Designation: E 1741 – 00

An American National Standard

Standard Practice for Preparation of Airborne Particulate Lead Samples Collected During Abatement and Construction Activities for Subsequent Analysis by Atomic Spectrometry[1]

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 Water[2]

D 1193 Specification for Reagent Water[2]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[3]

D 3335 Test Method for Concentrations of Lead, Cadmium, and Cobalt in Paint by Atomic Absorption Spectroscopy[4]

D 4185 Practice for Measurement of Metals in Workplace Atmosphere by Atomic Absorption Spectrophotometry[3]

D 4309 Practice for Sample Digestion Using Closed Vessel Microwave Heating Technique for the Determination of Total Recoverable Metals in Water[2]

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.

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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.

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[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 m^3/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.

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[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.

6.3.2 *Class A Pipets*, volumetric and graduated (1 to 10 mL).

6.3.3 *Class A Micropipets*, 10 to 1000 µL.

6.3.4 *Powderless Vinyl Gloves*.

6.3.5 *Tweezers*.

6.3.6 *Wash Bottle*.

7. Reagents

7.1 *Calibration Stock Solution*—1000 µg Pb/mL. Commercial certified reagent grade nitrate standards; alternatively, dissolve 1.00 g reagent grade Pb metal in a minimum volume of nitric acid and dilute to 1 L with 1 % (v/v) nitric acid. Store in polyethylene bottles.

7.2 *Hydrochloric Acid*, concentrated; reagent grade, specific gravity 1.19 (for microwave digestions).

7.3 *Hydrogen Peroxide*, 30 % (v/v); reagent grade (for hot plate digestions).

7.4 *Nitric Acid*, concentrated; reagent grade, specific gravity 1.42.

7.5 *Water*, ASTM Type I or Type II (conforming to Specification D 1193).

8. Hazards

8.1 Hot plate digestions must be conducted in a fume hood in order to prevent fumes from contaminating laboratory air.

8.2 In hot plate digestions, the applied power (and therefore temperature) should be increased slowly to prevent spattering.

8.3 Microwave units should be operated in accordance with the manufacturer's recommended operating and safety precautions. (**Warning—**It is not recommended to place a microwave unit in a fume hood, where it is surrounded by acid fumes which can cause corrosion of the equipment. Acid fumes inside the oven cavity should be air swept away from the cavity to a hood. Closed vessels used in microwave digestions should be operated in accordance with the manufacturer's recommended operating and safety instructions.)

9. Procedure

9.1 *Laboratory Records*—Record all reagent sources (lot numbers) used for sample preparation in a laboratory notebook. Record any inadvertent deviations, unusual events, or observations during sample preparation. Use these records to supplement analytical data concerning lead when reporting the final results.

9.2 *Hot Plate Digestion*:

9.2.1 Open the filter holders (cassettes) and transfer the samples (filters + collected particulate) and blanks to clean beakers.

9.2.2 Add 3 mL concentrated nitric acid and 1 mL 30 % hydrogen peroxide and cover with a watch glass.

NOTE 2—Start method (reagent) blanks at this point.

9.2.3 Heat on a hot plate at 140°C (85 to 100°C initially) until the volume is reduced to about 0.5 mL.

NOTE 3—A lower temperature should be applied during the initial stages of the digestion to prevent spattering of the beaker contents.

9.2.4 Rinse the watch glass and baker walls with 3 to 5 mL of 10 % nitric acid. Allow the solution to evaporate to 0.5 mL. If solid particulate remains, use 2 mL concentrated nitric acid and 1 mL 30 % hydrogen peroxide.

9.2.5 Repeat 9.2.3 and 9.2.4 as necessary until the solution is clear.

9.2.6 Cool each beaker to room temperature.

9.2.7 Transfer the solutions quantitatively to 10 mL Class A volumetric flasks and dilute to volume with ASTM Type I or II water.

9.3 *Microwave Digestion*:

9.3.1 *Procedure for Seven to Twelve Vessel Digestions*:

NOTE 4—For fewer than seven samples, see 9.3.2.

9.3.1.1 Perform an instrument power check as outlined in Appendix X1.

9.3.1.2 Open the filter holders (cassettes) and transfer the samples (filters + collected particulate) and blanks into clean vessel.

