



# Standard Test Method for Determination of Beryllium in the Workplace Using Field- Based Extraction and Fluorescence Detection<sup>1</sup>

This standard is issued under the fixed designation D 7202; 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 ( $\epsilon$ ) 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 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  $\mu\text{M}$ ), 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- $\mu\text{m}$  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  $\text{M}\Omega\text{-cm}$  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 ( $\text{NH}_4\text{HF}_2$ ) solution (aqueous) for dissolution of beryllium in collected particulate matter. (**Warning**—Ammonium bifluoride will etch glass, so it is essential that all  $\text{NH}_4\text{HF}_2$  solutions are contained in plastic labware.)

7.4.8 *Detection Solution*—63.4  $\mu\text{M}$  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  $\mu$ 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  $\mu$ 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  $\mu$ 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.

8.5.2 *Instrument Calibration*—Using the calibration standard solutions prepared above, calibrate the instrument for fluorescence intensity versus the concentration of beryllium. A calibration curve using linear regression must be obtained between the fluorescent intensity and the concentration of beryllium. The correlation coefficient should be equal or greater than 0.999. Verify calibration by measuring the highest concentration standard which should yield a value of within 10 % of the known value. The calibration must be verified at a minimum of once every hour (for example, after completing the measurement of the unknowns) to ensure that calibration still holds.

NOTE 14—Changes in temperature can cause a drift in the readings, thus it is important to verify calibration periodically. In addition the samples must not be left in the instrument for longer than necessary for measurement, as that can also cause drift in temperature and consequent change in signal intensity.

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

8.5.4 *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 15—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 (for example, a surface wipe sample). Table 1 shows a correlation between various standards, calibration standard solutions and the amount of beryllium in the sampling medium.

## 9. Calculation

9.1 *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.

9.1.1 Prepare at least ten blank test solutions from unused filters or wipes, or both, of the same type used for sample collection. Follow sample extraction and filtration procedures used to prepare sample test solutions (see 8.2).

9.1.2 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.

9.2 *Calculation of Beryllium Concentration in Samples*—From the calibration curve, obtain the solution concentration for each sample,  $C_s$  (µg/L), and the average media field blank,  $C_b$  (µg/L).

NOTE 16—Alternatively, the mass of beryllium per sample can be read directly from the calibration curve using the procedure described in 8.5, where  $W_s$  and  $W_b$  are the amounts of beryllium in micrograms on the sample and the blank filter, respectively.

9.2.1 *Air Filter Samples*—Using the solution volumes of sample,  $V_s$  (mL), and media blank,  $V_b$  (mL), calculate the concentration,  $C$  (µg/m<sup>3</sup>) of Be in the air volume sampled,  $V$  (L), while accounting for the dilution factor  $DF$  (assuming equal dilution factors for samples and blanks):

$$C = DF \times [C_s V_s - C_b V_b] / V, \mu\text{g Be}/\text{m}^3. \quad (1)$$

NOTE 17—Alternatively,  $C = (W_s - W_b)/V_m$ , µg Be/m<sup>3</sup>, where  $W_s$  and  $W_b$  are the amounts of beryllium in micrograms on the sample and the average blank filter, respectively, and  $V_m$  is volume of the air sampled in m<sup>3</sup> (1000 L).

9.2.2 *Surface Samples*—Using the solution volumes of sample,  $V_s$  (mL), and average media blank,  $V_b$  (mL), calculate the concentration,  $C$  (µg/100 cm<sup>2</sup>) of Be in the sample obtained from wiping or vacuuming an area  $A$  (in dm<sup>2</sup> or 100 cm<sup>2</sup>), while accounting for the dilution factor  $DF$  (assuming equal dilution factors for samples and blanks):

$$C = DF \times [C_s V_s - C_b V_b] / A, \mu\text{g Be}/100 \text{ cm}^2. \quad (2)$$

NOTE 18—Alternatively,  $C = (W_s - W_b)/A$ , µg Be/100 cm<sup>2</sup>, where  $W_s$  and  $W_b$  are the amounts of beryllium in micrograms in the sample and the blank medium, respectively.

NOTE 19—Vacuum samples can also provide gravimetric information, that is, mass of beryllium per total mass of sample collected; see Practice D 7144 for details.

## 10. Quality Control

10.1 *Laboratory and Field Blanks*—Carry reagent blanks (water and reagents) and media blanks (unspiked filters or wipes, or both) throughout the entire sample preparation and analytical process to determine whether the samples are being contaminated from laboratory activities. Field blanks shall also

**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 Media (Filter or Wipe) (µg) <sup>A</sup>
0.1 mL of 0 ppb standard + 1.9 mL of detection solution	0.0	Corresponds to 0 µg beryllium per sample
0.1 mL of 10 ppb standard + 1.9 mL of detection solution	0.5	Corresponds to 0.05 µg beryllium per sample
0.1 mL of 40 ppb standard + 1.9 mL of detection solution	2.0	Corresponds to 0.2 µg beryllium per sample
0.1 mL of 200 ppb standard + 1.9 mL of detection solution	10.0	Corresponds to 1 µg beryllium per sample
0.1 mL of 800 ppb standard + 1.9 mL of detection solution	40.0	Corresponds to 4 µg beryllium per sample

<sup>A</sup>Incorporating sample dilution factor for 5 mL of dissolution solution; note that volumes other than 5 mL will require a different appropriate dilution factor.

be obtained. Process reagent blanks and field blanks at a frequency of at least one per 20 samples, minimum of one per batch.

