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**A Tested Method to Minimize
Plutonium Assay Discrepancies Between
Laboratories**

**Raymond J. Seiler, Ronald L. Goss,
W. William Rodenburg, and Donald R. Rogers**

January 29, 1982

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Abstract

Plutonium assay differences are frequently observed between laboratories exchanging plutonium dioxide powders. These differences are commonly the result of chemical changes and/or nonhomogeneities in sampled materials. The irregularities are often caused by moisture absorption during sampling, packaging, shipment, and storage of the materials. A method is proposed which eliminates the effects of chemical change in samples, particularly moisture absorption, and minimizes sampling error. A nondestructive thermal watts/gram test on every preweighed sample and total dissolution of these samples for chemical assay are the primary features which make this method effective. Because this method minimizes the error related to exchange material, it is possible to design an interlaboratory exchange program which demonstrates the assay capabilities of the participants. Exchange participants can be confident that the samples they receive are representative of the source material within narrow limits of error.

In an experiment performed to demonstrate the effectiveness of this method, three PuO₂ batches of varying isotopic composition were synthesized at Mound to be used in the exchange tests. Powder sample aliquots from each batch were weighed directly into their vials under controlled atmospheric conditions. Calorimetric heat measurements were made on each vial to test homogeneity and verify sample weight. Six vials of each batch were chemically assayed at Mound and six at NBL (New Brunswick Laboratory). Both laboratories chose controlled-potential coulometry as the chemical assay technique because of its demonstrated precision and accuracy. Total dissolution of preweighed exchange samples eliminated the need for laborious and usually futile heating to return the material to its original condition. The mean chemical assay values obtained by Mound and NBL agree to within 0.01% for each of the compositions tested. Testing of both chemical assay and calorimetric data revealed no sampling error throughout the experiment.

Introduction

The reasons for plutonium assay discrepancies involving the exchange of PuO₂ powders for laboratory evaluation programs are often moisture absorption and/or chemical nonhomogeneity. When moisture absorption occurs during packaging, shipment, or storage of the sample or the bulk material, the sample is no longer representative of the bulk material. Sampling and subsampling errors caused by nonhomogeneities in plutonium concentration are frequently the result of water uptake, which is seldom uniform. Once water is absorbed, samples of most PuO₂ batches cannot be returned to their original plutonium concentration by heating at any temperature. Moisture absorption causes assay values of laboratory evaluation

samples to differ substantially from those analyzed to set the reference value. Frequently, the same phenomenon is responsible for errors in chemical assay between shipper and receiver laboratories. At the same time, it is possible that neither laboratory has analyzed a sample that is representative of the bulk shipment.

A method was developed to test for, and minimize, plutonium assay sampling error before the exchange of PuO₂ samples. This method was successfully demonstrated by an exchange of samples from three batches of reactor grade plutonium dioxide powder between Mound and NBL. To minimize sampling error, each batch was mechanically mixed in an inert atmosphere. To eliminate the effects of chemical change, specifically sorbed water samples for

this exchange were weighed into tared glass vials and sealed before removal from the inert box-line. Once the sample is weighed, chemical changes in the powder cannot affect the total plutonium content of the vial. Each sample vial was made up so that the thermal output would be approximately 10 mW. The weight of the samples ranged from 1.2 to 4.0 g depending on the isotopic composition. Each vial was tested calorimetrically for chemical nonhomogeneity and weight verification. No sample-to-sample nonhomogeneity could be detected in any of the three batches by testing the thermal watts/gram calorimetry data. Six sample vials of each composition were chemically assayed at Mound and six, at NBL.

Dissolution of the entire content of each vial prevented the possibility of subsampling error. Quantitative transfer of the powder from the vials was quick and simple. After the samples were poured in, both the vials and their Teflon gaskets were placed in the dissolution vessels. The mean chemical assay values obtained by Mound and NBL agree to within 0.01% for each of the three batches tested. The relative standard deviation was low (<0.07%) in all cases. The following sections provide details of: a) isotopic batch make-up, (b) sample preparation, c) nonhomogeneity and sampling error tests, and d) chemical assay procedures and results.

