



Designation: E2913/E2913M – 21

Standard Practice for Hotplate Digestion of Lead from Compositing Wipe Samples¹

This standard is issued under the fixed designation E2913/E2913M; 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 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.*

2. Referenced Documents

2.1 ASTM Standards:²

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

E631 Terminology of Building Constructions

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)³

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

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

2.2 ISO Standards:⁴

ISO 1042 Laboratory Glassware – One-mark Volumetric Flasks

ISO 3585 Borosilicate Glass 3.3 – Properties – Third Edition
ISO/IEC 17025:2017 General Requirements for the Competence of Testing and Calibration Laboratories

¹ 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.

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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), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

2.3 *Other Document*:⁵

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

3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, refer to Terminology **D1129**, Specification **D1193**, and Terminologies **E631** and **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 17025:2017

3.2.2.1 *Discussion*—The laboratory is responsible for validating **(1-4)**⁶ 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.

3.2.3

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 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 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 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 *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.

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

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

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

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

7.4 *Hydrogen Peroxide*—30 % (w/w), suitable for atomic spectrometric analysis, such as spectroscopic grade. See [Note 2](#).

8. Equipment Preparation

8.1 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.

8.2 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 allow to dry preferably in a fume hood.

9. Sample Preparation Procedure

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

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

9.2.1 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.

9.2.2 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.

9.2.3 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 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.

9.3 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.)

9.4 Remove from the hotplate and allow to cool.

9.5 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 3—Care should be exercised when removing the watch glass. Lead contamination problems may be avoided by placing the watch glass upside down on new clean laboratory wipes.

9.6 Cover the beaker with the watch glass 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.

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

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

NOTE 4—The undiluted digestate is a strong acid solution and may dissolve the filter paper if poured through it.

9.9 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.

9.10 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.

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

9.12 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.

9.13 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 5—The wipe material may or may not be completely solubilized. Many types of wipes contain materials that do not dissolve in nitric acid.

9.14 Analyze the digestate according to Test Method [E1613](#) or Practice [E2051](#).

10. Calculations

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

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

where:

C = digestate lead concentration analytically found; in micrograms of lead per millilitre, [$\mu\text{g Pb/mL}$],
 400 = final digestate volume, in millilitres, 400 mL, and
 $[\mu\text{g Pb}]$ = total leaded dust found on the surfaces sampled; in micrograms of lead, [$\mu\text{g Pb}$].

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

10.2.1 Determine the total area sampled (up to four areas per composited sample) by summing the individual areas sampled.