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RDT Standard

FAST FLUX TEST FACILITY
CERAMIC GRADE PLUTONIUM DIOXIDE

JUNE 1971

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FAST FLUX TEST FACILITY CERAMIC GRADE PLUTONIUM DIOXIDE

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RDT STANDARD

UNITED STATES ATOMIC ENERGY COMMISSION
DIVISION OF REACTOR DEVELOPMENT AND TECHNOLOGY

RDT E 13-1T

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FAST FLUX TEST FACILITY CERAMIC GRADE PLUTONIUM DIOXIDE

1. SCOPE

This standard establishes the requirements for plutonium dioxide, ceramic grade, for use in the fabrication of mixed oxide fuel pellets that meet the requirements of RDT Standard RDT E 13-6, FFTF Driver Fuel Pin Mixed Oxide Fuel Pellet.

2. APPLICABLE DOCUMENTS

The following documents form a part of this standard to the extent specified herein. In the event of conflict between the documents referenced and this standard, the detail requirements of this standard shall be superseding.

2.1 AEC-RDT Standards

RDT F 2-4T (October 1969) - Quality Verification Program Requirements

2.2 WADCO

WHAN-IR-5, FFTF Analytical Chemistry Methods in Support of Driver Fuel Fabrication, August, 1970

3. TECHNICAL REQUIREMENTS

3.1 Particle Size

3.1.1 Sieve Analysis. Plutonium dioxide powder shall meet the following sieve analysis:

<u>Sieve, U.S. Standard, or Equivalent</u>	<u>Percentage PuO₂ Powder Passed</u>
170-mesh	100
200-mesh	99 min.
325-mesh	95 min.

3.1.2 Particle Distribution. The plutonium dioxide powder shall meet one of the particle size criteria listed below.

(a) Coulter Counter Method. At least 50% of particles shall be less than 20 microns.

(b) Fisher Sub-Sieve Sizer. Average particle size shall be less than 10 microns.

(c) M.S.A. Size Distribution. At least 50%, by weight, shall be less than 10 microns.

- 3.2 Surface Area. The surface area of the plutonium dioxide powder shall be equal to or greater than 2.5 m²/g.
- 3.3 Isotopic Content. Plutonium dioxide shall have the following isotopic content based upon total plutonium weight. The concentration of each isotope from ²³⁸Pu through ²⁴²Pu shall be reported. Plutonium isotopes of mass numbers less than ²³⁸Pu and greater than ²⁴²Pu need not be determined.

²³⁹ Pu plus ²⁴¹ Pu	[88.0 ± 0.5]%
²⁴¹ Pu	2.5% (max.)
²³⁸ Pu	0.15% (max.)
All other plutonium isotopes excluding ²⁴⁰ Pu	0.35% (max.)
²⁴⁰ Pu	Balance

- 3.4 Americium Content. The americium content of the plutonium dioxide at time of delivery to the Contractor shall meet one of the following requirements.

(a) The americium content shall not exceed 0.25 wt%.

(b) The americium content shall be controlled by the relationship between ²⁴¹Pu and the time since separation from irradiated fuel or since refining to remove americium. The product of the percentage of ²⁴¹Pu and months since separation or purification shall not exceed 60. If two or more batches of plutonium nitrate solution or plutonium dioxide having different separation dates are blended, a weighted average of the months since separation shall be used.

- 3.5 Impurity Levels. The impurity levels shall not exceed the maximum limits shown in Table 1 for plutonium dioxide which has been calcined in air at 950° ± 25°C (See Paragraph 6.3). Impurities may optionally be determined on the powder calcined at a lesser temperature without change of impurity limits.

(a) Total impurities listed in Table 1, exclusive of uranium and americium, shall not exceed 5000 ppm by weight.

(b) Uranium shall not exceed 2000 ppm by weight.

- 3.6 Oxygen-to-Plutonium Ratio. The atom ratio of oxygen-to-plutonium shall be 1.95 to 2.00.

