

Designation: C 708 - 08

Standard Specification for Nuclear-Grade Beryllium Oxide Powder¹

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1. Scope

- 1.1 This specification defines the physical and chemical requirements of nuclear-grade beryllium oxide (BeO) powder to be used in fabricating nuclear components.
- 1.2 This specification does not include requirements for health and safety.^{2,3,4} It recognizes the material as a Class B poison and suggests that producers and users become thoroughly familiar with and comply to applicable federal, state, and local regulations and handling guidelines.²
- 1.3 Special tests and procedures are given in Annex A1 and Annex A2.
- 1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

- 2.1 ASTM Standards:⁵
- C 373 Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products
- C 859 Terminology Relating to Nuclear Materials
- C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- E 11 Specification for Wire Cloth and Sieves for Testing
- E 105 Practice for Probability Sampling Of Materials

2.2 ANSI Standard:

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities⁶

2.3 U.S. Government Standard:

Title 10, Code of Federal Regulations, Energy Part 50 (10CFR50) Domestic Licensing of Production and Utilization Facilities⁷

3. Terminology

- 3.1 Definitions:
- 3.1.1 Terms shall be defined in accordance with Terminology C 859 except as defined in 3.2.
 - 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 beryllium oxide powder—BeO that contains no hard aggregates larger than No. 20 sieve designation (850 µm).
 - 3.2.2 buyer—organization issuing the purchase order.
- 3.2.3 *calcination*—process of heating a material to a high temperature but without fusing in order to drive off volatile matter or to effect physical, chemical, or crystallographic
- 3.2.4 powder lot—that quantity of beryllium-oxide powder processed such that samples taken in accordance with the procedures of Section 8 can be considered as representative of the entire powder lot.
 - 3.2.5 *seller*—beryllium oxide powder supplier.

4. Ordering Information

- 4.1 The beryllium oxide powder must meet the chemical (Section 5) and physical (Section 6) requirements of this specification, based upon samples taken in accordance with Section 8. Deviation from these procedures and requirements or additions to them must be agreed upon between the buyer and the seller.
- 4.2 To be consistent with the requirements in 4.1, the buyer may specify the following information on the order:
 - 4.2.1 Quantity (weight of delivered product),
 - 4.2.2 Lot size,

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² ICC Regulations, Tariff 12, Code of Regulations, Title 49, Office of the Federal Register, National Archives and Record Services, General Services Administration, Washington, DC.

³ Breslin, A. J., arid Harris, W. B., "Health Protection in Beryllium Facilities: Summary of 10 Years of Experience," Health and Safety Laboratory, Vol 26, May

⁴ Ferreira, L. E., "Recommended Practices for Safe Handling of Beryllium Oxide Ceramics," Symposium on Materials and Electron Device Processing, ASTM STP 300, ASTTA, 1961.

⁵ 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.

⁶ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

⁷ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http:// www.access.gpo.gov.

- 4.2.3 Degree of calcination (optional),
- 4.2.4 Nominal particle size range and applicable tolerances in accordance with Specification E 11 (U.S. Standard Sieve Series). For particle sizes smaller than No. 270 sieve designation (53 µm), the particle size distribution shall be agreed upon between the buyer and the seller, and
 - 4.2.5 Sampling requirements.

5. Chemical Composition

- 5.1 Beryllium-oxide powder is used for a variety of nuclear applications. Depending upon end use, the purity requirements may be quite variable.
- 5.2 Use analytical methods as agreed upon between the buyer and the seller.
- 5.3 Mass loss on drying a 5 to 10-g sample of the powder at 110 ± 5 °C for 1 h shall be 0.50 % maximum. After drying, the mass loss on ignition of the sample at $1000 \pm 50^{\circ}$ C for 3 h shall be 1.0 % maximum.
- 5.4 Impurity concentrations before drying (see 5.3) shall not exceed the limits given in Table 1.
- 5.5 Sample material (see 5.3) shall not exceed the impurity limits given in Table 2.
- 5.6 Other elements, in addition to those listed in Table 2, may be included in the buyer's impurity content requirements by mutual agreement between the buyer and the seller.
- 5.7 The total equivalent boron content (EEC) of the impurities shall not exceed 9 µg/g on a mass basis relative to BeO. The method of performing the calculation shall be as indicated in Practice C 1233. The individual EBC values are calculated as follows:

EBC of impurity = (EBC factor) \times (µg of impurity/g BeO)

EBC factor = (atomic mass of boron) \times (σ a impurity)/ (atomic mass or impurity) \times (σ a boron), and

= atomic absorption cross section in barns. σa 5.7.1 Should the EBC of additional elements not listed in

Practice C 1233 be of concern, their inclusion in the summation and their respective EBC factors must be mutually agreed upon between the buyer and the seller.

