



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 specification for sinterable uranium dioxide powder. It recognizes the diversity of manufacturing methods by which uranium dioxide powders are produced and the many special requirements for chemical and physical characterization which may be imposed by the end use of the powder in a specific reactor system. It is, therefore, anticipated that the buyer may supplement this specification with additional requirements for specific applications.

1. Scope

1.1 This specification covers nuclear-grade, sinterable uranium dioxide (UO_2) powder. It applies to uranium dioxide powder containing uranium 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 uranium 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 uranium. 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 calcined 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 *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

- B 329 Test Method for Apparent Density of Metal Powders and Compounds by the Scott Volumeter
- C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets
- C 859 Terminology Relating to Nuclear Materials
- C 996 Specification for Uranium Hexafluoride Enriched to Less than 5 % ^{235}U
- C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials
- E 11 Specification for Wire-Cloth and Sieves for Testing Purposes
- E 105 Practice for Probability Sampling of Materials

2.2 ANSI Standard:³

- ANSI/ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications

2.3 Federal Regulation:⁴

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.

⁴ Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098.

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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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 Terminology C 859.

4. Chemical Requirements

4.1 *Uranium Content*—The uranium 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 C 696 or a demonstrated equivalent.

4.3 *Impurity Content*—The impurity content shall not exceed the individual element limit specified in Table 1 on a uranium weight basis. The summation of the contribution of each of the impurity elements listed in Table 1 shall not exceed 1500 µg/gU. 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. 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.

4.4 *Moisture Content*—The moisture content shall not exceed 0.40 weight percent of the powder.

4.5 *Isotopic Content:*

4.5.1 For UO₂ powder with an isotopic content of ²³⁵U between that of natural uranium and 5 %, the isotopic limits of Specification C 996 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 C 996 shall apply. The specific isotopic measurements required by Specification C 996 may be waived, provided that the seller can demonstrate compliance with Specification C 996, for instance, through the

seller’s quality assurance records. A ²³⁶U content greater than that specified in C 996 for Enriched Commercial Grade UF₆ may be agreed between the buyer and the seller since it is not a safety concern.⁵

4.5.2 For UO₂ powder, not having 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 uranium weight 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 C 1233. For fast reactor use, the above limitation on EBC does not apply.

4.7 *Cleanliness and Workmanship*—The powder shall be visually free of foreign material such as metallic particles and oil.

5. Physical Requirements

5.1 *Particle Size*—Based on visual observation, all of a representative sample of the UO₂ shall pass through a 425-µm (No. 40) standard sieve conforming to Specification E 11. Particle size distribution and method of determination shall be as agreed upon between the buyer and seller. Alternatively, as agreed upon between the buyer and the seller, the fraction not passing through a 425-µm (No. 40) standard sieve shall be reported to the buyer.

5.2 *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 kg/m³ as determined by Test Method B 329, or an agreed upon alternative.

NOTE 1—For powder prepared as a press feed, a minimum bulk density of 1.8 g/cm³ is recommended.

5.3 *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.

⁵ The intent of the C 996 isotope limits is to indicate possible presence of reprocessed UF₆. Acceptance of UO₂ pellets with ²³⁶U content above that specified for Enriched Commercial Grade UF₆, shall be based on fuel performance evaluation.

TABLE 1 Impurity Elements and Maximum Concentration Limits

Element	Maximum Concentration Limit of Uranium, µg/gU
Aluminum	250
Carbon	100
Calcium + magnesium	200
Chlorine	100
Chromium	200
Cobalt	100
Copper	250
Fluorine	100
Iron	250
Lead	250
Manganese	250
Molybdenum	250
Nickel	200
Nitrogen	200
Phosphorus	250
Silicon	300
Tantalum	250
Thorium ^A	10
Tin	250
Titanium	250
Tungsten	250
Vanadium	250
Zinc	250

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

6.5 UO₂ 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 C 696 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 thief. Practice E 105 is referenced as a guide. Acceptance shall be on a lot basis and shall be contingent upon the material properties meeting the requirements of Sections 4 through 6.

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 distribution, and
- 8.2.8 Bulk density.

9. Packaging and Package Marking

9.1 Uranium dioxide 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 ANSI/ASME NQA-1 are referenced as guides.

11. Keywords

- 11.1 nuclear fuel; powder; urania; 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 UO₂ 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 lubricant or additive to produce at least fifteen green pellets within a predetermined density range approximating the buyer's target range. The density of each pellet shall vary no more than $\pm 0.5\%$ theoretical density (TD) from the average of the required test pellets. The type of pellet press, pressing conditions, die taper, use and quantity of any lubricant or additive (if used), powder

preconditioning, dwell time, and any other relevant pressing conditions shall be approved by the buyer. Report the average green density and pressing pressure for each pellet.

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

X1.2.3 Fire the pellets as one batch in 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 unfired and fired, but unground pellets as follows:

X1.3.1.1 *Diameter*—Record the average of four 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 vertically 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.

X1.3.2 Alternatively, the density of the 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 $\pm 2.0\%$ of the average and not less than 94.0 % 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 fired 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 C 696. 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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