Designation: C 753 – 99

Standard Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder[1]

This standard is issued under the fixed designation C 753; 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.

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 powder. It applies to uranium dioxide powder containing uranium of any ^{235}U concentration 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 unreprocessed 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 Special tests and procedures are given in Annex A1.

1.5 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, sampling procedures or allowable impurity contents, or both, may need to be modified by agreement between the buyer and the seller.

1.6 The following safety hazards caveat pertains to the test methods portion in the annexes of this specification: *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 limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

B 329 Test Method for Apparent Density of Powders of Refractory Metals and Compounds by the Scott Volumeter[2]

C 696 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Uranium Dioxide Powders and Pellets[3]

C 859 Terminology Relating to Nuclear Materials[3]

C 996 Specification for Uranium Hexafluoride Enriched to Less than 5 % ^{235}U[3]

C 1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

E 105 Practice for Probability Sampling of Materials[4]

2.2 *ANSI Standard:*[5]

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

2.3 *Federal Regulation:*[6]

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.

[1] This specification is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.02 on Fuel and Fertile Material Specifications.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[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 Standardization Documents, Order Desk, Building 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111–5094, Attn: NPODS.

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 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/g. 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 g/100 g uranium dioxide (UO_2).

4.5 *Isotopic Content*:

4.5.1 For UO_2 powder with an isotopic content of ^{235}U 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 ^{236}U content is greater than enriched commercial grade UF_6 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. ^{236}U content greater then one specified in C 996 for Commercial grade UF_6 may be agreed between the buyer and the seller since it is not a safety concern.[7]

[7] The intent of the C 996 isotope limits is to indicate possible presence of reprocessed UF_6. Acceptance of UO_2 pellets with ^{236}U content above that specifed for Commercial Enriched UF_6, 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 ^{233}U.

4.5.2 For UO_2 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.

5. Physical Requirements

5.1 *Particle Size*— Based on visual observation, all of a representative sample of the UO_2 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 UO_2 powder will depend on the processing method. Unless otherwise agreed upon between the buyer and seller, the bulk density shall be a minimum of 625 kg/m^3 as determined by Test Method B 329, or an agreed upon alternative.

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 Annex A1 is presented as a guide.

6. Lot Requirements

6.1 A lot is defined as a quantity of UO_2 powder that is uniform in isotopic, chemical, physical, and sinterability characteristics.

6.2 The identity of a lot shall be retained throughout.

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. A suggested sampling procedure is given in Annex A2.

7. Test Methods

7.1 The seller shall test the sample obtained per Annex A2 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 prepared in accordance with Annex A2. 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 NQA-1 are referenced as guides.

11. Keywords

11.1 nuclear fuel; powder; urania; uranium dioxide

ANNEXES

(Mandatory Information)

A1. SINTERABILITY TEST

A1.1 Purpose

A1.1.1 The purpose of the sinterability test is to verify the fabricability of each lot of UO_2 powder. It is not intended to simulate the buyer's pellet fabrication process. A suggested sinterability test follows.

A1.2 Fabrication of Test Pellets

A1.2.1 *Preparation of Test Pellets*—Cold press powder to produce at least four pellets with the addition of an agreed upon quantity of lubricant or additive, or both, to green pellets within a predetermined density range. The density of each pellet shall vary no more than ±0.5 % theoretical density (TD) from the average of the required test pellets. Report the type of pellet press, density, pressing conditions, due taper, identification of any die lubricant (if used), pressing pressure, dwell time, and any other relevant pressing condition.

A1.2.2 The diameter of the unfired test pellets shall be approximately that of the unfired production pellet. The length-to-diameter ratio shall not be less than that of the unfired production pellet. Hold the variation in length of the unfired pellets to ±0.5 mm (±0.02 in.).

A1.2.3 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 ±25°C). Report the type of furnace used, time at sintering temperature, sintering temperature, and the atmosphere including dew point. The actual firing schedule and parameters shall be as agreed upon between the buyer and the seller.

A1.2.4 Once established, all of the parameters of this test shall remain unchanged throughout all lots of the order.

A1.3 Density Determination

A1.3.1 Determine the geometrical density of the fired and unfired pellets as follows:

A1.3.1.1 *Diameter*—Record the average of four readings taken at equally spaced intervals along a 180° helix 1.6 mm (1⁄16 in.) from each end of the pellet using a blade micrometer and reading to the nearest 0.0051 mm (0.0002 in.).

A1.3.1.2 *Length*—Record the average of three readings taken from end to end of the pellet at equally spaced intervals along a vertically bisecting plane using a micrometer and reading to the nearest 0.0127 mm (0.0005 in.).

A1.3.1.3 *Weight*—Weight to the nearest 0.001 g.

A1.3.1.4 *Density*—Calculate the density of each pellet to the nearest 0.01 g/cm^3.

A1.3.2 Alternatively, the density of the pellet may be obtained by an immersion technique as agreed upon between the buyer and the seller.

A1.4 Sintered Pellet Performance Test

A1.4.1 Make the sinterability test using at least four pellets produced from each lot sample obtained according to the sampling procedure in Section 6. Determine the density of each pellet, and verify that each pellet is within + 2.0 % of the average and not less than 94.0 % TD. The TD of stoichiometric UO_2 enriched to less than 5 % ^{235}U may be taken as 10.96 g/cm^3.

A1.4.2 *(Optional)* 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 endcapping, cracks, chips, etc., and report the result in accordance with the method and standard as agreed upon between the buyer and the seller.

A1.4.3 *(Optional)* Place the ground sintered pellets in a drying oven, and allow the pellets to be at a temperature of 100°C for 1 hr. 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.

A1.4.4 Once established, all of the parameters of this sinterability test shall remain unchanged throughout all lots of the order.

A2. SAMPLING

A2.1 Uranium oxide 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.

A2.2 Take a representative sample of powder from each lot for the purpose of determining chemical and physical properties and for sintering performance testing.

A2.3 A lot sample shall be of sufficient size to perform quality assurance testing at the seller's plant, acceptance testing at the buyer's plant, and referee tests in the event they become necessary.

A2.4 Package the lot sample for acceptance testing at the buyer's plant in a separate container, clearly identify by lot number, and ship with the lot. Clearly identify the referee sample and retain it at the seller's plant until the lot has been formally accepted by the buyer.

A2.5 Prepare the lot sample by blending and splitting the container samples.

A2.6 To obtain a container sample, take specimens with a thief at random locations along a randomly chosen vertical traverse through each container selected at random to be sampled. Then blend the thief samples from the selected containers and split down to the required size.

A2.7 The number of containers so sampled shall be $5 + (n/10)$, where n is the total number of containers per lot rounded to the nearest decade. If there are five or fewer containers per lot, each container shall be so sampled.

A2.8 Alternatively, an auto-sampler can be used to obtain samples during emptying or filling of the container.