Exchange details

Three batches of PuO_2 , having different isotopic compositions, were prepared to simulate the abundance of low, medium, and high burn-up commercial reactor fuel. Plutonium chemical assay measurements were performed by analytical personnel at Mound and NBL. The overall experimental flow chart including material preparation steps for each batch is shown in Figure 1.

Preparation of isotopic blend batches

Calculated quantities of plutonium nitrate solutions of known concentration and isotopic composition were combined to yield a solution of desired isotopic composition. The resulting isotopic composition was checked using mass spectrometry and alpha counting before further processing. The isotopic compositions of these batches are listed in Table 1.

Table 1 - ISOTOPIC COMPOSITION OF PuO_2 BATCHES

Isotope	wt %		
	Batch 1	Batch 2	Batch 3
Pu-238	0.06	0.25	1.1
Pu-239	86.6	73.2	70.3
Pu-240	11.7	22.6	24.1
Pu-241	1.5	3.0	3.2
Pu-242	0.2	1.0	1.2
Am-241	0.01	0.06	0.02
Thermal Power (mW/g Pu)	2.88	4.66	9.70

Sub-batches of solution containing 20-30 g of plutonium nitrate were converted to plutonium dioxide by precipitating the plutonium as oxalate and igniting it at 800°C for 2 hr. Four or five of these sub-batches were combined and mixed in an inert atmosphere for 2 hr, with a twin-shell blender. Analytical samples were taken after blending to check isotopic composition and impurity levels.

Preparation of exchange samples

The paper gaskets from the Bakelite caps of 1-dram glass vials were replaced with Teflon gaskets. Next the caps, gaskets, and vials were cleaned and carefully dried in a vacuum oven. At least 14 samples were divided from each batch of PuO_2 using a cone-and-quartering method (ASTM E300-30.5.1), in an inert atmosphere boxline. The powder aliquots were weighed directly into the tared glass vials on a Mettler H-18 balance. The samples were made up to have a thermal output of approximately 10 mW each. The approximate sample weights were 4.0, 2.5 and 1.25 g for Batches 1, 2, and 3, respectively. The vials were then capped and taped for Batches 2 and 3. Two samples from Batch 1 delivered to NBL were compromised when their caps loosened during shipment. Taping the caps for the next two shipments prevented any further problems.

FIGURE 1 - Individual batch preparation and sampling at Mound.

Calorimetric watts/gram test

After the exchange sample vials of each batch were withdrawn from the boxline, the thermal output of each sample was measured. The relative standard deviation (RSD) of the thermal measurements was estimated to be 0.03% based on eight measurements of Mound heat standards over a 6-mo period. The relative standard deviation of the weight measurements was estimated to be 0.009%. The watts/gram (W/M) values from each batch of samples were compiled and tested for homogeneity and sample weighing error. The following model was used:

$$S^2_{W/M} = (W/M)^2 \left(\frac{S_W}{W} \right)^2 + (W/M)^2 \left(\frac{S_M}{M} \right)^2 + S^2_{\text{sam}} + S^2_I \quad (1)$$

where:

$S^2_{W/M}$ = the variance in the watts/gram determination of the samples.

$(W/M)^2 \left(\frac{S_W}{W} \right)^2$ = the variance due to the wattage measurement.

$(W/M)^2 \left(\frac{S_M}{M} \right)^2$ = the variance due to the measurement of Mass, M.

S^2_{sam} = the variance potentially due to sampling which could be the result of chemical or isotopic inhomogeneity within the isotopic blend batch.

S^2_I = the variance due to chemical and other interferences to the power measurement.

An F-ratio was used to test for the significance of the variance due to sampling or interferences to the power measurement, S^2_{sam} and S^2_I , respectively:

$$F = \left(\frac{W}{M} \right)^2 \frac{S^2_{W/M}}{\left[\frac{S_W^2}{W^2} + \frac{S_M^2}{M^2} \right]} \quad (2)$$

The results of the three tests for sample homogeneity are summarized in Table 2.

