Designation: E1644 – 17

Standard Practice for
Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead[1]

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 standard. No other units of measurement are included in this standard.

1.2.1 *Exception*—Informational inch-pound units are provided in Note 3.

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

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for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)[3]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 *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, 1987[4]

NIOSH Manual of Analytical Methods, NIOSH 7082 and 7105, Eller, P.M., Ed., 3rd ed., 1984[4]

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.

3.2.3.1 *Discussion*—The same reagents and processing conditions that are applied to field samples within a batch are also applied to the method blanks so that analysis results 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.

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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 National Technical Information Service (NTIS), 5301 Shawnee Rd., Alexandria, VA 22312, http://www.ntis.gov.

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 that it has no sample placed into the digestion vessel.

3.2.5.1 *Discussion*—The same reagents and processing conditions that are applied to field samples within a batch are also applied to the reagent blank so that analysis results 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 µL, 500 µL, 250 µL, and 1000 µL.

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.[5] 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.[6]

7.5 *Calibration Stock Solution*—100 µg/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.

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[5] *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. (USP), Rockville, MD.

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

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.

8.1.2.3 If the sample container is a flexible plastic bag 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 residue in the container for later reporting with the results of the lead analysis.

8.1.3 Add 25 mL of 1:1 nitric acid:water to the beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85 to 100°C using the hot plate and reflux for 10 to 15 min without boiling. Estimate and monitor the temperature of the sample solution by having a thermometer inside a beaker or flask containing a small volume (about 25 mL) of water on the hot plate (see Note 4).

NOTE 4—A hot plate surface temperature of 120 to 140°C will yield a sample digestate temperature of 85 to 100°C.

8.1.4 Using a glass rod, push the wipe down into the digestion solution periodically to effect efficient extraction. (**Warning—**Some wipes break down into a gelatinous residue that readily bumps or spatters, or both. Heating should be slowed with these materials.)

8.1.5 Allow the sample to cool to near room temperature, add 10 mL of concentrated nitric acid, replace the watch glass, and reflux for 30 min without boiling.

8.1.6 Remove the watch glass and allow the solution to evaporate to approximately 10 mL without boiling (see Notes 5 and 6). Allow the sample to cool to near room temperature after evaporation to approximately 10 mL.

NOTE 5—Exercise care when removing the watch glass. Avoid lead contamination problems by placing it upside down on new clean laboratory wipes.

NOTE 6—Boiling of the sample should be avoided because of potential sample losses due to splattering and cross-contamination problems. The same problems can be experienced if samples are allowed to evaporate to dryness.

8.1.7 After the sample has cooled to near room temperature, add 5 mL of water and 5 mL of 30 % hydrogen peroxide. Cover the beaker with the watchglass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Take care to ensure that losses do not occur due to vigorous effervescence during heating. Heat until effervescence subsides and cool the beaker to near room temperature.

8.1.8 Remove the watch glass and continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 10 mL (see Notes 5 and 6).

8.1.9 Allow the digestate to cool to near room temperature, rinse the beaker walls and bottom of the watch glass with water, and quantitatively transfer through a funnel equipped with a fast filter into a 100-mL volumetric flask (see Note 6). If a large amount of undissolved material remains in the beaker, rinse this residue as many times as necessary to transfer solubilized lead into the 100 mL volumetric flask. Dilute to volume with water and swirl to mix.

8.1.9.1 The diluted digestate solution contains approximately 10 % (v/v) nitric acid. Calibration standards used for instrumental measurement should be made with this level of nitric acid.

NOTE 7—The wipe material may or may not be completely solubilized. Many types of wipes contain materials that do not dissolve in nitric acid.

9. Quality Assurance

9.1 *Quality Control Samples:*

9.1.1 *Reagent Blank Samples*—Process reagent blanks according to the frequency listed in Table 1. Submit reagent blanks to the entire dust-wipe sample preparation and digestion process to determine through subsequent lead analysis if the samples are being contaminated from laboratory activities.

9.1.2 *Non-Spiked, Spiked, and Spiked Duplicate Samples*—Process non-spiked, spiked, and spiked duplicate samples (that is, blank wipes) at the frequency listed in Table 1, expressed through subsequent analysis as percent lead recovery relative to the true spiked value. Use blank wipes provided by field personnel who submitted the dust wipe samples (see Note 8). If the number of blank wipes provided by field personnel is insufficient, use wipes that conform to Specification E1792.

9.1.2.1 To the extent possible, the brand or type of blank wipe used should be the same as that used for the collection of the dust wipe samples.

9.1.2.2 Since wipe samples cannot be split uniformly, blank wipes are used for non-spiked, spiked, and spiked duplicate samples.

9.1.2.3 *Preparation of Spiked and Spiked Duplicate Samples*—For a blank wipe targeted for spiking, add an appropriate volume of a lead standard stock solution to a beaker that contains a blank wipe (see Note 9). In the absence of other information, add 2 mL of the 100 mg/mL calibration stock solution (that is, 200 mg of lead) to the beaker with the blank wipe.

TABLE 1 Quality Control Samples

QC Samples	Frequency
Non-spiked sample or method blank	1 per 20 samples, a minimum of 1 per batch
Reagent blank	1 per batch
Spiked sample	1 per 20 samples, a minimum of 1 per batch
Spiked sample duplicate	1 per 20 samples, a minimum of 1 per batch
Certified reference material (CRM)	1 per batch of samples

NOTE 8—Laboratory personnel generally have little control over the number of blank wipes that are submitted along with a sample batch. Nevertheless, to the extent possible, laboratory personnel should advise the individuals responsible for the field sampling to submit a sufficient number of blank wipes to permit preparation and digestion of the quality assurance samples at the frequency listed in Table 1.

NOTE 9—The appropriate volume will be dependent on the anticipated average lead level in the wipe samples within a given batch. The optimum spike addition is one that will match the average lead level in the batch of wipes. Use of multiple spike levels such as 400 mg and 800 mg may also be useful for sample batches containing a wide range of lead levels.

9.1.2.4 Perform the digestion of the non-spiked, spiked, and spiked duplicate samples according to 8.1.3 – 8.1.9.

9.1.3 *Certified Reference Material (CRM) Samples*—Process CRM samples according to the frequency listed in Table 1, to determine an estimate of method accuracy on the sample batch, expressed through subsequent analysis as percent lead recovery relative to the certified value.

9.1.3.1 Use a CRM that has a matrix similar or identical to dust with a certified lead concentration level.

9.1.3.2 *Preparation of CRM Samples*—Place a known quantity of the CRM into a blank wipe and process along with other samples. The brand or type of blank wipe used should be the same as that used for the collection of the dust wipe samples. If the brand or type of blank wipe is unknown, use a wipe conforming to Specification E1792 (see Note 8).

9.1.3.3 Perform the digestion of CRM samples according to 8.1.3 – 8.1.9.

10. Laboratory Records

10.1 Laboratory records made during the preparation and digestion of dust wipe samples are used to add supplemental information when reporting results of subsequent lead analysis.

10.2 Record all information regarding preparation and digestion of both dust wipe samples and quality assurance samples including:

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

10.2.2 For each entry, the date(s) and identification and signature(s) of the person(s) making the entry.

10.2.3 Inadvertent deviations, unusual occurrences, or observations on a real-time basis as samples are processed. Use the records to add supplemental information when reporting results.

11. Record Keeping

11.1 Records shall be maintained in accordance with Practice E2239.

12. Keywords

12.1 digestion; lead; sample preparation; wipes