6. Physical Requirements

- 6.1 All of the beryllium-oxide shall be passed through a No. 20 sieve designation (850 µm) or equivalent.
- 6.2 The powder shall be at least 99.5 mass % less than a No. 100 sieve designation (150 µm) determined by the wet sieve test in Annex A1 or other test mutually agreed upon between the buyer and the seller.

TABLE 1 Impurity Limits for Beryllium-Oxide Powder Before Drying^A

Element	Maximum Concentration (μg/g BeO)
S	1500
F	500
CI	100
С	500
Р	300
S+F+CI+C+P	2000

A After drying sample in accordance with 5.3.

TABLE 2 Impurity Limits for Beryllium-Oxide Powder After Ignition

Element	Maximum Concentration (μg/g BeO)
Al	100
В	3
Cd	2
Ca	50
Cr	20
Co	5
Cu	10
Fe	50
Li	3
Mg	100
Mn	10
Ni	15
Si	150

- 6.3 Beryllium-oxide powders may exhibit different bulk or tap densities, or both, due to different production methods. The acceptable density values for nuclear grade BeO powder are as follows:
 - 6.3.1 Bulk Density—0.15 to 0.50 g/cm³, and
 - 6.3.2 Tap Density—0.30 to 1.00 g/cm³.
- 6.4 Sinterability—Test pellets shall be produced and measured in accordance with a sintering performance test agreed upon between the buyer and the seller. A sinterability performance test described in Annex A2 is presented as a guide.

7. Cleanliness

7.1 The powder lot shall be handled in a manner to avoid contamination by foreign matter such as dust, cleaning agents and organics, and materials such as plastics and paper used in packaging. Cleaning solutions, if used, shall be free of halides and nonvolatile additives and shall be removed from the powder prior to sampling and packaging.

8. Sampling

- 8.1 Sampling plans to meet acceptance criteria and inspection and measurement procedures that describe the method of compliance with this specification shall be established by the seller and submitted to the buyer for approval. The degree of sampling where not specified in this specification, varies with the application and for this reason should be specified on the purchase order. Practice E 105 is referenced as a guide.
- 8.2 Each sample taken shall be sufficient for quality verification tests, acceptance tests, referee tests, and achieve tests as needed.
- 8.3 Archive samples shall be retained for a period of time specified by the buyer and be delivered to the buyer upon
 - 8.4 Each sample shall be plainly marked with the following:
 - 8.4.1 Beryllium-oxide powder,
 - 8.4.2 Purchase order number,
 - 8.4.3 Purchase order specification,
 - 8.4.4 Gross, net, and tare weights,
 - 8.4.5 Lot number, and
 - 8.4.6 Name of seller.
- 8.5 Lot samples shall be prepared by blending and splitting the container samples.
- 8.5.1 To obtain a container sample, take specimens with a thief at random locations along a randomly chosen vertical

traverse through each container selected at random to be sampled. Then blend the thief samples from the selected containers and split down to the required size.

- 8.5.2 The number of containers so sampled shall be 5 + (n/10), where n is the total number of containers per lot rounded to the nearest decade. If there are five or fewer containers per lot, each container shall be so sampled.
- 8.6 Package the lot sample so that no foreign material is introduced into the powder during storage or shipment. The method of packaging shall be agreed upon between the buyer and the seller.
- 8.7 Acceptance of the powder shall be on a lot basis provided the material meets the requirements of this specification.

9. Inspection and Certification

- 9.1 The seller shall inspect the material covered by this specification and shall furnish the buyer with certificates of tests specified under Sections 5 and 6 showing the results of testing and inspection performed for a powder lot.
- 9.2 The results of all tests carried out by or on behalf of the seller including material sampling, size range, density, chemical and impurity levels, visual appearance, sinterability, etc., in conformance with the requirements of this specification shall be certified by the seller.

