



Standard Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable¹

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^{ε1} NOTE—Section 4.6 was changed editorially in June 2011.

INTRODUCTION

This specification is intended to provide the nuclear industry with a general standard for sinterable plutonium dioxide powder. It recognizes the diversity of manufacturing methods by which plutonium dioxide powders are produced, and the many special requirements for chemical and physical characterization that 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 more stringent or additional requirements for specific applications.

1. Scope

1.1 This specification covers nuclear grade plutonium dioxide, sinterable powder obtained by the oxalate precipitation route, calcined above 500°C, or any other equivalent process acceptable to the buyer. Included is plutonium dioxide 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 national and local regulations on processing, shipping, or using source or special nuclear materials. 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-7016², and “Handbook of Nuclear Safety”, H. K. Clark, U.S. Atomic Energy Commission Report, DP-532².

1.3 The PuO₂ 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 the 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

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

C1233 Practice for Determining Equivalent Boron Contents of Nuclear Materials

C1295 Test Method for Gamma Energy Emission from Fission Products in Uranium Hexafluoride and Uranyl Nitrate Solution

2.2 ANSI Standard:

ANSI/ASME NQA-1 Quality Assurance Requirements for Nuclear Facility Applications⁴

2.3 U.S. Government Documents:

Code of Federal Regulations, Title 10, Nuclear Safety Guide, U.S. Atomic Energy Commission Report TID-7016²

“Handbook of Nuclear Safety,” Clark, H. K., U.S. Atomic Energy Commission Report, DP-532²

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² Available from Superintendent of Documents, U.S. Government Printing Office, 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.

⁴ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

2.4 ISO Standard:

ISO 8300 Determination of Pu Content in Plutonium Dioxide (PuO₂) of Nuclear Grade Quality, Gravimetric Method⁴

3. Isotopic Content

3.1 Concentrations and homogeneity ranges of the plutonium isotopes shall be as specified by the buyer.

3.2 The isotopic composition of the final product shall be determined by mass spectrometry and shall be reported on a weight basis.

4. Chemical Composition

4.1 *Plutonium Content*—The minimum Pu content shall be 86.0 weight % as sampled on the date of sampling and 87.5 weight % after heating 4 h to 950°C.

4.2 *Uranium Content*—The uranium content of the plutonium dioxide shall be measured and reported on a plutonium basis.

4.3 *Americium Content*—The americium content shall be measured and reported on a plutonium basis. The maximum acceptable americium content shall be agreed upon between the buyer and the seller.

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

4.5 *Impurity Content*—The impurity content shall not exceed the individual element limit specified in Table 1. Total non-volatile oxide impurity content excluding Am shall not exceed 6000 µg/g Pu. 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.

4.6 *Equivalent Boron Content*—For thermal reactor use, the total equivalent boron content (EBC) shall not exceed 20.0 µg/g on a plutonium weight basis. For the purpose of EBC calculation Eu, Dy, and Sm 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.

4.7 *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 10⁵ MeV·Bq/g Pu.

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

5. Physical Properties

5.1 *Cleanliness and Workmanship*—The PuO₂ powder shall be free of visible fragments of foreign matter.

5.2 *Particle Size*—All the PuO₂ powder shall be capable of passing a 100 µm sieve and 95 % of the powder by weight shall be capable of passing a 44 µm sieve.

5.3 *Surface Area*—The specific surface area shall not be less than 5 m²/g and not greater than 30 m²/g based on a Brunauer, Emmett, Teller (BET) adsorption method. See Notes 1 and 2.

NOTE 1—A specific surface area as low as 2 m²/g has been demonstrated to be acceptable when subsequently blended with other powder(s) of greater specific surface area. Therefore, powder with a specific surface area as low as 2 m²/g may be acceptable if agreed upon between the supplier and the user.

NOTE 2—Consideration should be given to the compatibility of powders with widely different surface areas within the intended process.

6. Sampling

6.1 Plutonium oxide 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.

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

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

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

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

6.3 Sampling procedures, including the frequency and time period for conducting analyses, shall be agreed upon between buyer and seller in accordance with quality assurance requirements.

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

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

7. Methods of Chemical and Isotopic Analysis

7.1 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 plutonium dioxide of nuclear grade quality.

TABLE 1 Impurity Elements and Maximum Concentration Limits

Element	Maximum Concentration Limit of Uranium, µg/gPu
Boron	3
Cadmium	3
Carbon ^A	200
Chlorine	300
Chromium	200
Fluorine	200
Iron	300
Gadolinium	3
Nickel	100
Nitride Nitrogen	200
Thorium	200

^A Sample may be heated prior to carbon analysis.

8. Quality Assurance

8.1 Quality assurance requirements shall be agreed upon between buyer and seller. CFR Title 10, Part 50, Appendix 50 and ANSI/ASME NQA-1 are referenced as guides.

9. Rejection and Rehearing

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

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

10. Certification

10.1 The seller shall test the sample described in the Sampling section to ensure conformance of the oxide to the requirements of the Isotopic Content, Chemical Composition, and Physical Properties Sections.

10.2 The seller shall provide the buyer documents certifying that the oxide meets all the requirements of the Isotopic Content, Chemical Composition, and Physical Properties Sections.

10.3 The seller shall make available, as requested by the buyer, records of all data from tests used to meet the requirements in the Isotopic Content, Chemical Composition, and Physical Properties Sections.

11. Packaging and Package Marking

11.1 Plutonium dioxide powder shall be packaged in sealed metal 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 mutually agreed upon between the buyer and the seller, and in conformance with all applicable regulations.

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

- 11.2.1 Seller's name,
- 11.2.2 Lot number,
- 11.2.3 Gross, tare, net oxide weights,
- 11.2.4 Plutonium weight, and
- 11.2.5 A unique container reference number.

12. Keywords

- 12.1 nuclear fuel; plutonium; plutonium dioxide

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