# Standard Test Method for
# Determination of Beryllium in Soil, Rock, Sediment, and Fly Ash Using Ammonium Bifluoride Extraction and Fluorescence Detection[1]

## 1. Scope*

1.1 This test method is intended for use in the determination of beryllium in samples of soil, rock, sediment, and fly ash. 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, rock, sediment, or fly ash are collected using appropriate and applicable ASTM International standard practices.

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 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.

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

1.6 All observed and calculated values shall conform to the guidelines for significant digits and rounding established in Practice D6026.

1.6.1 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 design.

1.7 This test method contains notes that are explanatory and not part of mandatory requirements of the standard.

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 and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards associated with performance of this test method are described in Section 9.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D1193 Specification for Reagent Water
D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
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 in Geotechnical Data
D7202 Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection

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[1] This test method is under the jurisdiction of ASTM Committee D18 on Soil and Rock and is the direct responsibility of Subcommittee D18.01 on Surface and Subsurface Characterization.

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

***A Summary of Changes section appears at the end of this standard**

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 Pipettes[4]

## 3. Terminology

3.1 *Definitions*—For definitions of common technical terms in this standard, see Terminology D653.

## 4. Summary of Test Method

4.1 This standard test method is used to determine the beryllium content of soil, rock, sediment, and fly ash. Samples are collected in the field using procedures described in ASTM International standards (see Guide D5730 for listings of appropriate sample collection standards). A 0.5-gram 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.

4.2 The limit of quantification of this test method is 0.013 milligrams beryllium per kilogram of sample, based on a 0.5-gram sample (**1**).[5]

## 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 (**2, 3**). Measurement of beryllium in matrices such as soil, rock, sediment, and fly ash is important in environmental remediation projects involving beryllium contamination (**4**) and for establishment of background levels of beryllium at sites where anthropogenic beryllium may have been used (**2**). 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.

NOTE 1—The quality of the result produced by this standard is dependent on the competence of the personnel performing it, and the suitability of the equipment and facilities used. Agencies that meet the criteria of Practice D3740 are generally considered capable of competent and objective testing, sampling, inspection, and so forth. 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 highly 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 highly 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 dissolution 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 is found in the ASTM International standards referenced in Guide D5730.

7.2 *Instrumentation:*

7.2.1 *Ultraviolet/Visible (UV/Vis) Fluorometer,* with irradiance excitation lamp or light-emitting diode (excitation $\lambda = 380$ nm) and time-integrating visible detector (400–700 nm, $\lambda max \approx 475$ nm).

7.2.2 *Laboratory Balance,* capable of measuring to the nearest 0.1 mg.

7.2.3 *Laboratory Oven,* capable of maintaining a temperature of 90 ± 2°C.

7.3 *Laboratory Supplies:*

7.3.1 *Centrifuge Tubes,* plastic, 15-mL (plus 50-mL, if necessary).

7.3.2 *Polypropylene Bottles,* 60 to 100 mL, with screw caps.

7.3.3 *Syringe Filters,* 0.45-µm or smaller pore size filters, for example, 13 or 25-mm diameter nylon filters, in plastic housings.

7.3.4 *Syringes,* plastic, 5-mL or 10-mL.

7.3.5 *Pipetters,* mechanical, of assorted sizes as needed, with tolerances in accordance with EN ISO 8655-2.

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[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 the list of references at the end of this standard.

7.3.6 *Pipet Tips,* plastic, disposable, of assorted sizes as needed, with tolerances in accordance with EN ISO 8655-2.

7.3.7 *Fluorescence Cuvettes,* disposable, low fluorescence, 10-mm path length, transparent to UV/Visible radiation.

7.3.8 *Labware,* plastic (for example, beakers, flasks, graduated cylinders, and so forth), of assorted sizes as needed.

7.3.9 *Forceps,* plastic or plastic-coated.

7.3.10 *Personal Protective Wear,* for example, respirators, masks, gloves, lab coats, safety eyewear, and so forth, as needed.

7.3.11 *Thermometer,* to at least 100°C (accuracy ± 1°C).

