



Designation: D7458 – 21

Standard Test Method for Determination of Beryllium in Soil and Sediment Using Ammonium Bifluoride Extraction and Fluorescence Detection¹

This standard is issued under the fixed designation D7458; 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 test method is intended for use in the determination of beryllium in samples of soil and sediment. This test method can be used for purposes such as environmental remediation projects where beryllium is a contaminant of concern. It is also useful for characterization of levels of beryllium in soil at sites where beryllium is in mining or manufacturing applications, and for determination of background levels of beryllium in soil.

1.2 This test method assumes that samples of soil or sediment are collected using appropriate and applicable methods.

1.3 This test method includes a procedure for extraction (dissolution) of beryllium in dilute ammonium bifluoride, followed by analysis of aliquots of the extract solution using a beryllium-specific fluorescent dye.

1.4 For a 500 mg sample, the lower limit of the working range is approximately 0.04 mg Be/kg (5 $\times$ dilution) or 0.1 mg Be/kg (20 $\times$ dilution). The working range extends to concentrations of at least 500 mg Be/kg.

1.5 No detailed operating instructions are provided because of differences among various makes and models of suitable fluorometric instruments. Instead, the analyst shall follow the instructions provided by the manufacturer of the particular instrument. This test method does not address comparative accuracy of different devices or the precision between instruments of the same make and model.

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

1.7 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice [D6026](#).

¹ This test method 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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1.7.1 For purposes of comparing a measured or calculated value(s) with specified limits, the measured or calculated value(s) shall be rounded to the nearest decimal of significant digits in the specified limit.

1.7.2 The procedures used to specify how data are collected/recorded, or calculated, in this standard are regarded as the industry standard. In addition, they are representative of the significant digits that generally should be retained. The procedures used do not consider material variation, purpose for obtaining the data, special purpose studies, or any considerations for the user's objectives; and it is common practice to increase or reduce significant digits of reported data to be commensurate with these considerations. It is beyond the scope of this standard to consider significant digits used in analytical methods for engineering data.

1.8 *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.*

1.9 *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](#)

[D1193 Specification for Reagent Water](#)

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

[D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as](#)

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.

Used in Engineering Design and Construction

D4753 Guide for Evaluating, Selecting, and Specifying Balances and Standard Masses for Use in Soil, Rock, and Construction Materials Testing

D4840 Guide for Sample Chain-of-Custody Procedures

D5730 Guide for Site Characterization for Environmental Purposes With Emphasis on Soil, Rock, the Vadose Zone and Groundwater (Withdrawn 2013)³

D6026 Practice for Using Significant Digits and Data Records in Geotechnical Data

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

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

2.2 Other Standards:

ISO 594-2 Conical Fittings with a 6 % (Luer) Taper for Syringes, Needles and Certain Other Medical Equipment—Part 2: Lock Fittings

EN ISO 8655-2 Piston-Operated Volumetric Pipettes—Part 2: Piston Pipettes⁴

3. Terminology

3.1 Definitions—For definitions of common technical terms used in this standard, refer to Terminology **D653** and **D1356**.

4. Summary of Test Method

4.1 This standard test method is used to determine the beryllium content of soil or sediment. Samples are collected in the field using appropriate and applicable procedures, for example, those described in ASTM International standards; see Guide **D5730** for listings of appropriate sample collection standards. A 0.5 g aliquot is extracted using 3 % ammonium bifluoride solution heated at 90 °C for 40 hours. The presence of active fluoride ions (HF by dissociation of ammonium bifluoride in acidic medium) enables dissolution of refractory forms of beryllium, including silicates, borosilicates, and oxides. The extraction solution produced from each sample is then filtered and an aliquot of this extract is added to a pH-adjusted detection solution which contains a beryllium-specific fluorescence reagent. The fluorescence of this final solution is then measured on a calibrated fluorometer to quantify the amount of beryllium in the sample. This standard test method is adapted from Test Method **D7202**.

