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for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)[3]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

[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 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.