

*Materials Identification and Surveillance  
Project Item Evaluation*

*Item: Impure Plutonium Oxide (TS707001)*

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*Andreas Toupadakis*

*Charles Davis*

*Lynn Foster*

*David Horrell*

*Richard Mason*

*Jeremy Trujillo*

## CONTENTS

ABSTRACT .....	1
1.0 INTRODUCTION .....	2
2.0 EXPERIMENTAL METHODS .....	6
2.1 Materials .....	6
2.2 Procedures .....	6
3.0 RESULTS AND DISCUSSION .....	12
4.0 CONCLUSIONS .....	16
REFERENCES .....	16

## FIGURES

Fig. 1. Photograph of the canister at 0 degrees.....	7
Fig. 2. Radiograph of the canister at 0 degrees.....	8
Fig. 3. The as received can and inner can opened.....	9
Fig. 4. Characterization of the impure plutonium oxide TS707001.....	10

## TABLES

Table I. Rocky Flats Plutonium Oxide Legacy.....	5
Table II. Water Uptake Adsorption Measurements for the Impure Oxide TS707001.....	12
Table III. Percent Mass Loss During Calcination of Sample TS707001.....	12
Table IV. LOI Analyses Results of the Impure Oxide TS707001.....	13
Table V. Elemental Analysis of As-Received Powder S <sup>a</sup> and Calcined Powder S <sup>b</sup> .....	13
Table VI. Specific Surface Area Results for the Impure Oxide TS707001 (m <sup>2</sup> /g).....	15
Table VII. Particle Analysis Results of Precalcined Powder S <sup>a</sup> and Calcined Powder S <sup>b</sup> .....	15
Table VIII. Tap Density and Bulk Density of Calcined Powders S <sup>a</sup> and S <sup>b</sup> .....	16

# **MATERIALS IDENTIFICATION AND SURVEILLANCE PROJECT ITEM EVALUATION**

**Item: Impure Plutonium Oxide (TS707001)**

by

Andreas Toupadakis, Charles Davis, Lynn Foster,  
David Horrell, Richard Mason, Jeremy Trujillo

## **ABSTRACT**

In this report, we characterize properties relevant to storage of an impure plutonium oxide (86.26 mass % plutonium) in accordance with the Department of Energy (DOE) standard DOE-STD-3013-96.<sup>1</sup>

Methods used to characterize the oxide include mass loss-on-calcination (LOC) measurements, mass loss-on-ignition (LOI) measurements, water uptake adsorption measurements, elemental analysis, plutonium isotopic analysis, particle analyses measurements, tap and bulk density measurements, surface-area analyses, and x-ray powder diffraction. Methods used to characterize the container include x-ray radiography and photography.

Loss-on-ignition values are low. The material does satisfy DOE-STD-3013-96 criteria for plutonium content and LOI.

## 1.0 INTRODUCTION

Stabilization of pure and impure oxide materials destined for DOE-STD-3013 containment throughout the complex will be evaluated by the Materials Identification and Surveillance (MIS) project. The MIS project evaluates pure and impure plutonium metal and oxide legacy materials in existence, primarily at Rocky Flats, Savannah River, Hanford, and Los Alamos. The MIS project sponsors materials stabilization and packaging studies to confirm the suitability of material processing methods and packaging for long-term storage using DOE-STD-3013. Studies include characterization of plutonium oxide and assessment stabilization processing at 950°C for two hours. Experimental tasks and surveillance monitoring are carried out to evaluate the behavior of the oxide and packaging materials under storage conditions. Additional activities are sponsored to develop alternate methods for measuring volatiles on the oxide, determine the thermal conditions during storage, and model the pressure generation in sealed containers.

Rocky Flats has sent 24 items to Los Alamos for evaluation and verification that these materials can be stored safely for 50 years after thermal treatment. Material from all six categories were shipped; however, not all IDCs were represented in the samples.

### A. Oxide Source and Characterization Data

The plutonium oxide inventory at Rocky Flats has been divided into the following source categories:

#### **(1) Plutonium oxidation [IDCs 057, 060, 061, 083, 319, 653]**

This category consists of oxide obtained from metal brushing, chip burning, hydride oxidation, and certain pyrochemical operations. All oxide is believed to have been stabilized at temperatures above 500°C. The plutonium content will vary, but the impurities are expected to be metal oxides.

#### **(2) Aqueous processing [060, 061, 080, 081, 082, Y61]**

The aqueous processes involve dissolution of plutonium oxide followed by precipitation of peroxide, oxalate, or hydroxide salts. These salts are then calcined to form plutonium oxide. This category also includes oxide from the OY Leach process that will contain uranium as well as plutonium oxide. The efficiency of the process usually results in plutonium contents ranging from 75-88%. Individual items with lower plutonium content

are also included. Material may contain residual salts such as sulfates resulting from an incomplete calcination process. Impurities present may impact stabilization and LOI measurements.

**(3) Plutonium-uranium metal and hydride oxidation [U61, Y61]**

Plutonium oxide in this category contain uranium oxide and was prepared by a hydride process to separate plutonium and uranium. Oxide was formed by oxidation of the hydride at about 500°C. Uranium concentration in Y61 varies widely and ranges from 1- 100%. Stabilization of this material should not be a problem. However, accurate measurement of an LOI using gravimetric methods may be a problem and must be demonstrated.

**(4) Pyrochemical cell cleanout material [067, 086]**

This material was generated during the cleaning of pyrochemical cells. The material is expected to contain Na, K, and Mg chloride salts along with plutonium oxide. **This material has not been previously stabilized.** Problems should be expected during stabilization because of the volatile nature of the inorganic salts.