NOTE 5—Follow the manufacturer's suggested vessel cleaning instructions to avoid possible sample contamination. If the sample is to be analyzed by inductively coupled plasma (ICP), direct current plasma (DCP), or flame atomic absorption spectrophotometry (FAAS), add 3 mL of concentrated nitric acid and 2 mL of concentrated hydrochloric acid. If the sample is to be analyzed by graphite furnace atomic absorption spectrophotometry (GFAAS), add 5 mL of concentrated nitric acid. Install a safety pressure relief valve and cap on the vessel and seal to the manufacturer's recommended torque. Attach a vent tube if required by the manufacturer's operating instructions.

9.3.1.3 Repeat 9.3.1.2 until the turntable contains twelve vessels. It is recommended that a reagent blank be digested and analyzed along with the samples (see Table 1). If less than twelve samples are to be digested, fill the remaining vessels with an equal volume of acid mixture (either 3 mL HNO_3 + 2 mL HCl or 5 mL HNO_3, depending on the instrumental analytical method). It is critical to the procedure that each vessel contains an equal volume of acid. This is necessary to ensure uniform heating of all vessel solutions.

TABLE 1 Quality Control Samples

QC Samples	Definition	Frequency
Method blank or non-spiked sample	A blank filter carried through sample preparation along with other samples. Should reflect the maximum treatment given any one sample within the batch	One per 20 samples, minimum of one per batch.
Reagent blank	*ASTM Type I or II Water*—Digest as a sample with addition of all reagents. Should reflect the maximum treatment given any one sample within the batch.	One per batch.
Spiked sample	A blank filter fortified with all the target analytes before preparation.	One per 20 samples, minimum of one per batch.
Spiked duplicate sample	A blank filter fortified with all the target analytes before preparation (for filters, duplicates cannot be obtained in the field).	One per 20 samples, minimum of one per batch.
Reference material (standard reference material)	A material of known composition where the analyte levels are certified by the manufacturer.	One per batch.

9.3.1.4 Turn the microwave instrument exhaust on to the maximum fan speed. Activate the turntable so that it is rotating or alternating 360°.

9.3.1.5 For instruments delivering a measured power of 575 to 635 W, program the instrument time for 50 min and the power to 100 %. For instruments with a measured power of 635 to 700 W, program the instrument time for 30 min and the power to 100 %. Instruments delivering greater than 700 W must be operated at reduced powers in order to reduce the sample heating rates. Depress the start key and allow the sample mixtures to heat for the programmed time period.

9.3.1.6 At the end of the digestion period, remove the vessels from the microwave and allow the sample solutions to cool to room temperature. Shake the vessels to mix the sample solutions and vent to atmosphere any gas pressure that may be present in the vessels. (**Warning—**Shake the vessels with caution to prevent any rapid outgassing of vapor or liquid, or both, that may cause acid burns of the exposed skin of the operator.)

9.3.1.7 Detach the vent tubing and remove the vessel assembly from the turntable.

9.3.1.8 Transfer the contents of each vessel to 10 mL volumetric flasks (Class A) and bring to volume with ASTM Type I or II water. The diluted solutions are now ready for analysis. Further dilution of samples may be required in instances where the lead loadings on digested filters are very high.

NOTE 6—Prior to dilution, it may be necessary to filter or centrifuge digested samples that contain silicates or other insoluble materials.

9.3.2 *Procedure for One to Six Vessel Digestions*:

9.3.2.1 Perform an instrument power check as outlined in Appendix X2.

9.3.2.2 Transfer the samples to clean vessels as discussed in 10.3.1.2.

9.3.2.3 Repeat 9.3.2.2 until the turntable contains six evenly spaced vessels. A reagent blank is to be digested along with the samples. If fewer than six samples are to be digested, fill remaining vessels with the appropriate acid mixture (5 mL HNO_3 or 3 mL HNO_3 + 2 mL HCl). It is critical that each vessel contains an equal volume of acid to ensure uniform heating of all vessel solutions.

9.3.2.4 Turn the microwave instrument exhaust on to the maximum fan speed. Activate the turntable so that it is rotating or alternating 360°.

9.3.2.5 For instruments with a measured power of 575 to 635 W, program the instrument time for 30 min and 75 % power. For instruments with a measured power of 635 to 700 W, program the instrument time for 25 min and 75 % power. Instruments delivering greater than 700 W must be operated at further reduced powers so that the sample heating rates are not excessive. Depress the start key and allow the sample mixtures to heat for the programmed time period.

9.3.2.6 At the end of the digestion period, remove the vessels from the microwave and allow the sample solutions to cool to room temperature. Shake the vessels to mix the sample solutions, and vent to atmosphere any gas pressure that may be present in the vessels. (**Warning—**Shake the vessels with caution to prevent any rapid outgassing of vapor or liquid, or both, that may cause acid burns on the exposed skin of the operator.)

