Designation: C 1066 – 97

Standard Specification for Nuclear Grade Zirconium Oxide Pellets[1]

This standard is issued under the fixed designation C 1066; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification applies to pellets of stabilized zirconium oxide used in nuclear reactors.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles[2]

C 859 Terminology Relating to Nuclear Materials[3]

C 1065 Specification for Nuclear Grade Zirconium Oxide Powder[3]

E 105 Practice for Probability Sampling of Materials[4]

2.2 *ANSI Standard:*

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facility Applications[5]

2.3 *U.S. Government Document:*

Code of Federal Regulations, Title 10, Part 50–Energy (10 CFR 50) Domestic Licensing of Production and Utilization Facilities[6]

3. Terminology

3.1 Terms shall be defined in accordance with Terminology C 859, except for the following:

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

3.1.2 *pellet*—a fabricated geometric shape of zirconiumoxide having a chemical composition as described in Section 4.

3.1.3 *pellet lot*—the pellets produced from one zirconium oxide powder lot using one set of process parameters. Pellet lot size shall be agreed upon between the seller and the buyer.

3.1.4 *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 articles fabricated from such materials.

3.1.5 *powder lot*—a specified quantity of zirconium-oxide powder with stabilizing additive, blended together such that samples taken in accordance with Section 7 can be considered as representative of the entire quantity.

3.1.6 *seller*—the zirconium oxide pellet supplier.

3.1.7 *stabilizing additive*—A material which, when present in sufficient concentration in the subject material exhibiting the phase transformation, produces a stabilized crystalline phase that does not undergo a transformation or inversion at any temperature within the expected fabrication or usage regime of the manufactured pellet. The potentially deleterious volume change is therefore avoided.

4. Chemical Composition

4.1 The starting zirconium oxide powder shall be in accordance with Specification C 1065.

4.2 A stabilizing additive shall 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 %.

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

4.4 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.

4.5 The moisture concentration is included in the total hydrogen limit (see Table 1).

5. Physical Requirements

5.1 *Physical Dimensions*:

5.1.1 Dimensional requirements shall be in accordance with applicable drawings and purchase order documents.

5.1.2 Pellet dimensions shall be measured to ensure compliance with the buyer's requirements. Sampling plans to meet the acceptance criteria shall be agreed upon between the buyer and the seller to ensure that the pellets represented by the sample are within the required tolerance.

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[1] This specification is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C 26.03on Neutron Absorber Materials Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 12.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

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

[6] Available from U.S. Government Printing Office, Washington, DC 20402.

TABLE 1 Impurity Concentration Limits

Element	Maximum Concentration Limit (µg/g pellet)
Hf	200
B	100
Gd	50
Gd + Sm + Eu + Dy	200
Co	100
Si	2000
Fe	1000
Ca[A]	3000
Mg	1200
Al	1500
Ti	100
Th	400
F	30
F + Cl + Br + I	100
H (total hydrogen from all sources)	2

[A]This number will be higher if used as a stabilizing addition.

5.2 *Density*:

5.2.1 Pellet density limits shall be specified by the buyer. The incorporation of a stabilizing additive will introduce a change in the theoretical density of the fabricated pellets and should be taken into account. The method of establishing the theoretical density value shall be agreed upon by the buyer and seller.

5.2.2 The method of density measurement shall be Test Method C 559 or an alternative method submitted by the seller for approval by the buyer. Sampling plans to meet the acceptance criteria shall be agreed upon between the buyer and the seller. The method of density measurement and the method of compliance with 5.2.1 shall be submitted by the seller to the buyer for approval.

5.3 *Mechanical Properties*—Required mechanical properties and test methods shall be mutually agreed upon between the buyer and the seller. A compressive test at 69 MPa (10 000 psi) to ensure pellet integrity is recommended as a suitable test if the pellet is not to be subjected to service temperatures above 1100°C (2012°F). At higher temperatures, a thermal cycling test as given in Appendix X1 followed by a compressive test may be agreed upon between buyer and seller; or alternately, a powder X-ray diffraction pattern may be used to determine that the material has been stabilized in the correct crystalline phase.