TABLE 1: IMPURITY LIMITS FOR PLUTONIUM DIOXIDE CALCINED AT $950 \pm 25^{\circ}\text{C}$

<u>Impurity</u>	<u>Limits in ppm by Weight of Plutonium Dioxide, Max.</u>
Aluminum	250
Beryllium	20
Boron	10
Cadmium	20
Calcium	500
Carbon	200
Chlorine	50
Chromium	200
Cobalt	20
Fluorine	25
Iron	350
Lithium	10
Magnesium	100
Nickel	300
Nitride Nitrogen	200
Phosphorus	200
Potassium	200
Sodium	300
Vanadium	200
Sulfur	300
Tantalum	400
Tungsten	100
The sum of Copper, Zinc, Silicon, and Titanium	800
The sum of Silver, Manganese, Molybdenum, Lead and Tin	200
The sum of Samarium, Europium, Gadolinium, and Dysprosium	100

3.7 Plutonium Content. The plutonium content in the plutonium dioxide shall meet both of the following requirements:

- (a) The minimum plutonium content of a representative sample at the time of packaging shall be 85.0% by weight.
- (b) The minimum plutonium content of a representative sample when calcined to 950°C shall be 87.5% by weight.

3.8 Loss on Ignition. The loss on ignition shall not exceed 2.5% by weight.

3.9 Sinterability. Plutonium dioxide shall be sinterable to a minimum average density of 90% of the theoretical density of PuO_2 when

sintered in accordance with the sinterability performance test in Appendix A (See Paragraph 6.2).

3.10 Process

3.10.1 Process Sequence. The process sequence shall be prepared by the Manufacturer and shall be submitted to the Purchaser for approval. No changes shall be made to the process sequence until they are submitted to and approved by the Purchaser. The process sequence shall include a process flow diagram and description of containers for plutonium dioxide.

3.10.2 Screening. The plutonium dioxide powder shall be passed through a 170-mesh U.S. Standard Sieve, or equivalent.

3.10.3 Lotting. A description of lotting procedures shall be submitted to the Purchaser for approval. Plutonium dioxide lots shall be uniform in terms of isotopic, chemical, and physical characteristics.

3.10.4 Blending. Plutonium dioxide powder lots having a minimum and a maximum of 87% and 89%, respectively, of ^{239}Pu plus ^{241}Pu may be blended.

3.10.5 Storage. Plutonium dioxide powder shall be stored in containers marked as specified in "Labeling," Paragraph 5.2.

3.11 Analytical Samples

3.11.1 Archive Sample. Unless otherwise specified, at least 50 grams of plutonium dioxide from each lot shall be provided for an archive sample. The archive sample shall meet all requirements of this standard and shall be identified with lot number and purchase order number. The archive samples shall be delivered to the Purchaser.

3.11.2 Referee Sample. Unless otherwise specified, at least 50 grams of plutonium dioxide from each lot shall be provided for a referee sample. The referee sample shall meet all requirements of this standard and shall be identified with lot number and purchase order number. The referee samples shall be retained by the Manufacturer.

3.11.3 Contractor Sample. Unless otherwise specified, at least 50 grams of plutonium dioxide from each lot shall be provided for the Contractor sample. The Contractor sample shall meet all requirements of this standard and shall be identified with lot number and purchase order number. The Contractor samples shall be delivered to the Contractor.

4. QUALITY ASSURANCE PROVISIONS

4.1 Quality Assurance Program Requirements. The Manufacturer shall plan, establish, implement, and maintain a documented quality assurance program that fulfills all requirements of RDT F 2-4 and any additional quality assurance requirements imposed by contract.

- 4.2 Quality Assurance Documentation. The Manufacturer is responsible for the preparation of all quality assurance documentation required by RDT F 2-4. The documents identified in Table 2 shall be submitted to the Purchaser for the applicable purpose at the indicated time. The granting of any process or procedural approvals by the Purchaser shall not be construed to relieve the Manufacturer in any way or to any extent from the full responsibility for delivering items conforming with all requirements of this standard.
- 4.3 Standards of Manufacture, Manufacturing Process, and Production. All standards or specifications shall be as established in Paragraph 2, Applicable Documents. Other additional standards or specifications or modifications of said standards and specifications prepared by the Manufacturer shall be submitted to the Purchaser for approval.
- 4.4 Quality Records. The Manufacturer shall submit a copy of final inspection data and test results to the Contractor at the time of product delivery.
- 4.5 Responsibility. Unless otherwise specified, the Manufacturer shall be responsible for the performance of all tests and inspections required prior to submitting any plutonium dioxide powder to the Purchaser. Except as otherwise specified, the Manufacturer may use either his own or any commercial laboratory acceptable to the Purchaser. Records of all tests and examinations shall be kept complete and available to the Purchaser as specified in the contract or purchase order.

