Designation: E 1726 – 95

An American National Standard

Standard Practice for Sample Digestion of Soils for the Determination of Lead by Atomic Spectrometry[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]

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.

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 all the 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.) Analysis results from reagent blanks provide information on the level of potential contamination experienced by samples processed within the batch.

3.1.8 *reference material*—a material of known composition where the lead level is certified by the manufacturer.

3.1.9 *spiked sample and spiked duplicate sample*—each is a portion of a single homogenized sample to which the same known amount of analyte is added (spiked) before sample digestion. 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 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 paint in 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.

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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.

Current edition approved Aug. 15, 1995. Published October 1995.

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

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

5.2 This practice is to 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 preparation of soil samples collected from some other environments.

5.3 This practice is intended to be used to prepare samples that have been collected for risk 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 between 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.

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, it is intended that all reagents shall conform to the specifications for the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] 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 be understood to mean reagent water as defined by Type 1 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 moisture removal), and place in a drying oven for a minimum of 6 h at a temperature of 110 ± 10°C (see Note 4).

NOTE 4—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. Soil samples should not cake or exhibit packing characteristic of moisture, but should flow freely through the sieve (see 8.1.6) when broken apart.

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

8.1.6 Don a pair of plastic gloves and push the soil sample through a clean 4.7 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

[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.

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 4.7 mm sieve.

NOTE 6—In order to minimize small particle size soil losses, it is not recommended to perform this step inside a fume hood.

8.1.7 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.

8.1.8 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 8.1.9, delay cleaning until this retained material for the sample is re-ground as described in 8.1.10. 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.

8.1.9 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 8.1.8.

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.

8.1.10 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.

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

8.1.12 Transfer sieved portion to a labeled Griffin beaker and place in a drying oven overnight or for a minimum of 12 h, or to constant mass at a temperature of 110 ± 10°C (see Note 9). Remove from oven and allow to cool to room 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.

8.1.13 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 ± 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). 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).

NOTE 10—The appropriate volume will be dependent on the anticipated lead level in the targeted sample split. The optimum spike addition is one which will double the amount of lead in the targeted sample split.

8.2.2 Add 10 mL of 1:1 nitric acid:water to each beaker, gently swirl to mix, and cover with a watch glass. Gently heat the sample to 85 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.

8.2.3 Allow the sample to cool, add 5 mL of concentrated nitric acid, replace the watch glass, and reflux for 30 min without boiling.

8.2.4 Allow the sample to cool, add 5 mL of concentrated nitric acid, replace the watch glass, and reflux for 30 min without boiling. (This is a repeat of 8.2.3 to ensure complete oxidation.)

8.2.5 Remove the watch glass and allow the solution to evaporate to approximately 5 mL without boiling, while maintaining a covering of solution over the bottom of the beaker. When removing watch glasses, care must be exercised to avoid losses by rinsing them with a minimum amount of water (rinsed into the sample beaker) and avoiding contamination by placing them upside down on new clean laboratory wipes. During this step, minimize the activity in the hood area and increase the separation between beakers. Allow the sample to cool after evaporation to approximately 5 mL.

8.2.6 After the sample has cooled, add 2 mL of water and 3 mL of 30 % hydrogen peroxide. Cover the beaker with a watch glass and return the covered beaker to the hot plate for warming and to start the peroxide reaction. Care must be taken during heating to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker.

8.2.7 Continue to add 30 % hydrogen peroxide in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mL of 30 % hydrogen peroxide even if effervescence has not been reduced to a minimal level.

8.2.8 Remove the watch glass and continue heating the acid-peroxide digestate carefully until the volume has been reduced to approximately 5 mL. Follow the same precautions for sample evaporation as outlined in 8.2.5.

8.2.9 Allow the digestates to cool, rinse the beaker walls and bottom of the watch glass with water, and quantitatively

transfer to a 100 mL volumetric flask. Dilute to volume with water.

