Designation: ~~E1726 – 16~~ E1726 – 20

Standard Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis[1]

This standard is issued under the fixed designation E1726; 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 covers drying, homogenization, and acid digestion of soil samples and associated quality control (QC) samples using a hot plate type method for the determination of lead using laboratory atomic spectrometry analysis techniques such as ~~Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), and Graphite Furnace Atomic Absorption Spectrometry~~inductively coupled plasma atomic emission spectrometry (ICP-AES), flame atomic absorption spectrometry (FAAS), and graphite furnace atomic absorption spectrometry (GFAAS).

1.2 This practice is based on U.S. EPA SW 846, Test Method 3050.

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

1.4 The values stated in SI units are to be regarded as standard. The values given in parentheses ~~are mathematical conversions to inch-pound units that~~ after SI units are provided for information only and are not considered standard.

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~~ safety, health, and ~~health~~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
D1356 Terminology Relating to Sampling and Analysis of Atmospheres
E288 Specification for Laboratory Glass Volumetric Flasks
E1605 Terminology Relating to Lead in Buildings

2.2 *U.S. Government Analytical Method:*[3]

U.S. EPA SW 846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods

[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,~~Analysis of ~~Lead;~~Lead for Exposure and Risk Assessment.

Current edition approved ~~Oct. 1, 2016~~Sept. 1, 2020. Published ~~November 2016~~September 2020. Originally approved in 1995. Last previous edition approved in ~~2009~~2016 as ~~E1726 – 01~~E1726 – 16.~~(2009).~~ DOI: ~~10.1520/E1726-16.~~10.1520/E1726-20.

[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 U.S. Government Publishing Office, 732 N. Capitol St., NW, Washington, DC 20401-0001, http://www.gpo.gov.

2.3 *ISO Standards:*[4]
ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials
ISO 1042 Laboratory ~~glassware—One-mark~~ glassware — One-mark volumetric flasks
ISO 8655 Piston-operated volumetric apparatus ~~--~~— Part 1: Terminology, general requirements and user recommendations

3. Terminology

3.1 ~~*Definitions:*~~*Definitions*—For definitions of terms relating to the preparation of dried paint samples that are not given here, refer to ~~Terminology~~Terminologies D1129, D1356, or ~~Terminology~~ E1605.

3.1.1 ~~*batch*—~~*batch, n*—a group of field or quality control samples that are processed together using the same reagents and equipment.

3.1.2 ~~*digestate*—~~*digestate, n*—the acidified aqueous solution that results from digestion of the sample.

3.1.3 ~~*digestion*—~~*digestion, n*—high temperature sample preparation process that involves chemical breakdown to solubilize targeted analytes present in a sample, to result in an acidified aqueous solution called the digestate.

3.1.4 *method* ~~*blank*—~~*blank, n*—a sample, devoid of analyte, that is analyzyed to determine its contribution to the total blank (background) reading.

3.1.5 *non-spiked* ~~*sample*—~~*sample, n*—a sample, devoid of analyte, that is targeted for addition of analyte but is not fortified with all target analytes prior to sample preparation.
3.1.5.1 *Discussion*—
Analysis results for this sample are used to correct for background levels in the blank medium that is used for spiked and spiked duplicate samples.

3.1.6 *reagent* ~~*blank*—~~*blank, n*—a digestate that reflects the maximum treatment given any one sample within a batch of samples, except that it has no sample placed initially 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.)
3.1.6.1 *Discussion*—
Analysis results from this sample provide information on the level of potential contamination resulting from only laboratory sources that are experienced by samples processed within the batch.

3.1.7 *reference material (certified reference material)* ~~*(CRM)—*~~*(CRM), n*—reference material accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realization of the unit in which the property values are expressed; each certified value is accomplished by an uncertainty at a stated level of confidence (ISO Guide 30).

3.1.8 *spiked sample or spiked duplicate* ~~*sample*—~~*sample, n*—a blank medium that contains no purposely added analyte to which a known amount of analyte is added before preparation.
3.1.8.1 *Discussion*—
Analysis results for these samples are used to provide information on the precision and accuracy of the overall process.

4. Summary of Practice

4.1 A representative soil sample is dried and homogenized, and then digested (in a batch mode with other samples) on a hot plate using nitric acid and hydrogen peroxide. The digestate is diluted for final volume prior to lead measurement.

5. Significance and Use

5.1 There is a need to monitor the lead content in and around buildings and related structures in order to determine the potential lead hazard. Hence, effective and efficient methods are required for the preparation of soil samples for determination of their lead content.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

5.2 This practice may be used for the digestion of soil samples that are collected during various construction and renovation activities associated with lead abatement in and around buildings and related structures. The practice is also suitable for the digestion of soil samples for lead analyses collected from other locations, such as near roads and steel structures.

