



Designation: D 7202 – 05

Standard Test Method for Determination of Beryllium in the Workplace Using Field- Based Extraction and 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.

1.3 This test method includes a procedure for on-site 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 fluorescent dye.

1.4 The procedure is targeted for on-site use in the field for occupational and environmental hygiene monitoring purposes.

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.

1.7 This test method contains notes which 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.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specifications for Reagent Water

D 1356 Terminology Related to Sampling and Analysis of Atmospheres

D 4840 Guide for Sampling Chain-of-Custody Procedures

D 5337 Practice for Flow Rate Calibration of Personal Sampling Pumps

D 6966 Practice for Collection of Settled Dust Samples Using Wipe Sampling Methods for Subsequent Metals Determination

D 7035 Test Method for Determination of Metals and Metalloids in Airborne Particulate Matter by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

D 7144 Practice for Collection of Surface Dust by Microvacuum Technique for Subsequent Determination of Metals

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E 882 Guide for Control Charts

E 1792 Specification for Wipe Sampling Materials for Lead in Surface Dust

3. Terminology

3.1 *Definitions*—For definitions of terms not appearing here, see Terminology **D 1356**.

3.2 *Definition of Terms Specific to This Test Method:*

3.2.1 *wipe, n*—a disposable towelette that is moistened with a wetting agent such as water (**E 1792**; **D 6966**).

3.2.1.1 *Discussion*—These towelettes are used for collecting samples of dust, potentially containing beryllium, from surfaces.

4. Summary of Test Method

4.1 Particles comprising 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 on-site using an acidic extraction solution. The presence of active fluoride ions (HF by dissociation of ammonium bifluoride in acidic medium) enables dissolution of refractory materials such as beryllium oxide. The

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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 field-portable fluorometer to quantify the amount of beryllium in the sample.

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). Sampling and analytical methods for beryllium are needed in order to meet the challenges relating to exposure assessment and risk reduction. Field-portable sampling and analysis methods, such as the procedure described in this test method, are desired in order to facilitate on-site measurement of beryllium. On-site beryllium analysis results can then be used as a basis for management of protection of human health.

6. Interferences

6.1 This test method is highly specific for beryllium. Other solvated metal ions are either bound by ethylene diamine tetraacetic acid (EDTA) in the detection solution, or they precipitate out due to the high alkalinity of the detection solution.

6.2 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 **D 7035**.

7.1.2 *Wipe Sampling*—Use wipe sampling apparatus for collecting surface dust samples as described in Practice **D 6966**.

7.1.3 *Vacuum Sampling*—If wipe sampling is not advisable, use vacuum sampling apparatus collecting surface dust samples as described in Practice **D 7144**.

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*, shaker or rotator.

NOTE 1—An ultrasonic bath is an acceptable alternative.

7.3 Laboratory Supplies

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

7.3.2 *Syringe filters*, 0.45- μm nylon, 13- or 25-mm diameter, in plastic housings

7.3.3 *Syringes*, plastic, 5-mL

7.3.4 *Pipettors*, mechanical, of assorted sizes as needed

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

7.3.6 *Fluorescence cuvettes*, disposable, 10-mm diameter, 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 Other general laboratory supplies as needed.

7.4 Reagents

7.4.1 *Water*—Unless otherwise indicated, references to water shall be understood to mean reagent as defined by Type I of Specification **D 1193** (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 Ethylenediamine tetraacetic acid disodium salt dihydrate (EDTA)

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 (NH_4HF_2) solution (aqueous) for dissolution of beryllium in collected particulate matter. (**Warning**—Ammonium bifluoride will etch glass, so it is essential that all NH_4HF_2 solutions are contained in plastic labware.)

7.4.8 *Detection Solution*—63.4 μM 10-hydroxybenzo[h]quinoline-7-sulfonate (10-HBQS) (3) / 2.5 mM ethylene diamine tetraacetic 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 ethylenediamine tetraacetic 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.

NOTE 2—It is recommended to prepare the extraction and detection solutions in a fixed-site laboratory 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 **D 7035**, using personal sampling pumps calibrated in accordance with Practice **D 5337**.

8.1.2 *Wipe Samples*—Collect surface wipe samples for beryllium in accordance with Practice **D 6966**.

8.1.3 *Vacuum Samples*—If wipe sampling is inadvisable for surface dust sampling, collect surface vacuum samples for beryllium in accordance with Practice **D 7144**.

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 D 4840.

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 Samples*

8.2.1.1 Don clean gloves and open the samplers. Using forceps, remove the filters from the cassette and place them into 15-mL centrifuge tubes.