7.3.12 Other general laboratory supplies as needed.

## 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. 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.

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 MΩ-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*—3 % ammonium bifluoride ($NH_4HF_2$) solution (aqueous) for dissolution of beryllium in collected particulate matter. The solution may be prepared by dissolving 30 ± 0.3 g of solid $NH_4HF_2$ in water to a total volume of 1000 mL. (**Warning—**Ammonium bifluoride will etch glass, so it is essential that all $NH_4HF_2$ solutions be contained in plastic labware.)

8.9 *Detection Solution*—63.4 µM 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS) (**5**) / 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 water added to a total of 50 mL.

## 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.

## 10. Sample Collection and Transport

10.1 *Sample Collection*—Collect samples in accordance with applicable ASTM International standards for the type of media (for example, soil, rock, sediment, fly ash) and the location being sampled (for example, surface, vadose zone). Refer to Guide D5730 in selecting applicable ASTM International standards for sample collection.

10.2 *Sample Transport*—If applicable (that is, if samples are transported to a different location prior to sample preparation and analysis), follow sampling chain-of-custody procedures to document sample traceability. Make sure that the documentation that accompanies the samples is suitable for a chain of custody to be established in accordance with Guide D4840.

## 11. Procedure

11.1 *Specimen Preparation*—Wear appropriate personal protection during specimen preparation and analysis activities. Perform specimen preparation and analysis in a clean area that is well removed from any possible beryllium contamination.

11.1.1 *Digestion of Samples:*

11.1.1.1 Accurately weigh (to the nearest 0.1 mg) a 0.5-gram aliquot of sample into a polypropylene bottle. Close the lid and gently tap the lid to make sure that all of the aliquot falls to the bottom of the bottle. The bottle size is typically 10 to 50 mL larger than the liquid to be added.

NOTE 2—Care should be taken to make sure that the aliquot(s) used are representative of the soil, rock, sediment, or fly ash from which the samples were taken. Guidance may be found in references (**6-9**).

11.1.1.2 Remove the lid and, using a graduated cylinder, add 50 ± 0.5 mL of 3 % ammonium bifluoride ($NH_4HF_2$) extraction solution '(see 8.8). Replace the lid, making sure the lid is tight.

11.1.1.3 Swirl the bottle to make sure that the specimen is completely wetted.

11.1.1.4 Repeat steps 11.1.1.1 through 11.1.1.3 for all specimens.

11.1.1.5 Place specimen bottles in a laboratory oven, preheated to 90 ± 2°C, for 40 hours.

NOTE 3—The 40-hour heating step may be reduced by using a more concentrated solution (up to 10 %) of $NH_4HF_2$. After extraction using concentrations at or above 5 % $NH_4HF_2$, dilution with water is required to maintain a pH of 12 or higher when mixed with the dye solution; this pH is required to achieve quantitative recovery (**10**). Method evaluation should consider the sample media, particle physical characteristics (such as shape and size) and the inertness of beryllium-containing compounds in the specimens being analyzed.

NOTE 4—The specimen size of 0.5 grams is typically required to sufficiently account for the heterogeneous nature of soil, rock, sediment, and fly ash samples (**11**). In cases where the matrix is sufficiently homogenous to allow a smaller specimen size (for example, 50 mg), the amount of extraction solution may be reduced (for example, to 5 mL instead of 50 mL).

11.1.1.6 Remove specimens from the oven and allow them to cool to ambient temperature.

11.1.2 *Filtration*—Filter aliquots (for example, 5 mL) of extract solution through inert microfilters into 15-mL centrifuge tubes.

NOTE 5—The filtration process can be carried out by attaching a 25-mm diameter syringe filter to a 5 or 10-mL syringe with lock fitting (as described in ISO 594-2) and pouring the liquid contents into the syringe. The liquid is forced out through the filter into a separate 15-mL centrifuge tube.

11.1.3 *Preparation of Measurement Solution:*

11.1.3.1 Pipet 100 µL of filtered solution extracts into fluorescence cuvettes. To this pipet 1.9 mL of detection (dye) solution and make sure these are mixed well. This is a 20× dilution factor (DF). Use of a different DF will require a change in Eq 1 and Table 1.

11.1.3.2 Make sure that the measurement solution is colorless, or near colorless. If the solution is golden yellow, wait for a minimum of two hours for the solution to clear, and then re-filter the solution, as described in 11.1.2, into a clean 15-mL centrifuge tube.

