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National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1877

Beryllium Oxide Powder

This Standard Reference Material (SRM) is intended for use in laboratory analysis and health research for the development and validation of analytical methods and instruments used to determine beryllium, as well as for proficiency testing of laboratories involved in beryllium determinations [1,2]. A unit of SRM 1877 consists of one bottle containing 20 g of beryllium oxide powder.

Certified Value of Beryllium Mass Fraction: 0.3576 g/g ± 0.0024 g/g

The certified value is based on inductively coupled plasma optical emission spectrometry (ICP-OES) analysis of four sets of independently prepared samples. Each set was digested using a different digestion protocol. The ICP-OES instrument was calibrated with primary standards prepared gravimetrically from assayed, high-purity, beryllium metal.

The uncertainty in the certified value is calculated as

$$U = ku_c$$

where $k = 1.99$ is the coverage factor for a 95 % confidence interval, and u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [3]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effects of the uncertainty components associated with the ICP-OES results, possible bias among those results [4], and the uncertainty due to possible changes in moisture content during storage due to changing environmental conditions (see “Instructions for Use”).

Expiration of Certification: The certification of **SRM 1877** is valid, within the measurement uncertainty specified, until **15 March 2023**, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements leading to the certification of SRM 1877 was provided by M.R. Winchester and G.C. Turk of the NIST Analytical Chemistry Division; in cooperation with M.D. Hoover, A.B. Stefaniak, and G.A. Day (National Institute for Occupational Safety and Health, Morgantown, West Virginia) and L.D. Welch (B&W Technical Services, Y-12 National Security Complex, Oak Ridge, Tennessee); and with support from D.J. Weitzman (United States Department of Energy, Office of Health, Safety and Security, Germantown, Maryland) and S.D. Johnson (United States National Nuclear Security Administration, Washington, District of Columbia).

This SRM was prepared and packaged according to NIST specifications by Brush Wellman, Inc. (Elmore, Ohio), under the direction of T. Civic and K. Kampfer (Brush Wellman, Inc., Elmore, Ohio).

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 18 September 2008
See Certification Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Sets of samples for ICP-OES analysis, including primary standards for calibration, were prepared by T.J. Oatts (BWXT Y-12 National Security Complex, Oak Ridge, Tennessee), C. Coleman (Savannah River Site, Aiken, South Carolina), and D. Nadratowski and R. Sud (Bureau Veritas North America, Inc., Novi, Michigan).

The ICP-OES analyses were performed by T.A. Butler and M.R. Winchester of the NIST Analytical Chemistry Division.

Characterization of the beryllium oxide powder with regard to gain or loss of moisture during storage was performed according to NIST specifications by D.C. Sbarra, A.B. Stefaniak, and M.D. Hoover (National Institute for Occupational Safety and Health, Morgantown, West Virginia).

Specific surface area and specific gravity were measured by D.C. Sbarra, A.B. Stefaniak, and M.D. Hoover (National Institute for Occupational Safety and Health, Morgantown, West Virginia). Count median diameter, mass median diameter, and the associated geometric standard deviations were determined by A.B. Stefaniak, M.G. Duling, and M.D. Hoover (National Institute for Occupational Safety and Health, Morgantown, West Virginia). Transmission electron microscopy was performed by R.M. Dickerson (Los Alamos National Laboratory, Los Alamos, New Mexico).

Statistical consultation was provided by S.D. Leigh of the NIST Statistical Engineering Division.

The development and certification of this SRM were sponsored by the United States National Nuclear Security Administration.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Traceability: Traceability of measurement results to a given reference must be established through an unbroken chain of comparisons, each having stated uncertainties [5]. Comparisons may be based on chemical measurements performed using various spectroscopic or classical methods of analysis. This SRM can be used to establish traceability of the results of beryllium measurements to NIST measurement results and standards. One approach is to use the SRM as a method validation sample.

Preparation[1]: The beryllium oxide powder was prepared using the following procedure: Beryllium hydroxide was reacted with sulfuric acid, and the reaction product was filtered, concentrated, and cooled to yield beryllium sulfate tetrahydrate crystals. These crystals were calcined in an oxygen atmosphere at a temperature between 1100 °C and 1200 °C to drive off volatile gases, yielding aggregated clusters of individual primary particles of crystalline beryllium oxide. The calcination helped to ensure that all constituent forms of beryllium were converted to the oxide. This type of beryllium oxide is often called "high-fired." The calcined material was size separated to pass a 20 mesh screen, blended, and bottled according to NIST specifications.