**(5) Dissolution residuals [062, 065, 289]**

This category consists of material that could not be dissolved when foundry and scrap oxide went through the oxide dissolution process. This material contains high fired plutonium oxide and other difficult to dissolve materials. Plutonium content varies widely. Stabilization of this material may be unpredictable because of the nature of impurities. Likewise, gravimetric LOI measurements may be inconsistent because of oxidation of material during heating.

**(6) Impure calcined oxide [ 159, 145, 146 , oxide from residue processing]**

This category is a catch-all for material with a questionable origin or composition. One type of material consists of screenings removed from metal-generated oxide stabilized above 500°C. Impurities either oxidize slowly or are resistant to oxidation. Plutonium content varies from 1-88%. The second type of material in this category are oxides that have failed 500°C LOI tests at the 1% level. This oxide is not pure and the source is unknown. A third type of oxide is expected to be produced by solution stabilization, pyrochemical salt processing, and other residue processing activities.



## **B. Oxide Sample Selection**

Category 1:	IDC-057	1 sample
	IDC-060	2 samples
	IDC-061	4 samples
	IDC-653	1 sample
Category 2:	IDC-061	1 sample
	IDC-080	1 sample
	IDC-081	1 sample
Category 3:	IDC-U61	1 sample
	IDC-Y61	3 samples
Category 4:	IDC-067	1 sample
	IDC-086	1 sample
Category 5:	IDC-062	1 sample
	IDC-289	2 samples
Category 6:	IDC-159	2 samples
	IDC-146	1 sample
	IDC-054	1 sample

**Table I. Rocky Flats Plutonium Oxide Legacy**

IDC	Description	Source	Items	Purity %Pu	Calcination °C
057	Oxide, Awaiting Spec	Metal oxidation, 15% unknown	164	49-87	450-1000
060	Oxide	Metal oxidation, aqueous processing, 15% unknown	352	14-88	450-500
061	Non-spec Oxide	metal oxidation, 45% other and unknown	680	0-88	450-1000
Y61	Pu/eU >10,000ppm U	Oxide from hydride separation process	544	0-55	500
U61	Pu/eU <10,000ppm U	Oxide from hydride separation process	45	75-85	500
062	High Purity Oxide Heel	Undissolved material from oxide dissolution	69	3-86	200-250
065	Oxide Heel, small stacker can	Undissolved material from oxide dissolution	20	6-86	200-250
067	Chlorinated Oxide	ER scrape out, may contain reactive metals	12	63-78	NONE
080	Peroxide Cake	Oxide from calcined peroxide	73	63-88	450
081	Impure Peroxide Cake	Oxide from calcined peroxide	36	29-83	450
082	Green Cake in small can (371)	Oxide from calcined peroxide	2	79	450
083	High Fired Oxide DOR	Probably metal oxidation	15	34-88	~800
086	Oxide ER Scrape Out	Same as 067, may contain reactive metals	22	53-76	NONE
145	Oxide - Failed 1 LOI	Unknown source	5	56-76	450
146	Oxide - Failed 2 LOI	Unknown source	22	53-73	450
159	Screenings from Oxide	Material left after screening during oxide stabilization	165	0-88	500
289	Low Purity Oxide Heel	Undissolved material from oxide dissolution	108	3-69	250-300
319	Oxide from Ta Crucibles	Oxide from crucible burnout	2	72 & 78	500
653	Oxide, Pu/Np	Oxide with Np impurity	86	78-88	500

Item ORF59-TS707001, the subject of this report, was listed in the Rocky Flats inventory as IDC 057; however the label on the can shows IDC 061. Both IDC's are part of category 1, described as oxide obtained from metal brushing, chip burning, hydride oxidation, and certain pyrochemical operations. It is believed to have been stabilized at temperatures above 500°C. The plutonium content of the category varies, but the impurities are expected to be metal oxides.

Los Alamos records indicate the following:

Total Plutonium: 996 g  
 Sample Power: 2.5 W  
 Date: September 11, 1997.

Pu-238:	0.01929 mass %	Pu-239:	93.74466 mass %
Pu-240:	5.98093 mass %	Pu-241:	0.23012 mass %
Pu-242:	0.02500 mass %	Am-241:	0.15143 mass %

## 2.0 EXPERIMENTAL METHODS

### 2.1 Materials

See introduction.

### 2.2 Procedures

The as-received can is shown in Fig. 1. The can was radiographed prior to opening, see Fig. 2. In Fig. 3, the material can be seen in the inner container. The sequence of sampling and testing is presented in Fig. 4. After samples were taken from the as-received material, (sample S), a portion of the powder was calcined at 600°C to produce sample S'. After samples were taken from the 600°C calcined powder, all of the remaining powder was calcined at 950°C to produce sample S''. The detailed step-by-step procedure for performing LOI measurements at Los Alamos is found in the Safe Operating Procedure (SOP) CST15-SOP-600-R00, "Materials Characterization of Radioactive Oxides". A summary of the procedure follows: The LOI crucibles used in the LANL procedure are cleaned using an ultrasonic cleaner, and after excess water is wiped off, they are dried in a muffle furnace at 200°C for about 1 h. Crucibles are stored in a desiccator under vacuum prior to use. The powder to be analyzed is introduced to the clean crucibles with lids. Platinum crucibles are used for analysis of samples with plutonium content  $\geq 80\%$ ; otherwise they are made of alumina. Weighed samples (5-10 g) are split and placed in two different crucibles, which are covered and placed in the furnace. The loss-on-ignition run is initiated, and when the heating cycle is completed, the furnace maintains a 200°C waiting period until the samples are removed. The samples are heated isothermally at 1000°C for 2 h. The crucibles are removed from the muffle furnace and placed in a desiccator under argon for 15 min until they cool. The cooled loaded crucibles are weighed again as quickly as possible, and an average weight loss is calculated.

X-Ray analyses are performed on a Scintag XRD2000 diffractometer. The most common types of analyses are chemical constituent identification on powder samples, lattice constant determinations on metallic samples and combinations of the two. The powder samples are laced with an add-mixture of fine silver powder to serve as a secondary calibration standard. The diffraction angles of the silver has been calibrated with respect to a primary standard of lanthanum boride ( $\text{LaB}_6$ ) obtained from the National Bureau of Standards (NBS).

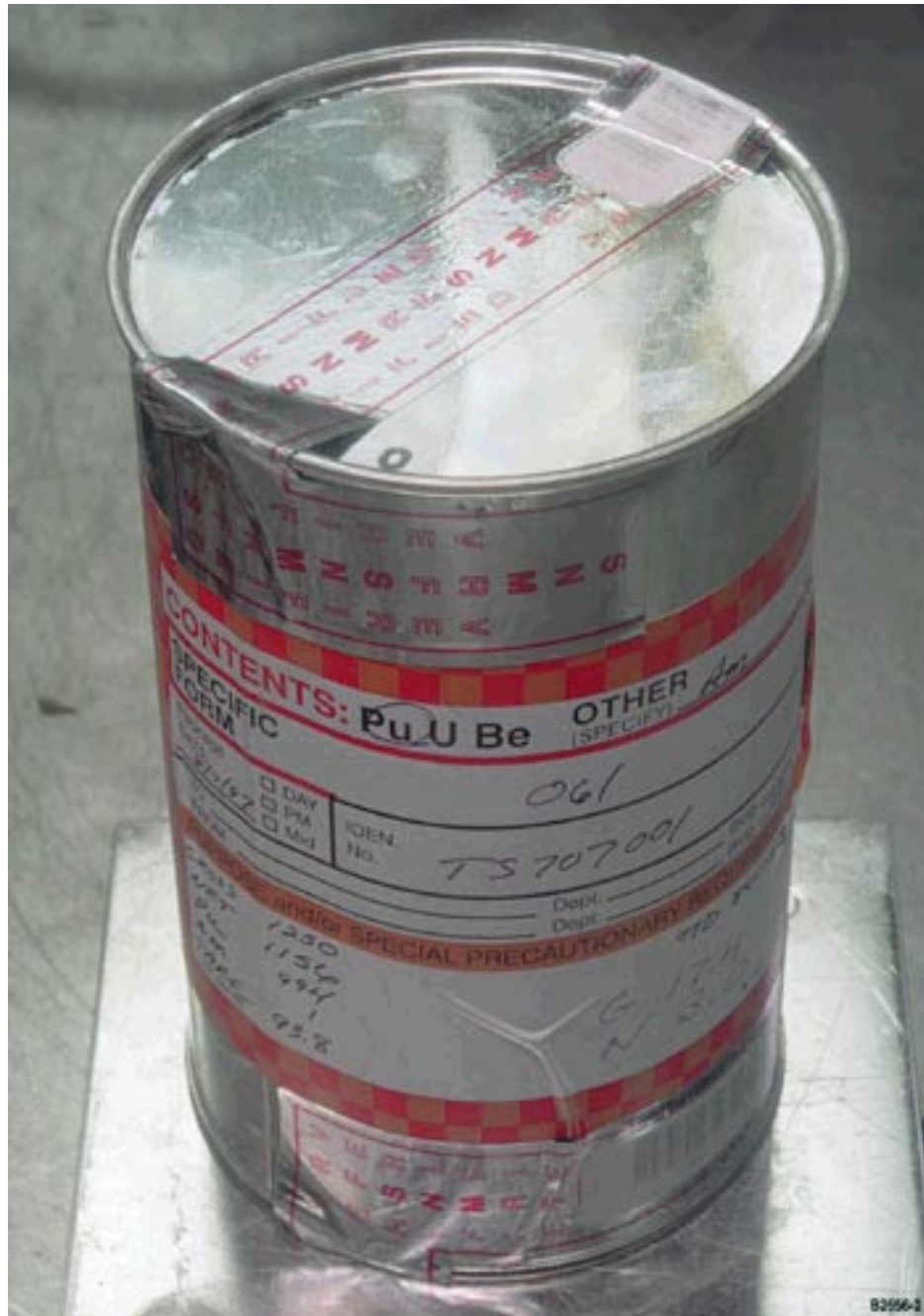


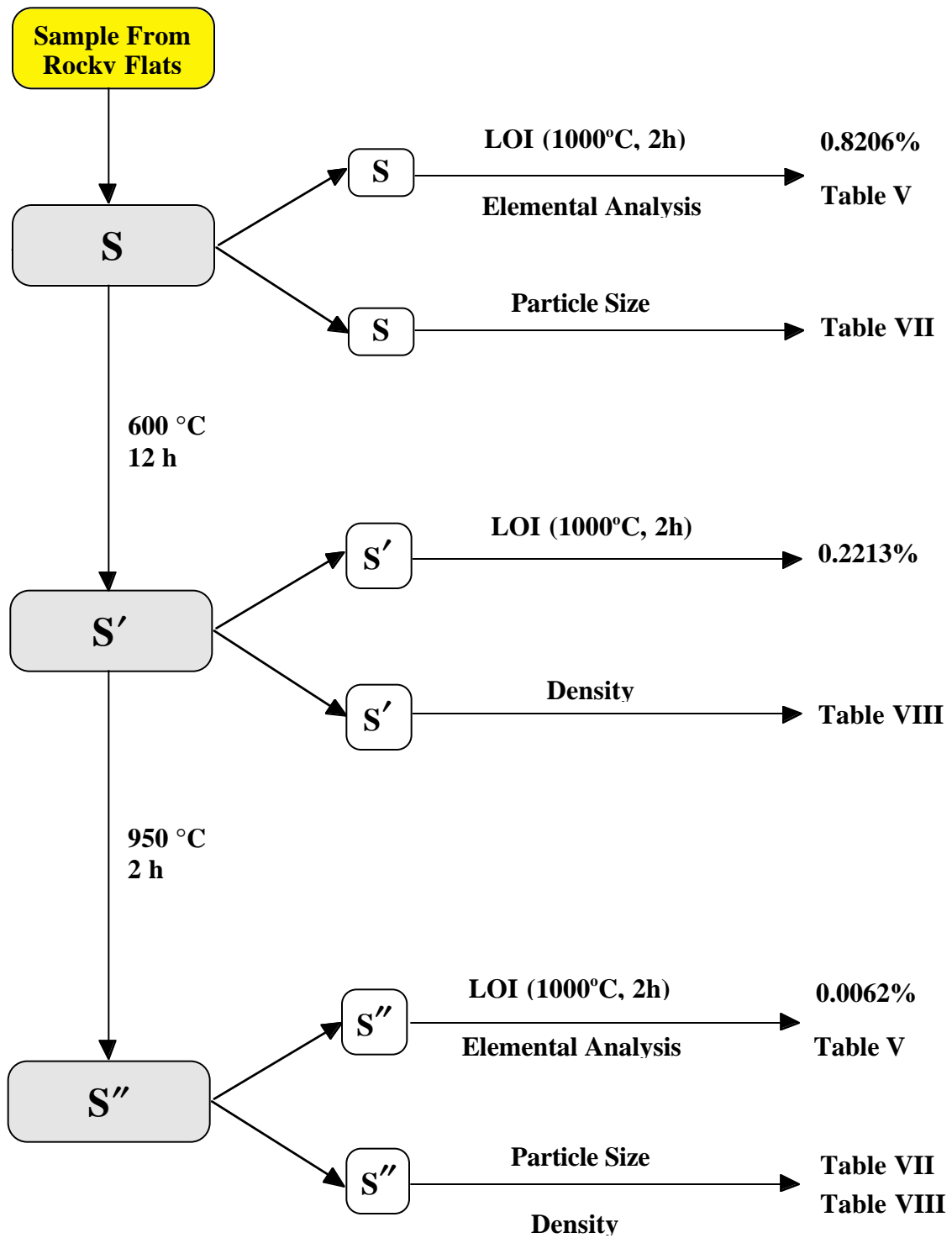
Fig. 1. Photograph of the canister at 0 degrees.



**Fig. 2. Radiograph of the canister at 0 degrees.**



**Fig. 3. The as-received can and inner can opened.**



**Fig. 4. Characterization of the impure plutonium oxide TS707001.**

Powder, metal, and ceramic samples are received from various customers over the trolley system in the PF-4 facility. For powder samples an eighth inch thick plastic washer is placed inside a metallography or met mount mold. The center hole in the plastic washer is one half inch in diameter. The powder is poured into the center hole of the plastic washer and the mold is then filled with epoxy. After the epoxy has hardened, the edges of the epoxy mount are ground to remove all rough or sharp edges. The metal samples are placed in the met mount mold and epoxy poured in to cover the metal piece. After the epoxy has hardened, the mount is ground and polished. After the met mount is prepared, the mount is thoroughly washed with alcohol or PF5070 to ensure that all loose contamination is removed from the surfaces. The met mount is then wrapped in commercial "glad wrap", carried to the diffractometer, and mounted in the sample holder. X-ray diffractometer then examines the surface for 1 to 8 hours. The metal samples are mechanically polished and subsequently electro-polished to remove mechanical work induced phase changes.

For chemical composition identification, the diffraction angles are commonly measured over ranges of two theta of  $5^\circ$  (or  $10^\circ$ ) to  $40^\circ$ . This range of data recording is usually sufficient for analysis of the major constituents in a mixture of crystalline phases. For lattice constant determinations, measurements over a greater range of angles,  $5^\circ$  to  $70^\circ$ , yields high accuracy data often precise to five significant figures. The diffractometer software