The computed F ratios are well below the critical value, $F_{0.95}$. Therefore, the data support the hypothesis that there are no significant sampling errors. The data obtained from the thermal power measurements of individual samples are compiled in Tables 1, 2, and 3 of the Appendix.

Mound plutonium assay

Dissolution

The contents of the analytical sample vials were emptied into a 200-ml Teflon dissolution vessel (Figure 2). Next, the glass vial and the Teflon gasket from the cap were also placed in this vessel. After the addition of 80 ml of 16 M HNO_3 - 0.1 M HF solution, the vessels were placed in an oven controlled at a temperature of 105°C overnight (approximately 18 hr). The solution was then transferred to a tared 4-ounce polyethylene bottle with dilute nitric acid.

Sample preparation and titration

From the 4-ounce polyethylene bottle, two weighed aliquots were taken for coulometry; aliquots were also taken for spectrophotometric iron determination, impurity analysis, and isotopic analysis. The flow of chemical samples within the Mound analysis scheme is shown in Figure 3. After dilution of the coulometry aliquots, three subaliquots (each containing about 8 mg of plutonium) were weighed into glass titration cells. These subaliquots were fumed to dryness twice with H_2SO_4 and titrated according to a Mound standard analytical procedure [1]. This controlled potential coulometric procedure was adapted from one described by Rodden [2]. The procedure is performed in a medium of 0.5 M H_2SO_4 with a platinum working electrode made from 45 mesh gauze. Along with each day's samples, at least five aliquots of a standard solution made from NBS 949e plutonium metal were titrated to calibrate the coulometry system. Both samples and standards were corrected for atomic mass, plutonium decay, and iron concentration. The three titration values were averaged to yield one value for each solution aliquot taken. The plutonium assay results derived from the titration of these solution aliquots are listed in full in Tables 4, 5, and 6 of the Appendix.

(DIMENSIONS IN INCHES)

FIGURE 2 - Solid teflon dissolution vessel, suitable for the dissolution of up to 8 g of PuO_2 or $(\text{Pu}, \text{U})\text{O}_2$. The maximum recommended solution volume is 100 ml.

Table 2 - SAMPLE HOMOGENEITY

Batch	$\frac{S^2_w}{M}$	$\left(\frac{W}{M}\right)^2$	$\left(\frac{S_w}{W}\right)^2$	$\left(\frac{S_M}{M}\right)^2$	F	ν_1, ν_2	$F_{0.95}$
1	1.00×10^{-12}	6.42×10^{-6}	9.0×10^{-8}	0.8×10^{-8}	1.58	9, 7	3.68
2	4.41×10^{-12}	1.68×10^{-5}	9.0×10^{-8}	0.8×10^{-8}	2.68	10, 7	3.64
3	5.29×10^{-12}	7.28×10^{-5}	9.0×10^{-8}	0.8×10^{-8}	0.74	11, 7	3.61

NBL plutonium assay

Dissolution and titration

The contents of each sample vial were emptied into a 250-ml Teflon beaker. The vial was then rinsed with 8 M HNO₃ and placed with the gasket in another 250-ml Teflon beaker with 8 M HNO₃. Dissolution of the powder portion was accomplished by adding approximately 100 ml of 8 M HNO₃-HF solution and heating below the boiling point. After the sample was completely dissolved, the two solutions were combined, taken to low volume, and transferred into a 500-ml tared bottle. Two solution aliquots were then weighed from a polyethylene squeeze bottle and fumed twice to dryness with H₂SO₄. Each aliquot was then purified using an anion-exchange separation procedure [3]. The eluate was collected and fumed twice to dryness with H₂SO₄ in the glass titration cell. The aliquots were then titrated by the controlled-potential coulometric method developed at NBL [4,5]. This titration is performed at the surface of a gold electrode in 0.9 M HNO₃ acid medium. Plutonium assay values are based upon electrical calibration of the M-T Model 3 coulometric system [6]. Chemical standards made from NBS-944 plutonium sulfate tetrahydrate were analyzed to monitor instrument performance. Figure 4 outlines the flow of samples at NBL. The plutonium assay results derived from the titration of these aliquots are compiled in Tables 4, 5, and 6 of the Appendix. The comparison of Mound vs. NBL chemical assay results is shown in Table 3.

