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TITLE

SPECIFICATION FOR NUCLEAR GRADE MIXED OXIDES (UO_2-PuO_2), HANFORD SALT CYCLE COMPACTIBLE POWDER

ISSUING FILE

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FOREWORD

The proposed specification is designed to help an internal effort in fabricating PRTR fuel elements containing nuclear fuel prepared by the "salt cycle" process. It recognizes the diversity of pilot plant processes, and chemical and physical characteristics that may be imposed by the final end use in a specific reactor system.

Since all these conditions cannot be met simultaneously by a standard product, a minimum standard is proposed. More stringent specifications or additional conditions can be added as needed.

1. Scope

1.1 This specification defines the physical and chemical characteristics required of the high density UO_2 particles enriched with PuO_2 (mixed oxides) to be used in the packed-particle nuclear fuel elements manufactured by either the vibrational compaction process[1] or a similar process. The high density particles are formed from nuclear grade UO_2 and PuO_2 powders that have been prepared by the "salt cycle" process,[2] then granulated and sieved. Specifications of UO_2 and PuO_2 are detailed in previous reports.[3,4,5]

2. Lot Sizes

2.1 A lot is defined as that quantity of material that has been blended as an entity before packaging or use. A lot of high density particles of mixed oxides shall be formed from "salt cycle" material that has been crushed, sieved, and thoroughly blended to assure homogeneity. A lot size shall generally be composed of material required to produce one fuel element.

3. Sampling

3.1 A representative sample of powder from each lot shall be taken for the purpose of determining chemical and physical properties.

3.2 The representative sample of material usually 1.5 wt% which passes through a -6 mesh U.S. standard sieve shall be taken from each lot of material. Sub-sampling of the lot after screening and mesh sizing of material in a lot shall be taken by coning and quartering. During preliminary remote control situations, grab sampleing may be used.

3.3 The sample size shall be sufficient to perform the following:

3.3.1 All quality control tests associated with the fabrication of the mixed oxides.

3.3.2 All acceptance tests for chemical and physical properties.

3.3.3 Reference tests in event it becomes necessary to re-examine the material.

3.3.4 Usually, a 1.5 wt% sample of the blended (mesh) lot is taken for storage.

4. Testing

4.1 All chemical and physical tests shall be performed on portions of the representative sample prepared in accordance with 3.

4.2 All chemical and physical testing methods shall be standardized methods of analyses to assure adequate testing of the material.

4.3 Material that does not meet specifications and approved deviations shall be subject to rejection and returned to storage for later recovery processes.

5. Physical Properties

5.1 Samples for physical property tests shall be taken from the material lot as specified under Section 3.

5.2 Detection within the samples of greater than one percent by weight of any combination of the following materials shall be cause for rejection of all of the lot represented by the sample:

5.2.1 Particles, crystals, and inclusions of any material other than UO_2 or PuO_2.

5.5.2 Porous particles of UO_2 or PuO_2. Porous particles are defined as particles containing interstices that will allow absorption or passage of water.

5.3 The apparent density of the UO_2-PuO_2 shall be greater than 10.86 g/cc as measured by the vacuum-mercury displacement technique (Hg. penetrometer at S.T.P.).

5.3.1 The apparent density determination shall be performed with 6 gram representative samples composed of particles from the -6 +10 mesh fraction.

5.4 The percentage of mesh sizes shall be specified before fabrication into fuel elements. (Ref. U.S. Standard Sieve.)

5.5 To assure absence of foreign matter and large agglomerates, all of the lot shall pass a 4 mesh U.S. Standard Sieve. (Ref. ASTM-B 214-56 Sieving Practice.)

6. Chemical Properties

6.1 Samples for chemical tests shall be taken from the material lot as specified under Section 3.

6.1.1 These samples for chemical tests shall pass or be crushed to pass through a 200 mesh U.S. Standard or equivalent sieve, and thoroughly blended.

6.2 The minimum plutonium content shall be specified in weight percent ($\pm$1.0 percent analytical error); minimum uranium content shall be specified in weight percent ($\pm$1.0 percent analytical error). (Ref. Coulometric Titration Method.)

6.2.1 Materials shall have the following O/M ratios:

6.2.1.1 The O/U ratio shall be 2.00 +.02

6.2.1.2 The O/Pu ratio shall be 2.00 $\pm$.02

6.3 The isotopic composition of UO_2 and PuO_2 shall not be changed during processing, and shall conform to the specified compositions. (Ref. Mass Spectrometry.)

6.3.1 U^{235} = specified weight percent U^{235} in total U.

6.3.2 Pu^{240} = specified weight percent P^{240} in total Pu.

6.4 Moisture content shall not exceed 100 parts per million. (Ref. Solids Moisture Analyzer)

6.5 The carbon content shall not exceed 100 parts per million. (Ref. Combustion Method.)

6.6 Nitrogen (including uranium or plutonium nitrides) shall not exceed 200 parts per million. Nitrogen shall be determined by the modified Kjeldahl procedure using hydrochloric-hydrofluosilicic acids and cupric selenate dissolution techniques.(6)

6.7 Other impurity limits are listed only for those elements that appear to affect compatibility and in-reactor stability. An emission spectrographic analysis shall be completed and the impurities are reported for purposes of 6.8.