8.2.10 Remove any particulate in the digestate by filtration, by centrifugation, or by allowing the sample to settle prior to instrumental measurement. The diluted digestate solution contains approximately 5 % (v/v) nitric acid. Use this level of nitric acid for calibration standards during instrumental measurement.

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. Process method blanks according to the frequency listed in Table 1.

9.1.2 *Non-Spiked Samples, Spiked Samples and Spiked Duplicate Samples*—Process these samples on a routine basis to estimate method accuracy on the sample batch, expressed as percent recovery relative to the true spiked value. Choose samples to use for spikes and spike duplicates at random. After sample pre-treatment (see 8.1) steps are completed, use three portions of the selected sample (one portion for the non-spike, one portion for the spike, and one portion for the spike duplicate sample). Proportion the selected samples using the splitting procedure listed below. Process spiked samples according to the frequency listed in Table 1.

9.1.3 *Procedures to Split Samples for Non-Spiked Samples, Spiked Samples, Spiked Duplicate Samples, and Duplicate Samples*—Use the following procedure to generate sample portions:

9.1.3.1 To mix the dried, sieved, and homogenized soil sample stored inside the air-tight sample containers, turn or roll the container repeatedly for about one min.

9.1.3.2 To split the homogenized soil sample stored inside the sample container, transfer the entire homogenized soil sample to a flat dry acid cleaned glass or plastic plate. Use of a new piece of plastic wrap or wax paper between the plate and sample will help reduce cross-contamination. Form the soil into a conical pile using a clean plastic spoon and flatten the pile to a uniform thickness and diameter by pressing down the apex of the pile. Divide the flattened pile into quarters using a plastic spoon. Using the spoon, scoop two opposite quarters of the sample to be spiked into separate labeled sample containers. Scoop the third quarter for the non-spiked sample into a separate labeled sample container and store the fourth quarter in another labeled sample container for permanent storage (if needed).

9.1.3.3 To alleviate cross-contamination, discard the piece of plastic wrap or wax paper (if used) and clean the glass plate with acetone and a laboratory wipe following each sample. Do not clean plastic plates with acetone.

9.1.4 *Duplicate Samples*—Process duplicate samples on a routine basis to determine an estimate of homogenization and analysis precision, expressed as percent relative range. Process duplicate samples according to the frequency listed in Table 1.

9.1.5 *Standard Reference Materials*—Process standard reference materials (SRMs) 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 into each analytical batch according to the frequency listed in Table 1. Use an SRM which has a matrix similar or identical to soil with a certified lead concentration level.

9.2 *Laboratory Records*—Record all information regarding the preparation of samples (both QC samples and those submitted to the analyst) in a laboratory notebook as described below.

9.2.1 Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Record any inadvertent deviations, unusual happenings, or observations on a real-time basis as samples are processed. Use these records to add supplemental lead data when reporting results.

9.2.2 Laboratory notebooks shall be bound with prenumbered pages. All entries on sample data forms and laboratory notebooks shall be made using ink with a signature and date of entry. Any entry errors shall be corrected by using only a single line through the incorrect entry (no scratch outs) accompanied by the initials of the person making the correction and the date of correction (see Note 11).

NOTE 11—These procedures are important to properly document and trace laboratory data.

10. Keywords

10.1 digestion; lead; sample preparation; soils

TABLE 1 Quality Control (QC) Samples

QC Samples	Definition	Frequency
Reagent blank	Type I Water—Digest as a sample with addition of all reagents. Reflects the maximum treatment given any one sample within the batch.	1 per 20 samples, a minimum of 1 per batch
Duplicate sample	A second portion of a homogenized sample digested in the same batch as the first portion.	1 per 20 samples, a minimum of 1 per batch
Spiked sample	A portion of a homogenized sample is spiked with lead before sample digestion.	1 per 20 samples, a minimum of 1 per batch
Spiked duplicate sample	A portion of the same homogenized sample used for the spiked sample is spiked with lead before sample digestion.	1 per 20 samples, a minimum of 1 per batch
Reference material (standard reference)	A material of known composition, where the analyte levels are certified by the manufacturer.	1 per batch of samples