Designation: E2914 – 14

Standard Practice for Ultrasonic Extraction of Lead from Composited Wipe Samples[1]

This standard is issued under the fixed designation E2914; 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 (ε) 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 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 SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4.1 *Exception*—Inch-pound units are provided in Note 6 and Note 8 for information.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

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

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for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)[3]

E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities

2.2 *ISO Standards:*[4]

ISO/IEC 17025:2005 General Requirements for the Competence of Testing and Calibration Laboratories

ISO 3585 Borosilicate Glass 3.3 – Properties – Third Edition

2.3 *Other Document:*[5]

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

3.2.2 *validation*—the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled ISO 17025:2005 (5.4.5.1).

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[1] This practice is under the jurisdiction of ASTM Committee E06 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Hazards Associated with Buildings.

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

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

[5] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

3.2.3 The laboratory is responsible for validating[6,7,8,9] 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.

NOTE 1—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 Practice E1728 to collect settled dust from equally-sized areas in the same space and composited as one sample. 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 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 2—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 *Reagent Water*—ASTM Type I water as given in Specification D1193 with minimum resistance of 16.67 megaohm-cm, or equivalent.

7.2 *Nitric Acid:*

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

NOTE 3—Suitable purity means having undetectable lead content in the extracted composited sample blanks.

7.2.2 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 ½ to ¾ 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.

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[6] Kennedy, Ph.D., Eugene R., Fischbach, Thomas J., Song, Ph.D., Ruiguang, Eller, Ph.D., Peter M., and Schulman, Ph.D., Stanley A., *Guidelines for Air Sampling and Analytical Method Development*, DHHS (NIOSH), Publication No. 95-117, 1995.

[7] Green, J. Mark, *A Practical Guide to Analytical Method Validation*, Analytical Chemistry, 1996, (68) 305A-309A.

[8] The Fitness for Purpose of Analytical Methods-A Laboratory Guide to Method Validation and Related Topics, EURACHEM Guide 1st Ed, 1998.

[9] Harmonized Guidelines for Single Laboratory Validation of Methods of Analysis (IUPAC Technical Report) Pure Appl. Chem., Vol. 74, No. 5, pp. 835–855, 2002.

(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 4—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 5—Centrifuge a portion of the extract 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. Calculations

9.1 To report the total lead found, calculate the total leaded dust found on the surfaces sampled, [μg Pb].

$$[\mu g\ \ Pb] = (C)(50) \qquad (1)$$

where:

C = extract lead concentration analytically found; in micrograms per millilitre, [μg Pb/mL]
50 = final digestate volume, in millilitres, 50 mL
[μg Pb] = total amount of lead found on the surfaces sampled; in micrograms, [μg Pb]

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

9.2.1 Determine the total area sampled (up to four areas per composited sample) by summing the individual areas sampled. All measurements are to be done in centimetres, [cm], with resulting areas in square centimetres, [cm^2].

$$A_T = a_1 + ... + a_n \qquad (2)$$

where:

A_T = total area sampled, in square centimetres, [cm^2]
a = area sampled by wipes to be composited, in square centimetres, [cm^2], where the maximum total number of wipes is four ($n \leq 4$)

9.2.2 Divide the mass of lead found [μg Pb] by the total area sampled A_T to determine the leaded dust loading as determined by composited wipe sampling.

$$[\mu g\ \ Pb/\ cm^2] = [\mu g\ \ Pb]/\ \ A_T \qquad (3)$$

where:

[μg Pb] = total leaded dust found on the surfaces sampled
[μg Pb/cm^2] = leaded dust loading as determined by composited wipe sampling

NOTE 6—To determine the average leaded dust loading for the areas sampled, the individual areas sampled must be the same size, for example, 929 cm^2 (1 ft^2) each. The use of English units is for informational purposes.

NOTE 7—Consult the laws, regulations and requirements of the local authority having jurisdiction for any required method of calculation of lead loading.

NOTE 8—In the United States of America (per 40 CFR Part 745), the average lead loading for a space is to be reported in units of micrograms of lead per square foot, [μg Pb/ft^2]. For example: For a three-wipe composited sample with each collected from a 929 cm^2 (1 ft^2) area, divide the reported results in units [μg Pb/cm^2] by 2787 (= 3 x 929) since 929 cm^2 = 1 ft^2. The use of English units is for informational purposes.

10. Records

10.1 Each analyst and technician shall record their sample preparation activities in a laboratory notebook or an electronic record.

10.1.1 Laboratory notebooks, if not electronic records, shall be bound with pre-numbered pages.

10.1.2 All entries in laboratory notebooks, if not electronic records, shall be made using ink with signature and date of entry.

10.1.3 Any entry errors in laboratory notebooks shall be corrected by using only a single line through the incorrect entry (no scratch outs) accompanied by the initials of the person making the correction and the date of correction.

10.1.4 If an electronic record is used, all corrections made shall be indicated in the record with the original data entry available for recovery. These changes shall be in some way indicated in the final record and final report as a change to the original entry.

10.2 Record all information regarding the preparation of all samples in a laboratory notebook or an electronic record.

10.2.1 Record all reagent sources (lot numbers) used for sample preparation.

10.2.2 Record any inadvertent deviations or unusual circumstances, events, and observations on a real-time basis as samples are processed.

10.3 Records shall be maintained in accordance with Practice E2239, and shall include a copy of the laboratory final report.

11. Reporting

11.1 Report the following information:

11.2 Report results as total lead found in units of micrograms of lead, [µg Pb], or as lead found per unit area in units of micrograms of lead found per square centimetre, [µg Pb/cm^2], or both. See Notes 7 and 8.

12. Keywords

12.1 lead; sample preparation; settled dust; ultrasonic extraction; wipe sample

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