NOTE 6—If iron is present in high excess (typically more than 20 µM) in the sample, the resulting measurement solution may be golden-yellow. Waiting for two hours allows the iron to precipitate and the solution to clarify to colorless/near colorless.

11.2 *Fluorometer Set-Up*—Set up the fluorometer for excitation radiation from 360 to 390 nm and measurement of emission in a spectral window selected from a range of (at least) 440 to 490 nm. Allow appropriate warm-up of the system prior to analysis (follow manufacturer's instructions).

NOTE 7—For fluorescence measurement, an emission band pass filter with peak transmission wavelength at ~475 nm and with a full width at half maximum (FWHM) of less than ±20 nm have been shown to be effective (**10, 12**).

11.3 *Preparation of Calibration Standards*—Using calibration stock solution and detection solution, prepare at least four standards covering the concentration range of interest.

NOTE 8—For example: To measure from about 1 to 80 mg of beryllium in samples, calibration standards from 0 to 800 parts per billion (ppb) are recommended (see Table 1). Alternatively, a different range of calibration standards, covering the range of interest, may be used provided that a linear calibration curve can be produced.

11.4 *Calibration and Specifications:*

11.4.1 *Calibration Blank and Calibration Stock Standard Solutions Preparation*—Calibration blank is prepared by adding the 0 ppb standard and the detection solution at a proportion of 1:19 (by volume) into a cuvette suitable for fluorescence measurements. Calibration standard solutions are also made in a similar fashion where the calibration standard and the detector solution are mixed in a volumetric ratio of 1:19 (a 20× dilution). At least four standard measurement solutions, plus a blank, must be made for calibration. Make sure that these are mixed properly.

NOTE 9—For routine beryllium analyses, calibration stock standard solution concentrations of 0, 10, 40, 200 and 800 ppb are recommended; see Table 1 for an example. Concentrations may be adjusted based on anticipated beryllium content. Solutions may be made up in 3 % ammonium bifluoride; alternatively, commercially available solutions using 1 % ammonium bifluoride may be used. Use of either 1 % or 3 % ammonium bifluoride has been determined to be acceptable (**10**).

11.4.2 *Instrument Calibration:*

11.4.2.1 Using the calibration standard solutions prepared above, calibrate the instrument for fluorescence intensity versus the concentration of beryllium. A calibration curve using linear regression shall be obtained between the fluorescent intensity and the concentration of beryllium. Samples shall not be left in the instrument for longer than necessary for measurement. The instrument should be programmed to display the concentrations (in ppb) of the calibration solutions. The correlation coefficient should be equal or greater than 0.999.

NOTE 10—Leaving samples in the instrument for longer than necessary can cause changes in sample temperature and consequent change in signal intensity.

11.4.2.2 Verify calibration by measuring the highest concentration standard which should yield a value of within 10 % of the known value. The calibration shall be verified at a minimum of once every two hours (for example, after completing the measurement of the unknowns) to make sure that calibration still holds.

NOTE 11—Changes in temperature can cause a drift in the readings;

**TABLE 1 Preparation of Calibration Standards (Example)**

| Concentration of Beryllium Used in Calibration Standards | Final Concentration of Beryllium (ppb) in Calibration Standard Solutions | Corresponding Amount of Beryllium (Be) in Sample (mg/kg)[A] |
|---|---|---|
| 0.1 mL of 0 ppb standard + 1.9 mL of detection solution (DF = 20) | 0.0 | Corresponds to 0 mg/kg beryllium per sample when DF = 20 |
| 0.1 mL of 10 ppb standard + 1.9 mL of detection solution (DF = 20) | 0.50 | Corresponds to 1 mg/kg beryllium per sample when DF = 20 |
| 0.1 mL of 40 ppb standard + 1.9 mL of detection solution (DF = 20) | 2.0 | Corresponds to 4 mg/kg beryllium per sample when DF = 20 |
| 0.1 mL of 200 ppb standard + 1.9 mL of detection solution (DF = 20) | 10.0 | Corresponds to 20 mg/kg beryllium per sample when DF = 20 |
| 0.1 mL of 800 ppb standard + 1.9 mL of detection solution (DF = 20) | 40.0 | Corresponds to 80 mg/kg beryllium per sample when DF = 20 |

[A] Incorporating sample dilution factor for 50 mL of dissolution solution and 0.5 g of sample; note that volumes other than 50 mL and/or a different sample size will require a different appropriate dilution factor.

thus, it is important to verify calibration periodically.