6.7.1 Fluorine = 10 ppm maximum. Chlorine = 100 ppm maximum. (Ref. Pyro-hydrolysis method; Fluoride by lanthanum-alizarin coulometric analysis, Chloride by coulometric titration to an amperometric end-point.)

6.7.2 Lithium and Potassium = 100 ppm, each, maximum.

6.8 Total impurity content shall not exceed the "Equivalent Boron Content" (EBC) of 4 ppm on a weight basis. An emission spectrographic analysis shall be completed and the impurities reported used to calculate the EBC. (See attached tabulation of EBC.)

6.8.1 EBC shall be calculated by the following formula:

$$EBC^{*} = \frac{(\text{Atomic Weight of Boron}) \times (\alpha_i \text{ Impurity})}{(\text{Atomic Weight of Impurity}) \times (\alpha_i \text{ Boron})} \times (\text{ppm impurity})$$

6.8.2 The following listed elements shall be included in the calculation of the total EBC, but are not necessarily all the elements to be considered as the total impurity content: Ag, B, C, Ca, Cl, Cr, Cu, Cd, F, Fe, Mg, Mn, N, Ni, P, Pb, Si, Sn, V, An, and Dy, Eu, Gd, and Sm.

6.9 If previously irradiated material is to be included in a lot, additional analyses required before use are, Np less than 0.03 ppm, decontamination factors on rare earth fission products, and total gamma.

6.10 The total gas release exclusive of H_2) shall not exceed 0.10 cc/g at S.T.P. (Ref. The total gas release shall be measured on -200 mesh material by vacuum outgassing at 1×10^{-6} mm Hg. pressure for 30 minutes at 1000 C.)

*Footnote: Where atomic weight is on the physical scale, and α_1 is the thermal cross-section (BNL-325).

7. Test Reports

7.1 Original reports shall be filed in a log book and shall contain the following information:

7.1.1 Chemical and physical test results.

7.1.2 Isotopic data.

7.1.3 Calculated macroscopic absorption cross-section (EBC).

7.1.4 A statement that the lot material does or why it does not meet the specifications.

7.1.5 The lot number, and weight of material in each container.

7.1.6 Date of blending and packing of lot, and/or date of inter-lab transfer.

8. Packing and Marking

8.1 The material shall be packed in a manner which will prevent material loss or contamination during transit and storage. Containers shall comply with all safety regulations applicable to the transport and/or storage of source and fissionable materials.

8.2 Each container shall be plainly marked with the following information:

8.2.1 Lot number.

8.2.2 Gross, tare, and net weight.

8.2.3 Enrichment (i.e., 2 wt% PuO_2).

8.2.4 Radiation level of containers.

8.2.5 Date of packaging or resealing.

8.2.6 Name of SS inventory holder.

8.2.7 Source of the plutonium and uranium.

REFERENCES

1. J. J. Hauth, US-AEC Patent No. 3042594.

2. W. R. Bond, G. Jansen, L. K. Mudge, "Hanford Salt Cycle Process, II. Engineering Development in a High Level Facility", HW-SA-3527, August 17, 1964.

3. H. J. Anderson and R. J. Anicetti, "Specification for High Density Uranium Dioxide (Nuclear Grade), "HW-74204, Rev. 1, September 9, 1963. Rev. 2, July 16, 1964.

4. H. J. Anderson, "Specification of Nuclear Grade PuO_2, Sinterable Powder," HW-76566, Rev. 1, September 9, 1963.

5. G. E. Benedict and R. A. Nixon, "Hanford Salt Cycle Process, I. Plutonium Chemistry," HW-SA-3622, July 17, 1964.

6. H. J. Anderson and J. C. Langford, "Determination and Control of Nitrogen as Uranium Nitrides in Fused Uranium Dioxide (UO_2)," HW-SA-2663, July 12, 1962.

BORON EQUIVALENTS FOR
IMPURITIES IN URANIUM

Impurity	σ_a(barns)(1)	Atomic Weight	EBC, parts per million
Aluminum	0.230	26.98	0.000122
Barium	1.170	137.34	0.000122
Beryllium	0.010	9.01	0.000015
Boron	755	10.81	0.999999
Calcium	0.43	40.08	0.000153
Cadmium	2550	112.40	0.325097
Carbon	0.00373	12.01	0.000004
Chromium	2.90	52.00	0.000799
Cobalt	38.00	58.93	0.009239
Copper	3.85	63.54	0.000868
Iron	2.62	55.85	0.000672
Lead	0.170	207.19	0.000011
Magnesium	0.069	24.32	0.000040
Manganese	13.20	54.93	0.003443
Molybdenum	2.70	95.94	0.000403
Nickel	4.60	58.71	0.001122
Nitrogen	1.88	14.00	0.001924
Oxygen	0.0002	16.00	--
Phosphorus	0.19	30.97	0.000087
Silicon	0.13	28.09	0.000066
Silver	62.0	107.87	0.008236
Tin	0.60	118.70	0.000072
Tungsten	19.20	183.85	0.001496
Vanadium	5.00	50.94	0.001406
Zinc	1.10	65.37	0.000241
Zirconium	0.185	91.22	0.000029
Samarium	5500	150.35	0.524575
Europium	4600	152.00	0.433973
Gadolinium	46000	157.26	4.194580
Dysprosium	1100	162.51	0.097064

(1) Brookhaven National Laboratory Publication BNL-325, Second Edition, July 1958, and Supplement #1, January, 1960.