



Standard Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder¹

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INTRODUCTION

This specification is intended to provide the nuclear industry with a general standard for sinterable uranium dioxide (UO_2) powder. It recognizes the diversity of manufacturing methods by which UO_2 powders are produced and the many special requirements for chemical and physical characterization that may be applicable for a particular fuel pellet manufacturing process or imposed by the end user of the powder in a specific reactor system. It is, therefore, anticipated that the buyer may supplement this specification with more stringent or additional requirements for specific applications.

1. Scope

1.1 This specification covers nuclear-grade, sinterable UO_2 powder. It applies to UO_2 powder containing uranium (U) of any ^{235}U concentration in the production of nuclear fuel pellets for use in nuclear reactors.

1.2 This specification recognizes the presence of reprocessed U in the fuel cycle and consequently defines isotopic limits for commercial grade UO_2 . Such commercial grade UO_2 is defined so that, regarding fuel design and manufacture, the product is essentially equivalent to that made from unprocessed U. UO_2 falling outside these limits cannot necessarily be regarded as equivalent and may thus need special provisions at the fuel fabrication plant or in the fuel design.

1.3 This specification does not include provisions for preventing criticality accidents or requirements for health and safety. Observance of this specification does not relieve the user of the obligation to be aware of and conform to all international, national, or federal, state, and local regulations pertaining to possessing, shipping, processing, or using source or special nuclear material.

1.4 This specification refers expressly to UO_2 powder before the addition of any die lubricant, binder, or pore former. If powder is sold with such additions or prepared as press feed, sampling procedures, allowable impurity contents, or powder physical requirements may need to be modified by agreement between the buyer and the seller.

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

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- B243 Terminology of Powder Metallurgy
 - B329 Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter
 - C696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets
 - C859 Terminology Relating to Nuclear Materials
 - C996 Specification for Uranium Hexafluoride Enriched to Less Than 5 % ^{235}U
 - 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 ASME Standard:³
- ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications

¹ This specification is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.02 on Fuel and Fertile Material Specifications.

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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 Society of Mechanical Engineers (ASME), ASME International Headquarters, Two Park Ave., New York, NY 10016-5990, <http://www.asme.org>.

2.3 Federal Regulation:⁴

Code of Federal Regulations, Title 10, Chapter 1, Nuclear Regulatory Commission, Applicable Parts

3. Terminology

3.1 *Definitions*—Definitions of terms are as given in Terminologies B243 and C859.

4. Chemical Composition

4.1 *Uranium Content*—The U content shall be determined on a basis to be agreed upon between the buyer and seller.

4.2 *Oxygen-to-Uranium Ratio (O/U)*—The O/U ratio may be specified as agreed upon between the buyer and seller. The determination of the O/U ratio shall be in accordance with Test Methods C696 or a demonstrated equivalent.

4.3 *Impurity Content*—The impurity content shall not exceed the individual element limit specified in Table 1 on a U basis. Total non-volatile oxide impurity content (see Table 1 and other impurity elements not having associated limits in Table 2) shall not exceed 1500 µg/gU. Some other elements such as those listed in Table 2 may also be of concern for the buyer and should be measured and reported if requested. If an element analysis is reported as “less than” a given concentration, this “less than” value shall be used in the determination of total impurities. Impurity elements measured and their associated limits may differ from what is listed in this specification agreed upon between the buyer and seller.

⁴ Available from U.S. Government Printing Office, Superintendent of Documents, 732 N. Capitol St., NW, Washington, DC 20401-0001, <http://www.access.gpo.gov>.

TABLE 1 Impurity Elements and Maximum Concentration Limits

Element ^B	Maximum Concentration Limit of Uranium, µg/gU
Aluminum (Al)	300
Carbon (C)	100
Calcium (Ca) + magnesium (Mg)	200
Chlorine (Cl)	100
Chromium (Cr)	200
Cobalt (Co)	100
Copper (Cu)	250
Fluorine (F)	100
Iron (Fe)	250
Lead (Pb)	250
Manganese (Mn)	250
Molybdenum (Mo)	250
Nickel (Ni)	200
Nitrogen (N)	200
Phosphorus (P)	250
Silicon (Si)	300
Tantalum (Ta)	250
Thorium ^A (Th)	10
Tin (Sn)	250
Titanium (Ti)	250
Tungsten (W)	250
Vanadium (V)	250
Zinc (Zn)	250

^A Thorium is primarily of concern because of the reactor production of ²³³U.

^B Any additional potential impurities, added by the fabrication process for example, beyond those listed here shall be evaluated (for example, in terms of equivalent boron) and associated limits established and agreed upon between the buyer and seller.