TABLE 2: DOCUMENTATION SUBMITTAL REQUIREMENTS

Item	Description	Reference Paragraph of RDT F 2-4	Submit to Purchaser for Approval	Information	Time of Submittal to Purchaser
1	Quality Verification Plan	2.1.1	X		Thirty days after contract award
2	Sampling Plans	2.1.2	X		Prior to implementation
3	Certification	5.5		X	With delivery
4	Corrective Action Reports	8		X	As generated
5	Audit Reports	10		X	As generated

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- 4.6 Inspection Requirements. Plutonium dioxide powder shall be inspected for conformance with Section 3 in accordance with Table 3. Other analytical methods may be substituted for the reference test method provided that equivalent results have been demonstrated; i.e., the alternate method has been qualified and provides a measurement error not greater than that typical of the reference method, and that the method shall have been approved by the Purchaser prior to implementation.
- 4.7 Inspection or Test Level. Acceptability of a lot shall be determined by lot qualification tests on representative samples taken in accordance with the Sampling Procedure, Paragraph 4.8. Lots shall be accepted only when all specified requirements are met.
- 4.8 Sampling
- 4.8.1 Sampling Requirements. At least one representative sample from each lot shall be taken. Sampling shall be as follows:
- (a) To obtain a lot sample, approximately equal size specimens shall first be taken with a thief at a random location along any vertical traverse through each container. The container specimens shall then be blended and split down to the required size to form the lot sample.
 - (b) All sample containers shall be traceable to information specified in Paragraph 5.2.
 - (c) Sample containers shall preserve the integrity of the sample, particularly preventing the sorption of water.
- 4.8.2 Sample Size. The lot sample shall be of a sufficient size to permit breaking down into smaller samples of at least 50 grams each for the following tests.
- Analytical testing at Manufacturer's plant
 - Acceptance tests at Contractor's plant (See Paragraph 3.11.3)
 - Referee tests (See Paragraph 3.11.2)
 - Archive sample (See Paragraph 3.11.1)
- Use of this type of composite sampling is expected to enable better comparison of chemical analytical results.
- 4.9 Referee Methods for Analytical Results. In the event of a dispute in analytical results, the Purchaser and the Contractor shall agree to third party referee methods.
- 4.10 Lot Records. Plutonium dioxide lot records, traceable to feed material lots showing complete processing, sequence of operation, chemical analysis, and sinterability test results shall be supplied to the Purchaser. Documentation shall be readily available and traceable to individual package identity number.

TABLE 3: QUALITY CONFORMANCE INSPECTION

Characteristic	Requirement	Sampling*	Reference Test Method
4.6.1 Sieve Analysis	3.1.1	1	Screen 100g PuO ₂ powder sample with 170-mesh, 200-mesh, and 325-mesh U.S. Standard Sieves, or equivalent. (This sample may be returned to the PuO ₂ lot.)
4.6.2 Particle Distribution	3.1.2	1	Determine distribution by one of the following methods: (a) <u>Coulter Counter Method.</u> Determine particle size and distribution. (See general guide, Operating Manual for Coulter Counter, Coulter Electronics Industrial Division of Coulter Electronics, Franklin Park, Illinois). (b) <u>Fisher Sub-Sieve Sizer.</u> Measure average particle size. (See Operating Manual for Fisher Sub-Sieve Sizer, Fisher Scientific Co., Pittsburgh, Penn.) (c) <u>M.S.A. Size Distribution.</u> Measure particle size distribution in accordance with Method 30.3, WHAN-IR-5.
4.6.3 Surface Area	3.2	2	Determine surface area by modified BET Method, No. 30.2, WHAN-IR-5.
4.6.4 Plutonium Isotopic Content	3.3	1	Determine isotopic composition on lot sample by mass spectrometry according to Method 10.2, WHAN-IR-5.

