Private Use  
\$300



Recycled/Recyclable Printed on paper that contains a minimum of  
50% postconsumer fiber content processed chlorine free