



Designation: E 1979 – 98

## Standard Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead<sup>1</sup>

This standard is issued under the fixed designation E 1979; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers an ultrasonic extraction procedure for the extraction of lead from environmental samples of interest in lead abatement and renovation (or related) work, for analytical purposes.

1.2 Environmental matrices of concern here include dry paint films, settled dusts, soils, and air particulates.

1.3 Samples subjected to ultrasonic extraction are prepared for subsequent determination of lead by laboratory analytical methods.

1.4 This practice includes, where applicable, descriptions of procedures for sample homogenization and weighing prior to ultrasonic extraction.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *This practice does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this practice to establish appropriate safety and health practices and determine the applicability of regulatory limitation prior to use.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 5438 Practice for Collection of Floor Dust for Chemical Analysis<sup>3</sup>

E 1553 Practice for Collection of Airborne Particulate Lead During Abatement and Construction Activities<sup>4</sup>

E 1605 Terminology Relating to Abatement of Lead Hazards<sup>4</sup>

E 1613 Test Method for the Analysis of Digested Samples for Lead Determination by Atomic Spectrometry<sup>4</sup>

E 1726 Practice for Sample Digestion of Soils for Lead Determination<sup>4</sup>

E 1727 Practice for Field Collection of Soil Samples for Lead Determination<sup>4</sup>

E 1728 Practice for Field Collection of Settled Dust Samples Using Wipe Sampling Methods for Lead Determination<sup>4</sup>

E 1729 Practice for Field Collection of Dried Paint Samples for Lead Determination<sup>4</sup>

E 1775 Guide for Evaluating the Performance of On-Site Extraction and Field-Portable Electrochemical or Spectrophotometric Analysis for Lead<sup>4</sup>

E 1792 Specification for Wipe Sampling Materials for Lead in Surface Dust<sup>4</sup>

PS 46 Provisional Practice for the Collection of Surface Dust by Air Sampling Pump Vacuum Technique for Subsequent Lead Determination<sup>4</sup>

### 3. Terminology

3.1 *Definitions:* For definitions of terms relating to this practice that do not appear in this section, refer to Terminology E 1605.

3.1.1 *extraction*—the dissolution of target analytes from a solid matrix into a liquid form.

3.1.1.1 *Discussion*—During sample digestion or extraction, target analytes are extracted (solubilized) into solution to enable subsequent determination by analytical techniques (for example, see Test Method E 1613).

3.1.2 *ultrasonic extraction*—the use of ultrasonic energy and acidic or basic solution to extract targeted analytes from samples.

3.1.2.1 *Discussion*—The extract solution is subsequently analyzed for the determination of targeted analytes.

### 4. Summary of Practice

4.1 Samples of paint, settled dust (wipe or vacuum), soil, or air particulate, obtained by ASTM sample collection methods (see Specification E 1729; Practice E 1728, Practice D 5438 and Provisional Practice PS 46; Practice E 1727; and Practice E 1553, respectively), are subjected to ultrasonic extraction in dilute nitric acid for a delineated time period.

4.2 Paint samples are ground, homogenized and weighed, if necessary, prior to ultrasonic extraction.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Abatement of Lead Hazards.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.03.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 04.07.

4.3 Soil and bulk dust vacuum samples are sieved, homogenized and weighed prior to ultrasonic extraction.

4.4 If applicable, dust filter samples are weighed prior to ultrasonic extraction.

4.5 Sample extracts are diluted with ASTM Type I water (see Specification D 1193) in preparation for subsequent determination of lead by analytical techniques (for example Test Method E 1613, Guide E 1775, and NIOSH methods 7082, 7105, and 7300 (1)<sup>5</sup>).

4.6 This practice was developed based on an EPA standard operating procedure (ultrasonic extraction followed by colorimetric lead analysis (2)) and published protocols (ultrasonic extraction followed by anodic stripping voltammetric determination of lead) (3)(4).

## 5. Significance and Use

5.1 Ultrasonic extraction using dilute nitric acid is a simpler and easier method for extracting lead from environmental samples than are traditional digestion methods that employ hot plate or microwave digestion with concentrated acids (1), (3), (5), (6). Hence, ultrasonic extraction may be used in lieu of the more rigorous strong acid/high temperature digestion methods (for example, see Ref (1) and Test Method E 1613), provided that the performance is demonstrated using accepted criteria as delineated in Guide E 1775.

5.2 In contrast with hot plate or microwave digestion techniques, ultrasonic extraction is field-portable, which allows for on-site sample preparation.

## 6. Apparatus and Materials

6.1 *Sonicator*, 50 W minimum power; proper operation of the sonicator shall be confirmed prior to initial use and periodically thereafter.

6.2 *Plastic Centrifuge Tubes*, 50 mL, with screw caps.

6.3 *Analytical Balance*, accuracy better than or equal to  $\pm 0.002$  g.

6.4 *Mortar and Pestle*, pug mill, or wiggiebug.

