



Designation: E1644 – 04

Standard Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead¹

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1. Scope

1.1 This practice covers the acid digestion of surface dust samples (collected using wipe sampling practices) and associated quality control (QC) samples for the determination of lead.

1.2 The values stated in SI units are to be regarded as the standard.

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

1.4 *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](#)

[E1724 Guide for Testing and Certification of Metal, Ore, and Metal-Related Reference Materials](#)

[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](#)

[E2239 Practice for Record Keeping and Record Preservation for Lead Hazard Activities](#)

2.2 *Other Documents:*

[EPA SW 846, Method 3050](#), “Acid Digestion of Sediments, Sludges, and Soils.” This method is found in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, U.S. EPA SW 846, 3rd Edition, Revision 1, 1987³

[NIOSH Manual of Analytical Methods](#), NIOSH 7082 and 7105, Eller, P.M., Ed., 3rd ed., 1984³

3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, refer to Terminologies [D1129](#) and [E1605](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *blank wipe*—an unused, unspiked dust wipe that is only removed from its packaging immediately before use.

3.2.1.1 *Discussion*—Blank wipes are used to prepare non-spiked, spiked, and spiked duplicate quality assurance samples.

3.2.2 *dust wipe sample*—surface dust collected on a wipe.

3.2.3 *method blank*—a digestate that reflects the maximum treatment given any one sample within a sample batch except that only the sampling medium (such as a blank wipe) is initially placed into the digestion vessel. (The same reagents and processing conditions that are applied to field samples within a batch are also applied to the method blanks.)

3.2.3.1 *Discussion*—Analysis results from method blanks provide information on the level of potential contamination resulting from the laboratory and sampling medium sources that are experienced by samples processed within the batch.

3.2.4 *non-spiked sample*—a portion of a homogenized sample that was targeted for the addition of analyte but is not fortified with the target analytes before sample preparation.

3.2.4.1 *Discussion*—For wipe samples, a non-spiked sample is equivalent to a method blank. Analysis results for this sample are used to correct for background levels in the blank wipes used for spiked and spiked duplicate samples.

3.2.5 *reagent blank*—a digestate that reflects the maximum treatment given any one sample within a sample batch except

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 Available from National Technical Information Service (NTIS), U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161.

that it has no sample placed into the digestion vessel. (The same reagents and processing conditions that are applied to field samples within a batch are also applied to the reagent blank.)

3.2.5.1 *Discussion*—Analysis results from reagent blanks provide information on the level of potential contamination resulting from only laboratory sources that are experienced by samples processed within the batch.

3.2.6 *spiked sample and spiked duplicate sample*—a blank wipe that is spiked with a known amount of analyte (that is, lead) before hot plate digestion and subsequent lead analysis.

3.2.6.1 *Discussion*—Analysis results for these samples are used to provide information on accuracy and precision of the overall analysis process.

4. Summary of Practice

4.1 A dust wipe sample is digested using hot plate type heating with nitric acid and hydrogen peroxide. The digestate is diluted to final volume prior to lead measurement.

NOTE 1—The procedure in this practice is based on U.S. EPA SW846 Method 3050, NIOSH 7082, and NIOSH 7105.

5. Significance and Use

5.1 This practice is intended for the digestion of lead in dust wipe samples collected during various lead hazard activities performed in and around buildings and related structures.

5.2 This practice is also intended for the digestion of lead in dust wipe samples collected during and after building renovations.

5.3 This practice is applicable to the digestion of dust wipe samples that have or have not been collected in accordance with Practice E1728 using wipes that may or may not conform to Specification E1792.

5.4 This practice is applicable to the digestion of dust wipe samples that were placed in either hard-walled, rigid containers such as 50-mL centrifuge tubes or flexible plastic bags.

NOTE 2—Due to the difficulty in performing quantitative transfers of some samples from plastic bags, hard-walled rigid containers such as 50-mL plastic centrifuge tubes are recommended for sample collection.

5.5 Digestates prepared according to this practice are intended to be analyzed for lead concentration using spectrometric techniques such as Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) and Flame Atomic Absorption Spectrometry (FAAS) (see Test Method E1613), or using electrochemical techniques such as anodic stripping voltammetry (see Practice E2051).

5.6 This practice is not capable of determining lead bound within matrices, such as silica, that are not soluble in nitric acid.

5.7 This practice is capable of determining lead bound within paint.

6. Apparatus and Materials

6.1 Borosilicate Glassware:

6.1.1 *Class A Volumetric Flasks with Stoppers*, 100 mL and other sizes needed to make serial dilutions,

6.1.2 *Griffin Beakers*, 150 mL or 250 mL,

6.1.3 *Watch Glasses*, sized to cover Griffin beakers,

6.1.4 *Class A Pipets*, as needed to make serial dilutions, and
6.1.5 *Glass Rods*.

6.2 *Funnels*—Plastic or porcelain or borosilicate funnels sized to fit into a 100-mL volumetric flask.

6.3 *Filter Paper*—Fast filtering, suitable for metals analysis.

6.4 *Thermometers*—Red alcohol or thermocouple, that covers a range of 0 to 150°C.

6.5 *Electric Hot Plate*—Suitable for operation at temperatures up to at least 100°C (see Note 3).

NOTE 3—Provided that the hot plate is capable of handling the extra heating required, use of a 12 to 25-mm (0.5 to 1-in.) thick aluminum plate placed on the burner head can help reduce the presence of hot spots common to electric hot plates.

6.6 *Vinyl Gloves*—Powderless.

6.7 *Micropipettors with Disposable Plastic Tips*—Sizes needed to make reagent additions, and spike standards. In general, the following sizes should be readily available: 1 to 5 mL adjustable, 100 μ L, 500 μ L, 250 μ L, and 1000 μ L.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this practice. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening accuracy of the determination.

7.2 *Nitric Acid*—Concentrated, suitable for atomic spectrometry analysis such as spectroscopic grade.

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

7.4 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 of Specification D1193.⁵

7.5 *Calibration Stock Solution*—100 μ g/mL of Pb in dilute nitric acid.

8. Sample Preparation Procedure

8.1 Sample Extraction:

8.1.1 Treat each sample in a batch equally.

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

8.1.2.1 Carefully open the container, remove the folded wipe using a new pair of plastic gloves or plastic forceps, or both, and place it into the Beaker.

8.1.2.2 If the sample container is a hard-walled, rigid containers such as a plastic centrifuge tube, rinse out the inside of the container into the beaker with two small volumes (2 to 3 mL) of water using a squirt bottle filled with ASTM Type I water.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁵ ASTM Type I Water: Minimum resistance of 16.67 megohm-cm, or equivalent.