



Designation: E1726 – 21

Standard Practice for Preparation of Soil Samples by Hotplate Digestion for Subsequent Lead Analysis¹

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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 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 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, health, and 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

2.3 ISO Standards:⁴

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

ISO 1042 Laboratory glassware — One-mark volumetric flasks

ISO 8655 Piston-operated volumetric apparatus — Part 1: Terminology, general requirements and user recommendations

3. Terminology

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

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

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

3.1.3 *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, n*—a sample, devoid of analyte, that is analyzed to determine its contribution to the total blank (background) reading.

3.1.5 *non-spiked 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.

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 of Lead for Exposure and Risk Assessment.

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

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

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, 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), 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, 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.

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 represen-

tative 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°C to 120°C.

6.1.3 *Electric Hot Plate*, capable of maintaining a temperature of 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 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°C to 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 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