



Designation: C 1065 – 93

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 the standard.

2. Referenced Documents

2.1 *ASTM Standards*:

C 859 Terminology Relating to Nuclear Materials²

E 11 Specification for Wire-Cloth Sieves for Testing Purposes³

E 105 Practice for Probability Sampling of Materials³

2.2 *ANSI Standard*:

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities⁴

2.3 *U.S. Government Standard*:

Code of Federal Regulations, Title 10, Part 50, Energy (10 CFR 50) Domestic Licensing of Production and Utilization Facilities⁵

3. Terminology

3.1 Terms shall be defined in accordance with Terminology C 859 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 may 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 E 11). For particle sizes less than 280 mesh, 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 (Y₂O₃). The recommended additive concentration in the case of CaO stabilization is 4 to 8 weight %. In the case of Y₂O₃ 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 any other elements not listed in Table 1.

5.4 The total equivalent boron content (EBC) of those impurities listed in Table 2 shall not exceed 500 µg/g on a mass

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² *Annual Book of ASTM Standards*, Vol 12.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ Available from the American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁵ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.



TABLE 1 Impurity Concentration Limits

Element	Maximum Concentration Limit (µg/g Powder)
Hf	200
B	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.

TABLE 2 Equivalent Boron Content Factors

Element	Neutron Absorption Cross Section ^A , (Barns) at 2200 m/s	Atomic Weight ^B	EBC Factor
Boron	764.0 ^C	10.81	1.0000
Cadmium	2520.0	112.41	0.3172
Calcium	0.43	40.08	0.0002
Cesium	29.0	132.91	0.0031
Chlorine	33.5	35.45	0.0134
Chromium	3.07	52.00	0.0008
Cobalt	37.2	58.93	0.0089
Copper	3.78	63.54	0.0008
Dysprosium	940.0	162.50	0.0818
Europium	4565.0	151.97	0.4250
Gadolinium	48890.0	157.25	4.3991
Hafnium	104.1	178.49	0.0083
Iron	2.56 ^D	55.85	0.0006
Lithium	70.6 ^E	6.94	0.1439
Manganese	13.3	54.94	0.0034
Molybdenum	2.55 ^D	95.94	0.0004
Nickel	4.49 ^D	58.69	0.0011
Nitrogen	1.90	14.01	0.0019
Samarium	5670.0	150.36	0.5336
Silver	63.3	107.87	0.0083
Tantalum	20.6	180.95	0.0016
Titanium	6.1	47.88	0.0018
Tungsten	18.4	183.85	0.0014
Vanadium	5.08	50.94	0.0014
Zinc	1.11	65.39	0.0002

^A Holden, N. E., "Neutron Cross Sections," Parts A and B, Vol 1, Academic Press, NY, 1981 and 1984.

^B Holden, N. E., and Martin, R. L., Pure and Applied Chemistry, Vol 56, 1984.

^C Cross Section is primarily due to a single isotope, whose isotopic abundance is variable in nature. Value can vary between 733 and 779 barns depending upon the source. (See Holden, N. E., "Neutron Capture Cross Section Standards for BNL-325," Fourth Edition, BNL-NCS-51388, January 1981.)

^D In the absence of other data, the neutron capture cross section for a Maxwellian flux is used.

^E Cross Section is primarily due to a single isotope, whose isotopic abundance is variable in nature. Value can vary between 69 and 72 barns depending upon the source. (See Holden, N.E., "Neutron Capture Cross Section Standards for BNL-325," 4th ed., BNL-NCS-51388, January 1981.)

basis relative to ZrO₂. The total EBC is the sum of the individual EBC values. The EBC value for each impurity in Table 2 shall be calculated using the following equation:

$$\text{EBC of impurity} = (\text{EBC factor}) (\mu\text{g of impurity/g ZrO}_2) \quad (1)$$

6. Physical Requirements

6.1 The particle size distribution shall be agreed upon between the buyer and the seller.

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 or 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 to perform the following in the event they are necessary or desired by the buyer:

8.2.1 Quality verification tests by the seller,

8.2.2 Acceptance tests by the buyer,

8.2.3 Referee tests, in the event these become necessary, and,

8.2.4 Retention of archive samples by the seller.

8.3 Archive samples shall be retained for a period of time specified by the buyer and be delivered to the buyer upon request.

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:

