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.

Designation: D7202 – 22

Standard Test Method for Determination of Beryllium in the Workplace by Extraction and Optical Fluorescence Detection[1]

This standard is issued under the fixed designation D7202; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is intended for use in the determination of beryllium 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.

1.9 *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 Micro-vacuum 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

[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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[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.

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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 beryllium. 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, $\lambda max \approx 475$ 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.