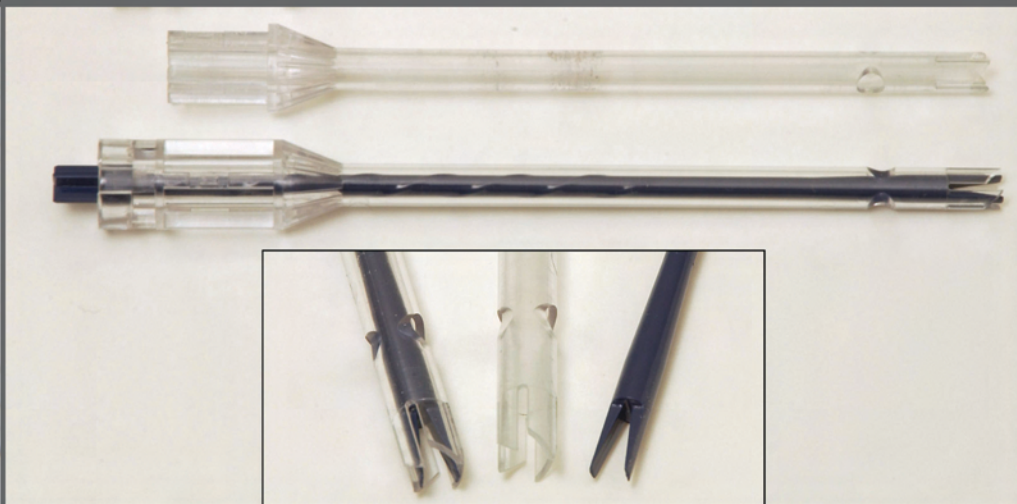


## Standard Operating Procedure for the Grinding and Extraction of Lead in Paint Using Nitric Acid and a Rotor/Stator System Powered by a High-Speed Motor

SCIENCE





# **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**

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### **Disclaimer**

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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.<sup>1</sup> The Federal regulated lead 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 lead 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 are that the test kit procedure 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 lead in paint are very sensitive but do not provide quantification of the lead 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 lead 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 lead 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.

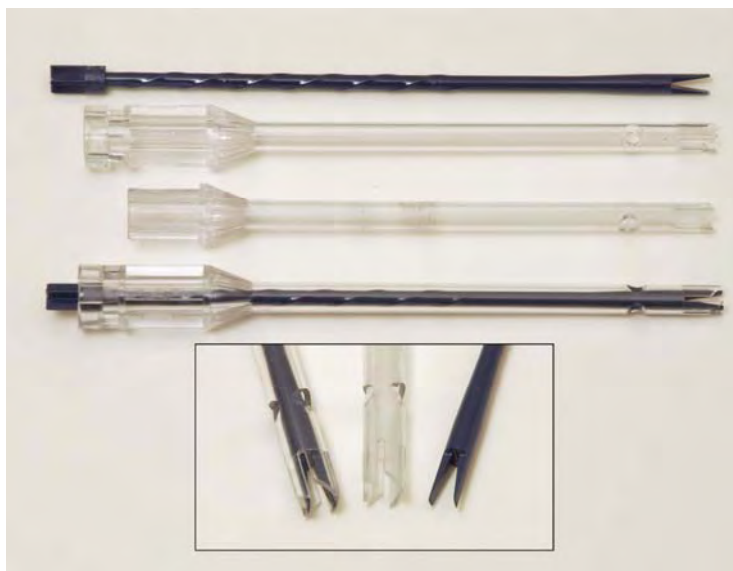
This standard operating procedure (SOP) describes a new, rapid, and relatively inexpensive one-step procedure that grinds the paint samples removed from the substrate and simultaneously quantitatively extracts the Pb from the paint in only one step in preparation for quantitative analysis. This method has been applied successfully in the laboratory 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 Rotor/Stator Grinding and Extraction

Typically a single chip of paint 1 cm<sup>2</sup> in area is collected from a painted surface and analyzed. Under certain circumstances, however, the paint sample collected may be in small pieces or an irregular chip. If the area is unknown and cannot be determined, then the sample is to be weighed to within  $\pm 0.001$  g ( $\pm 1$  mg). Simple scales for use in the field are available for this purpose, for example, the Gempro500 from Precision Weighing Balances, Bradford, MA. In the laboratory, a standard analytical balance is used to weigh the paint samples. No matter the physical form, the paint sample is crushed by hand, cut into small pieces with a scalpel or knife, or pulverized during sampling with an approach that grinds the paint as it removes it from the surface as per EPA "Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit and Variable-Speed Portable Electric Drill,"<sup>5</sup> prior to the grinding/extraction procedure described in this SOP.