5.3 This practice is intended to be used to prepare samples that have been collected for hazard assessment purposes.

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

5.5 This practice includes drying and homogenization steps in order to help assure that reported lead results are representative of the sample and are independent of potential differences in soil moisture levels among different sampling locations or changing weather conditions.

6. Apparatus

6.1 *Equipment:*

6.1.1 *Analytical Balance,* capable of accurately determining the mass to the nearest 0.001 g.

6.1.2 *Drying Oven,* capable of maintaining a temperature of ~~100~~100°C to 120°C.

6.1.3 *Electric Hot Plate,* capable of maintaining a temperature of ~~80~~80°C to 100°C as measured with a thermometer placed into a beaker or flask filled with water sitting on the hot plate head. When required to reduce the presence of hot spots in the electrical hot plate, place a 2 cm to 2.5 cm (0.75 in to 1 in.) thick aluminum plate on the burner head.

6.1.4 *Grinding Apparatus*—Mortar and pestle (porcelain or agate), shatter box, or mixer mill.

6.1.5 *Micropipettors with Disposable Plastic Tips* conforming to ISO 8655, sizes needed to make reagent additions, and spiking standards (see Note 1).

NOTE 1—In general, the following sizes should be readily available: 1 mL to 5 mL adjustable, ~~1000, 500, 250,~~ 1000 µL, 500 µL, 250 µL, and 100 µL.

6.1.6 *Sieves,* 4.75 mm (U.S. Standard No. 4), 2 mm (No. 10), and 500 µm (No. 35), plastic or stainless steel (see Note 2). When sieves containing soldered joints are used, then all solder joints shall be coated with epoxy resin prior to use to protect samples from potential lead contamination originating in the solder. Visually inspect prior to use for the presence of bare metal.

NOTE 2—Plastic or stainless steel sieves are better for use instead of brass sieves to alleviate possible lead contamination of the soil samples from contact with lead solder common to brass sieves.

6.1.7 *Thermometers,* red alcohol, that cover a range from ~~0~~0°C to ~~110°C.~~110°C

6.2 *Glassware and Supplies:*

6.2.1 *Borosilicate Glassware*—Volumetric flasks with stoppers meeting Specification E288 or conforming to ISO 1042, 100 mL; Griffin beakers, ~~100,~~ 100 mL, 150 mL or 250 mL; watch glasses sized to cover Griffin beakers.

6.2.2 *Plastic Gloves,* powderless.

6.2.3 *Air-Tight Sample Containers*—1 L (1 qt) or 4 L (1 gal) re-sealable plastic bags, or plastic 50 mL centrifuge tubes.

6.2.4 *Volumetric Flasks*—meeting Specification E288 or conforming to ISO 1042, 100 mL and other sizes as needed to make dilutions of sample digests or lead standards used for fortification of spiked samples.

6.2.5 *Tongs,* metal.

6.2.6 *Spoon,* stainless steel.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this practice. Unless otherwise indicated, all reagents shall conform to the specifications for the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades shall not be used unless 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),~~ mass fraction 30 %, suitable for atomic spectrometry analysis such as spectroscopic grade.

7.4 *Acetone,* reagent, spectroscopic grade.

7.5 *Water*—Unless otherwise indicated, references to water shall mean reagent water as defined by Type I of Specification D1193. (ASTM Type I ~~Water:~~water: minimum ~~resistance~~resistivity of 16.7 megohm·cm, or equivalent.)

7.6 *Calibration Stock Solution,* 100 µg/mL of lead (Pb) CRM in dilute (typically 2 %) nitric acid.

8. Sample Preparation Procedure

8.1 *Sample Pre-Treatment:*

8.1.1 Treat each sample in a processing batch equally.

8.1.2 If possible before removal, break up the soil sample[6,7,8,9] within the original containers containing the samples (see Note 3).

NOTE 3—This will not be possible for wet soil samples.

8.1.3 Label an acid-cleaned ~~100, 150,~~ 100 mL, 150 mL, or 250 mL Griffin beaker (or other vessel suitable for oven drying of soils that will not contaminate the sample with lead) with a high temperature wax pen or any other marker that will be visible after exposure to the drying oven.

8.1.4 Transfer the entire soil sample to the labeled Griffin beaker. Cover with a watch glass (tip to one side to permit moisture removal), and place in a drying oven for a minimum of 6 h at a temperature of ~~110~~110°C ± 10°C. Samples that cake or plug the sieve require additional drying (see Note 4).

NOTE 4—Soil samples should not cake or exhibit packing characteristic of moisture, but should flow freely through the sieve (see 8.1.7) when broken apart.

8.1.5 If the received soil sample is excessively large, then any attempts to sub-sample prior to drying and sieving are likely to cause bias. If possible, use a larger beaker to contain the entire sample. If not, then use multiple beakers followed by re-combining after drying. Samples that cake or plug the sieve require additional drying (see Note 4).