11.4.3 The calibration of fluorescence intensity due to the amount of beryllium present can be accomplished in either of two ways: by examining instrument response due to *(a)* the concentration of beryllium in calibration solutions, or *(b)* in terms of the amount of beryllium in the media; see Table 1.

11.4.4 *Fluorescence Measurement*—Place the cuvette in the calibrated fluorometer and read the value of the fluorescence intensity that is measured by the instrument. Follow manufacturer instructions on applicable integration times to be used for fluorescence intensity measurements.

NOTE 12—The intensity calibration on the instrument may have been carried out in terms of absolute intensity or one of the following if the instrument automatically prepared a correlation using linear regression fit of concentration of beryllium in calibration standards, concentration of beryllium in calibration standard solutions or in terms of amount of beryllium in the medium (soil, rock, sediment, or fly ash). Table 1 shows a correlation between various standards, calibration standard solutions and the amount of beryllium in the sampling medium.

11.4.5 *Measurement of Off-Scale Specimens*—If a measurement solution exceeds the value of the highest standard used, re-prepare the measurement solution and re-analyze using the following steps:

11.4.5.1 Pipet 0.1 mL of the filtered solution obtained in step 11.1.2, and 0.9 mL of dissolution solution (this is a 10× dilution) into a clean 15-mL centrifuge tube, and shake well. A higher dilution may be used if needed.

11.4.5.2 Pipet 100 mL of the solution prepared in step 11.4.5.1 into a fluorescence cuvette. To this pipet 1.9 mL of the detection solution and make sure these are mixed well.

11.4.5.3 Repeat the measurement, using step 11.4.4, taking into account the additional dilution factor from 11.4.5.1.

## 12. Calculation

12.1 *Estimation of Method Detection Limit*—Estimate the method detection limit (MDL) under the working analytical conditions, and repeat this exercise whenever experimental conditions (for example, integration time) are changed.

12.1.1 Prepare at least ten blank test solutions from the lowest non-zero calibration solution used. Follow sample extraction and filtration procedures used to prepare sample test solutions (see 8.2).

12.1.2 For a selected integration time, make fluorescence measurements on the test solutions and calculate the MDL as three times the sample standard deviation of the mean concentration value.

12.2 *Calculation of Beryllium Concentration in Samples:*

12.2.1 From the calibration curve, obtain the solution concentration for each sample, $W_s$ (ppb), and the average media field blank, $W_b$ (ppb).

NOTE 13—Alternatively, the mass of beryllium per sample can be read directly from the calibration curve using the procedure described in 11.4, where $W_s$ and $W_b$ are the amounts of beryllium in ppb in the sample and the blank, respectively.

12.2.2 Using the values of $W_s$ and $W_b$ from 11.1.1, calculate the concentration $C$ (mg of beryllium per kg sample) by using Eq 1:

$$C = 2 \times D\left[W_s - W_b\right] \qquad (1)$$

NOTE 14—In Eq 1, the factor of two accounts for the sample size of 0.5 g, and $D$ represents the additional dilution factor (see 11.4.5.3).

NOTE 15—In Eq 1, the number of significant digits associated with the concentration, $C$, will be governed by the instrumentation used. Typical instrumentation provides results in either two or three significant digits.

## 13. Quality Control

13.1 *Laboratory and Field Blanks*—Carry reagent blanks (water and reagents) throughout the entire sample preparation and analytical process to determine whether the samples are being contaminated from laboratory activities. Process reagent blanks at a frequency of at least one per 20 samples, minimum of one per batch.

13.2 *Calibration Verification Standards*—Analyze calibration verification standards, using standards near the midpoint of the calibration range, to verify freedom from excessive drift. Process calibration verification standards at a frequency of at least one per 20 samples, minimum of one per batch.

13.3 *Certified Reference Materials (CRMs)*—If available, certified reference materials (CRMs) for beryllium shall be analyzed prior to or during routine use of the sample preparation and analytical method to establish whether the percent recovery relative to the certified value is satisfactory.