In the method described in this SOP, the collected paint sample is placed in a 15-mL, conical bottom, plastic centrifuge tube containing 2 mL of 25% (v/v) nitric acid (HNO<sub>3</sub>). The paint in the acid is ground using a rotor/stator system (Figure 1) powered by a high-speed (15,000- to 30,000-rpm) motor. The paint is ground for about 3 min with 30-s on and 15-s off cycles. The rotor/stator configuration is common in high-shear grinders and mixers. This high-shear process is at the heart of the efficiency of this approach. When the system is operated, the small pieces of paint are drawn up into the center of the rotor and then spun out with great force. The small pieces are caught between the rotating and



**Figure 1. Photograph of rotor and stator separated and combined as normally used. Also shown is stator (clear plastic piece) with 12 mm removed to work with Rotozip motor as described below. (Rotor/stator probe shown manufactured by Omni International, Marietta, GA).**

static “blades” and are cut into smaller pieces. With continued grinding, the pieces become smaller and smaller. The combination of small particles, 25% (v/v)  $\text{HNO}_3$ , and intensive mixing results in Pb recoveries that are consistently greater than 95% for real-world paints, National Institute of Standards and Technology’s (NIST’s) Standard Reference Materials (SRMs), and audit samples from the American Industrial Hygiene Association’s (AIHA) Environmental Lead Proficiency Analytical Testing (ELPAT)<sup>6</sup> Program.

## 2.2 Method Performance

The method has been evaluated using a series of reference materials and real-world paints from the RTI repository of paint for the ELPAT program.<sup>6</sup> Included were six samples of certified NIST Research Material (RM) 8680 Paint on Fiberboard<sup>7</sup> (actually collected by RTI for EPA/NIST in the 1990s) and four standard reference paint films prepared by RTI for EPA that consist of uniform sheets of lead-nitrate-spiked, water-based paint that were characterized by sampling and acid extraction/inductively coupled plasma atomic emission spectroscopy (ICP-AES) measurement.<sup>8,9</sup> The average recovery with these samples always exceeded 95%, as shown in the data presented in Appendix 1.

Method performance also was determined as the recovery based on the amount of Pb extracted versus that found in any undissolved residue following the rotor/stator-based grinding and extraction. Residue analysis performed with the RTI-prepared paint films yielded a mean recovery of  $97.3\% \pm 0.53\%$  (0.54% RSD), while residue analysis performed with real-world paints yielded average recoveries exceeding 95%, as demonstrated in Appendix 2.

On the basis of all the tests done with both real-world field samples and reference samples, the rotor/stator method of grinding and extracting Pb in paint as described in this SOP consistently produces recoveries of Pb greater than 95% from real-world paints.

[Note: Some paints form a gooey material on grinding that sticks to the rotor/stator system. The reason for this formation remains unknown. To check the significance of this, several paint samples that form gooey material were ground/extracted using the rotor/stator method. As demonstrated in Appendix 3, the recovery was still above 95% for these samples, and, therefore, the material does not

negatively affect the recovery of these real-world paints. Because the cleaning method effectively removes the gooey material, it should be used to prepare the rotor/stator for reuse. The potential for gooey material formation requires that several rotor/stator systems be available for efficient work with multiple samples.]

### **3.0 DEFINITIONS, ACRONYMS, AND ABBREVIATIONS**

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

- AIHA – American Industrial Hygiene Association
- ELPAT – Environmental Lead Proficiency Analytical Testing Program
- EPA – U.S. Environmental Protection Agency
- ICP-AES – inductively coupled plasma atomic emission spectroscopy
- LBP – lead-based paint
- NIST – National Institute of Standards and Technology
- Pb – elemental or ionic lead
- RM – NIST Reference Material
- RRP – Lead; Renovation, Repair, and Painting Program; Final Rule
- RTI – RTI International
- SOP – standard operating procedure
- SRM – NIST Standard Reference Material
- v/v – volume-to-volume ratio

### **4.0 HEALTH AND SAFETY WARNINGS**

#### **4.1 Safety with Nitric Acid**

The component of this procedure requiring the greatest care is  $\text{HNO}_3$ , which 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 those that follow.

- Safety goggles (or safety glasses with side shields)
- Acid-resistant gloves
- 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.

## 4.2 Use of Equipment

The equipment used in this method does not present any major hazard to the user. However, using the rotor/stator does require several precautions to avoid personal injury or equipment damage.

- Be sure the high-speed motor is fastened securely to the holding device.
- The rotor/stator probe may warp slightly if it overheats; do not touch when still hot. When warping occurs, this indicates a need to insert a new rotor/stator probe and discard the warped one.
- Be sure the rotor/stator probe is secure and centered in the test tube so that it does not contact the sides of the test tube.

## 4.3 Paint Preparation

- If the paint chips are cut into small pieces using a knife or scalpel, caution is required to avoid cutting oneself.
- If the paint is ground using the modified drill bit sampling method, care must be taken to avoid any inhalation of this dust.
- Any lead-based paint or paint dust must be washed from the hands before eating or drinking.