*Minimum number of analyses per lot or sub-lot

TABLE 3: QUALITY CONFORMANCE INSPECTION (continued)

Characteristic	Requirement	Sampling*	Reference Test Method
4.6.5 Americium Content			
(a) Analysis	3.4 (a)	1	Determine by anion exchange and alpha analysis in accordance with Method No. 20.10, WHAN-IR-5.
(b) Calculation	3.4 (b)	1	Calculate on basis of ^{241}Pu content and months since separation.
4.6.6 Impurity Levels			Calcine sample in air at $950^\circ \pm 25^\circ\text{C}$ until constant weight is obtained. (See Paragraph 6.3) Impurities may optionally be determined on powder calcined at a lower temperature provided impurity limits requirements of Table 1 are met. Determine impurity content by use of appropriate methods listed in Section 20 of WHAN-IR-5.
(a) C, Cl, F	3.5	2	
(b) Impurities, all other	3.5	1	
(c) Total Impurities	3.5 (a)	0	Summation of (a) and (b), above
(d) Uranium	3.5 (b)	1	
4.6.7 Oxygen-to-Plutonium Ratio			
(a) Oxalate and Nitrate	3.6	0	No test required. The oxygen-to-plutonium ratio for air calcined oxalate and nitrate source PuO_2 powders is assumed to be 2.00.

* Minimum number of analyses per lot or sub-lot

TABLE 3: QUALITY CONFORMANCE INSPECTION (continued)

Characteristic	Requirement	Sampling*	Reference Test Method
(b) Burned Metal Source	3.6	1	Determine O/Pu ratio for burned-plutonium source PuO ₂ as prescribed in Method 10.3 of WHAN-IR-5.
4.6.8 Plutonium Content			
(a) Packaged Material	3.7(a)	2	Measure as prescribed in Method 10.1 of WHAN-IR-5.
(b) After Calcining	3.7(b)	1	Calcine sample at $950^{\circ} \pm 25^{\circ}\text{C}$ until constant weight is obtained (See Note 6.3). Measure as prescribed in Method 10.1 of WHAN-IR-5.
4.6.9 Loss on Ignition	3.8	2	Measure loss on ignition by heating a test sample to $950^{\circ} \pm 25^{\circ}\text{C}$ in air to constant weight (See Note 6.3) and determine the weight difference.
4.6.10 Sinterability	3.9	1	Conduct sinterability performance test in accordance with Appendix A.
4.6.11 Process Sequence	3.10.1	Certification	Manufacturer's records
4.6.12 Screening	3.10.2	Certification	Manufacturer's records

*Minimum number of analyses per lot or sub-lot.

Continued

TABLE 3: QUALITY CONFORMANCE INSPECTION (continued)

Characteristic	Requirement	Sampling*	Reference Test Method
4.6.13 Lotting	3.10.3	Certifi- cation	Manufacturer's records
4.6.14 Blending	3.10.4	Certifi- cation	Manufacturer's records
4.6.15 Storage	3.10.5	Certifi- cation	Manufacturer's records
4.6.16 Archive Sample	3.11.1	Certifi- cation	Manufacturer's records
4.6.17 Referee Sample	3.11.2	Certifi- cation	Manufacturer's records
4.6.18 Contractor Sample	3.11.3	Certifi- cation	Manufacturer's records

* Minimum number of analyses per lot or sub-lot.

5. PREPARATION FOR DELIVERY

5.1 Packaging and Shipping. A complete description of the containers, including drawings and packaging procedures, shall be submitted to the Purchaser for approval.

5.2 Labeling. Plutonium dioxide shall be shipped in containers and identified to show the following (See Paragraph 4.10):

- Purchase order number
- Identity of material in containers
- Gross, tare, and net weight
- Lot number
- Individual package identity number

6. NOTES

6.1 Intended Use. Plutonium dioxide described by this standard is to be used in the fabrication of mixed oxide fuel pellets for driver fuel pins which are to be assembled for loading into the FFTF.

6.2 Plutonium Dioxide Sinterability. The sinterability performance test as delineated in the Appendix is used to verify the sinterability of each lot of PuO_2 powder and is not intended to simulate a fuel fabricator's pellet fabrication process.