The beryllium oxide powder was obtained from a single lot of material from the beryllium oxide production line located at the Brush Wellman, Inc. facility in Elmore, Ohio. The Brush Wellman, Inc. commercial product designation for this material is "UOX grade beryllium oxide powder." This material has been utilized in studies described in publications that may be of interest to the user [6-10].

INSTRUCTIONS FOR USE

CAUTION: This SRM consists of beryllium oxide powder. Users of this SRM should be aware that exposure to particles, fumes, or solutions of beryllium-containing materials may cause chronic beryllium disease (CBD), a potentially disabling or even fatal respiratory disease [11]. The International Agency for Research on Cancer [12] and the National Toxicology Program [13] list beryllium as a known carcinogen. Controlling beryllium exposures to prevent CBD should also reduce the risk of lung cancer. ***Users of this SRM should read and thoroughly understand the provided Material Safety Data Sheet (MSDS) prior to use, and should take steps to prevent inhalation or skin exposure to the material.*** Questions or requests for additional information regarding the material and procedures for safe handling should be directed by telephone or in writing to the Brush Wellman, Inc. Product

[1] Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Stewardship Department listed on the Brush Wellman Material Safety Data Sheet at http://www.brushwellman.com. Product safety information, such as Safety Facts, is also available from the web site.

Given the small particle sizes of the material and the effects of static charge and ambient or user-generated air currents, special care must be taken to ensure that the powder does not become airborne during handling. The outside surfaces of the SRM bottle have been thoroughly cleaned of residual beryllium oxide powder prior to shipping. However, after being opened the outside of the SRM bottle may become contaminated. Opening of the bottle should take place inside a fume hood or other appropriate containment apparatus.

Drying: The certified beryllium mass fraction value was determined in the SRM material without drying of the material to remove moisture. In order to make use of the certified value, the material should be used without drying. The moisture content at the time of certification was approximately 0.2 % (*m/m*). It is not expected to change significantly when stored in the original closed SRM bottle under normal laboratory conditions, although absence of significant changes cannot be guaranteed. Consequently, the uncertainty given for the certified beryllium mass fraction includes a component of uncertainty to account for possible changes in moisture content.

Storage and Transport of a Partially Used SRM Bottle: A partially used SRM bottle should be tightly closed and stored inside a fume hood or other appropriate containment apparatus under normal laboratory conditions. If the SRM bottle is to be removed subsequently from the fume hood or containment apparatus for storage in another location, the bottle should first be cleaned externally and placed inside a clean outer container.

Information Values: A number of physical characteristics pertaining to this SRM have been measured. These are reported as information values in Table 1. A NIST Information Value is a value that may be of interest to the user, but for which insufficient information is available to assess the uncertainty [14]. Consequently, information values are reported without uncertainty estimates. Geometric standard deviation values are given for the count median diameter and mass median diameter to help describe the log-normal particle size distributions.

Table 1. Information Values

Measurement	Value	Geometric Standard Deviation
Specific surface area[(a)]	9.8 m^2/g	--
Specific gravity[(b)]	3.01	--
Count median diameter (CMD) and size distribution of the primary beryllium oxide particles[(c)]	0.12 μm	1.5
Mass median diameter (MMD) and size distribution of the primary beryllium oxide particles[(d)]	0.17 μm	1.4

[(a)] Specific surface area was measured by the Brunauer, Emmett, and Teller (BET) nitrogen gas adsorption method in accordance with ASTM B922-02: Standard Test Method for Metal Powder Specific Surface Area by Physical Adsorption (ASTM, 2002). Triplicate measurements were made on duplicate samples taken from each of two bottles of the SRM. Prior to BET analysis, the samples were outgassed under vacuum (1.7 Pa) for a minimum of 3 h at 200 ºC.

[(b)] Specific gravity was measured by helium gas pycnometry. Triplicate measurements were made on duplicate samples taken from each of two bottles of the SRM. The standard deviation of the set of all twelve measurements was 0.04.

[(c)] Count median diameter and geometric standard deviation of the primary beryllium oxide particle size distribution was determined from measurement of the Feret's diameter of 100 primary particles by transmission electron microscopy.