# 5.0 EQUIPMENT, SUPPLIES, AND REAGENTS

## 5.1 Apparatus

### 5.1.1 High-Speed Motor To Operate Rotor/Stator System

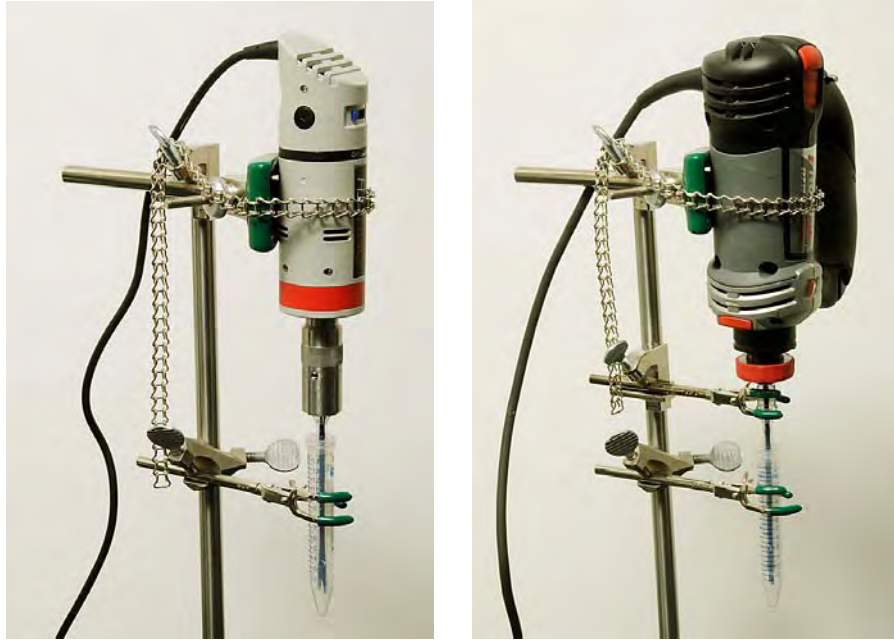
Motors currently identified are the Rotozip spiral saw system by Bosch and the Omni tissue homogenizer by Omni International; either motor may be used. Other suitable motors also may be available and equivalent.

#### 5.1.1.1 Rotozip RZ10 Spiral Saw System by Bosch<sup>10</sup>

- Variable Speed (15,000 to 30,000 rpm)
- Modification of the Omni rotor/stator probe required to fit Rotozip chuck. Stator must be reduced in length 12 mm at top and held in place by separate clamp.
- Rotozip tool can be set up on a timer (see below) that runs 30 s on and then 15 s off for a total time of 3 min.
- System to hold motor, rotor/stator probe, and sample preparation tube in place during grinding such as a ring stand with appropriate clamps as shown in Figure 2. The components of this support system for the Rotozip motor include the following four items.
  - (1) Ring stand, VWR 60010-105, or equivalent
  - (2) Chain clamp, VWR 21573-275, or equivalent
  - (3) Two clamp holders, VWR 21572-501, or equivalent
  - (4) Two miniature three-prong clamps, VWR 21572-805, or equivalent

#### 5.1.1.2 Omni Tissue Homogenizer Model TH115 by Omni International<sup>11</sup>

- Speed is variable from 5,000 to 35,000 rpm.
- Chuck provides for mounting of both Omni rotor and stator.
- Homogenizer can be set up on a timer (see below) that runs 30 s on and then 15 s off for a total time of 3 min.



**Figure 2. Omni (left) and Rotozip (right) high-speed motors fitted with Omni rotor/stator probes and 15-mL plastic centrifuge tubes. [Note: Clamp not needed to hold stator when using Omni motor.]**

- The manufacturer of that motor offers a support stand specifically made for the Omni motor.

### **5.1.2 Rotor/Stator System**

**5.1.2.1** Omni Inc.—Hard Tissue Omni Tips (7 x 110 mm) Model 30750H—includes rotor and stator.

- See Figure 1.
- The rotor and stator typically can be used 10 times before they need to be replaced.

**5.1.2.2** Omni-supplied chuck to hold stator and rotor or chuck to fit in another motor to hold rotor with another device such as a clamp on a ring stand to hold the stator.

[Note: As noted above, 12 mm of the top of the stator purchased from Omni must be cut off using a hacksaw or equivalent if the Omni rotor/stator probe is to be used in a Rotozip motor (see Figure 1). The stator then is supported with a clamp as shown in Figure 2b because the chuck on the Rotozip tool only allows insertion of the rotor.]

### **5.1.3 Power Timer To Control 30 s on/15 s off for Rotor/Stator Motor**

- VWR Controller/Timer, VWR 23609-188, or equivalent

### **5.1.4 Pregrinding Paint Preparation Tools**

If EPA “Standard Operating Procedure for Surface Paint Sample Collection Using a Modified Wood Drill Bit and Variable-Speed Portable Electric Drill”<sup>5</sup> is not used to collect/pregrind the paint materials, obtain the following supplies.

**5.1.4.1** Field scale balance with readability of  $\pm 0.001$  g, Gempro500 from Precision Weighing Balances, Bradford, MA, or equivalent

**5.1.4.2** Small cutting board that can be washed between samples; 8.5 x 11 in (22 x 28 cm), Chefmate white poly kitchen cutting board, Target or equivalent

**5.1.4.3** Weighing paper for sample handling and crushing, VWR WL565223-10A, or equivalent

**5.1.4.4** Scalpel or knife for cutting up rubbery samples, VWR 25853-003, or equivalent

### **5.1.5 Materials and Supplies**

**5.1.5.1** One 500-mL plastic beaker to serve as wash basin for cleaning rotors and stators, VWR 13917-550, or equivalent

**5.1.5.2** Two 1-L polyethylene bottles—one for storage of 25% v/v nitric acid and the other for storage of 2% trisodium phosphate solution for cleaning the rotor/stator probe, VWR 83009-063, or equivalent

**5.1.5.3** Squirt bottle for rinsing down rotor/stator during withdrawal from the centrifuge tube, VWR 16651-143, or equivalent