### 10.2 Quality Control Samples

10.2.1 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 filters or wipes, or both, 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.

10.2.2 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 E 882 for general guidance on the use of control charts.

10.3 *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.

NOTE 20—Typically, recoveries of 100 % ± 15 % are desired. However, for certain sample matrices, wider performance limits may be deemed acceptable.

10.4 *External Quality Assessment*—If the laboratory carries out analysis of beryllium in workplace air or wipe samples, or both, on a regular basis, it is recommended to participate in relevant external quality assessment and proficiency testing schemes.

## 11. Records

11.1 Records shall be maintained in accordance with Test Method D 7035 (for air samples) and Practice D 6966 (for wipe samples), and shall include a copy of the field sample collection report.

11.1.1 *Laboratory Records*—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.

## 12. Report

12.1 Data to report shall include, at a minimum, the following:

12.1.1 All sample receipt and chain-of-custody information, if applicable;

12.1.2 All sample analysis results;

12.1.3 Applicable quality assurance and quality control data;

12.1.4 Information on instrumentation and equipment used;

12.1.5 Instrument parameters used;

12.1.6 Identity of laboratory and analyst(s);

12.1.7 Any other information deemed appropriate.

## 13. Precision and Bias

13.1 *Interlaboratory Evaluation*—An interlaboratory evaluation of the method was carried out using mixed cellulose ester (MCE) membrane filters and Whatman 541 filters that were spiked with beryllium standard solutions so that the filters spanned the range ≈0.05 – ≈0.5 µg Be per sample (5). Beryllium nitrate solutions were used for spikes.

13.1.1 Data for interlaboratory precision (in terms of repeatability and reproducibility) were processed in accordance with Guide E 691. 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  $(S_R)^2 = (S_r)^2 + (S_L)^2$ , where  $S_L$  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 repeatability and reproducibility ( $RSD_r$  and  $RSD_R$ , respectively) are then computed by dividing the standard deviations  $S_r$  and  $S_R$  by the mean interlaboratory test result for a particular performance evaluation material. Precision data from the interlaboratory study are summarized in Table 2.

13.1.2 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  $i^{\text{th}}$  beryllium loading level in each performance evaluation sample. Bias data from the interlaboratory study are summarized in Table 3 (5).

**TABLE 2 Repeatability and Reproducibility for Beryllium Measurements from Performance Evaluation MCE and Whatman 541 Filters, as Computed Using Values Reported by Laboratories (n=8) Participating in the Interlaboratory Evaluation.**

Media Beryllium Level	Average (µg Be)	$S_r$	$S_R$	$RSD_r$	$RSD_R$
<i>MCE Filters</i>					
Low	0.052	0.0034	0.0051	0.065	0.098
Medium Low	0.10	0.0052	0.0071	0.052	0.071
Medium High	0.21	0.012	0.020	0.057	0.095
High	0.43	0.0080	0.033	0.019	0.077
<i>Whatman 541 Filters</i>					
Low	0.054	0.0027	0.0039	0.050	0.072
Medium Low	0.11	0.0068	0.012	0.062	0.11
Medium High	0.21	0.012	0.014	0.057	0.067
High	0.41	0.012	0.025	0.029	0.061

**TABLE 3 Bias Estimates for Beryllium Measurements from Performance Evaluation MCE and Whatman 541 Filters, Computed Using Mean Values from Tables 1-3 Reference Values for Beryllium Loadings on the Filters are Given in Parentheses.**

Media	Low (0.05 µg Be)	Medium low (0.10 µg Be)	Medium high (0.20 µg Be)	High (0.40 µg Be)
MCE filters	0.040	0.00	0.050	0.075
Whatman 541 Filters	0.080	0.10	0.050	0.025

NOTE 21—Laboratory studies of the method have obtained beryllium recoveries of 85-95 % from filter and wipe media spiked with BeO (4).

#### 14. Keywords

14.1 air; beryllium; filter; fluorescence; wipe; workplace

#### REFERENCES

- (1) Code of Federal Regulations, 10 CFR Part 850, Chronic Beryllium Disease Prevention Program. U.S. Department of Energy: Washington, DC (1999).
- (2) American Conference of Governmental Industrial Hygienists, Threshold Limit Values & Biological Exposure Indices. ACGIH: Cincinnati, OH (2005); updated annually.
- (3) Matsumiya, H., Hoshino, H., Yotsuyanagi, T., A novel fluorescence reagent, 10-hydroxybenzo[h]quinoline-7-sulfonate, for selective determination of beryllium(II) ion at pg cm<sup>-3</sup> levels, *Analyst*, Vol. 126, pp. 2082-2086 (2001).
- (4) 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].
- (5) Ashley, K., McCleskey, T. M., Brisson, M. J., Goodyear, G., Cronin, J., Agrawal, A., Interlaboratory evaluation of a portable fluorescence method for the measurement of trace beryllium in the workplace, *Journal of ASTM International*, Vol. 2, No. 9 [JAI 13156].

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