



Designation: D7202 – 22

Standard Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection¹

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1. Scope

1.1 This test method is intended for use in the determination of beryllium by sampling workplace air and surface dust.

1.2 This test method assumes that air and surface samples are collected using appropriate and applicable ASTM International standard practices for sampling of workplace air and surface dust. These samples are typically collected using air filter sampling, vacuum sampling or wiping techniques. See Guide [E1370](#) for guidance on air sampling strategies, and Guide [D7659](#) for guidance on selection of surface sampling techniques.

1.3 Determination of beryllium in soil is not within the scope of this test method. See Test Method [D7458](#) for determination of beryllium in soil samples.

1.4 This test method includes a procedure for extraction (dissolution) of beryllium in weakly acidic medium (pH of 1 % aqueous ammonium bifluoride is 4.8), followed by field analysis of aliquots of the extract solution using a beryllium-specific-optically fluorescent dye.

1.5 The procedure is suitable for on-site use in the field for occupational and environmental hygiene monitoring purposes. The method is also applicable for use in fixed-site laboratories.

1.6 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.

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

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

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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1.9 *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.*

1.10 *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](#)
- [D4840 Guide for Sample Chain-of-Custody Procedures](#)
- [D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps](#)
- [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 Determination of Metals and Metalloids](#)
- [D7296 Practice for Collection of Settled Dust Samples Using Dry Wipe Sampling Methods for Subsequent Determination of Beryllium and Compounds](#)
- [D7458 Test Method for Determination of Beryllium in Soil and Sediment Using Ammonium Bifluoride Extraction and Fluorescence Detection](#)
- [D7659 Guide for Strategies for Surface Sampling of Metals and Metalloids for Worker Protection](#)

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.

- D7707** Specification for Wipe Sampling Materials for Beryllium in Surface Dust
- D8358** Guide for Assessment and Inclusion of Wall Deposits in the Analysis of Single-Stage Samplers for Airborne Particulate Matter
- 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
- E1154** Specification for Piston or Plunger Operated Volumetric Apparatus
- E1370** Guide for Air Sampling Strategies for Worker and Workplace Protection

3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, see Terminology **D1356**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *wipe sample, n*—sample collected by wiping a representative surface of known area, as determined by Practice **D6966**, or equivalent method, with an acceptable wipe material as defined in Specification **D7707**.

4. Summary of Test Method

4.1 Particles potentially containing beryllium from workplace air or surfaces, or both, are collected in the field using procedures described in ASTM International standards. To extract (or dissolve) beryllium in the collected samples, the media in or on which the samples are collected (that is, air sample, vacuum sample or wipe) are treated using an acidic extraction solution containing dilute ammonium bifluoride, NH_4HF_2 (**1**).³ The presence of active fluoride ions (HF by dissociation of ammonium bifluoride in acidic medium) enables dissolution of refractory materials such as “high-fired” beryllium oxide. 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 optical fluorescence reagent (**1**, **2**). The fluorescence exhibited by this final solution is then measured on a calibrated fluorometer to quantify the amount of beryllium in the sample (**3**).

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 (**4-7**). 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 on-site and fixed-site laboratory measurement of trace beryl-

lium. Beryllium analysis results can then be used as a basis for exposure assessment and protection of human health.

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 (**1**). In case the sample is suspected of having fluorescent organic contaminants that are suspected to be present, then their presence can be checked and removed (**8**).

6.2 If the samples are suspected of having a contaminant that fluoresces and has excitation and emission spectra that overlap with that of the signal produced by the fluorescent dye bound to beryllium, then this contaminant needs to be removed. The presence of such a contaminant can be verified by subjecting the filtered sample to fluorescence excitation after the extraction step (without adding the fluorescent dye). If a fluorescence signal is detected, then that signal is ascribed to the presence of a fluorescent contaminant. To remove the contaminant, high-purity activated charcoal is added to the beryllium extraction solution and the extraction procedure is carried out at elevated temperature (80 to 90 °C for at least 45 minutes). If the beryllium extraction procedure has already been performed, then after the addition of activated charcoal, the extraction process is repeated at the elevated temperature. The solution is filtered to remove the activated charcoal before making the measurement solution. The measurement solution is made by the addition of the fluorescent dye solution to an aliquot of the extraction solution. Details of this process have been published (**8**).

6.3 If iron is present in high excess in the sample (typically more than 20 μM), the resulting measurement solution may appear golden-yellow. In this case the solution should be left for an hour or more for the iron to precipitate. The solution should then be 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:*

7.1.1 *Air Sampling*—Use air samplers and filters for collecting personal air samples as described in Test Method **D7035** and Guide **D8358**.

7.1.2 *Wipe Sampling*—Use wipe sampling apparatus for collecting surface dust samples as described in Practice **D6966** (or Practice **D7296** in special cases), using wipes meeting the specifications described in Specification **D7707**.

7.1.3 *Vacuum Sampling*—If wipe sampling is not advisable for surface sample collection, use vacuum sampling apparatus for collecting surface dust samples as described in Practice **D7144**.

