



Designation: D8344 – 20

Standard Practice for Ammonium Bifluoride and Nitric Acid Digestion of Airborne Dust and Dust-Wipe Samples for the Determination of Metals and Metalloids¹

This standard is issued under the fixed designation D8344; 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 digestion of airborne and surface dust samples (collected using air and wipe sampling practices) and associated quality control (QC) samples for the determination of metals and metalloids by means of a mixture of dilute ammonium bifluoride and nitric acid.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 This practice contains notes which are explanatory and not part of mandatory requirements of the standard.

1.4 *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.*

1.5 *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

[D1193 Specification for Reagent Water](#)

[D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)

[D4185 Test Method for Measurement of Metals in Workplace Atmospheres by Flame Atomic Absorption Spectrophotometry](#)

[D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers](#)

[D4840 Guide for Sample Chain-of-Custody Procedures](#)

[D6062 Guide for Personal Samplers of Health-Related Aerosol Fractions](#)

[D6785 Test Method for Determination of Lead in Workplace Air Using Flame or Graphite Furnace Atomic Absorption Spectrometry](#)

[D6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Determination of Metals](#)

[D7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry \(ICP-AES\)](#)

[D7144 Practice for Collection of Surface Dust by Microvacuum Sampling for Subsequent Metals Determination](#)

[D7202 Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection](#)

[D7296 Practice for Collection of Settled Dust Samples Using Dry Wipe Sampling Methods for Subsequent Determination of Beryllium and Compounds](#)

[D7439 Test Method for Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma-Mass Spectrometry](#)

[D7659 Guide for Strategies for Surface Sampling of Metals and Metalloids for Worker Protection](#)

[D7707 Specification for Wipe Sampling Materials for Beryllium in Surface Dust](#)

[D7822 Practice for Dermal Wipe Sampling for the Subsequent Determination of Metals and Metalloids](#)

[E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory](#)

[E1370 Guide for Air Sampling Strategies for Worker and Workplace Protection](#)

[E1605 Terminology Relating to Lead in Buildings](#)

[E1613 Test Method for Determination of Lead by Inductively Coupled Plasma Atomic Emission Spectrometry \(ICP-AES\), Flame Atomic Absorption Spectrometry](#)

¹ This practice is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality. Current edition approved Sept. 1, 2020. Published September 2020. DOI: 10.1520/D8344-20.

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.

(FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Techniques

E1644 Practice for Hot Plate Digestion of Dust Wipe Samples for the Determination of Lead

E1728 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Lead Determination

E1792 Specification for Wipe Sampling Materials for Lead in Surface Dust

E2051 Practice for the Determination of Lead in Paint, Settled Dust, Soil and Air Particulate by Field-Portable Electroanalysis (Withdrawn 2010)³

E3193 Test Method for Measurement of Lead (Pb) in Dust by Wipe, Paint, and Soil by Flame Atomic Absorption Spectrophotometry (FAAS)

E3203 Test Method for Determination of Lead in Dried Paint, Soil, and Wipe Samples by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

2.2 Other Standards:

ISO/IEC 17011 Conformity assessment — Requirements for accreditation bodies accrediting conformity assessment bodies⁴

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories⁴

3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, refer to Terminologies **D1356** and **E1605**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *blank wipe, n*—an unused, unspiked dust wipe that is only removed from its packaging immediately before use.

3.2.1.1 *Discussion*—Blank wipes are used to prepare non-spiked, spiked, and spiked duplicate quality assurance samples.

3.2.2 *dust wipe sample, n*—surface dust collected on a wipe.

3.2.3 *method blank, n*—a digestate that reflects the maximum treatment given any one sample within a sample batch except that only the sampling medium (such as a blank wipe) is initially placed into the digestion vessel.

3.2.3.1 *Discussion*—The same reagents and processing conditions that are applied to field samples within a batch are also applied to the method blanks so that analysis results provide information on the level of potential contamination resulting from the laboratory and sampling medium sources that are experienced by samples processed within the batch.

