Designation: E 1644 – 98

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

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1. Scope

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

1.2 This practice is based on U.S. EPA SW846 Method 3050, NIOSH 7082 and NIOSH 7105.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 1129 Terminology Relating to Water[2]

D 1193 Specification for Reagent Water[2]

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures[3]

E 1724 Guide for Testing and Certification of Reference Materials[4]

E 1792 Specification for Wipe Sampling Materials for Lead in Surface Dust[3]

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[5]

NIOSH Manual of Analytical Methods, NIOSH 7082 and 7105, Eller, P.M., Ed., 3rd ed., 1984[5]

3. Terminology

3.1 *Definitions*—For definitions of terms relating to this practice that do not appear in this section, refer to Terminology D 1129 and E 1605.

3.1.1 *batch*—a group of field or quality control (QC) samples that are processed together using the same reagents and equipment.

3.1.2 *certified reference material (CRM)*—reference material accompanied by a certificate, of which one or more of its property values are certified by a procedure that establishes its traceability to an accurate realization of the unit in which the property values are expressed. **E 1724**

3.1.3 *digestate*—an acidified aqueous solution that results from digestion of the sample.

3.1.4 *digestion*—the sample preparation process that solubilizes (extracts) targeted analytes present in the sample and results in an acidified aqueous solution called the digestate; equivalent to extraction.

3.1.5 *dust wipe sample*—a settled dust sample collected on a moistened disposable towellette (see *wipe*).

3.1.6 *extraction*—the dissolution of target analytes from a solid matrix into a liquid form. During sample digestion, target analytes are extracted (solubilized) into an acid solution.

3.1.7 *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.) 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.

3.1.8 *non-spiked sample*—a blank wipe sample that was targeted for addition of analyte but was not fortified with all the target analytes before sample preparation.

3.1.8.1 *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.

3.1.9 *reagent blank*—a digestate that reflects the maximum treatment given any one sample within a sample batch except that it has no sample 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 reagent blank.)

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[1] This practice is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.23 on Lead Paint Abatement.

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[2] *Annual Book of ASTM Standards,* Vol 11.01.

[3] *Annual Book of ASTM Standards,* Vol 04.11.

[4] *Annual Book of ASTM Standards,* Vol 03.06.

[5] Available from National Technical Information Service, 5285 Port Royal Rd., Springfield, VA 22161.

3.1.9.1 *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.

3.1.10 *spiked sample and spiked duplicate sample*—a spiked sample (or spiked duplicate sample) is a blank wipe that is spiked with a known amount of analyte before preparation.

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

3.1.11 *wipe*—a disposable, porous paper (cellulosic) towellette that is moistened with a wetting agent. **E 1792**

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 for final volume prior to lead measurement.

5. Significance and Use

5.1 This practice is intended for the determination of lead in dust wipe samples that have been collected during various construction and renovation practices in and around buildings and related structures.

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

5.3 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 as measured by a thermometer inside a solution-filled container placed on the surface of the hot plate (see Note 1) or a hot plate surface temperature of up to 150°C.

NOTE 1—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, 1000 µL, 500 µL, 250 µL, and 100 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.[6] 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 D 1193.[7]

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.

8.1.2 Quantitatively transfer the contents of the sample container to the labeled beaker as described as follows:

8.1.2.1 For blank wipe targeted for spiking (see Section 9), add the appropriate volume of a lead standard stock to the beaker (see Note 2). In the absence of other information, add 200 µg of lead to each beaker containing the blank wipes targeted for spike duplicates (2 mL of 100 µg/mL Pb stock solution).

NOTE 2—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 µg and 800 µg may also be useful for sample batches containing a wide range of lead levels.

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

8.1.2.3 If the sample container is a plastic centrifuge tube, then 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.

8.1.2.4 If the sample container is a plastic bag and material appears to be left behind in the container and the sample container, then attempt to transfer the material into the beaker using shaking or mechanical removal with a clean laboratory tool such as a spatula. Document observations in laboratory records of potential lost sample remaining in the container for later reporting with laboratory analysis results. Due to the difficulty in performing quantitative transfers from plastic bags for some samples, hard containers such as plastic 50-mL centrifuge tubes are recommended for sample collection.

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[6] *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.