performs two important data manipulations. It subtracts broad background x-ray scattering to produce a data file of sharp diffraction peaks. In addition, it analyses these peaks to yield a tabulation of the angles, widths and areas of the major peaks in any spectrum. These two data files are exported as text files from the laboratory by e-mail to an office location for subsequent analysis.

The text files are read and converted for spread-sheet manipulation using Excel. Much of the initial data manipulation is now performed by Excel programming macros. The background corrected raw data is converted to a graphical display to facilitate the sorting of critical lines from background noise. The peak tabulation file receives extensive manipulation depending on the type of sample analyzed. If a secondary standard has been used, the average deviation of the data is calculated and all the major lines calibrated to the standard. If the major compound(s) is cubic in crystal structure, a Nelson-Riley analysis is performed to determine the lattice constant and standard deviation of the data by a least squares analysis. The results are presented graphically for observational verification of the fitting reliability. There are about a half dozen macros available to perform these operations efficiently, some written specifically for the most common samples requiring analysis. The macros are limited in scope to permit operator intervention at critical decision points.



Qualitative chemical analysis is based on the x-ray data base maintained by the International Centre for Diffraction Data (Newtown Square, Pennsylvania) which contained in excess of 100,000 crystal phase entries. We are using the 1996 PDF-2 Database Sets 1 - 46 powder diffraction file. To simplify the task of analyzing the samples most often encountered in PF4, much smaller data files are maintained and continuously updated in both electronic and hardcopy format. These files are segregated into common plutonium compounds, a list of compounds commonly found and a list of compounds it is anticipated are likely to be found. These latter files are usually sufficient for most analyses but are easily supplemented by the PDF files for more unusual identifications.

Reporting includes a summary of the major phases or compounds identified supported by the graph of the diffraction data, the tabulation of the major lines including assignment and calculations performed to determine lattice constants and precision.