7.2 *Instrumentation:*

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

7.2.2 *Mechanical Agitator or Heating Source*, shaker, rotator or ultrasonic bath; or heat block, oven or heating bath.

³ The boldface numbers in parentheses refer to a list of references at the end of this standard.

NOTE 1—For routine samples, a shaker, rotator, or ultrasonic bath is adequate. To achieve higher recoveries from beryllium oxide (especially “high-fired” BeO), a heat block, oven or heating bath is required.

7.3 Laboratory Supplies:

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

7.3.2 *Syringe filters*, 0.2 to 0.45- μm nylon, polyethersulfone or hydrophilic polypropylene, 13- or 25-mm diameter, in plastic housings.

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

7.3.4 *Pipettors*, mechanical, of assorted sizes as needed, meeting requirements of Specification E1154.

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

7.3.6 *Fluorescence cuvettes*, disposable, low fluorescence, 10-mm path length, transparent to UV/Vis radiation.

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

7.3.8 *Forceps*, plastic or plastic-coated.

7.3.9 *Personal protective wear*, for example, respirators, masks, gloves, lab coats, safety eyewear, etc., as needed.

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

7.3.11 Other general laboratory apparatus, as needed.

7.4 *Reagents*—Use only reagents of analytical spectroscopic grade or greater purity.

7.4.1 *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 M Ω -cm or equivalent)

7.4.2 *Calibration Stock Solution*—1000 ppm beryllium in dilute nitric acid or equivalent.

7.4.3 Ethylenediaminetetraacetic acid (EDTA) disodium salt dihydrate.

7.4.4 L-lysine monohydrochloride.

7.4.5 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS).

7.4.6 Sodium hydroxide.

7.4.7 *Extraction (or Dissolution) Solution*—1 % ammonium bifluoride (NH₄HF₂) solution (aqueous) for dissolution of beryllium in collected particulate matter. (**Warning**—Ammonium bifluoride will etch glass, so it is essential that all NH₄HF₂ solutions be contained in plastic labware.)

7.4.8 *Detection Solution*—63.4 μM 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS) / 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 10.7 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 (1-3). An alternative preparation of dye solution without lysine may be made by adding 1.104 g of EDTA and 64 μmoles of the 10-HBQS dye in 900 mL of water. After a clear solution is obtained, 114.5 mL of 2.5 N NaOH is added and mixed to obtain the final dye solution. The pH of the

dye solution is 13.2. The dye solution without lysine may be used for all analytical purposes and also provides superior detection limits (9).

NOTE 2—For on-site analysis, it is recommended that the extraction and detection solutions be prepared in a fixed-site laboratory or other such controlled environment prior to transport to the field.

8. Procedure

8.1 Sampling:

8.1.1 *Air Samples*—Collect workplace air samples for beryllium in accordance with Test Method D7035 and Guide D8358, using personal sampling pumps calibrated in accordance with Practice D5337.

8.1.2 *Wipe Samples*—Collect surface wipe samples for beryllium in accordance with Practices D6966 and D7296 using wipe materials that comply with Specification D7707.

NOTE 3—Practice D7296 should only be used when wetted wipe sampling is not physically feasible (for example, if the surface to be wiped would be compromised by use of wetted wipes). Otherwise, Practice D6966 should be used.

8.1.3 *Vacuum Samples*—If wipe sampling is inadvisable for surface dust sampling, collect surface vacuum samples for beryllium in accordance with Practice D7144.

8.1.4 *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. Ensure that the documentation that accompanies the samples is suitable for a chain of custody to be established in accordance with Guide D4840.

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

8.2.1 Extraction of Air Filter or Vacuum Samples:

8.2.1.1 Don clean gloves and open the samplers. Use either technique (1) or (2) to perform sample extraction on each collected sample:

(1) Using forceps, remove the filter from the cassette and place it into 15-mL centrifuge tube. The interior of the cassette shall be rinsed with extraction solution or wiped with another clean filter, and included in the centrifuge tube. For each vacuum sample, quantitatively transfer all loose dust into the centrifuge tube before removing the filter and then rinsing or wiping the inside walls of the sampler.

(2) Alternatively, the extraction shall be carried out directly within the sampling cassette (see Test Method D7035 and Guide D8358).

8.2.1.2 Pipet 5 mL of 1 % ammonium bifluoride extraction solution (see 7.4.7) into the centrifuge tubes or cassettes containing the air filter or vacuum samples.

8.2.1.3 Cap the centrifuge tubes or cassettes, and agitate or heat the samples:

(1) Activate the shaker, rotator, or ultrasonic bath, and agitate for a minimum of 30 minutes; or

(2) Preheat the heat block, oven or heating bath to 85 °C (± 5 °C), and heat the samples for a minimum of 30 minutes.

NOTE 4—Extraction is an example of a dissolution and solvating process. Method evaluation might indicate that for complete dissolution of