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

8.1.3 Add 25 mL of 1 : 1 nitric acid : water to each beaker, gently swirl to mix, and cover with a watchglass. Gently heat the sample to 85°C to 100°C and reflux for 10 to 15 min without boiling. Monitor the temperature by having a thermometer inside a beaker or flask containing a small volume of water on the hot plate.

NOTE 3—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. CAUTION—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 watchglass and allow the solution to evaporate to approximately 10 mL without boiling (see Notes 4 and 5). Allow the sample to cool to near room temperature after evaporation to approximately 10 mL.

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

NOTE 5—Boiling of the sample should be avoided because of potential sample splattering losses 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 a 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 excessively vigorous effervescence during heating. Heat until effervescence subsides and cool the beaker to near room temperature.

8.1.8 Remove the watchglass and continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 10 mL (see Notes 4 and 5).

8.1.9 Allow the digestates to cool to near room temperature, rinse the beaker walls and bottom of the watchglass with water, and quantitatively transfer through a funnel equipped with a fast filter into a 100-mL volumetric flask (see Note 6). Dilute to volume with water and swirl to mix. 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 6—The sample media (wipe material) may or may not be completely solubilized. Many types of wipes contain materials that do not dissolve in nitric acid. If a large amount of undissolved material remains, rinse the solids as many times as possible to transfer solubilized lead into the volumetric flask.

NOTE 7—Some wipes leave a significant amount of residue. It may be necessary to filter the digestate before dilution in order to remove this material.

9. Quality Assurance

9.1 *Quality Control Samples*—Quality control samples to process with each batch of samples are summarized in Table 1.

9.1.1 *Reagent Blanks*—Carry reagent blanks (water and reagents) throughout the entire sample preparation and analytical process to determine if the samples are being contaminated from laboratory activities. Process reagent blanks according to the frequency listed in Table 1.

9.1.2 *Non-Spiked Samples, Spiked Samples and Spiked Duplicate Samples*—Process non-spiked, spiked and spiked duplicate samples on a routine basis to estimate method accuracy on the sample batch, expressed as percent recovery relative to the true spiked value. Since wipe samples cannot be split uniformly, blank wipes are used for non-spike, spikes and spike duplicates. The brand or type of wipe used should be the same as that used for the collection of the samples. Field personnel should submit sufficient numbers of blank wipes to the laboratory to permit generation of these QC samples at the frequency listed in Table 1.

9.1.3 *Certified Reference Materials*—Process certified or Standard Reference Materials (CRMs) on a routine basis to determine an estimate of method accuracy on the sample batch, expressed as percent recovery relative to the certified value. Incorporate SRMs or CRMs into each analytical batch according to the frequency listed in Table 1. Use a CRM that has a matrix similar or identical to dust with a certified lead concentration level. Place a known quantity of the CRM into a blank wipe and process along with other samples. The brand or type of wipe used should be the same as that used for the collection of the samples. Field personnel should submit sufficient numbers of blank wipes to the laboratory to generate these QC samples.

9.2 *Laboratory Records*—Record all information regarding the preparation of samples (both QC samples and those submitted to the analyst) as follows:

9.2.1 Record all reagent sources (lot numbers) used for sample preparation. For each entry, include the date(s) and

TABLE 1 Quality Control Samples

QC samples	Definition	Frequency
Method blank or Non-spiked sample	A blank wipe carried through sample preparation along with other amples. Should reflect the maximum treatment given any one ample within the batch.	1 per 20 samples, a minimum of 1 per batch
Reagent blank	Type I water—digest as a sample with addition of all reagents. Should reflect the maximum treatment given any one sample within the batch.	1 per batch
Spiked sample	A blank wipe fortified with all the target analytes before preparation.	1 per 20 samples, a minimum of 1 per batch
Spiked sample duplicate	A blank wipe fortified with all the target analytes before preparation.	1 per 20 samples, a minimum of 1 per batch
Reference material	A material of known composition where the analyte levels are certified by the manufacturer.	1 per batch of samples

identification and signature(s) of the person(s) making the entry. Record any 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.

9.2.2 Laboratory notebooks must be bound with prenumbered pages, and all entries must be made in ink. Any entry errors must be corrected by using only a single line through the incorrect entry, accompanied by the initials of the person making the correction, and the date of the correction.

10. Keywords

10.1 digestion; lead; sample preparation; wipes