Designation: E 1726 – 01

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

This standard is issued under the fixed designation E 1726; 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 (GFAAS).

1.2 This practice is based on U.S. EPA SW846 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 the standard. The inch-pound units given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

2.2 *U.S. Government Analytical Method:*

U.S. EPA SW 846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods[3]

2.3 *Other Standards:*

ISO Guide 30 Terms and Definitions Used in Connection with Reference Materials[4]

3. Terminology

3.1 *Definitions:*

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

3.1.3 *digestion*—the sample preparation process that will solubilize (extract) targeted analytes present in the sample and results in an acidified aqueous solution called the digestate. *Discussion:* Digestion is a form of extraction (see 3.1.5).

3.1.4 *duplicate sample*—a second portion of a homogenized sample carried through sample digestion. Analysis results for these samples are used to provide information on the precision of the homogenization process.

3.1.5 *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.

3.1.6 *non-spiked sample*—a portion of a homogenized sample that is targeted for addition of analyte but that is not fortified (spiked) with lead before sample preparation. Analysis results for this sample are used to correct for background levels in soil that are used for the spiked and spiked duplicate samples.

3.1.7 *reagent blank*—a digestate that reflects the maximum treatment given any one sample within a batch of samples, except that it has no sample initially placed into the digestion vessel. (The same reagents and processing conditions which are applied to field samples within a batch are also applied to the reagent blank.) *Discussion:* Analysis results from reagent blanks provide information on the level of potential contamination experienced by samples processed within the batch.

3.1.8 *certified reference material*—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).

3.1.9 *spiked sample*—a portion of a single homogenized sample to which the same known amount of analyte is added (spiked) before sample digestion. *Discussion:* Analysis results for these samples are used to provide information on accuracy and precision of the overall analysis process.

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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 Paint Abatement.

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

[3] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

[4] Available from ANSI, 11 W 42nd St., 13th Floor, New York, NY 10036.

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.

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 to 120°C.

6.1.3 *Electric Hot Plate*, capable of maintaining a temperature of 80 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 to 2.5 cm (0.75 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*, sizes needed to make reagent additions, and spiking standards (see Note 1).

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

6.1.6 *Sieves*, 4.7 mm (U.S. Standard No. 4), 1.9 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—Stainless steel or plastic sieves must be used instead of the standard 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 to 110°C.

6.2 *Glassware and Supplies*:

6.2.1 *Borosilicate Glassware*—Volumetric flasks with stoppers, 100 mL; Griffin beakers, 100, 150 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*—Class A, 100 mL and other sizes as needed to make dilutions of sample digests or lead standards used for fortification of spiked samples.

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), 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 D 1193. (ASTM Type I Water: minimum resistance of 16.7 megohm·cm, or equivalent.)

7.6 *Calibration Stock Solution*, 100 µg/mL of lead (Pb) in dilute 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 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, 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

[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. (USPC), Rockville, MD.