13.4 *Quality Control Samples:*

13.4.1 Carry spiked media and spiked duplicate media throughout the entire sample preparation and analytical process to estimate the method accuracy on the sample batch, expressed as a percent recovery relative to the true spiked value. Spiked samples and spiked duplicate samples consist of an appropriate certified reference material (CRM) with known beryllium concentration, to which known amounts of beryllium have been added. Process these quality control samples according to a frequency of at least 1 per 20 samples, minimum of one per batch.

NOTE 16—Alternatively, when the initial beryllium concentration is not known, the method of standard additions may be used to determine initial beryllium concentration.

13.4.2 Monitor the performance of the method by plotting control charts of the relative percent recoveries and of the relative percent differences between the spiked samples and spiked duplicate samples. If quality control results indicate that the methods is out of control, investigate the reasons for this, take corrective action, and repeat the analyses. See Guide E882 for general guidance on the use of control charts.

NOTE 17—Typically, recoveries of 100 ± 15 % are desired. However, for certain sample matrices, wider performance limits may be deemed acceptable.

13.5 *External Quality Assessment*—If the laboratory carries out analysis of beryllium in soil, rock, sediment, or fly ash, on a regular basis, it is recommended to participate in relevant external quality assessment and proficiency testing schemes.

## 14. Report: Test Data Sheet(s)/Form(s)

14.1 The methodology used to specify how data are recorded on the test data sheet/form, as given below, is covered in 1.6.

14.1.1 Record as a minimum the following general information (data):

14.1.1.1 Name and address of the laboratory performing the test;

14.1.1.2 Identification of the report and the date issued;

14.1.1.3 Name and address of the client;

14.1.1.4 Identification of the project;

14.1.1.5 Description and identification of each test sample, including sample receipt, chain-of-custody information, and unique sample identification;

14.1.1.6 Date(s) when the test was performed;

14.1.1.7 Identification of the standard test method(s) used (that is, this standard test method) and notation regarding any deviations from the standard test method;

14.1.1.8 Identification of any test results obtained from tests performed by a subcontractor;

14.1.1.9 Name of the person accepting technical responsibility for the test report;

14.1.1.10 Applicable quality assurance and quality control data, such as laboratory and field blanks, calibration verification standards, and quality control samples;

14.1.1.11 Information on instrumentation and equipment used; and

14.1.1.12 Instrument parameters used.

14.1.2 Record as a minimum the following test specimen data:

14.1.2.1 Beryllium concentration in milligrams of beryllium per kilogram of sample. Data may alternatively be recorded in micrograms of beryllium per gram of sample. Refer to 1.6 and 12.2 for guidance on the number of significant digits to record; and

14.1.2.2 Any other test specimen data required by the client or by cognizant accreditation bodies.

## 15. Laboratory Records

15.1 Records shall be maintained in accordance with Practice D3740, and shall include a copy of the field sample collection report.

15.1.1 Record all reagent sources (lot numbers and vendors) used for sample preparation and analysis in a laboratory notebook. Record any inadvertent deviations, unusual happenings and notable observations on a real-time basis as the samples are processed. Use these records to add supplemental information when reporting the results.

15.2 *Electronic Laboratory Notebooks*—If electronic laboratory notebooks, or ELNs, are used in lieu of a written laboratory notebook, procedures shall be implemented to preserve the data for the appropriate length of time, and to protect the integrity of the data recorded, including prevention of falsification or other unauthorized changes, and regular backup of data.

## 16. Precision and Bias[6]

16.1 *Statement of Precision*—Data for interlaboratory precision (in terms of repeatability and reproducibility) were processed in accordance with Guide E691. Repeatability was calculated by averaging the squares of the standard deviations of within-laboratory results for each beryllium level, hence the average within-laboratory variance is given by the repeatability variance, $(S_r)^2$. Reproducibility variance is expressed by $(S_R)^2 = (S_r)^2 + (S_X)^2$, where $S_X$ is the sample standard deviation of the mean value estimated from the average of reported interlaboratory test results for a given performance evaluation material. Relative standard deviations (*RSDs*) for interlaboratory reproducibility are computed by dividing the standard deviation $S_X$ by the mean interlaboratory test result for a particular performance evaluation material and then multiplying the quotient by 100 %. Precision data from the interlaboratory study are summarized in Table 2.