Chemical data test

An F ratio was also used to test the chemical assay data for the significant sampling, transfer, and dissolution error. The following model was used:

$$S^2_B = S^2_{ws} + S^2_{samp} + S^2_D + S^2_T \quad (3)$$

where: S^2_B = variance of plutonium values between samples assayed.

S^2_{ws} = variance of plutonium values within samples assayed.

S^2_{samp} = variance potentially due to sampling which could be the result of chemical or isotopic nonhomogeneity within the blend batch.

S^2_D = variance potentially due to incomplete dissolution or loss of sample material during dissolution.

S^2_T = variance potentially due to loss of sample material during transfer in any chemical assay step.

FIGURE 3 - Flow of individual samples at Mound.

FIGURE 4 - Flow of individual samples at NBL.

Table 3 - COMPARISON OF Pu CHEMICAL ASSAY VALUES FROM MOUND AND NBL

Batch	Site	Mean (wt %) ^a	RSD (%)	SD of Mean (%)	N(R) ^b
1	Mound	88.056	± 0.058	± 0.015	6(2)
1	NBL	88.060	± 0.030	± 0.009	4(2)
	Difference (wt %)	+ 0.004			
2	Mound	87.965	± 0.049	± 0.012	6(2)
2	NBL	87.960	± 0.058	± 0.015	6(2)
	Difference (wt %)	- 0.005			
3	Mound	87.976	± 0.069	± 0.018	6(2)
3	NBL	87.978	± 0.069	± 0.018	6(2)
	Difference (wt %)	+ 0.002			

^aWithin batch Pu assay values, decay corrected to a common date.

^bN(R) represents (the number of sample vials analyzed) x (the number of replicates per vial).

An F ratio was used to test for significance of the variance due to sampling, dissolution, and transfer, S^2_{samp} , S^2_D , and S^2_T , respectively:

$$F = \frac{S^2_B}{S^2_{ws}} \quad (4)$$

The F ratios calculated from both Mound and NBL data for all three batches are shown in Table 4.

The computed F ratios are well below the critical value, $F_{0.95}$. Therefore, the chemical assay data support the hypothesis that there is no significant amount of material lost in the dissolution and transfer steps of the assay procedure and that the samples are homogeneous with respect to plutonium content.

Conclusions

The calorimetric and chemical assay data reported for the three batches of PuO_2 exchanged demonstrate a method that would eliminate interlaboratory exchange assay discrepancies caused by the reactive nature of

this material. Several design elements contributed to the success of this exchange. First, preweighed samples avoid the effects of chemical change and permit the exchange of blind samples. Second, total dissolution avoids subsampling errors without laborious calcining, heating or mathematical calculations. Third, a nondestructive watts/gram test on every sample validates sample weight measurements and tests for sample-to-sample chemical and isotopic variability.

The interlaboratory chemical assay of all three batches agrees within 0.01% relative. Statistical testing of both calorimetric and chemical data revealed no sampling error throughout the exchange.

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Major efforts for chemical analyses of these materials were provided by: B. P. Freeman, M. K. Holland, S. A. Malone, C. E. Pietri and J. R. Weiss of New Brunswick Laboratory and V. L. George, J. R. Tackett, K. M. Muldoon and W. E. Sheehan of Mound Facility.