6.3 Calcination of Test Samples. Plutonium dioxide obtained by low temperature ($\sim 500^\circ\text{C}$) calcination of plutonium oxalate or nitrate can absorb sufficient water to cause analytical error. The plutonium dioxide powder sample shall be recalcined at $950^\circ\text{C} \pm 25^\circ\text{C}$ until a constant weight is obtained for determining weight loss and to provide a base for analytical and processing calculations. The plutonium content, impurity level, and loss on ignition may be determined from the same calcined sample. The 950°C calcination is not a raw material requirement.

6.4 Definitions

FFTF - Fast Flux Test Facility

Contractor - The fuel pellet fabricator

Manufacturer - The plutonium dioxide producer

Purchaser - USAEC Richland Operations Office

APPENDIX A: SINTERABILITY PERFORMANCE TEST

A10. REQUIREMENT

A sinterability test shall be performed using ten pellets produced from the lot sample taken according to the sampling procedure in Paragraph 4.8. The density of each sintered test pellet shall be determined and shall not deviate from the ten-pellet average by more than 2.0% of the average. (The theoretical density of plutonium dioxide is 11.46 grams per cubic centimeter.)

A10.1 Density Determination. The density of each of the fired pellets shall be determined as follows:

- (a) For a given pellet, record the average of the maximum and minimum diameter measurements taken with equipment capable of being read to the nearest 0.0002 inch. (It is suggested that pellets be ground to minimize measurement error.)
- (b) Record pellet length taken with equipment capable of being read to nearest 0.0002 inch.
- (c) Weigh the given sample pellet to the nearest 0.001 gram.
- (d) Calculate the density of each given sample pellet to the nearest 0.01 g/cc.

A10.2 Performance Test Instructions. The sinterability test shall be performed on plutonium dioxide that meets all other requirements of this standard. The plutonium dioxide powder may be processed to modify material characteristics (grinding or calcining) provided the impurity levels of Paragraph 3.5 are not exceeded. All sinterability test processing variables, when once established, shall remain unchanged through production for a given order.

A10.2.1 Binder Addition. Pellets shall be fabricated without a binder.

A10.2.2 Granulation (Optional). Plutonium dioxide powder may be prepressed at a pressure not exceeding the forming pressure and the resulting pellets granulated to pass through a 14 to 20-mesh Tyler Screen sieve, or equivalent.

A10.2.3 Pressing

- (a) Green Pellet Dimensions. The diameter shall be 0.235 ± 0.020 inch and the length-to-diameter ratio shall be between 1.0 and 1.5.

(b) Forming Pressure. A pellet press forming pressure of 30,000 to 50,000 psi is suggested.

(c) Die Lubrication. A die lubricant may be used.

A10.2.4 Lubricant Removal (Optional). Lubricant removal from the green pellet is optional and its use depends upon the lubricant used and the type of sintering furnace used. In the event that the pellets are to be subjected to lubricant removal, the following are recommended:

- (a) Atmosphere: Inert gas, carbon dioxide, or air (preferred).
- (b) Suggested furnace temperature: 600°C
- (c) Time at temperature: As required depending upon degree of lubricant burnoff desired.

A10.2.5 Sintering. All test pellets shall be sintered at one time in the same zone within a furnace under the following conditions:

- (a) Atmosphere: Air, or inert gas (helium or argon) bubbled through 0-25°C water.
- (b) Rate-of-rise: The temperature rate-of-rise shall be no more than 250°C per hour as determined from 100°C to sintering temperature.
- (c) Sintering temperature: 1550 ± 50°C
- (d) Soak time at sintering temperature: 1 hour, minimum
- (e) Cooling rate: The cooling rate shall not exceed 300°C per hour.

A10.2.6 Stoichiometry Control. Test pellets sintered in the oxygen potential specified in A10.2.5 should have an O/Pu ratio of 2.00. An O/Pu determination need not be performed if the pellets are sintered in an oxidizing atmosphere.

A10.2.7 Records. The following sinterability test data shall be recorded and included with PuO₂ lot records:

- (a) Sintered pellet density
- (b) Sinterability test procedure or reference document
 - Pretreatment, if any
 - Description of equipment used for pretreatment
 - Press description and forming pressure
 - Sintering time, furnace temperature, and furnace rate-of-rise
- (c) Deviations from the reference sinterability test shall be included.

1 of 1 Complete Record

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