**5.1.5.4** Small brush for cleaning rotors and stators, if required, VWR 17080-004, or equivalent

**5.1.5.5** Three wide-mouth, 5-L, polyethylene carboys—one for acid waste, one for wash and rinse water waste, and one for storage of soapy water for washing, VWR 80094-464, or equivalent

**5.1.5.6** Indelible marker to label centrifuge tubes and containers used to collect waste

**5.1.5.7** Notebook (VWR 28196-346, or equivalent) or bound forms prepared specifically for this SOP

**5.1.5.8** Volumetric pipette to deliver 2.0 mL of  $\text{HNO}_3$  into 15-mL centrifuge tube, VWR 89003-490, or equivalent, plus manual, 2-mL pipette pump, VWR 53502-222, or equivalent

**5.1.5.9** Labeled 15-mL plastic centrifuge tubes, VWR Catalog No. 21008-918, or equivalent

## **5.2 Reagents**

### **5.2.1 25% (v/v), Trace Metal Grade Nitric Acid**

Pour three parts deionized water into a narrow-mouth, 1-L polyethylene container that can be capped. Carefully and slowly add one part concentrated, reagent grade, nitric acid. Allow the mixture to cool, cap the bottle, and mix thoroughly by rotating the container. Label the container with the name of the contents, the date of preparation, and the name of the preparer.

**5.2.1.1** Concentrated, reagent grade nitric acid, VWR EM-NX 0409-2, or equivalent

**5.2.1.2** Deionized or distilled water

### **5.2.2 Quality Control Materials**

- NIST SRMs 2580,<sup>12</sup> 2581,<sup>13</sup> and/or 2582.<sup>14</sup> Order online: [www.srmors.nist.gov/orderingSRMs.cfm](http://www.srmors.nist.gov/orderingSRMs.cfm).
- ELPAT samples. Order from American Industrial Hygiene Association, 2700 Prosperity Ave., Suite 250, Fairfax, VA.

### **5.2.3 Cleaning and Rinsing**

**5.2.3.1** 100-mL plastic beaker for rotor/stator cleaning, VWR 25384-152, or equivalent

**5.2.3.2** Laboratory detergent, Alconox, VWR 21835-032, or equivalent. Prepare wash water in 5-L carboy following instructions on detergent container.

**5.2.3.3** Trisodium phosphate for rotor/stator cleaning, VWR AAAL15052-36, or equivalent

**5.2.3.4** 2% trisodium phosphate rotor/stator cleaning solution. Mix 20 g trisodium phosphate in 1 L of deionized water in a plastic bottle.

**5.2.3.5** Deionized water for rinsing down rotor and stator as they are withdrawn from the centrifuge tube and corresponding sample dilution if required for the follow-up measurement method

**5.2.3.6** Laboratory wipe, Kimwipe, VWR 21905-026, or equivalent

### **5.3 Safety Equipment**

Safety equipment is required for protection from harm from nitric acid. Items to be used include

- safety goggles, VWR 10837-120, or equivalent
- eye wash bottle, VWR 56611-060, or equivalent
- acid resistant gloves, VWR 40101 (extra small to extra large), or equivalent
- laboratory apron, VWR 32891-268, or equivalent

## **6.0 QUALITY CONTROL AND QUALITY ASSURANCE**

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

- Use only clean, 15-mL plastic centrifuge tubes and rotor/stator systems. If a rotor and stator are not clean, they must be thoroughly washed in soapy water, rinsed thoroughly in deionized water, and then air dried or blotted dry with a laboratory wipe before use.
- Use only a clean cutting board for each sample; cutting boards are to be washed and dried between samples or wiped clean with a laboratory wipe.
- Use only reagent grade nitric acid.
- A blank should be prepared for analysis about every 20th sample. This consists of running acid alone with the rotor/stator system.
- A quality control sample should be prepared about every 20th sample. This will consist of nominally 0.1 g of a NIST paint reference material such as NIST SRM's 2580 at nominally 4% Pb,<sup>12</sup> 2581 at nominally 0.5%,<sup>13</sup> or 2582 at nominally 200 mg/kg.<sup>14</sup> Alternatively, excess ELPAT materials may be purchased from the AIHA<sup>15</sup> for this purpose.
- Carefully record the sample tube label number on a sampling log and/or in a laboratory notebook. Adequately describe the sample collection location and characteristics of the paint, such as estimated thickness, color, and number of layers.

## **7.0 PROCEDURE**

### **7.1 System Setup**

**7.1.1** Secure a rotor/stator probe in the Omni motor chuck (or the Rotozip motor or some equivalent chuck and stator clamp), so that the clear and blue bottom ends of the rotor and stator, respectively, are flush with each other. When the Rotozip is used, 12 mm of the top of the stator has to be cut off, so that the bottom of the rotor and stator are aligned horizontally.

**7.1.2** Be sure that the rotor and stator are aligned vertically to avoid any physical contact between them; such contact will generate heat and the eventual failure of the rotor/stator system.

### **7.2 Grinding and Extraction Procedure**

#### **7.2.1 Paint Preparation for Extraction**

Paint samples collected for analysis will be whole chips of a known area, small pieces of paint, and/or powdered paint. If a whole paint chip is relatively thick (>1mm), the mass of the sample may exceed the capacity of the 2 mL of 25% (v/v) of nitric acid to yield 95+% recovery of Pb. If

this situation is suspected, it's advisable to crush the sample into millimeter-sized particles on weighing paper placed on the cutting board and to weigh out a portion that is no more than 0.1 g. Simple scales for use in the field are available for this purpose, for example, the Gempro500 from Precision Weighing Balances, Bradford, MA.

