

Standard Specification for Nuclear-Grade Zirconium Oxide Powder¹

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1. Scope

- 1.1 This specification defines the physical and chemical requirements for zirconium oxide powder intended for fabrication into shapes, either entirely or partially of zirconia, for use in a nuclear reactor core.
- 1.2 The material described herein shall be particulate in nature.
- 1.3 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:²
- C117 Test Method for Materials Finer than 75-µm (No. 200) Sieve in Mineral Aggregates by Washing
- C371 Test Method for Wire-Cloth Sieve Analysis of Nonplastic Ceramic Powders
- C859 Terminology Relating to Nuclear Materials
- C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves
- E105 Practice for Probability Sampling of Materials
- 2.2 ANSI Standard:³
- ANSI/ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications
- 2.3 U.S. Government Standard:⁴
- Code of Federal Regulations, Title 10, Part 50, Energy (10 CFR 50) Domestic Licensing of Production and Utilization Facilities
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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.
- ³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.
- ⁴ 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.

3. Terminology

- 3.1 Terms shall be defined in accordance with Terminology C859 except for the following:
 - 3.2 buyer—the organization issuing the purchase order.
- 3.3 phase transformation—the rearrangement of the atomic ordering of a crystalline lattice as a material is cycled through a critical transformation or inversion temperature. The change from one crystalline phase to another may be accompanied by a volume change that could lead to cracks or defects in products fabricated from such materials.
- 3.4 powder lot—a specified quantity of zirconium oxide powder (with stabilizing additive, if applicable) blended together such that samples taken in accordance with the procedures of Section 8 can be considered as representative of the entire specified quantity.
 - 3.5 *seller*—the zirconium oxide processor.
- 3.6 stabilizing additive—a material which, when added in sufficient quantity to the subject material exhibiting the phase transformation, produces a stabilized crystalline phase that does not undergo a transformation at any temperature within the expected fabrication or usage regime of the manufactured product; the potentially deleterious volume change is therefore avoided.

4. Ordering Information

- 4.1 The buyer shall specify the following information on the order:
 - 4.1.1 Quantity (weight of delivered product).
- 4.1.2 Nominal particle size range and applicable tolerances in accordance with U.S. Standard Sieve Series (Specification E11). Test Method C371 and Test Method C117 and Specification E11 may be applied for particles larger than 37 μ m. For particle sizes less than 37 μ m, the particle size distribution will be determined using a method agreed upon between the buyer and the seller.
- 4.1.3 *Stabilizing Additive*—The amount and types of stabilizing additives (if any, including limits).
 - 4.1.4 Lot size.
 - 4.1.5 Sampling requirements.

5. Chemical Composition

5.1 A stabilizing additive may be used with the zirconium oxide. The recommended stabilizing additive is either calcium

oxide (CaO) or yttrium oxide (Y_2O_3) . The recommended additive concentration in the case of CaO stabilization is 4 to 8 weight %. In the case of Y_2O_3 stabilization, the recommended additive concentration is 14 to 20 weight %.

- 5.2 Use analytical chemistry methods as agreed upon between the buyer and the seller.
- 5.3 Impurity Concentration—The impurity concentration excluding the stabilizing additives shall not exceed 0.5 weight %. Individual element limits are specified in Table 1. The buyer may specify additional limits for elements not listed in Table 1.
- 5.4 The total equivalent boron content (EBC) shall not exceed 500 μ g/g on a mass basis relative to ZrO₂. The total EBC is the sum of the individual EBC values. The method of performing the calculation shall be as indicated in Practice C1233.

6. Physical Requirements

6.1 The nominal particle size, particle size distribution and applicable tolerances, shall be specified on the purchase order.

7. Cleanliness

7.1 The powder 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 approved by the buyer prior to use. The degree of sampling shall be specified on the purchase order. Practice E105 is referenced as a guide.
- 8.2 Sufficient sampling shall be taken, as needed, for quality verification tests, acceptance tests, referee tests and archive samples.

TABLE 1 Impurity Concentration Limits

Element	Maximum Concentration Limit (μg/g Powder)
Hf	200
В	100
Gd	50
Gd + Sm + Eu + Dy	200
Co	100
Si	2000
F	30
F + Cl + Br + I	100
Fe	1000
Ca ^A	3000
Mg	1200
Al	1500
Ti	100
Th	400

^A This number will be higher if used as a stabilizing additive.

8.3 Archive samples shall be retained and disposed in accordance with the buyer's instructions.

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 showing the results of testing and inspection performed on each powder lot.
- 9.2 The seller shall certify that each powder lot is in compliance with the provisions of this specification.

10. Rejection and Rehearing

- 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. The 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 Package Marking

- 11.1 The powder shall be packaged in sealed containers for shipment from the seller to the buyer. The seller will be responsible for using a shipping container which 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 Zirconium oxide powder plus stabilizing additive, if applicable,
 - 11.2.2 Purchase order number,
 - 11.2.3 Purchase order specification,
 - 11.2.4 Gross, tare, and net weight,
 - 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.

13. Keywords

13.1 CaO; powder; stabilizing additive; Y₂0₃; zirconium oxide



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