6.5 *Dry Ice* (CO<sub>2</sub>(s)).

6.6 *Cooler*, for storage of dry ice.

6.7 *Plastic or Glass Rods*, 0.6 to 1.0 cm diameter, 15 to 20 cm length; tapered at one end to conform to the shape of the bottoms of the 50 mL centrifuge tubes.

6.8 *ASTM Type I Water*, in accordance with Specification D 1193.

NOTE 1—Commercially available distilled or deionized water meets ASTM Type I water specifications.

6.9 *Extraction Solution*, 10 to 25 % v/v HNO<sub>3</sub>/H<sub>2</sub>O; prepared from reagent grade concentrated nitric acid (70 to 71 % HNO<sub>3</sub>) and ASTM Type I water.

6.10 *Powderless Plastic Gloves*.

6.11 *Tweezers*.

6.12 *Class A Pipets*, 5 or 10 mL or both, and other volumes as needed.

NOTE 2—Precision digital mechanical pipettors using replaceable tips

meet Class A requirements.

6.13 *Laboratory Wipes*, wet or dry, or both.

6.14 *Power Source for Sonicator*.

6.15 *Sieves*, Number 10 (1.9 mm) and 250  $\mu$ m.

6.16 *Collection Tray*.

6.17 *Rubber Mallet*.

6.18 *Nylon Brush*.

6.19 *Aluminum Pie Tins*.

6.20 *Dust Mask*.

6.21 *Glass or Plastic Stirring Rods*.

6.22 *Aluminum Foil* (conventional thickness for household use, that is, not heavy duty).

## 7. Procedure

7.1 *Testing of Sonicator*:

7.1.1 *Quarterly Testing*—Before initial use and at least quarterly thereafter, test ultrasonic extraction of lead from a bulk solid performance evaluation material that is traceable to a primary standard (for example, National Institute of Standards and Technology Standard Reference Materials, NIST SRMs) using the sonicator in the following manner:

7.1.1.1 Add a weighed amount of performance evaluation material (0.05 to 0.5  $\pm$  0.002 g) to a minimum of six 50 mL centrifuge tubes. Wear gloves while weighing is conducted.

7.1.1.2 Introduce nitric acid, either 5 mL of 25 % nitric acid or 10 mL of 10 % to 25 % HNO<sub>3</sub>, to each centrifuge tube containing weighed performance evaluation material, and the tubes capped.

7.1.1.3 Shake the centrifuge tubes briefly to ensure that no solid material remains stuck to the bottom of the tubes.

7.1.1.4 Place the centrifuge tubes in the sonicator in an upright position, and add water to the sonicator bath to a level at least 2.5 cm above the level of liquid within the tubes.

7.1.1.5 Turn on the sonicator for a period of at least 30 min to effect ultrasonic extraction of lead from the performance evaluation materials immersed in diluted nitric acid.

7.1.1.6 Then remove the centrifuge tubes from the sonicator bath.

7.1.1.7 Uncap the centrifuge carefully, and add ASTM Type I water to effect a final volume within the tubes of 50 mL. Shake tubes and allow to settle. The performance evaluation samples are now ready for the contents.

NOTE 3—The sample solutions may require filtration or centrifugation prior to analysis.

7.1.1.8 Determine lead in the performance evaluation samples according to Test Method E 1613 or an equivalent method.

7.1.1.9 The lead concentration as determined in 7.1.1.8 must be within 100  $\pm$  20 % of the certified lead concentration in the performance evaluation material (see Guide E 1775). If the lead recovery determined is outside this range, the sonicator performance does not meet the requirements of this standard.

7.1.2 *Monthly Testing*—At least monthly, demonstrate proper operation of the sonicator according to the following procedure:

<sup>5</sup> The boldface numbers given in parentheses refer to a list of references at the end of the text.

7.1.2.1 Fill the sonicator bath with warm water (ca. 45°C) to a level about half-full, and add a small amount (for example, three drops) of surfactant

7.1.2.2 Turn on the sonicator for a minimum of 5 min to degas the solutions. Turn off the sonicator.

7.1.2.3 Place aluminum foil (that is cut to a size conforming to  $\frac{1}{2}$  to  $\frac{3}{4}$  of the area of the bottom of the sonicator bath) on the bottom of the sonicator bath. Lower the foil at an angle to prevent the trapping of air beneath the foil. Ensure that a narrow layer of solution remains between the foil and the bottom of the sonicator bath. The foil shall be parallel and centered to the bottom of the sonicator.

7.1.2.4 Turn on the sonicator for a period of 45 s.

7.1.2.5 Examine the aluminum foil after sonication. The foil should be observed to contain a myriad of small holes and bumps, and may be torn apart. Also, the perforation observed should be uniform; that is, all portions of the foil should be observed to have a high density of holes and perhaps tears. If the foil is not affected in this manner, then the sonicator performance is inadequate for the purposes of this practice.

