



## Standard Specification for Nuclear-Grade Hafnium Oxide Powder<sup>1</sup>

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### 1. Scope

1.1 This specification defines the physical and chemical requirements for hafnium oxide powder intended for fabrication into shapes 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*:<sup>2</sup>

**C117** Test Method for Materials Finer than 75- $\mu$ m (No. 200) Sieve in Mineral Aggregates by Washing

**C371** Test Method for Wire-Cloth Sieve Analysis of Non-plastic Ceramic Powders

**C859** Terminology Relating to Nuclear Materials

**E11** Specification for Woven Wire Test Sieve Cloth and Test Sieves

**E105** Practice for Probability Sampling of Materials

2.2 *ANSI/ASME Standard*:

**NQA-1** ASME Quality Assurance Program Requirements for Nuclear Facility Applications<sup>3</sup>

2.3 *U.S. Government Standard*:

**Code of Federal Regulations, Title 10, Part 50, Energy (10 CFR 50), Domestic Licensing of Production and Utilization Facilities**<sup>4</sup>

### 3. Terminology

3.1 Terms shall be defined in accordance with Terminology **C859** except for the following:

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.03 on Neutron Absorber Materials Specifications.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

<sup>4</sup> 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>.

3.2 *buyer*—the organization issuing the purchase order.

3.3 *hafnium oxide powder*—hafnium oxide that contains no hard aggregates larger than 20 mesh (840  $\mu$ m).

3.4 *phase transformation*—the rearrangement of the atomic ordering of a crystalline lattice as 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.<sup>5,6</sup>

3.5 *powder lot*—a specified quantity of hafnium 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 quantity.

3.6 *seller*—the hafnium oxide powder processor.

3.7 *stabilizing additive*—material which, when added in sufficient concentration 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, particle size range, and applicable tolerances. Test Method **C371** and Test Method **C117** and Specification **E11** may be applied for particles larger than 37  $\mu$ m. For particle sizes less than 37  $\mu$ 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.

<sup>5</sup> Curtis, C. E., Doney, L. M., and Johnson, J. R., "Some Properties of Hafnium Oxide, Hafnium Silicate, Calcium Hafnate, and Hafnium Carbide," *Journal of American Ceramic Society*, Vol 37, 1954, pp. 458–465.

<sup>6</sup> Brown, L. M., and Mazdiyasi, K. S., "Characterization of Alkoxy-Derived Yttria-Stabilized Hafnia," *Journal of American Ceramic Society*, Vol 53, 1970, pp. 590–594.

#### 4.1.5 Sampling requirements.

### 5. Chemical Composition

5.1 A stabilizing additive may be utilized with the hafnium oxide. The recommended stabilizing additive is yttrium oxide ( $Y_2O_3$ ). The typical concentration range is 7 to 10 weight %  $Y_2O_3$  in the finished product. References to other stabilizing additives (such as calcium oxide (CaO) and magnesium oxide (MgO)) may be found in the literature and may be used if agreed upon between the buyer and the seller. However, it is cautioned that the buyer should be aware of potential destabilization during thermal cycling when MgO or CaO is used.

5.2 Use analytical chemistry methods as agreed upon between the buyer and the seller.

5.3 *Impurity Concentration*—The impurity concentration, excluding zirconia and the stabilizing additives, shall not exceed 0.5 weight %. Individual element limits are specified in **Table 1**. The buyer may, in addition, specify the limits for elements not listed in **Table 1**.

5.4 The zirconium concentration shall not exceed 4.5 weight % on the basis of Zr/(Zr + Hf).

### 6. Physical Requirements

6.1 The nominal particle size, particle size range, 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. 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 **E105** is referenced as a guide.

**TABLE 1 Impurity Concentration Limits**

Element	Maximum Concentration Limit, ( $\mu\text{g/g}$ Powder)
B	400
Gd	200
Gd + Sm + Eu + Dy	500
Co	100
Si	2000
F	30
F + Cl + Br + I	100
Th	400

8.2 Sufficient samples shall be taken, as needed, for quality verification tests, acceptance tests, referee tests and archive samples.

8.3 Archive samples shall be retained and disposed of 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. 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 that assures 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 Hafnium oxide powder plus stabilizing additive, if applicable,

11.2.2 Purchase order number,

11.2.3 Purchase order specification,

11.2.4 Gross, net, and tare weight,

11.2.5 Lot number, and

11.2.6 Name of seller.

### 12. Quality Assurance

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

### 13. Keywords

13.1 CaO; hafnium oxide; MgO; powder; stabilizing additive;  $Y_2O_3$

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