5.4 *Visual Appearance*—Visual examination shall be conducted on finished pellets in accordance with Section 7 on Sampling. The seller and the buyer shall agree on visual standards as representing the requirements of 5.4.1, 5.4.2, and 5.4.3. These standards shall be used as acceptance standards for the visual examination of the pellets. In the event of a dispute, the method of defect measurement shall be submitted by the seller for approval by the buyer. Maximum permissible defects are defined as follows:

5.4.1 *End Chips*—Pellet end surface shall not be chipped beyond 10 % of the end-face surface area, and no chip shall exceed 1.02 mm (0.040 in.) in depth.

5.4.2 *Circumferential Chips*—Pellet circumferential surfaces shall not be chipped beyond 5 % of the circumferential surface area. No single chip shall exceed a depth of 1.02 mm (0.040 in.).

5.4.3 *Cracks*—Cracks not exceeding 90° of circumference in length are acceptable provided other requirements of the specifications are met.

5.4.4 Fissures and other defects shall be evaluated with respect to the criteria of 5.4.1, 5.4.2, and 5.4.3.

6. Cleanliness

6.1 The finished pellets shall be handled in a manner to avoid contamination by grinding fluids and dust, cleaning agents, and organic 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 pellets prior to sampling and packaging.

7. Sampling

7.1 Sampling plans to meet the 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 varies with the application and for this reason should be specified on the purchase order. Practice E 105 is referenced as a guide.

7.2 Each powder and pellet sample taken shall be sufficient to perform the following in the event they are necessary or desired by the buyer:

7.2.1 Quality verification tests,

7.2.2 Acceptance tests,

7.2.3 Referee tests in the event these become necessary, and

7.2.4 Retention of archive sample by the seller.

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

8. Inspection and Certification

8.1 The seller shall inspect the material covered by this specification and shall furnish the buyer with certificates of test showing the results of testing and inspection performed on each pellet lot prior to shipment. The seller shall certify that each pellet lot is in compliance with the provisions of this specification.

9. Rejection

9.1 Unless the buyer and seller agree otherwise, rejection and acceptance shall be on a pellet lot basis.

9.2 Pellet lots that fail to conform to the requirements of this 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 he can demonstrate to the buyer that such remedy does not impair the function or preclude the certification of the rejected material.

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

10. Packaging and Shipping

10.1 The pellets shall be packaged in sealed containers to prevent loss or damage, or both, of material and contamination

from airborne or container materials. The exact size and type of packaging shall be as mutually agreed upon between the buyer and seller.

10.2 Each container shall be clearly marked with the following:

10.2.1 Zirconium oxide pellets plus stabilizing additive,

10.2.2 Purchase order number,

10.2.3 Gross, tare, and net weights,

10.2.4 Lot number, and

10.2.5 Name of pellet manufacturer.

11. Quality Assurance

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

12. Keywords

12.1 CaO; stabilizer; stabilizing additive; thermal cycling test; Y_2O_3; zirconium oxide

APPENDIX

(Nonmandatory Information)

X1. THERMAL CYCLING TEST

X1.1 The following thermal cycle is recommended as a guide to determine the thermal stability of zirconium-oxide pellets.

X1.1.1 Heat the pellets in air to 1100 ± 25°C (2012 ± 77°F) at a heating rate of at least 100°C/h (212°F/h).

X1.1.2 Hold at 1100 ± 25°C (2012 ± 77°F) for at least 1 h.

X1.1.3 Cool to 500°C (932°F) at a cooling rate of at least 100°C/h (212°F/h).

X1.1.4 Repeat steps X1.1.1-X1.1.3 two additional times.

X1.1.5 Cool to room temperature and remove for mechanical testing.