16.2 *Statement of Bias*—Estimates of analytical bias, $B$, were computed by simply dividing the difference between the measurand and the reference value by the reference value, $B = (\mu_i - R_i) / R_i$, where $\mu_i$ and $R_i$ are the mean and reference beryllium contents, respectively, for the $i$th beryllium loading level in each performance evaluation sample. Bias data from the interlaboratory study are summarized in Table 3 (**10, 13**).

16.3 *Interlaboratory Evaluation*—An interlaboratory evaluation of the method was carried out using samples of Canadian Till-1 standard soil that were spiked with beryllium oxide so that the samples spanned the range ≈2.4 – ≈240 mg Be per kg of sample (**9**). A total of six laboratories participated in the evaluation.

## 17. Keywords

17.1 beryllium; environment; fluorescence; fly ash; rock; sediment; soil

**TABLE 2 Precision Statistics for Beryllium Measurements from Performance Evaluation Soil Samples, as Computed Using Values Reported by Laboratories (n=6) Participating in the Interlaboratory Evaluation**

| Reference [Be], mg/kg | Interlaboratory Mean, mg/kg | $S_X$ (mg/kg) | $S_r$ (mg/kg) | $S_R$ (mg/kg) | $R_X$ (mg/kg) | $R_r$ (mg/kg) | Interlaboratory RSD (%) | Acceptable Range of Two Results (mg/kg)[A] |
|---|---|---|---|---|---|---|---|---|
| 2.4 | 2.43 | 0.217 | 0.0623 | 0.223 | 0.174 | 0.624 | 8.9 | 0.173 |
| 4.8 | 5.13 | 0.528 | 0.456 | 0.646 | 1.28 | 1.81 | 10.3 | 1.26 |
| 12.0 | 12.42 | 0.594 | 1.054 | 1.054 | 2.95 | 2.95 | 4.8 | 2.92 |
| 120 | 125.6 | 8.98 | 9.85 | 12.05 | 27.58 | 33.74 | 7.1 | 27.3 |
| 240 | 233.4 | 16.08 | 8.39 | 17.48 | 23.49 | 48.94 | 6.9 | 23.3 |

[A] As defined by Practice E177.

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[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D18-1016.

**TABLE 3 Bias Estimates for Beryllium Measurements from Performance Evaluation Soil Samples, Computed Using Mean Values from Tables 1 and 2**

NOTE 1—Reference values for beryllium loadings on the samples are given in parentheses.

| Native Soil (2.4 mg/kg Be) | Low spike (4.8 mg/kg Be) | Medium Spike (12.0 mg/kg Be) | High spike (120 mg/kg Be) | Very high spike (240 mg/kg Be) |
|---|---|---|---|---|
| 0.0125 | 0.0688 | 0.0350 | 0.0467 | -0.0275 |

# APPENDIX

## (Nonmandatory Information)

## X1. SPECIAL SAFETY PRECAUTIONS WHEN USING AMMONIUM BIFLUORIDE

X1.1 Ammonium bifluoride ($NH_4HF_2$) 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 acid-resistant gloves and eye protection) when working with $NH_4HF_2$.

X1.2 Because $NH_4HF_2$ does not have the high vapor pressure that hydrofluoric acid (HF) does, it does not pose the same hazard from fumes that is posed by HF. However, dermal exposure to $NH_4HF_2$ does pose hazards similar to those for HF.

X1.3 Ammonium bifluoride is dissociated into HF and $NH_4F$ in aqueous solution. Make sure that the nature and seriousness of hydrofluoric acid burns are understood before commencing work with this substance.

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**(12)** Minogue, E. M., Ehler, D. S., Burrell, A. K., McCleskey, T. M., Taylor, T. P., "Development of a Portable Fluorescence Method for the Detection of Beryllium on Surfaces," *Journal of ASTM International* , Vol 2, No. 9 [JAI 13168].

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## SUMMARY OF CHANGES

In accordance with Committee D18 policy, this section identifies the location of changes to the standard since the last edition (2008) that may impact the use of this standard.

*(1)* Referenced documents have been updated.
*(2)* Information on sample collection and transport has been placed into a new Section 10. Subsequent sections have been renumbered.
*(3)* Use of "sample" and "specimen" is now consistent with how they are defined in Terminology D653.
*(4)* Requirements for test data sheet(s)/form(s) in Section 14 have been updated.
*(5)* Information on electronic laboratory notebooks has been added to Section 15.

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