



Designation: E2914/E2914M – 21

# Standard Practice for Ultrasonic Extraction of Lead from Composited Wipe Samples<sup>1</sup>

This standard is issued under the fixed designation E2914/E2914M; 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 the extraction of lead (Pb) using ultrasonication, heat and nitric acid from a composited sample of up to four individual wipe samples of settled dust collected from equally-sized areas in the same space.

1.2 This practice contains notes which are explanatory and not part of mandatory requirements of the practice.

1.3 This practice should be used by analysts experienced in digestion techniques such as hot blocks. Like all procedures used in an analytical laboratory, this practice needs to be validated for use and shown to produce acceptable results before being applied to client samples.

1.4 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system are not necessarily exact equivalents; therefore, to ensure conformance with the standard, each system shall be used independently of the other, and values from the two systems shall not be combined.

1.5 *This standard 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 standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.12 on Sampling and Analysis of Lead for Exposure and Risk Assessment.

Current edition approved Sept. 1, 2021. Published October 2021. Originally approved in 2014. Last previous edition approved in 2019 as E2914 – 19. DOI: 10.1520/E2914\_E2914M-21.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D1129 Terminology Relating to Water
- D1193 Specification for Reagent Water
- D6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals
- E1605 Terminology Relating to Lead in Buildings
- E1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques (Withdrawn 2021)<sup>3</sup>
- E1728 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination
- E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust
- E1979 Practice for Ultrasonic Extraction of Paint, Dust, Soil, and Air Samples for Subsequent Determination of Lead
- E2051 Practice for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)<sup>3</sup>
- E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities
- E2271/E2271M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Multifamily Dwellings
- E3074/E3074M Practice for Clearance Examinations Following Lead Hazard Reduction Activities in Single Family Dwellings, in Individual Units of Multifamily Dwellings, and in Other Child-Occupied Facilities

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

## 2.2 ISO Standards:<sup>4</sup>

ISO 3585 Borosilicate glass 3.3 – Properties – Third edition  
ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories

## 2.3 Other Document:<sup>5</sup>

40 CFR Part 745 Lead-Based Paint Poisoning Prevention in Certain Residential Structures

### 3. Terminology

3.1 For definitions of terms not appearing here, refer to Terminology D1129, Specification D1193, and Terminology E1605.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *composited sample, n*—the single sample resulting from the combination of individual samples collected from different sections of the same area.

3.2.2 *validation, n*—provision of objective evidence that a given item fulfills specified requirements, where the specified requirements are adequate for an intended use.

#### ISO/IEC 17025:2017

3.2.2.1 *Discussion*—The laboratory is responsible for validating (1-4)<sup>6</sup> non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application. The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.

3.2.2.2 *Discussion*—It is the responsibility of the laboratory to carry out its testing activities in such a way as to satisfy the needs of the client, the regulatory authorities, and organizations providing recognition.

### 4. Summary of Practice

4.1 Up to four wipes, meeting the requirements of Specification E1792, are used according to Practices D6966 or E1728 to collect settled dust from equally-sized areas in the same space and composited as one sample as in Practices E2271/E2271M and E3074/E3074M. This composited sample is then extracted using ultrasonication, heat and nitric acid. The resulting extract solution is analyzed according to Test Method E1613 or Practice E2051.

### 5. Significance and Use

5.1 This practice is for use in the preparation of no more than four wipe samples collected from equally-sized areas in the same space combined to form a composited sample for subsequent determination of lead content.

5.2 This practice assumes use of wipes that meet Specification E1792 and should not be used unless the wipes meet Specification E1792.

5.3 This practice is capable of preparing samples for determination of lead bound within paint dust.

5.4 This practice may not be capable of preparing samples for determination of lead bound within silica or silicate matrices, or within matrices not soluble in nitric acid.

5.5 Adjustment of the nitric acid concentration or acid strength, or both, of the final extract solution may be necessary for compatibility with the instrumental analysis method to be used for lead quantification.

5.6 This sample preparation practice has not been validated for use and must be validated by the user prior to using the practice for client samples.

NOTE 1—Each combination of wipes (two wipes, three wipes, and four wipes) constitutes a different matrix and must be separately validated.

### 6. Apparatus and Materials

6.1 *Borosilicate Glass* (conforming to ISO 3585) or *Plastic Labware*:

6.1.1 *Bottles*, 125 mL, polypropylene or equivalent, with screw caps, or equivalent,

6.1.2 *Graduated Cylinders*, 100 mL, and,

6.1.3 *Stirring Rods*, sized to reach the bottom of the bottles.

6.2 *Tweezers*, non-metallic tweezers sized to remove wipes from sample shipping containers.

6.3 *Ultrasonic Bath*, 53 watts or greater output, 1 L or greater capacity, capable of heating to at least 60 °C.

6.4 *Thermometer*, calibrated, non-mercury thermometer that covers the range from 0 °C to at least 110 °C.

6.5 *Disposable Plastic Gloves*, impermeable and powder free, to avoid the possibility of contamination, and to protect from contact with toxic and corrosive substances.

6.6 *Pencil*, with graphite tip.

6.7 *Aluminum Foil*.

6.8 *Surfactant*, liquid.

### 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available (5). Other grades may be used provided that it can be demonstrated that they are of sufficiently high purity to permit their use without decreasing the accuracy of the determinations.

