



Standard Practice for Separation of Beryllium from Other Metals in Digestion and Extraction Solutions from Workplace Dust Samples¹

This standard is issued under the fixed designation D7441; 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 separation of beryllium from other metals and metalloids in acid solutions, by extraction chromatography, for subsequent determination of beryllium by atomic spectroscopy techniques such as inductively coupled plasma atomic emission spectroscopy (ICP-AES).

1.2 This practice is applicable to samples of settled dust that have been collected in accordance with Practices **D6966** or **D7296**.

1.3 This practice is compatible with a wide variety of acid digestion techniques used in digesting settled dust samples, such as those described in Test Method **D7035**.

1.4 This practice is appropriate for the preparation of settled dust samples where an unacceptable bias is suspected or known because of spectral interferences caused by other metals or metalloids present in the sample. This practice may also be appropriate for the analysis of other types of samples.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D1193 Specification for Reagent Water

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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 Met-

alloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D7296 Practice for Collection of Settled Dust Samples Using Dry Wipe Sampling Methods for Subsequent Determination of Beryllium and Compounds

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3. Terminology

3.1 For discussion of pertinent terms not discussed here, see Terminology **D1356**.

3.2 *Definitions:*

3.2.1 *digestion*—dissolution using a combination of acids and other reagents of solid materials into solution for subsequent instrumental analysis.

3.2.2 *eluate*—the effluent from a chromatography or resin column.

3.2.3 *extraction chromatography*—liquid chromatography applied to the separation of metal ions utilizing selective organic extractants as the stationary phase and the aqueous solution as the mobile phase (**1**)³.

3.2.3.1 *Discussion*—Extraction chromatography resins consist of inert porous beads coated with selective extractants.

3.2.4 *spectral interference*—an interference caused by the emission from a species other than the analyte of interest.

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3.2.5 *surface wipe*—refers either to a wetted wipe, as defined in Practice **D6966**, or to a dry wipe, as defined in Practice **D7296**, used to gather material from a surface for subsequent analysis.

3.2.5.1 *Discussion*—The terms wipe sampling, swipe sampling, and smear sampling describe the techniques used to assess surface contamination on the skin, work surfaces, and PPE surfaces (for example, gloves, respirators, aprons, etc.)

3.2.6 *vacuum box*—container used to maintain a vacuum on a resin or column sample in order to increase the rate of flow

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 April 1, 2013. Published April 2013. Originally approved in 2008. Last previous edition approved in 2008 as D7441 – 08. DOI:10.1520/D7441-08R13.

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 The boldface numbers in parentheses refer to a list of references at the end of this standard.

of liquid through the column. Other vacuum sources such as an aspirator may be used.

4. Summary of Practice

4.1 This practice is based on using extraction chromatography resin to separate beryllium ions from other metal ions in extracts and digestates of surface wipe samples.

4.2 Surface wipe samples are collected using Practice **D6966** or Practice **D7296**, and are then digested or extracted into solution by mineral acids.

4.3 The pH of the solution is adjusted to between 1 and 2 with sodium acetate.

4.4 The sample is then loaded onto the extraction chromatography resin column, where beryllium is retained. Matrix interferences are rinsed from the column with 0.2 M nitric acid.

4.5 Beryllium is selectively eluted from the resin with 4 M nitric acid and is available for analysis using the spectroscopic techniques such as ICP-AES (See Test Method **D7035**).