3.2.4 *non-spiked sample, n*—a portion of a homogenized sample that was targeted for the addition of analyte but is not fortified with the target analytes before sample preparation.

3.2.4.1 *Discussion*—For wipe samples, a non-spiked sample is equivalent to a method blank. Analysis results for this sample are used to correct for background levels in the blank wipes used for spiked and spiked duplicate samples.

3.2.5 *spiked sample and spiked duplicate sample, n*—a blank wipe that is spiked with a known amount of analyte (that is, lead) before hot plate digestion and subsequent lead analysis.

3.2.5.1 *Discussion*—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 An airborne dust or dust wipe sample is digested using hot block type heating or heated sonication with a mixture of dilute ammonium bifluoride and concentrated nitric acid. The digestate is diluted to final volume prior to measurement of metals and metalloids.

NOTE 1—The procedure in this practice is based on Test Methods **D4185**, **D6785**, **D7035**, **D7202**, Practice **E1644**, and NIOSH 7404 and 9110 of the *NIOSH Manual of Analytical Methods*.⁵

5. Significance and Use

5.1 This practice is intended for the digestion of metals and metalloids in airborne dust and dust wipe samples collected during various activities performed in and around workplaces, buildings and related structures.

5.2 This practice is applicable to the digestion of airborne dust and dust wipe samples collected in accordance with Test Method **D4532**, Guide **D6062**, Practice **D7144** or Guide **E1370** for airborne dust, and Practices **D6966**, **D7296**, **D7822**, or **E1728** using wipes that may or may not conform to Specifications **D7707** or **E1792**.

5.2.1 This practice is applicable to the digestion of airborne dust sample filters that have been removed from their sampling cassettes which have been wiped to collect all dust adhering to the side walls and included in the hard-walled containers as part of the collected samples.

5.2.2 This practice is applicable to the digestion of airborne dust samples that use acid-soluble cellulosic air sampling capsules with the entire contents of the cassettes transferred to hard-walled containers.

5.2.3 This practice is applicable to the digestion of settled dust samples collected using wipe materials in hard-walled containers.

5.3 Digestates prepared according to this practice are intended to be analyzed for metal and metalloid concentrations using spectrometric techniques such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES), graphite furnace atomic absorption spectrometry (GFAAS), and flame atomic absorption spectrometry (FAAS) (see Test Methods **D4185**, **D6785**, **D7035**, **D7439**, **E1613**, **E3193**, and **E3203**), or for lead using electrochemical techniques such as anodic stripping voltammetry (see Practice **E2051**), or for beryllium using optical fluorescence detection (see Test Method **D7202**).

5.4 Laboratories developing in-house test methods using this procedure shall determine precision and bias in accordance with the principles laid down by their accrediting agency.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

⁵ Ashley, K., and O'Connor, P.F., eds., *NIOSH Manual of Analytical Methods*, 5th ed., 2017.

6. Reagents and Materials

6.1 Laboratory Supplies:

6.1.1 *Ultrasonic bath or heat block*, controllable to maintain the specified digestion temperature in wells sized to accept the screw top plastic centrifuge tubes used for the digestion.

6.1.2 *Centrifuge tubes*, plastic, 50-mL, with screw top caps.

6.1.3 *Pipettors*, mechanical, of assorted sizes as needed.

6.1.4 *Pipet tips*, plastic, disposable, of assorted sizes as needed.

6.1.5 *Labware*, plastic (for example, beakers, flasks, graduated cylinders, etc.), of assorted sizes as needed.

6.1.6 *Forceps*, plastic or plastic-coated.

6.1.7 *Thermometer*, to at least 100 °C.

6.1.8 Other general laboratory apparatus, as needed.

6.1.9 *Syringes*.

NOTE 2—The filtration process can be carried out by attaching a 25-mm diameter syringe filter to a 5- or 10-mL Luer lock syringe and pouring the liquid contents into the syringe. The liquid is forced out through the filter into a separate 15-mL centrifuge tube.

6.1.10 *Microfilters*.