9.3.2.7 Detach the vent tubing and remove the vessels from the turntable.

9.3.2.8 Open the vessels and filter or centrifuge the samples, if required, to remove any silicates or other insoluble material. Transfer the samples to 10 mL Class A volumetric flasks and bring to volume with ASTM Type I or II water. The diluted samples are now ready for analysis. Further dilution may be necessary to ensure that the measurement of lead concentration is within the instrumental dynamic range.

10. Quality Assurance

10.1 *Quality Control Samples*—Quality control samples to be processed with each batch of samples are summarized in Table 1.

10.1.1 *Reagent Blanks*—Carry reagent blanks (water and reagents) throughout the entire sample preparation and analytical process to determine if the samples are being contaminated from laboratory activities. Process reagent blanks according to the frequency listed in Table 1.

10.1.2 *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. Since filter samples cannot be split uniformly, blank filters are used for non-spiked, spiked, and spiked duplicate samples. The brand or type of filter should be the same as that used for collection of samples. Field personnel should submit a sufficient number of blank filters to the laboratory to permit generation of these QC samples at the frequency listed in Table 1.

10.1.3 *Standard Reference Materials*—Process certified 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 that has a matrix similar or identical to dust with a certified lead concentration level. Place a known quantity of the SRM onto a blank filter and process along with the other samples. The brand or type of filter used should be the same as that used for sample collection. Field personnel should submit sufficient numbers of blank filters to the laboratory to generate these QC samples.

10.2 *Laboratory Records*—In a laboratory notebook, record all reagent sources (and lot numbers) used for sample preparation. Also, record sample receipt information, including submitter, number and type of samples, and so on. Record any inadvertent deviations, unusual occurrences, or observations in real time, as samples are processed. Use these records to add supplemental data when reporting results.

NOTE 7—Laboratory notebooks must be bound with pre-numbered pages, and all entries must be made in ink. Any entry errors must be corrected by using a single line through the incorrect entry, accompanied by the initials of the person making the entry, and the date of the correction.

11. Keywords

11.1 airborne particulate; digestion; hot plate; lead; microwave; sample preparation

APPENDIXES

(Nonmandatory Information)

X1. POWER CHECK AT 100 % INSTRUMENT POWER

X1.1 *Procedure:*

X1.1.1 Remove from the instrument cavity the turntable, drive lug, and all vessels.

X1.1.2 Adjust the instrument cavity exhaust to minimum air flow.

X1.1.3 Program the instrument for 4 min time and 100 % power.

X1.1.4 Transfer 2000 ± 2 mL of room temperature (19 to 25°C) water into a 2 L polypropylene beaker.

X1.1.5 Measure and record the initial water temperature (T_i) to the nearest 0.1°C.

X1.1.6 Place the beaker in the right front corner of the instrument cavity (facing the front of the instrument).

X1.1.7 Heat the water for the programmed time.

X1.1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature (T_f) to the nearest 0.1°C.

X1.1.9 Calculate the delivered power, W, as follows:

$$\text{Power} = \Delta T \times [(K \times C_p \times M)/t] \quad \text{(X1.1)}$$

where:

ΔT = $T_f - T_i$, where:
T_f = final water temperature, °C, and
T_i = initial water temperature, °C.
K = 4.2, the conversion factor for thermochemical calories to Watts,
C_p = 1.0, the heat capacity for water in $\text{cal}\cdot\text{g}^{-1}\cdot\text{deg}^{-1}$,
M = mass of water, g, and
t = time, s.

X2. POWER CHECK AT 75 % INSTRUMENT POWER

X2.1 *Procedure:*

X2.1.1 Remove from the instrument cavity the turntable, drive lug and all vessels.

X2.1.2 Adjust the instrument cavity exhaust to minimum air flow.

X2.1.3 Program the instrument for 4 min time and 75 % power.

X2.1.4 Transfer 2000 ± 2 mL of room temperature water (19 to 25°C) water in a 2 L polypropylene beaker.

X2.1.5 Measure and record the initial water temperature (T_i) to the nearest 0.1°C.

X2.1.6 Place the beaker in the right front corner of the instrument cavity (facing the front of the instrument).

X2.1.7 Heat the water for the programmed time.

X2.1.8 When the heating cycle is complete, immediately remove the beaker from the cavity, thoroughly stir the water to ensure even heat distribution, and measure the final temperature (T_f) to the nearest 0.1°C.

X2.1.9 Calculate the delivered power according to Eq X1.1.