NBL Program Office

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material C122

Plutonium Oxide (PuO_2) Plutonium Assay and Isotopic Standard in Powder Form

Plutonium	87.790 ± 0.039 Wt. %*
	(877.90 ± 0.39 $g \cdot kg^{-1}$)
Plutonium-238	0.0521 ± 0.0011 At. %*
Plutonium-239	87.305 ± 0.004 At. %*
Plutonium-240	11.539 ± 0.004 At. %*
Plutonium-241	0.9248 ± 0.0011 At. %*
Plutonium-242	0.1790 ± 0.0013 At. %*
Relative Atomic Weight	239.191*

*As of January 1, 1985. Refer to Table I for Quarterly Decay-Adjusted Values.

This Certified Reference Material (CRM) is an assay and isotopic standard primarily for use in the analysis of plutonium materials in process intermediate or finished product forms. Each unit of C122 consists of plutonium oxide (PuO_2) powder, nominally 1 gram, contained in a 1-dram (5-mL) glass vial.

NOTE-the vial and its outer plastic containment should be handled under proper radiologically-controlled conditions at all times.

The statistical uncertainty assigned to each certified value is the individual 95% confidence interval for the mean of the respective assay and isotopic measurements. The uncertainty assigned to the assay value is derived from random measurement variations; the uncertainties assigned to the isotopic measurements additionally take into account the uncertainty on each mass discrimination correction extrapolated from the mean of the $^{239}Pu/^{240}Pu$ ratios of NBS SRMs 946 and 948.

Preparation and packaging of C122 were carried out by the Los Alamos National Laboratory, under the direction of the CHM-1 Group headed by G. R. Waterbury. For certification, sample dissolutions and assay measurements were performed by M. I. Spaletto, NBL; isotopic measurements were performed by D. W. Crawford, NBL. Characterization studies and impurity measurements were performed by CHM-1, Los Alamos. Technical assistance was provided by J. E. Rein and J. W. Dahlby, Los Alamos and C. G. Cacic, NBL. Statistical assessment of the data for certification was performed by J. T. Bracey and M. D. Soriano, NBL. Overall direction and coordination of the preparation, certification and issuance of this CRM were provided by N. M. Trahey, NBL.

The PuO_2 material, prepared by calcination at 1250°C to obtain a stable near-stoichiometric product, was packaged prior to characterization and certification. CRM units were selected according to a statistical sampling plan, heated to constant weight at 120°C, then dissolved in separate acid mixtures, subsampled and purified for plutonium assay and isotopic analyses.[1,2,3] The plutonium content of each subsample was determined by the NBL coulometric method verified with NBS SRM 949f; plutonium isotopic composition for masses 238-242 was determined by thermal ionization mass spectrometry verified with NBS SRMs 946 and 948.[4] All other isotopes were determined to be at or below a detection limit of 0.0005 At. %. Metallic impurity content (excluding ^{241}Am and uranium) was determined by emission spectrography and spectrophotometry and is estimated to be 524 µg/g PuO_2 Uranium was determined by isotope dilution mass spectrometry and is estimated to be 23l µg/g PuO_2, as of January 1, 1985, with a calculated ingrowth of 41 µg/g PuO_2 per year; ^{241}Am was determined by radiochemistry and is estimated to be 2719 µg/g PuO_2 as of January 1, 1985, with a calculated ingrowth of 472 µg/g PuO_2 per year. *NOTE - Due to the levels of fixed and ingrowing impurities in C122, the material must be purified prior to use.*

C122 had a radioactivity of 3.44×10^{10} Bq (0.930 Ci) per unit as of January 1, 1985, which is dominated by ^{241}Pu.

Table I provides the decay-adjusted values for the certified assay content and isotopic composition of C122 at quarterly intervals for a five-year period. The half-life values (in years) used for the decay calculations are as follows: ^{238}Pu – 87.74; ^{239}Pu – 24,119; ^{240}Pu – 6,562; ^{241}Pu – 14.35; ^{242}Pu – 376,000.