8.1.6 Using tongs, remove the beakers containing the samples and allow them to cool to room temperature.

---

[5] ~~*Reagent Chemicals, American Chemical Society Specifications,*~~*ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials,* American Chemical Society, Washington, ~~DC, www.chemistry.org.~~ 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. Pharmacopeial Convention, Inc. ~~(USP),~~(USPC), Rockville, ~~MD, http://www.usp.org.~~MD.

[6] U.S. Environmental Protection Agency, Soil Sampling Quality Assurance User's Guide, Second Edition, EPA/600/8-69/046, March 1989, available from http://www.epa.gov.

[7] Rohlf, F., Akcakaya, H., and Ferraro, S., "Optimizing Composite Sampling Protocols," *Environmental Science and Technology*, Vol 30, 1996, pp. 2899–2905.

[8] Australia National Environmental Health Forum Monograph, Soil Series No. 3, Composite Sampling, 1996, available from http://www.enhealth.nphp.gov.au.

[9] U.S. Environmental Protection Agency, Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, EPA/600/R-03/027, November 2003, available from http://www.epa.gov.

8.1.7 Don a pair of plastic gloves and push the soil sample through a clean 4.75 mm sieve (U.S. Standard No. 4) to remove any large objects or root material, or both. Discard material retained on the sieve (see Notes 5 and 6). Clean the sieve between samples by tapping or using forced air or other dry method to prevent cross-contamination. Perform this step in a location well removed from other samples in process and in an area where soil dust will not contaminate the laboratory operations such as in front of a fume hood.

NOTE 5—If the samples do not appear to contain any large objects or root material, it is not necessary to perform this step with the 4.75 mm sieve.

NOTE 6—In order to minimize small particle size soil losses, this step should not be performed inside a fume hood.

8.1.8 Don a pair of plastic gloves and push the soil sample through a clean 2 mm sieve (U.S. Standard No. 10) to remove coarse material (see Note 6). Discard material retained on the sieve. Clean the sieve between samples by tapping or using forced air or other dry method to prevent cross-contamination. Perform this step in a location well removed from other samples in process, and in an area where soil dust will not contaminate the laboratory operations, such as in front of a fume hood.

8.1.9 Grind the sample using a porcelain mortar and pestle or other appropriate homogenization apparatus such as a shatter-box or mixer mill. Clean the grinding apparatus between samples to prevent cross-contamination between samples by rinsing with water and drying. When any material is retained from 8.1.10, delay cleaning until this retained material for the sample is re-ground as described in 8.1.11. An acetone rinse will facilitate drying (see Note 7).

NOTE 7—Acetone should not be used on sieves since it can damage epoxy coatings which may be present to seal lead solder joints.

8.1.10 Place the ground up sample on a clean 500 µm sieve (U.S. Standard No. 35). Use a stainless steel spoon to help move material around until no more sample will pass through the sieve. Do not discard the retained material (see Note 8). Return any retained material for one more grinding as described in 8.1.9.

NOTE 8—A second re-grinding step is included for retained material to avoid inadvertent loss of larger pieces of material that can remain as a result of inadequate grinding.

8.1.11 Place the ground up retained sample material back on the clean U.S. Standard No. 35 (500 µm sieve) (see Note 6). Using a stainless steel spoon, help move material around until no more sample will pass through the sieve, adding the passed material to the previous sample material that passed through the sieve. Discard any retained material. Clean the sieve between samples by tapping or using forced air or other dry method to prevent cross-contamination. Perform this step in a location well removed from the samples in process.

8.1.12 Label acid-cleaned ~~100, 150,~~ 100 mL, 150 mL, or 250 mL Griffin beakers and watch glasses for performing the digestion of each soil sample and associated QC samples.

8.1.13 Transfer sieved portion to a labeled Griffin beaker and place in a drying oven overnight or for a minimum of ~~12 h,~~ 12 h, or to constant mass at a temperature of ~~110~~110°C ± ~~10°C (see~~10°C. Constant ~~Note 9).~~ mass for this procedure is defined as a less than 0.1 % change in mass for repeated measurements (a minimum of two) taken over a minimum of a 1 h interval. Remove from oven and allow to cool to room ~~temperature.~~temperature

~~NOTE 9—Constant mass for this procedure is defined as a less than 0.1 % change in mass for repeated measurements (a minimum of two) taken over a minimum of a 1 h interval.~~

8.1.14 Store the dried, homogenized, and sieved soil samples inside new labeled air-tight sample containers.

8.2 *Sample Digestion:*

8.2.1 Turn or roll the sample container repeatedly for about 1 min. Determine the mass of each dried homogenized sample to the nearest 0.001 g and transfer a 1.0 g ± 0.10 g portion of the sample to a labeled Griffin beaker. Record the mass of each sample. For sample portions targeted for spiking, add the appropriate volume of a lead standard stock to the beaker (see Note ~~10~~9). In the absence of other information, add 500 µg of lead to each beaker containing sample portions targeted for spikes and spike duplicates (5 mL of 100 µg/mL of Pb stock solution).