



Standard Specification for Nuclear-Grade Plutonium Dioxide Powder, Sinterable¹

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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 purchaser 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 purchaser. 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 Federal, state, and local regulations on processing, shipping, or using source or special nuclear materials. Examples of U.S. Government documents are N7.2 Radiation Protection in Nuclear Reactor Fuel Fabrication Plants², Code of Federal Regulations, Title 10 Nuclear Safety Guide, U.S. Atomic Energy Commission Report TID-7016³, and “Handbook of Nuclear Safety”, H. K. Clark, U.S. Atomic Energy Commission Report, DP-532³.

1.3 The PuO₂ shall be produced by the processor employing 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:

C 697 Test Method for Chemical, Mass Spectrometric, and Spectrochemical Analysis of Nuclear-Grade Plutonium Dioxide Powders and Pellets⁴

2.2 ANSI Standards:

N7.2 Radiation Protection in Nuclear Reactor Fuel Fabrication Plants²

ANSI-ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facilities²

2.3 U.S. Government Documents:

Code of Federal Regulations, Title 10, Nuclear Safety Guide, U.S. Atomic Energy Commission Report TID-7016³

“Handbook of Nuclear Safety,” Clark, H. K., U.S. Atomic Energy Commission Report, DP-532³

2.4 ISO Standard:

ISO 8300 Determination of Pu content in plutonium dioxide (PuO₂) of Nuclear Grade Quality, Gravimetric Method²

3. Isotopic Content

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

3.2 The isotopic composition of the final product shall be

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² Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

³ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁴ *Annual Book of ASTM Standards*, Vol 12.01.

determined by mass spectrometry and shall be reported on a weight basis.

4. Chemical Composition

4.1 The necessary chemical analyses shall be performed on portions of a representative sample taken in accordance with the Sampling section after sampling within an agreed period. Analytical results shall be reported on a plutonium basis.

4.1.1 The uranium content of the plutonium dioxide shall be measured and reported.

4.1.2 The thorium content of the plutonium dioxide shall not exceed 200 µg/g of plutonium.

4.1.3 The americium content shall be measured and reported. The maximum acceptable americium content shall be agreed upon between the purchaser and the seller.

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

4.2 Total non-volatile oxide impurity content excluding Am shall not exceed 6000 µg/g Pu.

4.2.1 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.

4.2.2 The carbon content shall not exceed 200 µg/g after heating.

4.2.3 The nitride nitrogen content shall not exceed 200 µg/g.

4.2.4 The chlorine content shall not exceed 300 µg/g and the fluorine content shall not exceed 200 µg/g.

4.2.5 The iron content shall not exceed 300 µg/g, the chromium content 200 µg/g and the nickel 100 µg/g.

4.2.6 The gadolinium (Gd), cadmium (Cd), and boron (B) content shall be measured and each of them shall not exceed 3 µg/g.

4.2.7 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. This measured gamma activity shall be multiplied by the appropriate mean energy per disintegration (MeV); the sum of these values shall be less than 10⁵ MeV·Bq/g Pu.

4.2.7.1 The mean energies per disintegration to be used in the calculations are in the following list:

Fission product	MeV/disintegration
Ru-105	0.210
Ce-144	0.0527
Sb-125	0.427
Cs-134	1.555
Cs-137	0.597

5. Physical Properties

5.1 All physical testing shall be performed on portions of the representative sample prepared in accordance with the Sampling section.

5.2 The PuO₂ powder shall be free of visible fragments of foreign matter. 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 The surface area shall not be less than 5 m²/g and not greater than 30 m²/g based on a Brunauer, Emmett, Teller adsorption method. In any one batch, the surface area determined on any one lot shall not exceed the surface area

determined on any of the other lots by more than a factor of two.

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, or both, of the sample.

6.2 A representative sample of powder shall be 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-frequency 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 Methods C 697 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.

8. Quality Assurance

8.1 Quality assurance requirements shall be agreed upon between buyer and seller. CFR Title 10, Part 50, Appendix 50 and 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 purchaser and seller.

9.2 The purchaser 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 purchaser 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 purchaser, 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 purchaser 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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