### 3.0 RESULTS AND DISCUSSION

It is interesting to notice that both powders the as-received and the calcined at 950°C show similar affinity for water.

**Table II. Water Uptake Adsorption Measurements for the Impure Oxide TS707001**

<b>Time &amp; Amount of Water</b>	<b>Powder S<sup>a</sup></b>	<b>Powder S''<sup>b</sup></b>
Time (days)	5	-
Mass (%)	0.002124	0.0001863

<sup>a</sup>Powder S is the as-received impure oxide.

<sup>b</sup>Powder S'' is obtained after impure oxide S is calcined first at 600°C for 12 h and then at 950°C for 2 h.

**Table III. Percent Mass Loss During Calcination of Sample TS707001**

<b>Sample</b>	<b>Calcination Temperature (°C)</b>	<b>Heating Time (h)</b>	<b>Mass Before Calcination (g)</b>	<b>Mass After Calcination (g)</b>	<b>Mass Loss (%)</b>
S'	600	12	1066.5	1057.3	0.863
S''	950	2	1106.5	1093.5	1.175

**Table IV. LOI Analyses Results of the Impure Oxide TS707001**

<b>LOI Conditions</b>	<b>Powder S<sup>a</sup> LOI (mass %)</b>	<b>Powder S<sup>b</sup> LOI (mass %)</b>	<b>Powder S<sup>c</sup> LOI (mass %)</b>
1000°C for 2 h	0.8206	0.2213	0.0062

<sup>a</sup>Powder S is the impure oxide as received.

<sup>b</sup>Powder S' is obtained after impure oxide S is calcined at 600°C for 12 h.

<sup>c</sup>Powder S'' is obtained after impure oxide S' is calcined at 950°C for 2 h.

For the as-received material it was found 86.26 mass % plutonium by calorimetry. Elemental analysis of the as-received and 950°C calcined powder was measured with ICP-MS.

**Table V. Elemental Analysis of As-Received Powder S<sup>a</sup> and Calcined Powder S<sup>b</sup>**

<b>Element</b>	<b>Powder S (µg/g)</b>	<b>Powder S'' (µg/g)</b>
Uranium	0.3679%	0.3707%
Lithium	<10	<10
Beryllium	<10	<10
Boron	<10	<10
Sodium	<100	<100
Magnesium	83	96
Aluminum	77	96
Silicon	565	550
Phosphorus	336	335
Potassium	<100	<100
Calcium	128	< 100
Scandium	<10*	<10*
Titanium	16	29
Vanadium	<10	<10
Chromium	163	154
Manganese	16	16
Iron	227	237
Cobalt	<10	<10
Nickel	170	103

Copper	116	32
Zinc	22	12
Gallium	697	599
Germanium	<10	<10
Arsenic	<10	<10
Selenium	<10	<10
Rubidium	<10	<10
Strontium	<10	<10
Yttrium	<10*	<10*
Zirconium	<10	<10
Niobium	<10	<10
Molybdenum	<10	<10
Ruthenium	<10	<10
Rhodium	<10	<10
Palladium	<10	<10
Silver	<10	<10
Cadmium	<10	<10
Indium	<10	<10
Tin	<10	23
Antimony	<10	<10
Tellurium	<10	<10
Cesium	<10	<10
Barium	<10	<10
Lanthanum	<10	<10
Cerium	<10	<10
Praeseodymium	<10*	<10*
Neodymium	<10	<10
Samarium	<10	<10
Europium	<10	<10
Gadolinium	<10	<10
Terbium	<10	<10
Dysprosium	<10	<10
Holmium	<10*	<10*
Erbium	<10	<10
Thulium	<10	<10
Ytterbium	<10	<10
Lutetium	<10	<10
Hafnium	<10	<10

Tantalum	65	108
Tungsten	36	41
Rhenium	<10	<10
Osmium	<10	<10
Iridium	<10	<10
Platinum	<10	<10
Gold	<10	<10
Mercury	<10	<10
Thallium	<10	<10
Lead	34	16
Bismuth	<10*	<10*
Thorium	<10	<10
Carbon	6900	80

<sup>a</sup>Powder S is the impure oxide as received.

<sup>b</sup>Powder S'' is obtained after impure oxide, S, is calcined at 600°C for 12 h and next at 950°C for 2 h.

\*Semi-quant estimate based on response curve.

**Table VI. Specific Surface Area Results for the Impure Oxide TS707001 (m<sup>2</sup>/g)**

As-received	Calcined at 600 °C & 950°C
6.09315	2.3464

The particle size distribution shifted to larger particles after calcination.

**Table VII. Particle Analysis Results of Precalcined Powder S<sup>a</sup> and Calcined Powder S''<sup>b</sup>**

Property	Powder S	Powder S''
Spherical	3.1	28.48
Equivalent Mean (µm)		
Diameter-by-Volume Mean (µm)	13.6	82.8

<sup>a</sup>Powder S is the impure oxide as received.

<sup>b</sup>Powder S'' is obtained after impure oxide, S, is calcined at 600°C for 12 h and next at 950°C for 2 h.

**Table VIII. Tap Density and Bulk Density of Calcined Powders S<sup>a</sup> and S<sup>b</sup>**

Property	Powder S'	Powder S''
Tap Density (g/cc)	2.25	2.98
Bulk Density (g/cc)	1.77	1.97

<sup>a</sup>Powder S' is obtained after impure oxide S is calcined at 600°C for 12 h.

<sup>b</sup>Powder S'' is obtained after impure oxide S' is calcined at 950°C for 2 h.