[(d)] Mass median diameter and geometric standard deviation of the primary beryllium oxide particle size distribution was determined by transformation of the count diameter distribution to a mass distribution under the assumption of smooth, spherical particle geometry.

PARTICLE MORPHOLOGY

The transmission electron micrograph in Fig. 1 illustrates the morphology of the beryllium oxide particles.

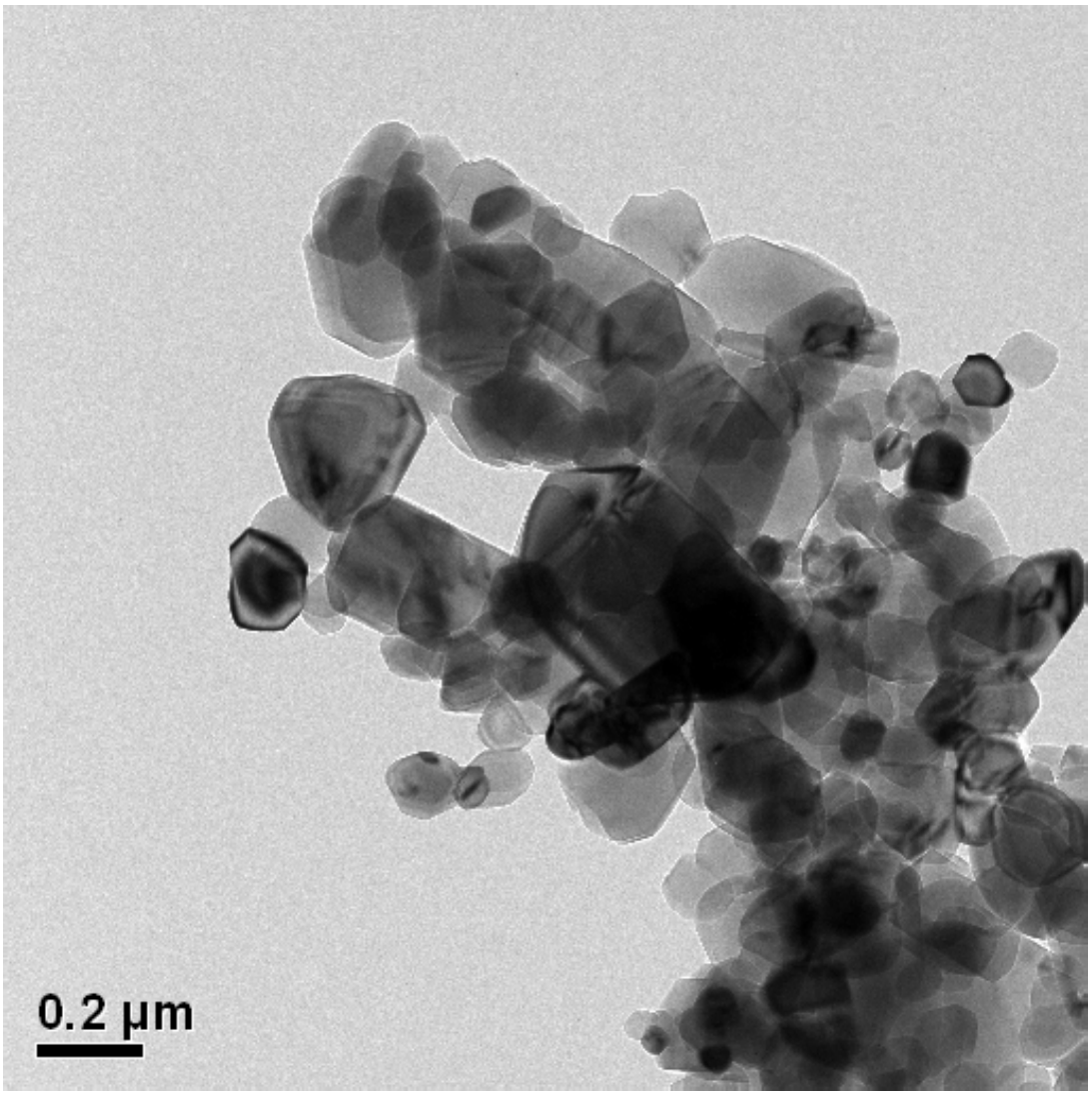

Figure 1

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Certificate Revision History: 18 September 2008 (Corrected technical contact name, clarified the directing of questions for material's safe handling, and editorial changes.); 20 May 2008 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.