Designation: C 1348 – 01

Standard Specification for Blended Uranium Oxides with a ^{235}U Content of Less Than 5 % for Direct Hydrogen Reduction to Nuclear Grade Uranium Dioxide[1]

This standard is issued under the fixed designation C 1348; 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 standard for such uranium oxide powders. It recognizes the diversity of conversion processes and the many special requirements for chemical and physical characterization that may be imposed by the end use of the powder. It is anticipated, therefore, that this specification may be supplemented with additional requirements by agreement between the buyer and the seller.

1. Scope

1.1 This specification covers blended uranium trioxide (UO_3), U_3O_8, or mixtures of the two, powders that are intended for conversion into a sinterable uranium dioxide (UO_2) powder by means of a direct reduction process. The UO_2 powder product of the reduction process must meet the requirements of Specification C 753 and be suitable for subsequent UO_2 pellet fabrication by pressing and sintering methods. This specification applies to uranium oxides with a ^{235}U enrichment less than 5 %.

1.2 This specification includes chemical, physical, and test method requirements for uranium oxide powders as they relate to the suitability of the powder for storage, transportation, and direct reduction to UO_2 powder. This specification is applicable to uranium oxide powders for such use from any source.

1.3 The scope of this specification does not comprehensively cover all provisions for preventing criticality accidents, for health and safety, or for shipping. Observance of this specification does not relieve the user of the obligation to conform to all international, national, state, and local regulations for processing, shipping, or any other way of using uranium oxide powders (see 2.2 and 2.3).

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

1.5 The following safety hazards caveat pertains only to the test methods portion of the annexes in 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 Metal Powders and Related Compounds Using 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 753 Specification for Nuclear-Grade, Sinterable Uranium Dioxide Powder[3]

C 799 Test Methods for Chemical, Mass Spectrometric, Spectrochemical, Nuclear, and Radiochemical Analysis of Nuclear-Grade Uranyl Nitrate Solutions[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]

C 1287 Test Method for Determination of Impurities in Uranium Dioxide by Inductively Coupled Plasma Mass Spectrometry[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:*

NQA-1 Quality Assurance Program, Requirements for Nuclear Facilities[5]

[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] *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.

2.3 *U.S. Government Document:*

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

3. Terminology

3.1 *Definitions*—Terms shall be defined in accordance with Terminology C 859, except the following.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Commercial-Grade Uranium Oxide*—uranium trioxide (UO_3), U_3O_8, or a mixture of the two, made from unirradiated uranium. It is recognized that some contamination with reprocessed uranium may occur during routine processing; this is acceptable, provided that the specification for Commercial-Grade Uranium Oxide, as set forth in 4.1, is met.

4. Isotopic Content

4.1 For Commercial-Grade Uranium Oxide with an isotopic content of ^{235}U between that of natural uranium and 5 %, the isotopic limits, including the requirements for reporting and measurements of Specification C 996 shall apply, unless otherwise agreed upon between the buyer and seller. 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 contents greater than that specified in C 996 for Enriched Commercial Grade UF6 may be agreed upon between the buyer and seller since it is not a safety concern.

4.2 For commercial uranium oxides not having an isotopic concentration in the range set forth in 4.1, the isotopic requirements shall be as agreed upon between the buyer and the seller.

5. Physical and Chemical Requirements

5.1 *Uranium Content*—The uranium content shall be determined using the procedures described in Test Methods C 696 or C 799, or as agreed upon between the buyer and the seller. The minimum total uranium content shall also be agreed upon between the buyer and the seller.

5.2 *Oxygen-to-Uranium Ratio (O/U)*—The O/U ratio of the Commercial-Grade Uranium Oxide shall be nominally between 2.67 and 3.0. The O/U ratio shall be determined by Test Methods C 696 or by another method as agreed upon by the buyer and the seller.

5.3 *Impurity Content*—The impurity content of the Commercial-Grade Uranium Oxide 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 1000 µg/gU. The impurity content shall be determined using the procedures described in Test Methods C 696 or C 1287, or both, or as agreed upon between the buyer and the seller.

5.4 *Equivalent Boron Content*—The total equivalent boron content (EBC) of the Commercial-Grade Uranium Oxide shall not exceed 2.0 µg/gU. The list of elements to be considered in the EBC calculation shall be as agreed upon between the buyer and the seller. The method of performing the calculation shall be as indicated in Practice C 1233.

TABLE 1 Impurity Elements and Maximum Concentration Limits

Element	Maximum Concentration Limit of Uranium, µg/gU
Aluminum	50
Barium	5
Beryllium	100
Bismuth	3
Calcium + magnesium	100
Carbon	100
Chlorine	100
Chromium	100
Cobalt	80
Copper	100
Fluorine	100
Iron	150
Lead	40
Manganese	50
Molybdenum	200
Nickel	80
Phosphorus	100
Silicon	200
Sodium	20
Tantalum	200
Thorium	10
Tin	50
Titanium	50
Tungsten	100
Vanadium	10
Zinc	20

5.5 If the concentrations of any of the elements used in the calculations in 5.3 are reported as a less-than value, this less-than value shall be used for any further calculations involving the concentration of this element.

5.6 *Bulk Density*—Unless otherwise agreed upon between the buyer and the seller, the bulk density of the Commercial-Grade Uranium Oxide shall be a minimum of 575 kg/m^3, as determined by Test Method B 329, or an alternative agreed upon between the buyer and the seller.

5.7 *Moisture Content*—The moisture content of the Commercial-Grade Uranium Oxide shall not exceed 1 % by weight unless otherwise agreed upon by the buyer and the seller.

5.8 *Ability to Flow*—The Commercial-Grade Uranium Oxide shall be sufficiently free-flowing to permit sampling and powder handling.