5. Significance and Use

5.1 Beryllium is an important analyte in industrial hygiene because of the risk of exposed workers developing Chronic Beryllium Disease (CBD). CBD is a granulomatous lung disease that is caused by the body's immune system response to inhaled dust or fumes containing beryllium, a human carcinogen (2). Surface wipe samples and air filter samples are collected to monitor the workplace. This practice addresses the problem of spurious results caused by the presence of interfering elements in the solution analyzed. The practice has been evaluated for all elements having emission spectra near the 313.042 and 313.107 nm beryllium lines, as well as elements of general concern including aluminum, calcium, iron and lead. Below is a table listing each possible spectrally interfering element:

Cerium	Chromium	Hafnium	Molybdenum
Niobium	Thorium	Titanium	Thulium
Uranium	Vanadium	Uranium	

Measurement of beryllium on the order of 1 ppb (0.003 µg Be/100 cm² wipe sample) has been successfully accomplished in the presence of spectrally interfering elements on the order of hundreds of ppm. This method has been validated on matrices containing 10 mg of each of the above elements. In some cases including interferents such as chromium and calcium, the single 2 mL beryllium extraction chromatography resin can handle >100 mg of total dissolved solids and still deliver >90 % beryllium yield. Should the matrix contain greater amounts of contaminants, additional resin may be used or, more likely, a combination of different resins may be used. (3,4).

6. Reagents and Materials

6.1 Equipment:

6.1.1 50-mL polypropylene centrifuge tubes,

6.1.2 2-mL cartridges of beryllium extraction chromatography resin⁴,

6.1.3 Syringe barrel (or similar size reservoir),

6.1.4 Vacuum box, with fittings compatible with the cartridges,

6.1.5 Delivery pipet, 5 mL or 10 mL,

6.2 Reagents:

(See **Note 1**.)

6.2.1 Deionized Water, Type I or Type II in accordance with Practice **D1193**,

6.2.2 Nitric Acid (HNO₃), concentrated, ρ~1.42 g/mL (~70 % m/m),

6.2.3 Boric Acid (H₃BO₃),

6.2.4 Ammonium Oxalate monohydrate (NH₄C₂H₄O),

6.2.5 Sodium acetate trihydrate (C₂H₃O₂Na·3H₂O),

6.2.6 Methyl Violet (indicator grade).

NOTE 1—Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society 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 the accuracy of the determination.

6.3 Solutions:

6.3.1 3.4 M Sodium acetate + 0.2 M Ammonium oxalate + (0.2 M Boric acid): To a 1-L volumetric flask, add 500 mL of deionized water. Add 12.37 grams of Boric acid and mix until dissolved. Add 28.42 grams of Ammonium oxalate monohydrate and mix until dissolved. Add 462.68 grams of Sodium acetate trihydrate and mix. Fill to 1 L with deionized water and mix until dissolved.

NOTE 2—Boric acid is added to combat the effect of HF used in digestion. If HF is not used, the Boric acid may be omitted.

6.3.2 *Ammonium Oxalate, 0.25 M*: To a 1-L volumetric flask, add 500 mL of deionized water. Add 35.53 grams of Ammonium oxalate monohydrate and mix until dissolved. Fill to 1 L with deionized water and mix until dissolved.

6.3.3 *Nitric Acid, 0.2 M*: To a 1-L volumetric flask, add 200 mL of deionized water. Add 12.5 mL of concentrated (70%) nitric acid (trace metal grade) and mix. Fill to 1 L with deionized water and mix thoroughly.

6.3.4 *Nitric Acid, 4.0 M*: To a 1-L volumetric flask, add 200 mL of deionized water. Add 250 mL of concentrated (70 %) nitric acid (trace metal grade) and mix. Fill to 1 L with deionized water and mix thoroughly.

6.3.5 *Methyl Violet, 0.1 % solution, in deionized water*: 0.1 gm Methyl Violet per 100 mL of water.

7. Procedure

7.1 Wipe Digest Preparation:

7.1.1 Prepare resin load solution in accordance with the chemical reagents normally used to digest beryllium surface wipes.

⁴ Beryllium extraction chromatography resin cartridge: 2 mL bed volume, 50-100 micron beads, bis (2-ethylhexyl) methanediphosphonic acid sorbed onto acrylic ester beads (3, 4, 5). Quality control parameters for the resin are stated in subsection 8.6.