7.2 *Reagent Water*—Unless otherwise indicated, reference to water shall be understood to mean Type II reagent water conforming to Specification D1193.

7.3 *Nitric Acid*:

7.3.1 Concentrated, of suitable purity for atomic spectrometric analysis, such as spectroscopic grade.

<sup>4</sup> Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

<sup>5</sup> Available from U.S. Government Printing Office (GPO), 732 N. Capitol St., NW, Washington, DC 20401, <http://www.gpo.gov>.

<sup>6</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

NOTE 2—Suitable purity means having undetectable lead content in the extracted composited sample blanks.

7.3.2 Dilute 35 % (v/v). Prepare by first adding 500 mL of reagent water to a 1 L volumetric flask and then carefully adding 350 mL concentrated nitric acid. Make to volume with reagent water.

## 8. Sample Preparation Procedure

### 8.1 Testing of Sonicator (Practice E1979):

8.1.1 Before use, ensure proper operation of the sonicator by employing one of the following diagnostic tests:

8.1.1.1 Turn on the sonicator and allow for a reasonable warm-up period, as recommended by the instrument manufacturer.

(1) Insert the tip of a graphite pencil into the bath.

(2) If the sonication device is operating properly, graphite in solution will be observed streaming off the tip of the pencil.

8.1.1.2 Alternatively, demonstrate proper operation of the sonicator according to the following procedure:

(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.

(2) Turn on the sonicator for a minimum of 5 min to degas the solution. Turn off the sonicator.

(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.

(4) Turn on the sonicator for a period of at least 45 s.

(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.

### 8.2 Sample Handling:

8.2.1 Don a pair of impermeable gloves.

8.2.2 Using clean tweezers and a clean stirring rod as needed, remove the wipes from the sample shipping container and place the wipes in a clean 125 mL bottle.

8.2.3 Using 15 mL 35 % (v/v) nitric acid, wash the inside of the sample shipping container, the tweezer tips, and the stirring rod into the bottle containing the wipes. Add the remainder of the acid to the bottle, ensuring that the wipes are immersed in the acid. Clean the tweezers and rods before and after use with laboratory wipes.

8.2.4 Cap the bottle and shake to assure that the wipes are well wetted with the acid. If bubbles are observed inside of the immersed wipe, apply pressure to the wipes with a clean stirring rod in order to force the bubbles up and out.

8.2.5 Repeat 8.2.1 to 8.2.4 for successive samples as needed, depending on the size of the ultrasonic bath.

NOTE 3—Depending on the size of the sonicator, many bottles may be immersed in the bath at one time. A custom rack for the bottles may be

purchased or constructed to allow for the regular and orderly placement of multiple bottles in the sonicator bath.

8.2.6 Preheat water in the sonicator bath to at least 60 °C.

8.2.7 Place the bottle (containing the wipes immersed in dilute acid) upright in the sonicator bath, and ensure that the water level in the bath is approximately 2.5 cm above the liquid level within the bottle.

8.2.8 Sonicate for at least 30 min at 60 °C or warmer.

8.2.9 Remove the bottles from sonicator bath and allow to cool.

8.2.10 When cool, use a clean glass or plastic stirring rod for each sample to push down on the wipes to force bubbles (formed during sonication) up and out.

8.2.11 Add 35 mL of reagent water to each bottle.

8.2.12 Recap and shake to mix.

8.2.13 Place the bottle into the sonicator such that the water in the sonicator bath is approximately 2.5 cm above the liquid level in the bottle.

8.2.14 Sonicate for at least 30 min at 60 °C or warmer. If bubbles are observed inside of the immersed wipe, apply pressure to the wipes with a clean stirring rod in order to force the bubbles up and out.

8.2.15 Remove from sonicator bath and allow to cool.

8.2.16 When cool, shake to mix. The extract is about 10 % (v/v) nitric acid.

NOTE 4—A portion of the extract may be centrifuged if it is excessively turbid or shows excess suspended solids, or both.

8.2.17 Analyze the extract according to Test Method E1613 or Practice E2051.

## 9. Equipment Preparation

9.1 Wash glassware and plastic equipment with laboratory detergent, rinse with tap water, soak for at least 4 hours in volume fraction 35 % nitric acid and water, rinse three times with ASTM Type I Water, and allow to dry preferably in a fume hood. Commercial, automatic systems are available that perform a similar process.

9.2 Alternatively, soak glassware and plastic equipment in volume/volume 1+1 nitric acid and water in a plastic tub preferably in a working hood with the hood sash down, rinse three times with ASTM Type I Water, and allowed to dry preferably in a fume hood.

## 10. Calculations

10.1 To report the total lead found, calculate the total leaded dust found on the surfaces sampled, [ $\mu\text{g Pb}$ ].

$$[\mu\text{g Pb}] = (C)(50) \quad (1)$$

where:

$C$  = extract lead concentration analytically found; in micrograms per millilitre, [ $\mu\text{g Pb/mL}$ ],

50 = final digestate volume, in millilitres, 50 mL, and

[ $\mu\text{g Pb}$ ] = total amount of lead found on the surfaces sampled; in micrograms, [ $\mu\text{g Pb}$ ].

10.2 To report the results as leaded dust loading per unit area: