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Standard Specification for Nuclear-Grade Plutonium Dioxide Powder for Light Water Reactors[1]

This standard is issued under the fixed designation C757; 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 plutonium dioxide (PuO_2) powder. It recognizes the diversity of manufacturing methods by which PuO_2 powders are produced and the many special requirements for chemical and physical characterization that may be applicable for a particular Mixed Oxide (MOX, that is (U, Pu)O_2) fuel pellet manufacturing process or imposed by the end user of the powder in different light water reactors. 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 PuO_2 powder. It applies to PuO_2 of various isotopic compositions as normally prepared by in-reactor neutron irradiation of natural or slightly enriched uranium or by in-reactor neutron irradiation of recycled plutonium mixed with uranium.

1.2 There is no discussion of or provision for preventing criticality incidents, nor are health and safety requirements, the avoidance of hazards, or shipping precautions and controls discussed. Observance of this specification does not relieve the user of the obligation to be aware of and conform to all applicable international, national, or federal, state, and local regulations pertaining to possessing, shipping, processing, or using source or special nuclear material. For examples in the U.S. Government, relevant documents are Code of Federal Regulations, Title 10 Nuclear Safety Guide, U.S. Atomic Energy Commission Report TID-7016[2], and "Handbook of Nuclear Safety", H. K. Clark, U.S. Atomic Energy Commission Report, DP-532[2].

1.3 The PuO_2 shall be produced by a qualified process and in accordance with a quality assurance program approved by the user.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

B243 Terminology of Powder Metallurgy

C697 Test Methods for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets

C859 Terminology Relating to Nuclear Materials

C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

C1274 Test Method for Advanced Ceramic Specific Surface Area by Physical Adsorption

C1295 Test Method for Gamma Energy Emission from Fission and Decay Products in Uranium Hexafluoride and

[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] Available from Superintendent of Documents, U.S. Government Printing Office, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20402.

[3] 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.

Uranyl Nitrate Solution
C1770 Test Method for Determination of Loose and Tapped Bulk Densities of Small Quantities of Plutonium Oxide
E105 Guide for Probability Sampling of Materials

2.2 *ASME Standard:*
ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications[4]

2.3 *U.S. Government Documents:*
Code of Federal Regulations, Title 10, Nuclear Safety Guide, U.S. Atomic Energy Commission Report TID-7016[2]
"Handbook of Nuclear Safety," Clark, H. K., U.S. Atomic Energy Commission Report, DP-532[2]

2.4 *ISO Standard:*[5]
ISO 8300 Determination of Pu Content in Plutonium Dioxide (PuO_2) of Nuclear Grade Quality, Gravimetric Method
ISO 9161 Uranium Dioxide Powder—Determination of Apparent Density and Tap Density
ISO 13463 Nuclear-grade Plutonium Dioxide Powder for Fabrication of Light Water Reactor MOX Fuel—Guidelines to Help in the Definition of a Product Specification

3. Terminology

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

4. Isotopic Content

4.1 Concentrations and homogeneity ranges of the plutonium (Pu) shall be as specified by the buyer.

4.2 The isotopic composition of the final product shall be determined by a method to be agreed upon between the buyer and seller and shall be reported on a Pu basis including the associated measurement uncertainties. The date of the determination will be indicated.

5. Chemical Composition

5.1 *Plutonium Content*—The minimum Pu content shall be 86.0 weight % including measurement uncertainties as sampled on the date of sampling.

5.2 *Uranium Content*—The uranium content of the PuO_2 shall be measured and reported on a Pu basis.

5.3 *Americium Content*—The americium (Am) content shall be measured and reported on a Pu basis. The maximum acceptable Am content shall be agreed upon between the buyer and seller.

5.4 The dates of analyses of U, Th and Am shall be recorded.

5.5 *Impurity Content*—The impurity content shall not exceed the individual element limit specified in Table 1 on a Pu basis. Total non-volatile oxide impurity content excluding Am shall not exceed 6000 µg/g Pu. 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 as agreed upon between the buyer and seller.

5.6 *Moisture Content*—The moisture content shall be measured and reported on a Pu basis. The maximum acceptable moisture content shall be agreed upon between the buyer and the seller.

5.7 *Equivalent Boron Content*—For thermal reactor use, the total equivalent boron content (EBC) shall not exceed 20.0 µg/g on a Pu basis. 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.

5.8 *Gamma Activity*—The gamma activity (Bq/g Pu) of the gamma emitting fission products whose isotopes have half lives of 30 days or greater shall be measured. The gamma radiation from fission products shall be less than 10^5 MeV·Bq/g Pu.

5.8.1 The list of nuclides and mean energies per disintegration found in Test Method C1295 are to be used in the calculations.

TABLE 1 Impurity Elements and Maximum Concentration Limits

Element[C]	Maximum Concentration Limit of Plutonium, µg/gPu
Aluminum (Al)	300
Boron (B)	3
Cadmium (Cd)	3
Carbon (C)[A]	500
Chlorine (Cl)	300
Chromium (Cr)	200
Dysprosium (Dy)	0.5
Europium (Eu)	0.5
Fluorine (F)	200
Iron (Fe)	500
Gadolinium (Gd)	3
Magnesium (Mg)	200
Molybdenum (Mo)	100
Nickel (Ni)	200
Nitrogen (N)	300
Samarium (Sm)	2
Silicon (Si)	200
Sodium (Na)	100
Titanium (Ti)	100
Thorium (Th)[B]	50
Tungsten (W)	100
Zinc (Zn)	100

[A] Sample may be heated prior to carbon analysis.
[B] Thorium is primarily of concern because of the reactor production of ^{233}U.
[C] 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.

[4] Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Two Park Ave., New York, NY 10016-5990, http://www.asme.org.
[5] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

TABLE 2 Additional Impurity Elements

Element	
Beryllium (Be)	Niobium (Nb)
Bismuth (Bi)	Phosphorous (P)
Calcium (Ca)	Potassium (K)
Cobalt (Co)	Silver (Ag)
Copper (Cu)	Sulfur (S)
Indium (In)	Tantalum (Ta)
Lead (Pb)	Tin (Sn)
Lithium (Li)	Vanadium (V)
Manganese (Mn)	Zirconium (Zr)
Neptunium (Np)	

6. Physical Properties

6.1 *Cleanliness and Workmanship*—The PuO_2 powder shall be free of visible fragments of foreign matter.

6.2 *Particle Size*—PuO_2 powder particle size limits and method of determination shall be agreed upon between the buyer and seller. As an example, in oxalic acid type precipitation processes, no particles should exist above 100 µm and at least 95 % of the particles are expected to be less than 50 µm.

6.3 *Tap Density*—The measured tap density of the PuO_2 powder will depend on the production process and measurement method. This measurement is often used in criticality calculations. The tap density limit and method of determination (for example, Test Method C1770 or ISO 9161) shall be agreed upon between the buyer and seller.

6.4 *Specific Surface Area*—The specific surface area limits and method of determination shall be agreed upon between the buyer and seller. As an example, in oxalic acid type precipitation processes the specific surface area of the purified PuO_2 powder is expected to be between 2 m^2/g and 30 m^2/g based on the Brunauer-Emmet-Teller, or BET, adsorption method (for example, in accordance with Guide C1274).

NOTE 1—Requirements relative to the physical properties of the PuO_2 will depend on the particular MOX fuel pellet fabrication process employed. For example, the unique physical properties of all of the PuO_2 (for example, particle size, tap density, specific surface area, etc.) entering into the MOX fuel pellet manufacturing process are essentially erased when the powder is milled in the case where this is a fuel manufacturing step. Nevertheless, even when no pass-fail criteria on physical property measurements is applicable, typical measurement values can be provided as an expected target and measurements can be provided for information in order to detect process drift, for example. Consistency between as-received PuO_2 batches, reflecting a stable and controlled PuO_2 powder manufacturing process, is desirable to minimize any potential impact on the MOX fuel pellet manufacturing process.

NOTE 2—For fuel pellet manufacturing processes that involve blending of different powders, consideration should be given to the compatibility of powders with widely different physical properties within the intended process.

7. Sampling

7.1 PuO_2 is hygroscopic and can absorb sufficient water during exposure to a moist atmosphere to cause detectable analytical errors. Sampling, weighing of the sample, and handling the sample shall be done under atmospheric conditions that do not alter the moisture or impurity content of the sample.

7.2 The necessary chemical and physical analyses shall be performed on portions of a representative sample taken from each lot.

7.2.1 A lot is defined as the quantity of material that is uniform in isotopic, chemical, and physical characteristics.

7.2.2 Lots may be formed by blending the powder to ensure homogeneity within each lot.

7.2.3 The mixing of two or more lots shall require the establishment of a new lot.

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

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

7.3 Sampling plans and procedures, including the frequency and time period for conducting analyses, shall be agreed upon between buyer and seller. Analytical confirmation of sampling plans shall be documented as part of the manufacturer's quality assurance and nuclear materials control and accountability program.

7.4 All sample containers shall be clearly identified by lot number and container number.

7.5 The sample material shall be packaged so that no foreign material is introduced into the powder during storage or shipment.

7.6 *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 and shall be contingent upon the material properties meeting the requirements of Sections 4 through 7.

8. Methods of Chemical and Isotopic Analysis

8.1 The analytical chemistry methods used shall be as described in Test Method C697 or other methods agreed upon between buyer and seller. See, for example, ISO 8300 for determination of Pu content in PuO_2 of nuclear grade quality.

9. Quality Assurance

9.1 Quality assurance requirements shall be agreed upon between buyer and seller. Code of Federal Regulations Title 10, Part 50, Appendix B and ASME NQA-1 are referenced as guides.

10. Rejection and Rehearing

10.1 Rejection and acceptance shall be by lot unless there is prior agreement to do otherwise between the buyer and seller.

10.2 The buyer and seller shall agree to a third party as a referee in the event of a dispute in analytical results.

11. Certification

11.1 The seller shall test the sample described in the Sampling section to ensure conformance of the oxide to the requirements of Sections 4, 5, and 6.

11.2 The seller shall provide the buyer documents certifying that the oxide meets all the requirements of Sections 4, 5, and 6.