



Standard Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry¹

This standard is issued under the fixed designation E 1741; 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 the preparation of airborne particulate samples collected during the abatement of lead hazards for lead analysis in and around buildings and structures.

1.2 This practice describes the digestion procedures for airborne particulate lead samples that are collected on cellulose ester membrane filters during abatement and construction activities. The practice is intended for use with airborne particulate lead samples that are prepared for subsequent analysis by laboratory-based quantitative analytical methods.

1.3 This practice covers the general considerations for quantitative sample digestion for total recoverable lead in airborne particulate using hot plate or microwave heating techniques.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 The following safety hazards caveat pertains only to the procedure section of this practice. *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.* Specific hazard statements are given in Section 8, 9.3.1.6, and 9.3.2.6.

2. Referenced Documents

2.1 ASTM Standards:

D 1129 Terminology Relating to Water²

D 1193 Specification for Reagent Water²

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres³

D 3335 Test Method for Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy⁴

D 4185 Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry³

D 4309 Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Recoverable Metals in Water²

E 1605 Terminology Relating to Abatement of Hazards from Lead-Based Paint in Buildings and Related Structures^{5,6}

2.2 U.S. Code of Federal Regulations:⁷

CFR 1910.1025, Volume 29; OSHA Standard for Lead in Construction

CFR 1030.10, Volume 21; U.S. Dept. of Health and Human Services Standard

CFR Volume 47, FCC Rule Part 18, Federal Communications Commission Standard

3. Terminology

3.1 *Definitions*—For definitions of terms relating to the preparation of atmospheric samples that are not given here, refer to Terminology D 1129, D 1356, or E 1605.

3.2 *Definitions of Terms Specific to This Standard:*

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

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

3.2.3 *digestion*—the sample preparation process which will solubilize (extract) targeted analytes present in the sample, and results in an acidified aqueous solution called the digestate.

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

3.2.5 *field blank*—a sampling device (filter holder containing filter) that is handled in the same manner as field samples, except that no air is drawn through it.

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 Jan. 10, 2000. Published April 2000. Originally published as ES 33 – 94. Last previous edition E 1741 – 95.

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

³ *Annual Book of ASTM Standards*, Vol 11.03.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 04.11.

⁶ *ASTM Standards on Lead-Based Paint Abatement in Buildings*, 1994, available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

⁷ Available from Office of the Federal Register, National Archives Records Administration, Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20401.

3.2.6 *filter holder*—a plastic holder that supports the filter medium upon which airborne particulate matter is collected. This device is also often referred to as a filter “cassette.”

3.2.7 *method (reagent) blank*—a reagent, without analyte added, that is analyzed to determine its contribution to the total blank (background) reading.

3.2.8 *non-spiked sample*—a blank filter sample that is targeted for addition of analyte but which is not fortified with all the target analytes before sample preparation. For filter samples, a non-spiked sample is equivalent to a method (reagent) blank. Analysis results for this sample are used to correct for background levels in the blank filters used for spiked and spiked duplicate samples.

3.2.9 *reagent blank*—a digestate that reflects the maximum treatment given any one sample within a sample batch, 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.) 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.

3.2.10 *reference material (standard reference material)*—a material of known composition where the lead level is certified by the manufacturer.

3.2.11 *sample set*—a group of samples (one or more).

3.2.12 *spiked sample and spiked duplicate sample*—a blank filter to which a known amount of analyte is added before preparation. Analysis results for these samples are used to provide information on the precision and accuracy of the overall analysis process.

4. Summary of Practice

4.1 Particulate matter containing lead, which has been collected from air on cellulose ester membrane filters (see Test Methods [D 4185](#)), is digested in a heated acidic mixture. The filter, which contains the collected particulate, may be digested on a hot plate (see Test Methods [D 4185](#)) or within a specially designed microwave apparatus (see Practice [D 4309](#)). The digestion procedure is meant to prepare the samples for subsequent analysis by atomic spectroscopic methods, such as atomic absorption spectroscopy (see Test Method [D 3335](#)) and inductively coupled plasma atomic emission spectrometry.

5. Significance and Use

5.1 This practice is to be used for the digestion of airborne particulate lead that has been collected during various construction and renovation practices associated with lead abatement and removal in and around buildings and related structures. It may also be used to treat samples from other workplace environments where airborne lead is suspected to be present, for example, battery recycling, smelting, firing ranges, etc.

5.2 This practice may be used to prepare samples that have been obtained in order to ensure compliance with OSHA permissible exposure limits (PELs) for airborne lead concentrations.⁸

6. Apparatus

6.1 Hot Plate Digestion:⁸

6.1.1 *Electric Hot Plate*, suitable for operation at temperatures up to at least 140°C.

6.1.2 *Borosilicate Glass Beakers*, 100 to 150 mL Griffin or Phillips beakers with watch glass covers.

6.1.3 *Laboratory Thermometer*, accurate to nearest 0.1°C, covering the range 0 to 200°C.

6.2 Microwave Digestion:⁹

6.2.1 *Laboratory Microwave Heating System*, capable of delivering 575 to 1000 W of power. The unit should be capable of 1 % power adjustment and 1 s time adjustment. The oven cavity should be fluorocarbon-coated and equipped with exhaust ventilation at 2.8 m³/min for acid vapor protection of the unit and operator. The unit must have a rotating or alternating turntable, capable of holding one to twelve digestion vessels, to ensure uniform sample heating. Safety interlocks, to shut off magnetron power output, must be contained in the oven door opening mechanism.

NOTE 1—Because of differences among various makes and models of satisfactory microwave instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument.

6.2.1.1 The unit must comply with U.S. Dept. of Health and Human Services Standards under CFR Part 1030.10, Subparts (C)(1), (C)(2), and (C)(3), for microwave leakage. The unit should have FCC-type approval for operations under FCC Rule Part 18.

6.2.2 *Closed Vessels*, capable of holding up to 100 mL of solution. The vessels must be transparent to microwave energy and capable of withstanding internal pressures of 100 psig and temperatures of 200°C. Each vessel must contain a safety pressure relief valve, rupture disc, or be connected to an external safety relief valve that will prevent possible vessel rupture or ejection of the vessel cap.

6.2.3 An apparatus for tightening the vessel system cap to the manufacturer’s specified torque.

6.3 Other Supplies:

6.3.1 *Class A Volumetric Flasks*, 10 to 100 mL.

⁸ Cassinelli, M. E. and Eller, P. M., Eds., *NIOSH Manual of Analytical Methods*, 4th ed.; Methods 7082, 7105, and 7300; National Institute for Occupational Safety and Health, Cincinnati, OH, 1994. Available from National Institute for Occupational Safety and Health, Publications Office, 4676 Columbia Pkwy., Cincinnati, OH 45226.

⁹ Environmental Protection Agency, *Standard Operating Procedures for Lead in Paint by Hotplate- or Microwave-Based Acid Digestions by Atomic Absorption or Inductively Coupled Plasma Emission Spectrometry*; U.S. EPA, Research Triangle Park, NC, 1991. Available from National Technical Information Services, 5285 Port Royal Rd., Springfield, VA 22161.