#### **7.2.1.1 Large mass paint samples**

- (1) Place a piece of weighing paper on the scale and determine the weight as per the manufacturer's instructions.
- (2) Place the paint on the paper and weigh again. The weight of paper plus paint minus weight of the paper is the weight of the paint.
- (3) Alternatively, cut a large chip into smaller chips of measurable area and use one of these for the rotor/stator extraction process. [Note: The risk of using a smaller sample is that it will be less representative of the true average value of the source.]

#### **7.2.1.2 Small mass paint samples**

- (1) If a paint sample is intact or consists of several pieces, place it on a weighing paper, being sure to not lose any of the sample during the transfer.
- (2) Fold the paper and crumble the paint sample in the folded paper as much as possible using your fingers, taking care to not touch the paint with your hands. If the paint is rubbery and unable to be crushed, carefully cut up the paint sample on a clean glass, polypropylene, Teflon, or equivalent cutting board (to approximately the size of grains of rice) using a scalpel. Take care not to lose any of the paint during the cutting process.

**7.2.1.3** If the paint sample has been ground as part of the sample collection method, then it is transferred directly without further pretreatment to the 15-mL, grinding/extraction tube.

**7.2.1.4** If the surface area of the paint sample is unknown and cannot be determined, then the sample is to be weighed to within  $\pm 0.001$  g ( $\pm 1$  mg). Simple scales for use in the field are available for this purpose, for example, the Gempro500 from Precision Weighing Balances, Bradford, MA.

- (1) Place a piece of weighing paper on the scale and determine the weight as per the manufacturer's instructions.
- (2) Place the paint on the paper and weigh again.
- (3) The weight of paper plus paint minus weight of the paper is the weight of the paint. This weight will yield the final value in percent Pb.

### **7.2.2 Paint Extraction**

**7.2.2.1** Place pieces of, or "pour", ground paint chip sample carefully into a labeled, 15-mL, plastic centrifuge tube.

**7.2.2.2** Tap the bottom of the tube lightly on a hard surface to bring all the particles of paint to the bottom of the tube.

**7.2.2.3** Using the volumetric pipette or pipette pump, transfer 2.0 mL of the 25% (v/v)  $\text{HNO}_3$  into the centrifuge tube.

**7.2.2.4** Secure the tube with a clamp at the base of the motor holder.

**7.2.2.5** Raise the tube containing the sample onto the rotor/stator such that the separation between the inner bottom of the centrifuge tube and the bottom of the rotor/stator is about 1 to 2 mm; be sure that the rotor/stator is centered in the tube.

**7.2.2.6** Turn on the motor on top speed (30,000 rpm) and allow it to run its timed course of 30 s on, 15 s off for approximately 3 min. This on-and-off process controlled by a suitable power



timer allows the particles to settle and then be pulled back up into the vortex and thus sheared. The resultant mixture will be cloudy.

**7.2.2.7** Partially lower the centrifuge tube containing the paint extract, trying to keep any remaining paint residue in the solution.

**7.2.2.8** With the rotor/stator above the liquid, rinse the rotor/stator with 1 to 2 mL of deionized water.

**7.2.2.9** Lower the centrifuge tube away from the rotor/stator.

**7.2.2.10** Carefully add deionized water to bring the total volume to 5 mL. [*Note:* Add water until the bottom of the meniscus is at the 5-mL mark on the tube.]

**7.2.2.11** Cap the labeled centrifuge tube and place it in a secure container for lead analysis.

[*Note:* If the lead concentration is to be determined using the EPA “Standard Operating Procedure for the Turbidimetric Determination of Lead in Paint Extracts”<sup>16</sup> or some other test method that requires the maximum concentration of lead in the extract, then do not rinse the rotor/stator. Instead, with the rotor/stator just above the liquid, tap the rotor/stator lightly to shake off any remaining droplets of solution. Then fully lower the centrifuge tube. Finally, cap the labeled centrifuge tube and place it in a secure container for lead analysis.]

### 7.3 Rotor/Stator Reuse

Experience has shown that the rotor/stator probe can be used 10 to 12 times before the components become worn to the point of not efficiently grinding the paint, or that the components become warped such that the rotor and stator come in contact during grinding, and excessive heating occurs.

As noted earlier, some paints form a gooey mass during the extraction process that is very difficult to remove from the rotor/stator by simple washing with soap and water. The method for cleaning is described below.

**7.3.1** Use a laboratory wipe to remove as much goo as possible.

**7.3.2** With the rotor/stator probe mounted in the motor, immerse the probe in about 50 mL of 2% trisodium phosphate solution in a 100-mL beaker; the probe should extend about 8 cm into the cleaning solution.