TABLE I

C122 Quarterly Decay-Adjusted Certified Values
(Assay in Wt. %; Isotopic Composition in At. %)

Date	Plutonium	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu	RAW
January 1, 1990	87.596	0.0502	87.484	11.558	0.7279	0.1794	239.188
April 1, 1990	87.588	0.0501	87.492	11.559	0.7194	0.1794	239.187
July 1, 1990	87.579	0.0500	87.500	11.560	0.7108	0.1794	239.187
October 1, 1990	87.571	0.0499	87.508	11.561	0.7023	0.1794	239.187
January 1, 1991	87.562	0.0499	87.515	11.562	0.6939	0.1795	239.187
April 1, 1991	87.554	0.0498	87.523	11.562	0.6857	0.1795	239.187
July 1, 1991	87.546	0.0497	87.530	11.563	0.6776	0.1795	239.186
October 1, 1991	87.538	0.0496	87.538	11.564	0.6695	0.1795	239.186
January 1, 1992	87.530	0.0495	87.545	11.565	0.6614	0.1795	239.186
April 1, 1992	87.522	0.0494	87.552	11.565	0.6536	0.1795	239.186
July 1, 1992	87.514	0.0493	87.559	11.566	0.6458	0.1795	239.186
October 1, 1992	87.506	0.0492	87.566	11.567	0.6381	0.1796	239.186
January 1, 1993	87.499	0.0491	87.573	11.567	0.6304	0.1796	239.186
April 1, 1993	87.491	0.0490	87.580	11.568	0.6230	0.1796	239.185
July 1, 1993	87.484	0.0489	87.587	11.569	0.6156	0.1796	239.185
October 1, 1993	87.476	0.0488	87.594	11.569	0.6082	0.1796	239.185
January 1, 1994	87.469	0.0487	87.601	11.570	0.6009	0.1796	239.185
April 1, 1994	87.462	0.0486	87.607	11.571	0.5938	0.1797	239.185
July 1, 1994	87.455	0.0486	87.614	11.571	0.5868	0.1797	239.185
October 1, 1994	87.447	0.0485	87.620	11.572	0.5797	0.1797	239.185
95% Confidence Interval (expressed as% of value):							
	±0.045	±2.0	±0.005	±0.031	±0.11	±0.74	

RECOMMENDED PROCEDURE FOR USING C122

Each CRM package unit contains 1 ± 0.05 g of PuO_2 powder and is intended *only* to store the material until use. For valid application of the CRM, this recommended procedure or an equivalent, must be followed.

Empty the vial unit contents into a Pt crucible, previously preheated to constant weight at 120°C. Proceed to dry the crucible and its contents at 120°C until a constant weight is achieved. (An initial 2-hr drying period followed by two successive 1-hr drying periods should be sufficient.) Transfer the dried PuO_2 to a Teflon beaker and reweigh the crucible in order to calculate the final sample weight. Add ~100 mL of 16 M HNO_3 - 0.05 M HF or 11 M HCl-0.1 M HF to the beaker and apply heat. Continue heating *in air* and replenish the acid mixture as necessary until the sample is dissolved. Cool the solution, quantitatively transfer to a preweighed bottle equipped with cap, and adjust the volume to the desired concentration. Cap the bottle tightly, vigorously shake to homogenize contents, and weigh. Distribute all the solution, as individually weighed portions, into suitable containers and purify each portion before use.

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[1]Spaletto, I., Cacic, C., and Crawford D., Annual Progress Report, NBL-311, October 1982-September 1983, pp. 14-15.

[2]Waterbury, G., Private Communication, November 12, 1980.

[3]Swinburn, K., and McGowan, f., "An Approach to the Use of Plutonium Dioxide as a Chemical Reference Standard for Plutonium," BNFL Report 205(W).

[4]NBL Procedures Manual (Internal Document).