NOTE 3—Filters with 0.2 to 0.45- μm pore size are acceptable. Preferred filters are made out of nylon, polyethersulfone, or hydrophilic polypropylene. Polytetrafluoroethylene (PTFE) filters are unsuitable because they may partially dissolve in ammonium bifluoride solution during filtration.

6.2 Reagents:

6.2.1 *Purity of Reagents*—Reagent grade chemicals shall be used in this practice. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,⁶ where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening accuracy of the determination.

6.2.2 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent defined as Type I in Specification **D1193** (ASTM Type I Water: minimum resistivity of 18 M Ω -cm or equivalent).

6.2.3 *Calibration Stock Solutions*—1000 $\mu\text{g}/\text{mL}$ of the metal, metals, or metalloids of interest in dilute nitric acid obtained from a commercial supplier accredited to ISO/IEC 17025 by an accreditation body complying with ISO/IEC 17011.

6.2.4 *Extraction (or Dissolution) Solution*—Mass fraction 1 % ammonium bifluoride (NH_4HF_2) solution (aqueous) for dissolution of metals and metalloids in collected particulate matter. (**Warning**—Ammonium bifluoride will etch glass, so it is essential that all NH_4HF_2 solutions be contained in plastic labware.)

6.2.5 *Nitric Acid*—Concentrated, suitable for atomic spectrometry analysis such as spectroscopic grade.

⁶ ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials, 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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7. Procedure

7.1 Sample Preparation:

7.1.1 Dust wipe samples should have been shipped from the sampling site to the laboratory in hard-walled containers such as plastic 50-mL centrifuge tubes. This digestion procedure is to be performed in the hard-walled containers as received by the laboratory.

7.1.2 Air samples should be removed from cassettes using forceps and transferred to hard-walled containers such as plastic 50-mL centrifuge tubes. Cassettes without acid-soluble cellulosic air sampling capsules should be wiped with a compatible wipe to collect all dust adhering to the side walls and the wipe included with the filter transferred to hard-walled containers. Cassettes with acid-soluble cellulosic air sampling capsules should have the entire contents of the cassettes transferred to hard-walled containers.

7.2 Pipet 5-mL of the 1 % ammonium bifluoride extraction solution into each of the plastic tubes containing the samples.

7.3 Carefully pipet 5-mL of concentrated nitric acid into each of the plastic tubes containing the samples.

7.4 Digest using hot block type heating or heated sonication.

7.4.1 Hot Block Heated Digestion:

7.4.1.1 Preheat the hot block to 95 °C (± 5 °C).

7.4.1.2 Reflux for 30-min allowing the reflux wetting band to reach no closer than 2-cm from the mouth of the tube.

7.4.1.3 Remove from the hot block and allow to cool to room temperature.

7.4.1.4 Pipet 5-mL of concentrated nitric acid into each of the plastic tubes containing the samples.

7.4.1.5 Reflux for 30-min allowing the reflux wetting band to reach no closer than 2-cm from the mouth of the tube.

7.4.1.6 Remove from the hot block and allow to cool to room temperature.

7.4.1.7 Make to 50-mL volume in the tube with water.

7.4.2 Heated Sonication Digestion:

7.4.2.1 Preheat the sonication bath to 85 °C (± 5 °C).

7.4.2.2 Sonicate for 30-min.

7.4.2.3 Remove from the bath and allow to cool to room temperature.

7.4.2.4 Pipet 5-mL of concentrated nitric acid into each of the plastic tubes containing the samples.

7.4.2.5 Sonicate for 30-min.

7.4.2.6 Remove from the bath and allow to cool to room temperature.

7.4.2.7 Make to 50-mL volume in the tube with water.

7.5 *Preparation of Calibration Standards*—Using calibration stock solution, concentrated nitric acid, and the 1 % aqueous ammonium bifluoride solution, prepare at least four standards covering the concentration range of interest.

7.6 Calibration:

7.6.1 *Preparation of Calibration Standards*—Using calibration stock solution, concentrated nitric acid, and the 1 % aqueous ammonium bifluoride solution as in 7.2 to 7.4 prepare at least four standards covering the concentration range of interest for each metal and metalloid of interest.