TABLE 2 Additional Impurity Elements

Element	
Beryllium (Be)	Lithium (Li)
Bismuth (Bi)	Niobium (Nb)
Boron (B)	Potassium (K)
Cadmium (Cd)	Silver (Ag)
Dysprosium (Dy)	Samarium (Sm)
Europium (Eu)	Sodium (Na)
Gadolinium (Gd)	Sulfur (S)
Indium (In)	Zirconium (Zr)

4.4 *Moisture Content*—The moisture content shall not exceed 0.50 weight percent of the powder.

4.5 Isotopic Content:

4.5.1 For UO₂ powder with an isotopic content of ²³⁵U below 5 %, the isotopic limits of Specification C996 shall apply, unless otherwise agreed upon between the buyer and the seller. If the ²³⁶U content is greater than Enriched Commercial Grade UF₆ requirements, the isotopic analysis requirements of Specification C996 shall apply. The specific isotopic measurements required by Specification C996 may be waived, provided that the seller can demonstrate compliance with Specification C996, for instance, through the seller’s quality assurance records.

4.5.2 For UO₂ powder that does not have an assay in the range set forth in 4.5.1, the isotopic requirements shall be as agreed upon between the buyer and the seller.

4.6 *Equivalent Boron Content*—For thermal reactor use, the total equivalent boron content (EBC) shall not exceed 4.0 µg/g on a U basis. For purpose of EBC calculation B, Gd, Eu, Dy, Sm, and Cd shall be included in addition to elements listed in Table 1. The method of performing the calculation shall be as indicated in Practice C1233. For fast reactor use, the above limitation on EBC does not apply.

5. Physical Properties

5.1 *Cleanliness and Workmanship*—The UO₂ powder shall be free of visible fragments of foreign matter.

5.2 *Particle Size*—UO₂ powder particle size limits and method of determination shall be as agreed upon between the buyer and seller. As an example, as agreed upon, the fraction of a representative sample not passing through a 425-µm (No. 40) standard sieve conforming to Specification E11 shall be reported to the buyer.

5.3 *Bulk Density*—The bulk density of UO₂ powder will depend on the processing method. Unless otherwise agreed upon between the buyer and seller, the bulk density shall be a minimum of 0.625 g/cm³ as determined by Test Method B329, or an agreed upon alternative.

5.4 *Sinterability*—Test pellets shall be produced and measured in accordance with a sintering performance test agreed upon between the buyer and seller. A sinterability performance test described in Appendix X1 is presented as a guide.

6. Sampling

6.1 A lot is defined as a quantity of UO₂ powder that is uniform in isotopic, chemical, physical, and sinterability characteristics.

6.2 The identity of a lot shall be retained throughout its processing history.

6.3 A powder lot shall form the basis for defining sampling plans used to establish conformance to this specification.

6.4 Sampling plans and procedures shall be mutually agreed upon by the buyer and the seller. Analytical confirmation of sampling plans shall be documented as part of the manufacturer's quality assurance and nuclear materials control and accountability program.

6.5 UO_2 may be hygroscopic and retain sufficient water after exposure to a moist atmosphere to cause detectable errors. Sample, weigh, and handle the sample under conditions that will ensure that the sample is representative of the lot.

7. Test Methods

7.1 The seller shall test the sample to ensure conformance of the powder to the requirements of Sections 4 and 5.

7.1.1 All chemical analyses shall be performed on portions of the representative sample. Analytical chemistry methods used shall be in accordance with Test Methods C696 or demonstrated equivalent methods agreed upon between the buyer and seller.

7.2 *Lot Acceptance*—Acceptance testing may be performed by the buyer on either the sample provided by the seller or a sample taken at the buyer's plant by sampling one or more individual containers with a sample thief. Practice E105 is referenced as a guide. Acceptance shall be on a lot basis, consistent with Section 6, and shall be contingent upon the material properties meeting the requirements of Sections 4 and 5.

7.3 *Referee Method*—The buyer and seller shall agree to a third party as a referee in the event of a dispute in analytical results.

8. Certification

8.1 The seller shall provide to the buyer documents certifying:

8.1.1 The isotopic content and identity of the starting material lot and

8.1.2 That the powder meets all the requirements of Sections 4 through 6.

8.2 Test data on the following characteristics shall be supplied upon request:

8.2.1 Uranium isotopic content,

8.2.2 Uranium content,

8.2.3 Individual impurity levels,

8.2.4 Moisture content,

8.2.5 Sinterability test results,

8.2.6 O/U ratio,

8.2.7 Particle size, and

8.2.8 Bulk density.

9. Packaging and Package Marking

9.1 UO_2 powder shall be packaged in sealed containers to prevent loss of material and undue contamination from air or the container materials. The exact size and method of packaging shall be as agreed upon by the buyer and seller.

