Designation: E2913 – 14

Standard Practice for
Hotplate Digestion of Lead from Composited Wipe Samples[1]

This standard is issued under the fixed designation E2913; 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 is similar to Practice E1644 and covers the hot, nitric acid digestion of lead (Pb) 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 7 and Note 9 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
E1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead
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
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for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)[3]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 1042 Laboratory Glassware – One-mark Volumetric Flasks
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.

Current edition approved May 1, 2014. Published June 2014. DOI: 10.1520/E2913-14

[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 digested using 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 Glassware:*

6.1.1 *Volumetric Flasks,* 400 mL, borosilicate, with Stoppers, conforming to ISO 1042 and ISO 3585,

6.1.2 *Griffin Beakers,* 250 mL,

6.1.3 *Watch Glasses,* sized to cover the Griffin Beakers,

6.1.4 *Graduated Cylinder,* 100 mL, and,

6.1.5 *Stirring Rods,* sized to reach the bottom of the Griffin Beakers.

6.2 *Funnels,* plastic, porcelain or borosilicate funnels sized to deliver filtrate into a 400 mL volumetric flask.

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

6.4 *Filter Paper,* fast filtering, suitable for metals analysis.

6.5 *Electric Hotplates,* suitable for operation at temperatures that allow digestion of nitric acid and hydrogen peroxide solutions encountered in this practice.

6.6 *Wash Bottles,* of suitable size and material to contain 1:1 (v/v) nitric acid solution or reagent water.

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

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 1:1 (v/v); prepared by carefully pouring a volume of concentrated acid into an equal volume of reagent water.

7.3 *Hydrogen Peroxide*—30 % (w/w), suitable for atomic spectrometric analysis, such as spectroscopic grade. See Note 3.

8. Sample Preparation Procedure

8.1 Don a new pair of impermeable gloves. Treat each composited sample in a batch equally.

8.2 Quantitatively transfer the contents of the dust-wipe sample containers to a labeled beaker as follows:

8.2.1 Carefully open each container, remove wipes using a clean tweezers and a clean stirring rod as needed, and place all the wipes comprising the composited sample into a clean 250 mL beaker.

8.2.2 If the sample containers are hard-walled, rigid containers such as a plastic centrifuge tubes, using a minimum volume of solution, wash the inside of the sample shipping container, the tweezer tips and the stirring rod with 1:1 (v/v) nitric acid into the beaker containing the wipes.

8.2.3 If the sample containers are flexible plastic bags and material appears to be left behind after wipe removal, attempt to transfer the residual material into the beaker by shaking or using mechanical removal with a clean laboratory spatula or

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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.

similar tool. Document observations of potential loss of sample due to any residue in the container for later reporting with the results of the lead analysis.

8.3 Slowly add 60 mL of concentrated nitric acid to the beaker. Assuring that all wipes are immersed, gently swirl to mix, and cover with a clean watch glass. Digest on a hotplate to a solution volume of about 8 mL. Do not digest to dryness. (**Warning—**Some wipes break down into a gelatinous residue that readily bumps or spatters, or both. Heating should be slowed with these materials.)

8.4 Remove from the hotplate and allow to cool.

8.5 When cool, carefully remove the watch glass and slowly add 40 mL of 30 % (w/w) hydrogen peroxide, assuring that all wipes are immersed.

NOTE 4—Exercise care when removing the watch glass. Avoid lead contamination problems by placing it upside down on new clean laboratory wipes.

8.6 Cover the beaker with the watchglass and return the covered beaker to the hot plate. Take care to ensure that losses do not occur due to vigorous effervescence during heating. Heat until effervescence subsides and then digest to a solution volume of about 8 mL. Do not digest to dryness.

8.7 Remove the digestate from the hotplate and allow to cool.

8.8 Assemble funnels, filter paper and volumetric flasks as needed to allow filtering of digestates into 400 mL volumetric flasks.

NOTE 5—Do not attempt to pour the undiluted digestate through the filter paper as the filter paper will dissolve in strong acid solutions.

8.9 When cool, use reagent water to wash the bottom of the watch glass, the inside walls of the beaker, and any remaining wipe material to the bottom of the beaker. Use about 80 mL of reagent water to accomplish the washing. Pour the diluted digestate through the filter paper.

8.10 Using reagent water and a clean glass stirring rod, break up and wash the mass of any remaining wipe material in the bottom of the beaker three times. Use enough reagent water each time to at least cover the remaining wipe material in the beaker. Use about 150 mL to accomplish the washing. Pour the first two washings through the filter paper, and use the third to wash any remaining wipe material from the beaker and onto the filter paper.

8.11 Using reagent water, wash the glass stirring rod and the beaker at least three times. Pour the washings through the filter paper.

8.12 Using reagent water, wash the filter paper, any remaining wipe material on the filter paper, the funnel, and the funnel stem into the volumetric flask.

8.13 Add reagent water to the volumetric flask to the mark, cap and invert to mix. The resulting digestate solution is about 2 % (v/v) nitric acid. Calibration standards used for instrumental measurement should be made with this level of nitric acid.

NOTE 6—The wipe material may or may not be completely solubilized. Many types of wipes contain materials that do not dissolve in nitric acid.

8.14 Analyze the digestate 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].

$$[\mu g\ \ Pb] = (C)(400) \tag{1}$$

where:

C = digestate lead concentration analytically found; in micrograms of lead per millilitre, [µg Pb/mL]

400 = final digestate volume, in millilitres, 400 mL

[µg Pb] = total leaded dust found on the surfaces sampled; in micrograms of lead, [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].

$$A_T = a_1 + ... + a_n \tag{2}$$

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.

$$[\mu g\ \ Pb/\ cm^2] = [\mu g\ \ Pb]/\ \ A_T \tag{3}$$

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 7—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 8—Consult the laws, regulations and requirements of the local authority having jurisdiction for any required method of calculation of lead loading.

NOTE 9—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 8 and 9.

12. Keywords

12.1 hotplate digestion; lead; sample preparation; settled dust; wipe sample

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