NOTE 3—If the entire contents of the sampler are regarded as part of the sample, the interior of the cassette should be rinsed with extraction solution, or wiped with another clean filter, and included in the centrifuge tube. Alternatively, the extraction can be carried out within the sampling cassette (see Test Method D 7035).

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

8.2.1.3 Cap the centrifuge tubes and place them in a mechanical shaker or agitator.

8.2.1.4 Activate the mechanical shaker or agitator and agitate 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 beryllium, it may be necessary for the dissolution process to be assisted by ultrasonic energy, heat or longer treatment periods to obtain acceptable recoveries. This will be dependent upon the sample media, particle physical characteristics (such as shape and size) and the inertness of beryllium-containing compounds. Heating can aid in the dissolution of refractory compounds such as beryllium oxide.

8.2.1.5 If the samples are heated during the extraction step, they shall be cooled to ambient temperature before aliquots are removed prior to addition of the detection solution.

8.2.2 *Extraction of Wipe Samples*

8.2.2.1 Don clean gloves and, using forceps, place the wipes into 15- or 50-mL centrifuge tubes.

NOTE 5—The size of the wipes used for sampling (8.1.2) will determine the size of the centrifuge tubes to use for extraction. Small wipe materials, such as 47-mm diameter filters, can be placed into 15-mL centrifuge tubes. Larger wipes, however, will require the use of larger tubes such as 50-mL volume.

8.2.2.2 Pipet 5 mL or 10 mL of 1 % ammonium bifluoride extraction solution (see 7.4.7) into the centrifuge tubes containing the wipe samples.

NOTE 6—The size of the wipes used for sampling (8.1.2) and the size of the centrifuge tubes used for extraction will determine the volume of extraction solution to add. Small wipes in 15-mL tubes will require only 5 mL of extraction solution, but larger wipes in 50-mL tubes will require a minimum of 10 mL of extraction solution to ensure complete wetting and effective extraction.

8.2.2.3 Cap the centrifuge tubes and place them in a mechanical shaker or agitator.

8.2.2.4 Activate the mechanical shaker or agitator and agitate for a minimum of 30 minutes.

NOTE 7—Extraction is an example of a dissolution and solvating process. Method evaluation might indicate that for complete dissolution of beryllium, it may be necessary for the dissolution process to be assisted by ultrasonic energy, heat or longer treatment periods to obtain acceptable recoveries. This will be dependent upon the sample media, particle physical characteristics (such as shape and size) and the inertness of beryllium-containing compounds. Heating can aid in the dissolution of refractory compounds such as beryllium oxide.

8.2.2.5 If the samples are heated during the extraction step, they shall be cooled to ambient temperature before aliquots are removed prior to addition of the detection solution.

8.2.3 *Filtration*—Filter aliquots (for example, 5 mL) of extract solution through inert microfilters.

NOTE 8—0.45-micrometre filters are acceptable. Preferred filters are made out of nylon.

NOTE 9—The filtration process can be carried out by attaching a 25-mm diameter syringe filter to a 5- or 10-mL luer lock syringe and pouring the liquid contents into the syringe. The liquid is forced out through the filter into a separate 15-mL centrifuge tube.

8.2.4 *Measurement solution preparation*: Pipet 100 μ L of filtered solution extracts into fluorescence cuvettes. To this add 1.9 mL of detection (dye) solution and ensure these are mixed well.

NOTE 10—If iron is present in high excess (typically more than 20 μ M) in the sample, the resulting measurement solution may be golden-yellow. In this case the solution should be left for an hour for iron to precipitate. The solution should then be re-filtered using the same procedure as for filtering the dissolution solution and then used for fluorescence measurement.

8.3 *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 11—For fluorescence measurement, a band pass filter with peak transmission wavelength at \sim 475 nm and with a full width at half maximum (FWHM) of less than \pm 10 nm have been shown to be effective (4).

8.4 *Preparation of Calibration Standards*—Using calibration stock solution and 1 % aqueous ammonium bifluoride solution, prepare at least four standards covering the concentration range of interest.

NOTE 12—*Example*: To measure the range of 0.02 to 4 μ g of beryllium in the samples, a recommended range of calibration standards is 0, 10, 40, 200 and 800 parts per billion (ppb).

8.5 *Calibration and Specifications*

8.5.1 *Calibration Blank and Calibration Standard Solutions Preparation*—Calibration blank is prepared by adding in a proportion of 1:19 (by volume) the 0 ppb standard and the detection solution in a cuvette suitable for fluorescent measurements. Ensure that these are mixed properly. 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. At least four standard measurement solutions, plus a blank, must be made for calibration.

NOTE 13—For most standard fluorescence cuvettes, 0.1 mL of the calibration standard is added to 1.9 mL of the detection solution.