10. Rejection

- 10.1 Unless the buyer and seller agree otherwise, rejection and acceptance shall be on a lot basis.
- 10.2 Powder lots that fail to conform to the requirements of the specification may be rejected by the buyer. The seller may petition the buyer to waive selected requirements for identified

out-of-specification lots. Decision to grant such waiver belongs to the buyer. The seller may also apply any remedy to bring rejected lots into specification providing the seller can demonstrate to the buyer that such remedy does not impair the function or preclude the certification of the rejected material.

10.3 In the event of disagreement over the results of analysis, samples shall be submitted to a mutually selected referee for resolution.

11. Packaging and Shipping

- 11.1 The powder shall be packaged in commercial vaportight nonhalogen bearing bags. The sealed bag shall be contained in an outer container for shipping purposes. The seller will be responsible for using a shipping container that ensures cleanliness, minimizes moisture pickup, provides adequate protection against damage during transportation, and ensures reasonable ease of unpackaging.
- 11.2 Each container shall be clearly marked with the following:
 - 11.2.1 Beryllium oxide powder,
 - 11.2.2 Purchase order number,
 - 11.2.3 Purchase order specification,
 - 11.2.4 Gross, net, and tare weights,
 - 11.2.5 Lot number, and
 - 11.2.6 Name of seller.

12. Quality Assurance

12.1 Quality assurance requirements shall be agreed upon between the buyer and the seller when specified in the purchase order. Code of Federal Regulations, Title 10, Part 50, Appendix B and ANSI/ASME NQA-1 are referenced as guides.

ANNEXES

(Mandatory Information)

A1. WET SIEVE TEST

- A1.1 Place approximately 20 g of beryllium-oxide powder onto a 51 mm (2 in.) diameter preweighed sieve No. 100 designation (150 μ m).
- A1.2 Both the powder and the sieve shall be dried at 150 \pm 5°C.
 - A1.3 Weigh the dry powder and sieve to nearest 1 mg.
 - A1.4 Wet the powder carefully with a small portion of

water from a wash bottle that has a fine dispensing tip and that contains 500 mL of warm water. Wash the beryllium-oxide through the sieve with the remaining water while gently shaking the sieve.

A1.5 Redry the sieve at 150 \pm 5°C and reweigh. The residue retained on the sieve shall not exceed 0.5 mass % of the initial sample.

A2. SINTERABILITY PERFORMANCE TEST

- A2.1 The purpose of the performance test is to verify the sinterability of the beryllium-oxide powder. It is not intended to simulate the buyer's fabrication process.
- A2.2 All parameters of the performance test, once mutually established, between the buyer and the seller shall remain unchanged throughout the order.
- A2.3 Performance test results shall be required for the lot sample pulled from the powder lot in accordance with the sampling procedure (Section 8). A minimum of five pellets shall be required to constitute a valid performance test for a powder sample.
- A2.4 Density of the fired test pellets shall be determined and shall not deviate from the five pellet average by more than 3.0 %. Results shall show a minimum density of 90 % of theoretical density for each pellet. (The theoretical density of BeO is 3.01 g/cm³.) Sintered pellet density shall be determined by Test Method C 373 or demonstrated equivalent methods agreed upon between the buyer and the seller.

- A2.5 Sinterability Test:
- A2.5.1 Cold press the sample. A binder or lubricant may be used in the pelletizing. Report the binder, lubricant, green density, compacting pressure, and dwell time.
- A2.5.2 Green dimensions of the test pellets shall be 12.7 \pm 0.375 mm diameter and the length/diameter ratio shall be 0.75 or greater. Report the type of press and pressing conditions.
- A2.5.3 Fire the pellets in one group (boat) in air for 2 h at a temperature controlled to ±25°C in the range from 1500 to 1625°C. Report the sintering temperature and heating rate used. After cooling to room temperature determine the density of the pellets.
- A2.5.4 The type of furnace used and atmosphere shall be identical throughout all performance tests. Report firing schedules and actually observed "at temperature" data. Indicate the source of the temperature measurement, that is, wall or boat temperature.

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