5. Significance and Use

5.1 Exposure to beryllium can cause a potentially fatal disease, and occupational exposure limits for beryllium in air and on surfaces have been established to reduce exposure risks

to potentially affected workers (**1**, **2**).⁵ Measurement of beryllium in matrices such as soil and sediment is important in environmental remediation projects involving beryllium contamination (**3**) and for establishment of background levels of beryllium at sites where anthropogenic beryllium may have been used (**2**, **4-6**). Sampling and analytical methods for beryllium are needed in order to meet the challenges relating to exposure assessment and risk reduction. Sampling and analysis methods, such as the procedure described in this test method, are desired in order to facilitate measurements of beryllium that can be used as a basis for management of remediation projects and protection of human health.

5.2 This test method can be used for purposes such as environmental remediation projects where beryllium is a contaminant of concern. It is also useful for characterization of levels of beryllium in soil at sites where beryllium is in mining or manufacturing applications, and for determination of background levels of beryllium in soil.

5.3 The limit of quantification of this test method varies with the dilution factor (see **13.6.1**). For the detection solution containing lysine the detection limit is 0.013 mg beryllium per kilogram of sample, based on a 0.5 g sample (**7**) extracted in a 50 mL extraction solution and analyzed using a dilution factor of 20×. When the lysine-free detection solution is used one may use a 20× dilution factor and obtain the same detection limit or use 5× dilution factor and obtain a detection limit of 0.004 mg/kg of sample.

NOTE 1—Users of this standard are cautioned that compliance with Practice **D3740** does not in itself assure reliable results. Reliable results depend on many factors; Practice **D3740** provides a means of evaluating some of those factors.

6. Interferences

6.1 This test method is specific for beryllium. Other solvated metal ions are either bound by ethylenediaminetetraacetic acid (EDTA) in the detection solution, or they precipitate out due to the high alkalinity of the detection solution. The fluorophore used for detection is specific for the beryllium divalent cation (Be^{+2}).

6.2 If iron or titanium are present in high excess in the sample (typically above 7 %), the resulting measurement solution may appear golden-yellow. In this case the solution is left for two hours or more for the iron or titanium (or both) to precipitate. The solution is then re-filtered using the same procedure as for filtering the extraction solution (after the dissolution step), prior to fluorescence measurement.

7. Apparatus

7.1 Sampling Equipment—Use sampling apparatus appropriate for the type of media being collected (for example, soil, rock, sediment, fly ash) and its location (for example, surface, subsurface, vadose zone). Guidance on selection of appropriate sampling apparatus can be found as referenced in Guide **D5730**.

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

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

⁵ The boldface numbers in parentheses refer to a list of references at the end of this standard.

7.2 Ultraviolet/Visible (UV/Vis) Fluorometer—A device with irradiance excitation lamp or light-emitting diode (excitation $\lambda = 365$ or 384 nm) and time-integrating visible detector (400 – 700 nm, $\lambda_{\text{max}} \approx 475$ nm).

7.3 Balance—Balances shall conform to Guide **D4753** and shall have a readability without estimation of 0.1 mg.

7.4 Oven—Vented, thermostatically controlled oven capable of maintaining a uniform temperature of $90\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ throughout the drying chamber.

7.5 Centrifuge Tubes—Plastic tubes with a capacity of 15 mL and if needed, 50 mL.

7.6 Polypropylene Bottles—Bottles with screw caps having a capacity between 60 mL to 100 mL.

7.7 Syringe Filters—Hydrophilic polypropylene or polyethersulfone filters in plastic housings with a pore size of 0.2 to $0.45\text{ }\mu\text{m}$ and having a diameter of 13 mm or 25 mm. A $0.2\text{ }\mu\text{m}$ pore size filter is preferred.

7.8 Syringes—Plastic syringes with a capacity of 5 mL or 10 mL.

7.9 Pipetters—Mechanical pipetters in an assortment of sizes as needed, with tolerances in accordance with EN ISO 8655-2.