The X-ray results are given in Appendix 1 and Appendix 2. In Appendix 1 the X-ray crystallographic analysis results are given for the as-received material, S. For this material only one phase was identified i.e. plutonium dioxide. This sample was cast into epoxy with an added mixture of secondary x-ray standard, silver powder, before analysis. This sample has a single component of plutonium dioxide with no traces of other compounds and it is very similar to the calcined one although the diffraction line widths are somewhat larger. Graph of x-ray line spectra with background removed. The lines near 20.6 and 20.8° are from the aluminum sample holder. Tabulation of major diffraction line assignments, line centers, peak intensity, line width and line area as determined by curve fitting routine. Also included are the calibration calculations making use of the added silver powder. The silver serves as a secondary standard for diffraction angle calibration and has been itself calibrated from an NBS supplied primary standard. In Appendix 2 the X-ray crystallographic analysis results are given for the calcined material, S''. For this material also only one phase was identified i.e. plutonium dioxide.

#### 4.0 CONCLUSION

Loss-on-ignition values are low. The material satisfies DOE-STD-3013-96 criteria for plutonium content and LOI.

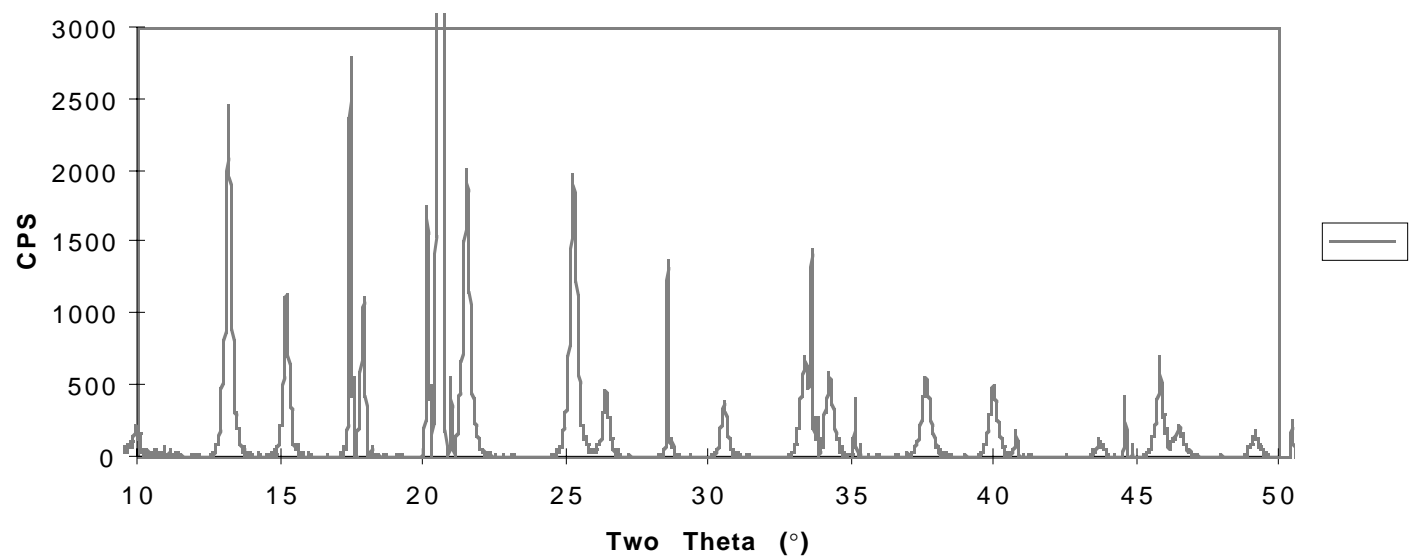
#### REFERENCES

1. "Criteria for Preparing and Packaging Plutonium Metals and Oxides for Long-Term Storage," Department of Energy document DOE-STD-3013-96 (September 1996).

**APPENDIX 1. X-ray results for the as-received powder TS707001.**

Scan Type: Normal	Ave Displacement	-.144
Start Angle: 5 deg.	Max Displacement	.056
Stop Angle: 70 deg.	Min Displacement	-.344
Num Points: 3251	Wavelength	0.7093
Step Size: 0.02 deg.		

**X-ray Crystallographic Spectrum**



Peaks:

ID	Position (Deg.)	Intensity (cps)	Std?	Disp (°)	Corrected		FWHM (L) (Deg.)	Exp. (L) (Deg.)	Area (cps-deg.)	
					Position (°)	d max A°				d min A°
	8.88	82			8.74	4.55	4.77	0.04	0	252.1
	9.09	75			8.95	4.45	4.65	0.02	0	346
	9.31	88			9.17	4.34	4.54	0.06	0	402.4
	9.50	62			9.35	4.26	4.45	0.06	0	573
	9.78	112			9.64	4.14	4.31	0.16	0	1370.9
	9.93	211			9.79	4.07	4.24	0.06	0	2587.7
	10.23	57			10.09	3.95	4.11	0.14	0	262.4
	10.92	66			10.78	3.71	3.85	0.1	0	302.5
PuO2	13.18	2458			13.04	3.08	3.17	0.06	0	30117.5
	14.78	54			14.64	2.75	2.82	0	0	83
PuO2	15.21	1114			15.07	2.67	2.74	0.06	0	13655.4
	17.3002		1	-.149						
Ag	17.45	2764		.000	17.30	2.33	2.38	0.02	0	12700
	17.91	1090		.000	17.76	2.27	2.32	0.12	0	10020.7
	18.20	73		.000	18.06	2.24	2.29	0.16	0	222.7
	19.9945		1	-.150						
Ag	20.14	1744		.000	20.00	2.02	2.06	0.1	0	8015.1
	20.60	12314		.000	20.46	1.98	2.02	0.06	0	132033
	20.95	556		.000	20.81	1.95	1.98	0.02	0	4258.6
PuO2	21.52	1984		.000	21.38	1.89	1.93	0.04	0	24317.8
PuO2	25.27	1925		.000	25.12	1.62	1.64	0.06	0	23590
PuO2	26.40	440		.000	26.25	1.55	1.57	0.02	0	5397.8
	28.422		1	-.144						