5.9 *Particle Size*—Based on visual observation, all of a representative sample of the Commercial-Grade Uranium Oxide shall pass through a 425-µm (No. 40) standard sieve conforming to Specification E 11, or as otherwise agreed upon between the buyer and the 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. Particle size, size distribution, and method of determination shall be as agreed upon between the buyer and the seller. Packing or agglomeration, or both, during shipping may be a concern.

5.10 *Reduction and Sinterability*—The Commercial-Grade Uranium Oxide shall be reduced to UO_2, and test pellets shall be produced and measured in accordance with a sintering performance test agreed upon between the buyer and the seller.

[6] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

Reduction and sinterability performance tests described in Annex A1 are presented as a guide.

6. Lot Requirements

6.1 A lot is defined as a quantity of Commercial-Grade Uranium Oxide powder that is uniform in isotopic, chemical, and physical 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 shall be mutually agreed upon between the buyer and the seller. A suggested sampling procedure is given in Annex A2.

7. Test Methods

7.1 The sample described in 6.4 shall be tested to ensure conformance of the powder to the requirements of Sections 4-6.

7.2 *Lot Acceptance*—Acceptance testing may be performed by the buyer on either the sample provided by the seller or on 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 4-6.

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

8. Certification

8.1 The buyer shall be provided documents certifying that the material meets all of the requirements of Sections 4-6.

8.2 Records of all data obtained from tests to certify that the material meets the requirements of Sections 4-6 shall be provided to the buyer as requested.

9. Packaging and Marking

9.1 The Commercial-Grade Uranium Oxide shall be packaged in sealed containers to prevent (*1*) loss of material and (*2*) contamination of oxide from humidity or the container materials. The exact size and method of packaging shall be as agreed upon between the buyer and the seller.

9.2 As a minimum, each container shall bear 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, and net oxide weights,

9.2.6 Uranium weight,

9.2.7 Purchase order number (or equivalent), and

9.2.8 Container ( ) of ( ).

10. Quality Assurance

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

11. Keywords

11.1 conversion; nuclear fuel; reduction; uranium dioxide; uranium oxide

ANNEXES

(Mandatory Information)

A1. REDUCTION AND SINTERABILITY TESTS

A1.1 Purpose

A1.1.1 The purpose of the reduction and sinterability tests is to verify the fabricability of each lot of Commercial-Grade Uranium Oxide powder. The tests are not intended to simulate the buyer's reduction and pellet fabrication process. Suggested reduction and sinterability tests follow.

A1.2 Reduction to UO_2

A1.2.1 Reduce the Commercial-Grade Uranium Oxide to UO_2 using dissociated ammonia as the reducing agent and a predetermined temperature (typically 600 to 800°C) or temperature profile. Report the type of reduction equipment used, time at temperature, and reduction atmosphere. The actual reduction conditions shall be as agreed upon between the buyer and the seller. Measure and record the O/U ratio, particle size distribution, bulk density, and any other properties agreed upon between the buyer and the seller.

A1.2.2 Prepare the powder for pellet pressing. The powder preparation steps shall be as agreed upon between the buyer and the seller, but they may include steps such as sieving, milling, slugging, and granulating. Record the powder preparation steps and conditions, including sieve size, slug density, and slugging pressure.

A1.3 Fabrication of the Test Pellets

A1.3.1 *Preparation of Test Pellets*—Cold press powder to produce at least five 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, die taper, identification of any die lubricant (if used), pressing pressure, dwell time, and any other relevant pressing condition.

A1.3.2 The diameter of the unfired test pellets shall be approximately that of the unfired production pellet. The length-to-diameter ratio shall be not less than the unfired production pellet. Hold the variation in length of the unfired pellets to ±0.5 mm (±0.02 in.).

A1.3.3 Fire the pellets in 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 atmosphere, including dew point. The actual firing schedule and parameters shall be as agreed upon between the buyer and the seller.

A1.3.4 Once established, all parameters of this test shall remain unchanged throughout all lots of the order.

A1.4 Density Determination

A1.4.1 Determine the geometrical density of the fired and unfired pellets as follows:

A1.4.1.1 *Diameter*—Record the average of four readings taken at equally spaced intervals along a 180° helix 1.5 mm (0.06 in.) from each end of the pellet, using a blade micrometer and reading to the nearest 0.005 mm (0.0002 in.).

A1.4.1.2 *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 0.01 mm (0.0005 in.).

A1.4.1.3 *Weight*—Weigh to the nearest 0.001 g.

A1.4.1.4 *Density*—Calculate the density of each pellet to the nearest 0.01 g/cm^3.

A1.4.2 Alternatively, the density of the pellet may be obtained by an immersion technique as agreed upon between the buyer and the seller.

A1.5 Sintered Pellet Performance Test

A1.5.1 Make the sinterability test using at least five pellets produced from each lot sample obtained in accordance with the sampling procedure of Section 6. Determine the density of each pellet, and verify that each pellet is within 1.5 % of the average and not less than 94 % TD. The TD of stoichiometric UO_2 enriched to less than 5 % ^{235}U may be taken as 10.96 g/cm^3.

A1.5.2 (Optional) Grind the cylindrical surface of the fired pellets using a centerless grinder, or equivalent, as agreed upon between the buyer and the seller. 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.

A1.5.3 (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 procedure described in Test Methods C 696. Report the individual pellet hydrogen results.

A1.5.4 Once established, all 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 error. 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 reduction and sinterability tests.

A2.3 A lot sample shall be of sufficient size to perform quality assurance testing at the seller's plant, acceptance testing at the purchaser's plant, and referee tests in the event that 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. Identify the referee sample clearly, and retain it at the manufacturer's plant, if the manufacturer is someone other than the seller, until the lot has been accepted formally 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 = 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.