7.10 Pipet Tips—Disposable plastic pipet tips in an assortment of sizes as needed, with tolerances in accordance with EN ISO 8655-2.

7.11 Fluorescence Cuvettes—Disposable, low fluorescence, 10 mm path length, transparent to UV/Visible radiation cuvettes.

7.12 Thermometric Device—A thermometric device capable of measuring the temperature range within which the test is being performed readable to $0.5\text{ }^{\circ}\text{C}$ or better and having an accuracy of at least $\pm 0.5\text{ }^{\circ}\text{C}$.

7.13 Miscellaneous Items—The following items may be needed: plastic beakers, plastic flasks, plastic graduated cylinders, plastic or plastic coated forceps, microfilters, respirators, masks, gloves, lab coats, safety eyewear.

7.14 pH Meter—For measurement of pH to within ± 0.1 pH unit.

8. Reagents

8.1 Purity of Reagents—Reagent grade chemicals shall be used. 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.⁶ 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 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.

8.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent as defined by Type I of Specification **D1193** (ASTM Type I Water: minimum resistance of $18\text{ M}\Omega\text{-cm}$ or equivalent).

8.3 Calibration Stock Solution— 1000 ppm beryllium in dilute nitric acid.

8.4 Ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate.

8.5 L-lysine monohydrochloride.

8.6 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS).

8.7 Sodium hydroxide (NaOH).

8.8 Extraction (or Dissolution) Solution—Mass fraction 3% ammonium bifluoride (NH_4HF_2) solution (aqueous) for dissolution of beryllium in collected specimens. The solution may be prepared by dissolving $30\text{ g} \pm 0.3\text{ g}$ of solid NH_4HF_2 in Type 1 water to a total volume of 1000 mL. **Warning**—Ammonium bifluoride will etch glass, so it is necessary that NH_4HF_2 solutions be contained in plastic labware.

8.9 Detection Solution— $63.4\text{ }\mu\text{M}$ 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS) (**8, 9**) / 2.5 mM ethylenediaminetetraacetic acid (EDTA)/ 50.8 mM lysine monohydrochloride (pH adjusted to 12.8 with NaOH): The aqueous detection reagent is prepared by the addition of 12.5 mL of 2.5 mM ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate and 25 mL of 107 mM L-lysine monohydrochloride to 3 mL of 1.1 mM 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS). The pH is adjusted to 12.85 with addition of sodium hydroxide and Type 1 water added to a total of 50 mL .

8.9.1 An alternative preparation of dye solution without lysine may be made by adding 1.104 g of EDTA and $64\text{ }\mu\text{moles}$ of the 10-HBQS dye in 900 mL of water. After a clear solution is obtained, 114.5 mL of 2.5 M NaOH is added and mixed to obtain the final dye solution.

8.9.1.1 Check the pH of the dye solution by using the pH meter; the measured pH should be 12.5 to 13.3 . If the pH requires adjustment to fall within the desired pH range, add 2.5 M NaOH dropwise until the measured pH is satisfactory.

NOTE 2—The pH of the dye solution was measured by Adams et al. (**7**) as 13.2 . The dye solution without lysine may be used for all analytical purposes and also provides superior detection limits.

9. Hazards

9.1 Ammonium bifluoride is highly corrosive, and is very toxic when in contact with the skin. Effects to the skin, including irritation and burns, may not be felt for several hours. Avoid exposure by contact with the skin. Use suitable personal protective equipment (including impermeable gloves and eye protection) when working with NH_4HF_2 . See **Appendix X1** for further pertinent safety information.

9.2 Hydrofluoric acid can cause serious medical issues. Upon contact with skin, it easily and quickly enters through the skin and into the tissues in the body where it damages cells. Upon inhalation, it can damage lung tissue and cause swelling and fluid buildup in the lungs. The seriousness of the health