**7.3.3** Operate the motor at 30,000 rpm for 1 min.

**7.3.4** Lower the beaker, rinse the probe with deionized water, and wipe the probe lightly with a paper towel.

**7.3.5** Rinse the rotor/stator probe with deionized water a second time.

## 8.0 DATA CALCULATION

This SOP describes a method for grinding and extracting lead from a known amount of paint. The collected paint sample may be measured by its area (cm<sup>2</sup>) or by its mass or weight (g). The analysis following this grinding and extraction will yield a value of total micrograms or milligrams of Pb in the extract. To calculate the concentration in weight per unit area, divide the total value for the Pb measured by the area of the paint sample. For example,

$$\text{mg Pb/cm}^2 = \text{mg Pb measured/area of paint sample in cm}^2.$$

To calculate the concentration in percent, divide the total micrograms or milligrams of lead measured by the mass of the paint sample and multiply by 100. For example,

$$\% \text{ Pb} = 100 \times (\text{mg Pb measured/mg of paint sample extracted}).$$

## 9.0 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 lead-in-paint treatment. Activities to be performed include those that follow.

- 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.
- Each paint sample shall be given an identifying name or number that is recorded along with a description of the sample. This description shall indicate the source of the paint and its physical attributes (color, estimated number of layers, estimated thickness, and brittleness).
- The label placed on the 15-mL extraction tube shall be the same as the name or number of the paint sample. If the tube is given a different label, an accurate record must be maintained that relates the tube label to the name or number of the paint sample placed in the tube.
- The date and time of the extraction shall be recorded in the notebook or on the form.
- The person performing the grinding and extraction shall be identified in the notebook or on the form.
- Records are to be maintained of unusual occurrences such as the formation of the goo, unusual amounts of residue, or unusual extract color.

## 10.0 WASTE MANAGEMENT

There are several forms of waste generated during the operation of this SOP. Each is discussed in the following subsections.

### 10.1 Nitric Acid

If nitric acid waste or waste extract is generated, carefully pour this material into a wide mouth, polyethylene carboy that is carefully 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.

### 10.2 Wash Water

The wash water from cleaning the rotor and stator is to be poured into a 5-L, polyethylene carboy that is carefully labeled with a description of the contents or can simply be poured down the drain since the Pb and acid content of the wash water will be minimal. Trisodium phosphate solution used to remove paint goo from a rotor/stator probe is to be poured into this vessel also.

### 10.3 Lead Paint

Excess paint chips or paint powders that are no longer needed are to be placed in a resealable plastic bag. The bag is to be labeled with a description of the contents, the source(s) of the paint, and the name of the person who has placed these materials in the bag. This waste paint shall be delivered to a commercial firm that specializes in removal of hazardous waste.

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## APPENDIX 1

### Results of Application of Rotor/Stator Extraction Procedure to Lead-In-Paint Reference Materials

The paint samples were removed from the certified NIST RM 8680 Paint on Fiberboard<sup>7</sup> pieces using the modified drill bit method<sup>5</sup> described in the aforementioned SOP, and these samples were then ground/extracted using the rotor/stator method. The RTI reference paint films were rubbery when made, but they have become somewhat brittle over approximately 10 years of storage.<sup>8,9</sup> When these “imbrittled” samples were hand crushed and put through the rotor/stator process, well-ground samples were produced. Results are shown in Table A-1, along with results for several ELPAT materials and samples of NIST SRM 2581 powdered paint.<sup>13</sup>

**Table A-1. Results of Rotor/Stator Grinding/Extraction and ICP-AES Analysis of Six NIST RM 8680 Reference Materials, Two RTI-Prepared Paint Films, and Six Reference Materials**

| NIST Fiber Board, RM 8680 <sup>a</sup> | ICP-AES Pb Conc. <sup>b</sup> (mg/cm <sup>2</sup> ) | NIST Value (mg/cm <sup>2</sup> ) | Recovery Based on Expected Value (%) |
|--|---|----------------------------------|--------------------------------------|
| KB2                                    | 1.20 ± 0.12   | 1.25 ± 0.35                      | 96.0                                 |
| TD5                                    | 1.48 ± 0.05   | 1.21 ± 0.38                      | 122                                  |
| DG2                                    | 1.13 ± 0.07   | 1.14 ± 0.32                      | 99.1                                 |
| HA3                                    | 1.28 ± 0.36   | 1.31 ± 0.34                      | 97.7                                 |
| MD2                                    | 1.09 ± 0.09   | 1.10 ± 0.30                      | 99.1                                 |
| JH1                                    | 1.57 ± 0.09   | 1.29 ± 0.40                      | 122                                  |
|  |   |                                  | <b>Av 106 ± 12 (11%)</b>             |

<sup>a</sup>Paint samples preground with wood drill bit during sample collection prior to rotor/stator grinding.

<sup>b</sup>Conc. = concentration

| RTI-Made Reference Film <sup>c</sup> | ICP-AES Pb Conc. (mg/cm <sup>2</sup> ) | Expected Value (mg/cm <sup>2</sup> ) | Recovery Based on Expected Value (%) |
|--------------------------------------|--|--------------------------------------|--------------------------------------|
| RTI-49-A1-B                          | 0.36                                   | 0.30                                 | 120                                  |
| RTI-49-A1-B                          | 0.36                                   | 0.30                                 | 120                                  |
| RTI-35-A6-T                          | 0.57                                   | 0.60                                 | 95                                   |
| RTI-35-A6-T                          | 0.52                                   | 0.60                                 | 86.7                                 |
|                                      |  |                                      | <b>Av 105 ± 17 (16%)</b>             |