#### 7.2 *Dry Paint Film Samples:*

NOTE 4—This practice assumes paint samples collected in accordance with Practice E 1729.

7.2.1 Don a pair of gloves.

7.2.1.1 If the desired reported lead concentration units are to be in terms of lead mass per unit area of sample, and the estimated sample mass is greater than or equal to 0.25 g, then quantitatively transfer the entire sample to an analytical balance and weigh to the nearest  $\pm 0.002$  g. Under these circumstances, do quantitative transfer and sample weighing prior to grinding and homogenization as described later in this practice.

7.2.1.2 If the desired reporting units of lead concentration are to be in terms of lead mass per unit mass of sample, or if the desired reporting units are to be in terms of lead mass per unit area and the estimated mass of the sample is less than 0.25 g, then quantitatively transfer the sample to a mortar and pestle or a labeled 50 mL centrifuge tube. Use tweezers if necessary for quantitative sample transfer. Clean tweezers before and after use with laboratory wipes.

#### 7.2.2 *Sample Grinding and Homogenization:*

7.2.2.1 Grind and homogenize paint film sample to a fine powder using a mortar and pestle, pug mill, or wiggiebug. Quantitatively transfer the ground and homogenized paint sample to a weighing vessel or weighing paper (see below). Thoroughly clean mortar and pestle, wiggiebug, or pug mill with moistened laboratory wipes prior to and following grinding, and then dry.

7.2.2.2 Alternatively, place paint film sample in a clean, dry 50 mL centrifuge tube, and place the tube in dry ice. Using a plastic or metal rod, grind and homogenize the super-cooled, brittle paint sample to a fine powder. Thoroughly clean rod with laboratory wipes prior to and following grinding procedure. Avoid condensation of water onto the sample by sealing the sample and allowing it to warm to ambient temperature before weighing.

NOTE 5—The use of dry ice as described assists greatly in the grinding and homogenization of dry paint film samples.

7.2.3 If the total sample mass is greater than or equal to 0.25 g, or if the total sample mass is less than 0.25 g and the desired reported lead concentration is to be in units of mass of lead per unit mass of sample, weigh 0.05 to 0.25 g of ground and homogenized paint sample to the nearest  $\pm 0.002$  g, and record the sample mass.

NOTE 6—If the desired reporting units are to be in terms of mass of lead per unit area of sample, and the sample mass is estimated to be less than 0.25 g, a larger sample may be collected in order to obtain more mass (thereby enabling the sample to be treated as described in 7.2.1.1).

7.2.4 If the total sample mass is less than 0.25 g and the desired reported lead concentration is to be in terms of mass of lead per unit area of collected sample, then the sample need not be weighed. However, the entire ground and homogenized sample must be placed in the tube for extraction as described below.

7.2.5 Place sample in a clean, dry, labeled 50 mL polypropylene centrifuge tube. To ensure quantitative sample transfer, rinse materials used for transferring samples with a minimum of dilute (5 %) nitric acid, and direct the rinsate into the centrifuge tube.

7.2.6 Using a class A pipet, introduce 10 mL of 10 to 25 %  $\text{HNO}_3$  or 5 mL of 25 %  $\text{HNO}_3$  to the centrifuge tube containing the ground and homogenized paint sample. Shake the centrifuge tube briefly to ensure that no solid paint material remains stuck to the bottom of the tube. Rinse the insides of the centrifuge tube with a minimum quantity of 5 %  $\text{HNO}_3$  in order to ensure immersion of all solid material.

7.2.7 Cap the centrifuge tube, and place in an upright position in the sonicator bath.

7.2.8 Ensure that the bath of the sonicator contains enough water so that the water level is at least 2.5 cm above the level of liquid within the centrifuge tube.

7.2.9 Repeat steps 7.2.1-7.2.8 as needed, depending on the size of the ultrasonic bath and the number of samples.

NOTE 7—Depending on the size of the sonicator, many centrifuge tubes may be immersed in the bath at one time. A custom rack for the centrifuge tubes may be purchased or constructed to allow for the regular and orderly placement of multiple tubes in the sonicator bath.

7.2.10 Apply ultrasonic energy to the crushed and acidified samples within the immersed centrifuge tubes for at least 30 min.

7.2.11 Remove centrifuge tubes from the bath. Keep tubes in upright position.

7.2.12 Remove caps from centrifuge tubes that contain samples that were subjected to ultrasonic agitation, and dilute acidified extracts to the 50 mL mark with ASTM Type I water.

7.2.13 Re-cap and then shake the tubes for 5 to 10 s, and allow the contents to settle. The samples are now ready for analysis for lead content.

NOTE 8—The sample solutions may require filtration or centrifugation prior to analysis.

#### 7.3 *Settled Dust Wipe Samples:*

NOTE 9—This assumes dust wipe samples collected in accordance with Practice E 1728 using wipes that conform to Specification E 1792.

7.3.1 Don a pair of gloves.