Ag	28.57	1346		.000	28.42	1.43	1.45	0.06	0	6183.6
PuO2	30.55	372		.000	30.41	1.34	1.36	0	0	4560.3
PuO2	33.38	701		.000	33.24	1.23	1.25	0.16	0	8587.6
	33.4664		1	-.139						
Ag	33.61	1431		.000	33.46	1.22	1.24	0.04	0	6576.1
	33.80	285		.000	33.66	1.22	1.23	0.02	0	1309.7
	34.23	561		.000	34.09	1.20	1.22	0.06	0	6868.6
	34.9964		1	-.144						
Ag	35.14	409		.000	35.00	1.17	1.19	0.02	0	1251.6
	35.34	95		.000	35.20	1.17	1.18	0.16	0	145.2
PuO2	37.62	543		.000	37.48	1.10	1.11	0.08	0	6651.1
PuO2	39.98	498		.000	39.84	1.04	1.05	0.06	0	6106.6
	40.6208		1	-.147						
Ag	40.77	180		.000	40.62	1.02	1.03	0.04	0	827.2
PuO2	43.70	115		.000	43.56	0.95	0.96	0.16	0	1231.3
	44.4551		1	-.145						
Ag	44.60	426		.000	44.46	0.93	0.94	0.16	0	1303.9
	44.86	98		.000	44.72	0.93	0.94	0.16	0	0
	45.6789		1	-.134						
Ag	45.81	716		.000	45.67	0.91	0.92	0.16	0	7673.2
	46.48	210		.000	46.33	0.90	0.91	0.12	0	2574
	49.15	163		.000	49.01	0.85	0.86	0.1	0	2000.7
	50.46	253		.000	50.32	0.83	0.84	0.16	0	1164.2
	50.76	55		.000	50.62	0.83	0.83	0.04	0	250.8
	51.05	104		.000	50.91	0.82	0.83	0.06	0	1276.5
	51.73	89		.000	51.59	0.81	0.82	0.06	0	1095.3
	53.75	237		.000	53.61	0.78	0.79	0	0	1087



56.01	170		.000	55.87	0.75	0.76	0.06	0	2088.3
56.51	72		.000	56.36	0.75	0.75	0.02	0	660.5
56.60	67		.000	56.46	0.75	0.75	0.16	0	408.5
58.93	169		.000	58.79	0.72	0.72	0.14	0	2067.9
60.67	127		.000	60.52	0.70	0.71	0.02	0	1550.2
61.93	161		.000	61.78	0.69	0.69	0.16	0	984.7
62.90	85		.000	62.76	0.68	0.68	0.16	0	261.7
		8	-0.143						
11.12	53						0.14	0	81.4
8.26	53						0.16	0	162.1
54.08	52						0.16	0	0
8.47	50						0.06	0	230.4
65.5	49						0.06	0	302.5
7.9	49						0.16	0	75
10.54	48						0.16	0	73.4
10.7	48						0.06	0	292.3
65.64	47						0	0	578.8
15.74	46						0.16	0	0
8.1006	44						0.12	0	203.5
8.6838	44						0.08	0	338.9
40.48	43						0.16	0	325.5
66.72	42						0.16	0	129.5
13.92	41						0.08	0	63.5
22.28	40						0.04	0	61.3
67.72	40						0.02	0	0
14.18	39						0.08	0	0
65.08	36						0.16	0	54.6

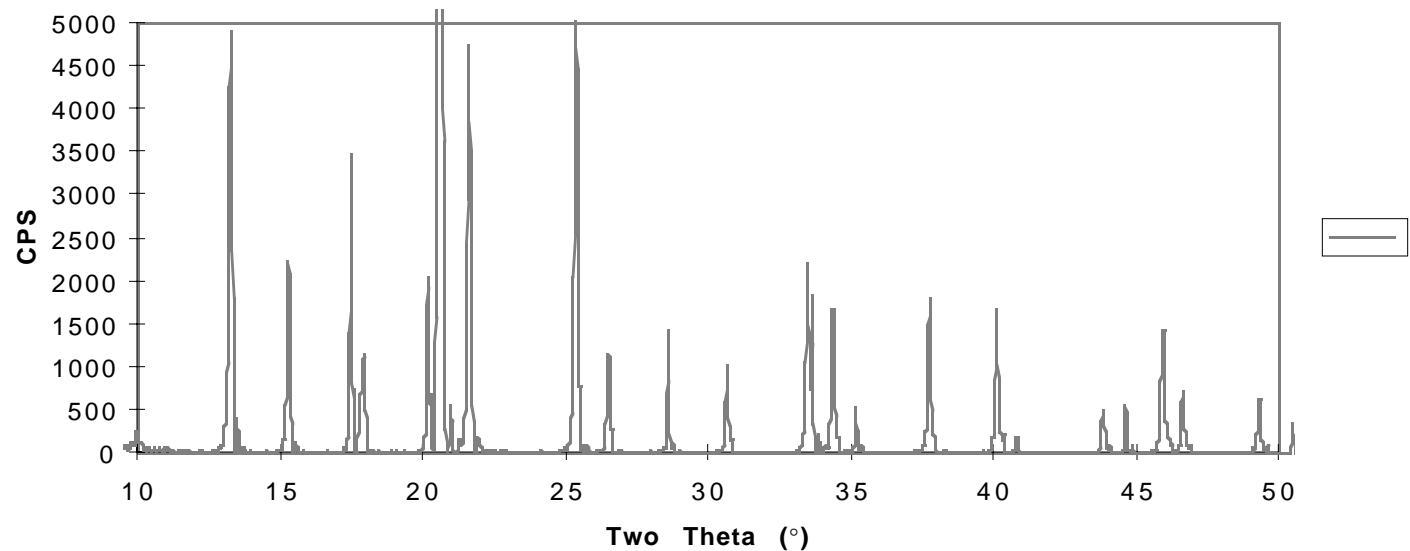
38.2	34
7.7	34

0.04	0	52.3
0	0	155.1

**APPENDIX 2. X-ray results for the calcined at 950°C powder TS707001.**

Scan Type: Normal	Ave Displacement	-.165
Start Angle: 5 deg.	Max Displacement	.035
Stop Angle: 70 deg.	Min Displacement	-.365
Num Points: 3251	Wavelength	0.7093
Step Size: 0.02 deg.		