9.2 Each container shall bear, as a minimum, a label on the lid and side with the following information:

9.2.1 Seller's name,

9.2.2 Material in container,

9.2.3 Lot number,

9.2.4 Uranium enrichment,

9.2.5 Gross, tare, net oxide weights,

9.2.6 Uranium weight,

9.2.7 Purchase order number, and

9.2.8 Container () of ().

10. Quality Assurance

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

11. Keywords

11.1 nuclear fuel; powder; uranium dioxide

APPENDIX

(Nonmandatory Information)

X1. SINTERABILITY TEST

X1.1 Purpose

X1.1.1 The purpose of the sinterability test is to verify the fabricability of each lot of UO_2 powder. Although not required, it is desirable to simulate the buyer's pellet fabrication process to improve the predictability of the powder. A suggested sinterability test follows.

X1.2 Fabrication of Test Pellets

X1.2.1 *Preparation of Test Pellets*—Cold press powder with the addition of agreed upon quantities of additive to produce at

least fifteen green pellets within a predetermined density range approximating the buyer's target range. The density of each green pellet shall vary no more than $\pm 0.5\%$ from the average density of the test pellets. The type of pellet press, pressing conditions, die geometry (that is, taper, dishing, etc.), use and quantity of any additive (if used), powder preconditioning, dwell time, and any other relevant pressing conditions shall be approved by the buyer. Report the green density and pressing pressure for each pellet.

X1.2.2 The diameter and length-to-diameter ratio of the green test pellets shall be approximately that of the buyer's green production pellets. The length of each green pellet shall vary no more than ± 0.5 mm (± 0.02 in.).

X1.2.3 Sinter the pellets as one batch in reducing atmosphere (for example, hydrogen, or dissociated ammonia) at a predetermined temperature set point (typically 1625 to 1750°C, with a temperature control of $\pm 25^\circ\text{C}$) for 2 to 6 h, approximating the buyer's sintering conditions. The type of furnace used, atmosphere including dew point, and actual sintering cycle shall be as agreed upon between the buyer and the seller.

X1.2.4 Once established, all of the parameters of this test shall remain unchanged throughout all lots of the order.

X1.3 Density Determination

X1.3.1 Determine the geometrical density of the green and sintered, but unground pellets as follows:

X1.3.1.1 *Diameter*—Record the average of three readings taken to the nearest 0.005 mm (0.0002 in.) at equally spaced intervals along the length of the pellet using a blade micrometer or equivalent gauge.

X1.3.1.2 *Length*—Record the average of three readings taken to the nearest 0.005 mm (0.0002 in.) from end to end of the pellet at equally spaced intervals along a longitudinally bisecting plane using a micrometer or equivalent gauge.

X1.3.1.3 *Weight*—Record the pellet weight to the nearest 0.001 g.

X1.3.1.4 *Density*—Calculate the density of each pellet to the nearest 0.01 g/cm³. The geometric density calculation should account for known biases due to pellet geometry effects and should be qualified by demonstrated equivalency with an immersion technique for sintered pellets.

X1.3.2 Alternatively, the density of the sintered pellet may be obtained by an immersion technique or demonstrated equivalent method as agreed upon between the buyer and the seller.

X1.4 Sintered Pellet Performance Test

X1.4.1 Make the sinterability test using at least fifteen pellets produced from each lot. Determine the density of each pellet, and verify that each pellet is within ± 2.0 % of the average and not less than 94.0 % of the theoretical density (TD). The minimum sintered density should account for test additives (if used) and approximate the buyer's nominal pellet density requirements, accounting for differences in processing parameters and use of process additives or recycle powders in the buyer's fabrication process. Report the average sintered density and standard deviation.

X1.4.2 Grind the cylindrical surface of the sintered pellets using a centerless grinder or equivalent as agreed upon by the buyer and the seller, and perform a visual inspection of the test pellets for surface defects such as pits, inclusions, endcapping, cracks, chips, etc. Report the result in accordance with the method and standard as agreed upon between the buyer and the seller.

X1.4.3 *Optional*—Place the ground sintered pellets in a drying oven, and allow the pellets to be at a temperature of 100°C for 1 h. Randomly select at least three pellets for individual pellet hydrogen testing in accordance with the method described in Test Methods C696. Report the individual pellet hydrogen results.

X1.4.4 Once established, all of the parameters of this sinterability test shall remain unchanged throughout all lots of the order.

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