<sup>c</sup>Paint samples prepared by hand crushing prior to rotor/stator grinding.

| Reference Materials <sup>d</sup> | ICP-AES Pb Conc. (%) | Expected Value (%) | Recovery Based on Expected Value (%) |
|----------------------------------|----------------------|--------------------|--------------------------------------|
| ELPAT 51P1                       | 2.18                 | 2.22 ± 0.13        | 97.8                                 |
| ELPAT 51P2                       | 1.47                 | 1.51 ± 0.11        | 96.1                                 |
| ELPAT 39P3                       | 0.53                 | 0.558 ± 0.039      | 98                                   |
| ELPAT 40P2                       | 0.50                 | 0.506 ± 0.032      | 109                                  |
| ELPAT 51P3                       | 0.48                 | 0.461 ± 0.035      | 102                                  |
| SRM 2581                         | 0.43                 | 0.449 ± 0.011      | 95.6                                 |
| SRM 2581                         | 0.44                 | 0.449 ± 0.011      | 98.0                                 |
|                                  |                      |                    | <b>Av 99.5 ± 4.7 (4.7%)</b>          |

<sup>d</sup>Paint samples in powdered form prior to rotor/stator grinding.

## APPENDIX 2

### **Results of Application of Rotor/Stator Extraction Procedure to Real-World, Lead-In-Paint Samples**

Extraction efficiency was determined by using ICP-AES to separately analyze the extract solution and any remaining residue following rotor/stator extraction; these two phases were separated using centrifugation. The microwave/aqua regia digestion method, which is used for preparing ELPAT paint materials,<sup>5</sup> was used to digest the solid residue remaining in the 15-mL centrifuge tube following extraction and centrifugation of the rotor/stator paint extract. The recovery in the extract, which was calculated as the amount measured in the extract divided by the total of the amounts in the extract and the residue, was found to be in the range of 92% to 96% as demonstrated in Table A-2 below.

**Table A-2. Determination of Lead Recovery from Real-World Paint Samples with the Rotor/Stator Grinding/Extraction Procedure as Determined by Comparison of the Lead in the Extract and Lead in Digested/Extracted Residue**

| Paint Material Identifier | Mixer/Grinder | Pb in Liquid Extract (µg) | Pb in Residue (µg) | Pb Recovery in Liquid Extract (%) |
|---------------------------|---------------|---------------------------|--------------------|-----------------------------------|
| <b>TEST 1</b>             |               |                           |                    |                                   |
| "0.3% Pb"                 | Omni          | 153                       | 4.38               | 97.2                              |
| "0.3% Pb"                 | Omni          | 151                       | 4.30               | 97.2                              |
| LGS                       | Omni          | 534                       | 18.2               | 96.7                              |
| LGS                       | Omni          | 389                       | 14.8               | 96.3                              |
|                           |               |                           |                    | <b>Av 96.9 ± 4.4 (4.5%)</b>       |
| <b>TEST 2</b>             |               |                           |                    |                                   |
| "0.3% Pb"                 | RotoZip       | 133                       | 5.00               | 96.4                              |
| "0.3% Pb"                 | RotoZip       | 181                       | 6.08               | 96.8                              |
| "0.3% Pb"                 | RotoZip       | 147                       | 4.56               | 97.0                              |
|                           |               |                           |                    |                                   |
| LGS                       | RotoZip       | 402                       | 15.7               | 96.2                              |
| LGS                       | RotoZip       | 430                       | 18.3               | 95.9                              |
| LGS                       | RotoZip       | 405                       | 15.4               | 96.3                              |
|                           |               |                           |                    | <b>Av 96.4 ± 4.0 (4.2%)</b>       |

**Table A-2. Determination of Lead Recovery from Real-World Paint Samples with the Rotor/Stator Grinding/Extraction Procedure as Determined by Comparison of the Lead in the Extract and Lead in Digested/Extracted Residue (cont'd.)**

| Paint Material Identifier | Mixer/Grinder | Pb in Liquid Extract (µg) | Pb in Residue (µg) | Pb Recovery in Liquid Extract (%) |
|---------------------------|---------------|---------------------------|--------------------|-----------------------------------|
| <b>TEST 3</b>             |               |                           |                    |                                   |
| "0.3% Pb"                 | RotoZip       | 173                       | 5.01               | 97.2                              |
| "0.3% Pb"                 | RotoZip       | 179                       | 5.38               | 97.1                              |
|                           |               |                           |                    |                                   |
| LGS                       | RotoZip       | 325                       | 14.5               | 95.7                              |
| LGS                       | RotoZip       | 392                       | 16.8               | 95.9                              |
|                           |               |                           |                    |                                   |
| DorDix                    | RotoZip       | 211                       | 4.67               | 97.8                              |
| DorDix                    | RotoZip       | 585                       | 19.2               | 96.8                              |
|                           |               |                           |                    |                                   |
| P926                      | RotoZip       | 72.5                      | 1.65               | 97.8                              |
| P926                      | RotoZip       | 104                       | 2.72               | 97.5                              |
|                           |               |                           |                    | <b>Av 97.0 ± 0.8 (0.8%)</b>       |
| <b>TEST 4</b>             |               |                           |                    |                                   |
| "0.3% Pb"                 | RotoZip       | 194                       | 10.9               | 94.7                              |
| "0.3% Pb"                 | RotoZip       | 166                       | 4.67               | 97.3                              |
|                           |               |                           |                    |                                   |
| LGS-74B                   | RotoZip       | 447                       | 15.2               | 96.7                              |
| LGS-74B                   | RotoZip       | 387                       | 16.9               | 95.8                              |
|                           |               |                           |                    |                                   |
| DorDix-P1108              | RotoZip       | 802                       | 18.7               | 97.7                              |
| DorDix-P1108              | RotoZip       | 689                       | 19.2               | 97.3                              |
|                           |               |                           |                    |                                   |
| OH-P926                   | RotoZip       | 36.0                      | 0.41               | 98.9                              |
| OH-P926                   | RotoZip       | 77.4                      | 2.02               | 97.5                              |
|                           |               |                           |                    | <b>Av 96.9 ± 1.3 (1.3%)</b>       |