## X-ray Crystallographic Spectrum



Peaks:

ID	Position (Deg.)	Intensity (cps)	Std?	Disp (°)	Corrected		d min A°	FWHM (L) (Deg.)	Exp. (L) (Deg.)	Area (cps-deg.)
					Position (°)	d max A°				
PuO <sub>2</sub>	9.74	109			9.57	4.16	4.34	0.1	0	998.9
	9.97	258			9.80	4.07	4.24	0.08	0	2762.3
	13.25	4908			13.09	3.07	3.16	0.1	0	37589.6
	13.46	391			13.29	3.02	3.11	0.02	0	1798.9

PuO2	15.29	2193			15.12	2.66	2.73	0.14	0	13435.6
	15.53	125			15.36	2.62	2.69	0.02	0	576.6
	17.3002		1	-.174						
Ag	17.47	3480			17.31	2.33	2.38	0.12	0	15990.9
Al	17.91	1133			17.74	2.27	2.33	0.14	0	12148.8
	19.9945		1	-.176						
Ag	20.17	2076			20.01	2.02	2.06	0.06	0	9539.6
Al	20.61	12863			20.44	1.98	2.02	0.08	0	137922
Al	20.96	571			20.79	1.95	1.98	0.06	0	4373.5
	21.32	157			21.15	1.91	1.95	0.14	0	1927.4
PuO2	21.60	4714			21.44	1.89	1.92	0.1	0	28881
	21.90	181			21.73	1.86	1.90	0.12	0	1108
PuO2	25.35	5030			25.19	1.61	1.64	0.12	0	38526.2
	25.72	106			25.55	1.59	1.62	0.06	0	813.8
PuO2	26.49	1126			26.32	1.55	1.57	0.1	0	8627.1
	28.422		1	-.166						
Ag	28.59	1432			28.42	1.43	1.45	0.06	0	6582.5
PuO2	30.65	1011			30.49	1.34	1.36	0.16	0	4646.1
	33.4664		1	-.164						
PuO2	33.47	2180			33.30	1.23	1.24	0.1	0	16700
Ag	33.63	1862			33.47	1.22	1.24	0.08	0	8554.5
	33.82	227			33.65	1.22	1.23	0.06	0	347.8
	33.94	114			33.77	1.21	1.23	0.1	0	174.9
PuO2	34.35	1647			34.19	1.20	1.21	0.06	0	12610.5
	34.9964		1	-.164						
Ag	35.16	535			34.99	1.17	1.19	0.06	0	1638.4
PuO2	37.74	1777			37.57	1.10	1.11	0.08	0	10889.7

PuO2	40.11	1633			39.94	1.03	1.04	0.06	0	12503.5
	40.6208		1	-.174						
Ag	40.79	182			40.63	1.02	1.03	0.02	0	835.1
PuO2	43.82	489			43.66	0.95	0.96	0.06	0	2994.7
	44.4551		1	-.165						
Ag	44.62	573			44.45	0.93	0.94	0.1	0	1756.5
PuO2	45.92	1436			45.76	0.91	0.92	0.14	0	13200.2
PuO2	46.62	690			46.45	0.90	0.90	0.08	0	5284.6
	46.84	105			46.67	0.89	0.90	0.06	0	1121.4
PuO2	49.29	631			49.13	0.85	0.86	0.08	0	4833.6
	49.58	100			49.41	0.85	0.85	0.1	0	153.8
	50.3202		1	-.160						
Ag	50.48	332			50.31	0.83	0.84	0.1	0	1017.4
	51.23	401			51.06	0.82	0.83	0.02	0	3068.4
	51.86	324			51.70	0.81	0.82	0.04	0	2484.8
	53.612		1	-.161						
Ag	53.77	271			53.61	0.78	0.79	0.02	0	1245.2
	54.34	116			54.17	0.78	0.78	0.04	0	532.1
	56.15	595			55.99	0.75	0.76	0.1	0	4556.8
	56.75	215			56.59	0.75	0.75	0.04	0	1647.2
	58.8064									
	59.09	529			58.93	0.72	0.72	0.1	0	4054.4
	60.81	575			60.64	0.70	0.70	0.1	0	5281.5
	61.7908		1	-.155						
Ag	61.95	173			61.78	0.69	0.69	0.1	0	1058.8
	65.27	134			65.10	0.66	0.66	0.1	0	822.7
	65.81	223			65.64	0.65	0.66	0.08	0	2047.5

	67.96	196			67.80	0.63	0.64	0.04	0	1497.5
	69.3864		1	-.163						
Ag	69.55	232			69.38	0.62	0.62	0.1	0	1776
			11	-0.1655						
	44.88	100						0.08	0	153.5
	9.575	100						0.1	0	767.4
	35.36	99						0.1	0	151.3
	62.92	88						0.12	0	270
	44.06	88						0.08	0	269.3
	8.88	87						0.04	0	533.6
	13.66	75						0.08	0	459.5
	56.4806	74						0.12	0	453.7
	9.0863	72						0.04	0	660.5
	9.3094	69						0.1	0	531.8
	66.74	66						0.1	0	202.3





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Los Alamos, New Mexico 87545