Designation: D8404 – 26a

# Standard Practice for Preparation of Soil Samples by Ammonium Bifluoride-Nitric Acid Digestion for Subsequent Analysis for Metals and Metalloids[1]

This standard is issued under the fixed designation D8404; 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 ammonium bifluoride-nitric acid digestion of soil samples and associated quality control (QC) samples for the determination of metals and metalloids using laboratory atomic spectrometry analysis techniques such as inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma atomic emission spectrometry (ICP-AES), flame atomic absorption spectrometry (FAAS), and graphite furnace atomic absorption spectrometry (GFAAS). For ammonium bifluoride-nitric acid digestion of airborne dust and dust-wipe samples for the determination of metals and metalloids, see Practice D8344.

1.2 This practice is based on U.S. EPA SW 846, Test Method 3050, Test Method D7202, and Practice D8344.

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]

D653 Terminology Relating to Soil, Rock, and Contained Fluids

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D3974 Practices for Extraction of Trace Elements from Sediments

D4840 Guide for Sample Chain-of-Custody Procedures

D7202 Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection

D8344 Practice for Ammonium Bifluoride and Nitric Acid Digestion of Airborne Dust and Dust-Wipe Samples for the Determination of Metals and Metalloids

D8457 Practice for Cleaning Glass and Plastic Labware Used in Metal and Metalloid Analyses

E288 Specification for Laboratory Glass Volumetric Flasks

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory (Withdrawn 2025)[3]

E1154 Specification for Piston or Plunger Operated Volumetric Apparatus and Operator Qualification

E1605 Terminology Relating to Lead in Buildings

E1727 Practice for Field Collection of Soil Samples for

[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.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

Subsequent Lead Determination

2.2 *U.S. Government Analytical Method:*[4]

U.S. EPA SW 846 Test Methods for Evaluating Solid Waste Physical/Chemical Methods

2.3 *ISO Standards:*[5]

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

ISO 1042 Laboratory glassware — One-mark volumetric flasks

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

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

## 3. Terminology

3.1 *Definitions*—For definitions of terms relating to the preparation of soil samples that are not given here, refer to Terminologies D653, 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.

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.

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 sample portion (split from an original sample) that is spiked with a known amount of analyte.

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) in a hot block or sonication bath using ammonium bifluoride and nitric acid. The digestate is diluted for final volume prior to measurement for metals and metalloids.

## 5. Significance and Use

5.1 There is a need to monitor the content of metals and metalloids to determine the presence of potential hazards. Hence, effective and efficient methods are required for the preparation of soil samples for determination of metals and metalloids present therein.

5.2 This practice may be used for the digestion of soil samples that are collected during various construction and renovation and hazard survey activities in and around buildings and related structures. The practice is also suitable for the digestion of soil samples for metal and metalloid analyses collected from other locations, such as near roads and steel structures. For other extraction procedures, see Practices D3974.

5.3 This practice is intended to be used to prepare samples that have been collected for hazard assessment purposes but may be used for other applications such as, for example, monitoring the effectiveness of remediation activities.

5.4 This practice may be capable of determining metals and metalloids bound within matrices, such as silica, that are not soluble in nitric acid alone.

5.5 This practice includes drying and homogenization steps to help assure that reported results are representative of the sample and are independent of potential differences in soil moisture levels among different sampling locations or changing weather conditions.

## 6. Reagents and Materials

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,* preferably a gravity convection type capable of maintaining a temperature of 100 °C to 120 °C.

6.1.3 *Grinding Apparatus*—Mortar and pestle (porcelain or agate), shatter box, or mixer mill.

6.1.4 *Micropipette with Disposable Plastic Tips* conforming to Specification E1154, sizes needed to make reagent additions, and spiking standards.

NOTE 1—In general, the following sizes should be readily available: 1 mL to 5 mL adjustable, 1000 mL, 500 mL, 250 mL, and 100 mL.

[4] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

[5] Available from International Organization for Standardization (ISO), ISO Central Secretariat, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, https://www.iso.org.