### **APPENDIX 3**

#### **Results of Application of Rotor/Stator Extraction Procedure to Paint Samples That Form a Gooney Material During the Extraction Procedure**

Some paints form a gooney material on grinding that sticks to the rotor/stator system. The reason for this formation remains unknown. To check the significance of this, several paint samples that form gooney material were ground/extracted using the rotor/stator method. Because the material sticks to the rotor/stator, the gooney material from each sample was scraped carefully and washed from the rotor/stator using a small spatula, small brush, and soap and water, which break up and apparently dissolve the goo. The wash water was quantitatively collected in a beaker. The solutions that were collected in this manner were then extracted using microwave/aqua regia digestion and analyzed for Pb by ICP-AES for the measurement. As demonstrated in Table A-3, the recovery remains above 95% with these types of samples, and it is apparent that the gooney material does not negatively affect the recovery of these real-world paints.

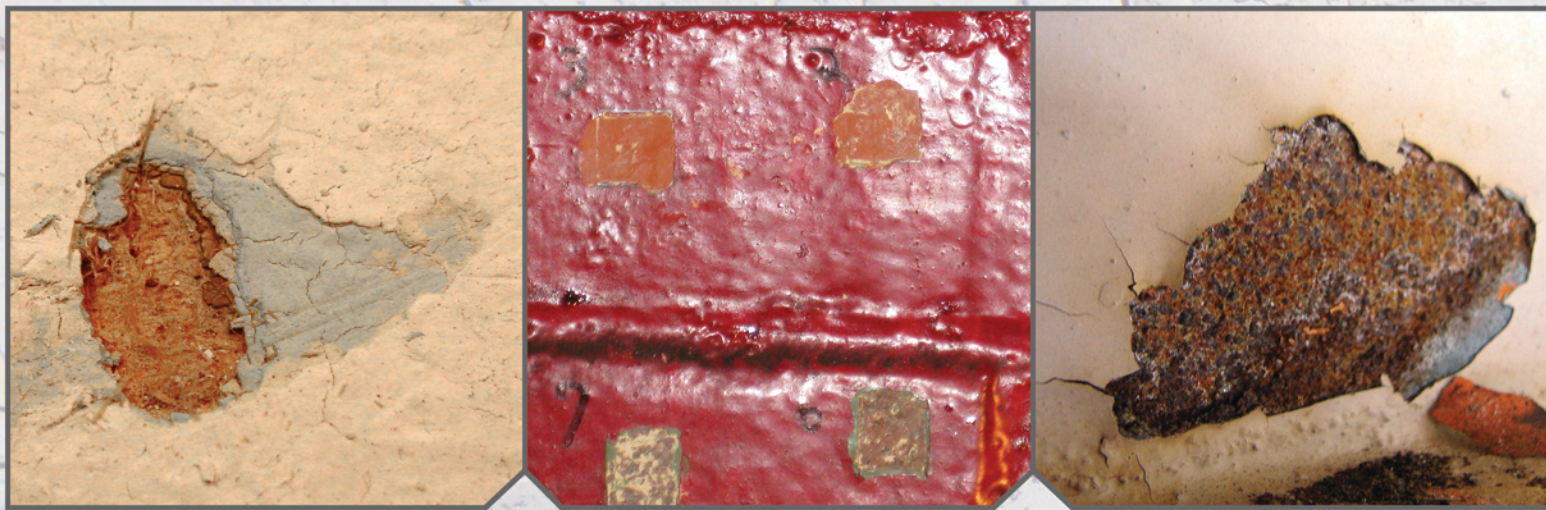
**Table A-3. Results of Analysis of Paint Samples That Form Gooney Material**

| <b>RTI Repository<br/>Paint Material</b> | <b>Weight<br/>(g)</b> | <b>Amount Wash<br/>Water Used (mL)</b> | <b>Liquid Extract,<br/>Lead, Pb (µg)</b> | <b>Rotor/Stator<br/>Wash Water,<br/>Pb (µg)</b> | <b>Pb in Wash<br/>Water (%)</b> |
|--|-----------------------|--|--|---|---------------------------------|
| P728                                     | 0.100                 | 18                                     | 43,126.60                                | 1,053   | 2.38                            |
| P1149                                    | 0.108                 | 24.5                                   | 40,389.90                                | 300   | 0.74                            |
| PB-5                                     | 0.100                 | 20.5                                   | 32,335.70                                | 192   | 0.59                            |









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