Table 4 - CHEMICAL ASSAY TEST FOR SAMPLE HOMOGENEITY PLUS TRANSFER AND DISSOLUTION ERROR

<u>Batch</u>	<u>Site</u>	<u>S^2_B</u>	<u>S^2_{ws}</u>	<u>F</u>	<u>ν_1, ν_2</u>	<u>$F_{0.95}$</u>
1	Mound	0.00230	0.00096	2.40	5, 6	4.39
1	NBL	0.00044	0.00053	0.83	3, 4	6.59
2	Mound	0.00168	0.00068	2.47	5, 6	4.39
2	NBL	0.00160	0.00221	0.72	5, 6	4.39
3	Mound	0.00314	0.00152	2.07	5, 6	4.39
3	NBL	0.00292	0.00185	1.58	5, 6	4.39

References

1. PD-80026, **Mound Facility Standard Analytical Procedures**, Operation 102, "Plutonium Assay by Controlled Potential Coulometry," Mound Facility, Miamisburg, Ohio, 36 pp.
2. C. J. Rodden (ed.), **Selected Measurement Methods for Plutonium and Uranium in the Nuclear Fuel Cycle**, U. S. Atomic Energy Commission Report TID-7029 (2nd ed.), pp. 314-326 (1972).
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4. M. K. Holland, J. R. Weiss and C. E. Pietri, *Anal. Chem.*, **50**, 239 (1978).
5. **Controlled-Potential Coulometric (CPC) Determination of Plutonium with Controlled-Potential Adjustment Technique**, NBL-PC-PA-6.
6. **M-T Model 3 Instrument Calibration Procedure**, NBL-PC-CP-1.

APPENDIX: Decay corrected data

Table 1 - MOUND CALORIMETRY DATA
FOR PLUTONIUM BATCH 1
(Common Decay Date 9/01/76)

Sample* ID	Thermal Power (W/g)	
	Sample Reading	Vial Av
1002	0.0025348 0.0025347	0.0025348
1006	0.0025345 0.0025352	0.0025348
1010	0.0025348 0.0025348	0.0025348
1014	0.0025356 0.0025358	0.0025357
1016	0.0025354 0.0025349	0.0025352
1017	0.0025344 0.0025347	0.0025346
1004	0.0025358 0.0025350	0.0025354
1007	0.0025342 0.0025337	0.0025340
1008	0.0025346 0.0025346	0.0025346
1011	0.0025326 0.0025319	0.0025322
<u>Mean</u>	0.0025346	
<u>Standard Deviation</u>		
Between Vials	± 0.0000010	0.038%, relative
Within Vial	± 0.0000003	0.012%, relative

*Only samples which were assayed
chemically are included in these data.

**Table 2 - MOUND CALORIMETRY DATA
FOR PLUTONIUM BATCH 2
(Common Decay Date 6/01/77)**

<u>Sample* ID</u>	<u>Thermal Power (W/g)</u>	
	<u>Sample Reading</u>	<u>Vial Av</u>
2015	0.0040962 0.0040969	0.0040966
2020	0.0000000 0.0000000	
2017	0.0041022 0.0041026	0.0041024
2010	0.0040969 0.0040969	0.0040969
2019	0.0040967 0.0040981	0.0040974
2007	0.0040960 0.0040984	0.0040972
2009	0.0041004 0.0040988	0.0040996
2001	0.0040972 0.0040960	0.0040966
2013	0.0040955 0.0040947	0.0040951
2005	0.0040980 0.0040971	0.0040976
2018	0.0040982 0.0040966	0.0040974
2012	0.0040959 0.0040935	0.0040947
<u>Mean</u>	0.0040974	
<u>Standard Deviation</u>		
Between Vials	0.0000021	0.051%, relative
Within Vial	0.0000010	0.025%, relative

*Only samples which were assayed chemically are included in these data.

**Table 3 - MOUND CALORIMETRY DATA
FOR PLUTONIUM BATCH 3
(Common Decay Date 9/01/77)**

<u>Sample* ID</u>	<u>Thermal Power (W/g)</u>	
	<u>Sample Reading</u>	<u>Vial Av</u>
3109	0.0085314 0.0085314	0.0085314
3105	0.0085339 0.0085339	0.0085339
3108	0.0085297 0.0085300	0.0085298
3106	0.0085261 0.0085261	0.0085261
3103	0.0085286 0.0085286	0.0085286
3117	0.0085299 0.0085299	0.0085299
3101	0.0085327 0.0085327	0.0085327
3111	0.0085295 0.0085295	0.0085295
3102	0.0085314 0.0085314	0.0085314
3115	0.0085274 0.0085274	0.0085274
3113	0.0085276 0.0085276	0.0085276
3107	0.0085303 0.0085303	0.0085303
<u>Mean</u>	0.0085299	
<u>Standard Deviation</u>		
Between Vials	0.0000023	0.027%, relative
Within Vial	0.0000000	0.00%, relative

*Only samples which were assayed chemically are included in these data.

Table 4 - CHEMICAL ASSAY DATA FOR PLUTONIUM BATCH 1
(Common Decay Date 9/01/76)

Mound				NBL			
Sample ID	Aliquot ID	wt % Pu		Sample ID	Aliquot ID	wt % Pu	
		Aliquot Value	Vial Av			Aliquot Value	Vial Av
1002	X0	88.100	88.101	1004	X0	88.081	88.055
	X1	88.102			X1	88.029	
1006	X0	88.128	88.112	1007	X0	88.082	88.080
	X1	88.096			X0	88.079	
1010	X0	88.125	88.082	1008	X0	88.085	88.072
	X1	88.038			X1	88.058	
1014	X0	88.014	88.020	1011	X0	88.020	88.034
	X1	88.026			X1	88.047	
1016	X0	88.006	88.002				
	X1	87.999					
1017	X0	88.043	88.016				
	X1	87.990					
<u>Grand Mean</u>			88.056	(Difference = +0.004)			88.060
<u>Standard Deviation</u>							
of Mean			± 0.015				± 0.009
between Vials			± 0.048				± 0.021
between Aliquots			± 0.031				± 0.023

Table 5 - CHEMICAL ASSAY DATA FOR PLUTONIUM BATCH 2
(Common Decay Date 9/01/76)

Mound				NBL			
Sample ID	Aliquot ID	wt % Pu		Sample ID	Aliquot ID	wt % Pu	
		Aliquot Value	Vial Av			Aliquot Value	Vial Av
2015	X0	87.966	87.934	2009	X0	87.896	87.898
	X1	87.902			X1	87.900	
2020	X0	87.946	87.950	2001	X0	88.015	87.995
	X1	87.954			X1	87.975	
2017	X0	88.019	88.038	2013	X0	88.010	87.956
	X1	88.058			X1	87.902	
2010	X0	87.969	87.981	2005	X0	87.967	87.937
	X1	87.993			X1	87.907	
2019	X0	87.947	87.927	2018	X0	88.006	87.966
	X1	87.907			X1	87.926	
2007	X0	87.968	87.960	2012	X0	88.035	88.007
	X1	87.952			X1	87.979	
<u>Grand Mean</u>			87.965	(Difference = -0.005)			87.960
<u>Standard Deviation</u>							
of Mean			± 0.012				± 0.015
between Vials			± 0.041				± 0.040
between Aliquots			± 0.026				± 0.047

Table 6 - CHEMICAL ASSAY DATA FOR PLUTONIUM BATCH 3
(Common Decay Date 9/01/77)

Mound				NBL			
Sample ID	Aliquot ID	wt % Pu		Sample ID	Aliquot ID	wt % Pu	
		Aliquot Value	Vial Av			Aliquot Value	Vial Av
3109	X0	87.982	88.014	3107	X0	87.957	88.014
	X1	88.046			X1	88.070	
3105	X0	87.991	87.948	3101	X0	87.979	88.006
	X1	87.904			X1	88.034	
3108	X0	88.039	88.027	3111	X0	88.037	88.033
	X1	88.015			X1	88.029	
3106	X0	87.947	87.939	3102	X0	87.860	87.892
	X1	87.931			X1	87.924	
3103	X0	87.885	87.896	3115	X0	87.968	87.988
	X1	87.908			X1	88.008	
3117	X0	87.996	88.032	3113	X0	87.949	87.933
	X1	88.067			X1	87.917	
<u>Grand Mean</u>			87.976	(Difference = +0.002)			87.978
<u>Standard Deviation</u>							
of Mean			±0.018				±0.018
between Vials			±0.056				±0.054
between Aliquots			±0.039				±0.043

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