Certificate of Analysis

Certified Reference Material 122

Plutonium Oxide (PuO_2) Plutonium Mass Fraction and Isotope Standard in Powder Form

Description: Certified Reference Material (CRM) 122 consists of nominally 1 gram of plutonium oxide (PuO2) powder contained in a 1-dram (5 mL) glass vial. Certified property values for the standard are provided in Table 1. Supplemental non-certified values are provided in Table 2.

Table 1. Certified Property Values and Uncertainties [a] [b]

	g g^{-1}			g mol^{-1}	
Plutonium Mass Fraction:	0.87790		Plutonium Molar Mass:	239.191	
Uncertainty:	0.00039		Uncertainty:	0.014	
Isotope-Amount	$n(^{238}Pu)/n(Pu)$	$n(^{239}Pu)/n(Pu)$	$n(^{240}Pu)/n(Pu)$	$n(^{241}Pu)/n(Pu)$	$n(^{242}Pu)/n(Pu)$
Fraction (•100):	0.0521	87.305	11.539	0.9248	0.1790
Uncertainty:	0.0011	0.004	0.004	0.0011	0.0013

[a] The statistical uncertainty assigned to each certified value is the individual 95 % confidence interval for the mean of the respective assay and isotope measurements. The uncertainty assigned to the mass fraction 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 CRMs 136 and 138 (formerly NBS SRMs 946 and 948).

[b] **Values are provided for a reference date of January 1, 1985.**

Intended use: This CRM is a plutonium mass fraction and isotope standard primarily for use in the analysis of plutonium materials in process intermediate or finished product forms.

Storage: This material should be stored in its original packaging under normal laboratory environmental conditions.

Period of validity: When stored in its original, unopened container, the certification of this material is valid indefinitely. The Nuclear Reference Material Program (NRMP) will notify customers should degradation be detected.

Minimum sample size: No minimum sample size is declared for this material.

Instructions for handling: The material in the unit is radioactive. This radioactive material should be handled only by qualified individuals. The primary container and its outer plastic containment should be handled under proper radiologically-controlled conditions at all times. To minimize personnel exposure, appropriate facilities and personal protective equipment should be used. Refer to the Safety Data Sheet for further information.

Recommended Procedure for Using CRM 122

Each CRM package unit contains 1 g ± 0.05 g of PuO_2 powder and is intended only to store the material until use. For valid application of the CRM, the following procedure or an equivalent procedure, 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 to calculate the final sample weight. Add ≈100 mL of mixed solution of 16 mol L^{-1} HNO_3, and 0.05 mol L^{-1} HF or 11 mol L^{-1} HCl and 0.1 mol L^{-1} 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 mass fraction (g g^{-1}). Cap the bottle tightly, vigorously shake to homogenize contents, and weigh. Distribute all the solution, as individually weighed portions, into suitable containers. Due to the levels of fixed and ingrowing impurities in CRM 122, the material must be purified prior to use.

Traceability statement: The certified isotope-amount fraction values are traceable to the SI unit of mole. The certified plutonium mass fraction values are traceable to the SI unit kilogram. The certified plutonium molar mass is traceable to the SI units mole and kilogram.

Additional information: Preparation and packaging of CRM 122 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, New Brunswick Laboratory (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 mass fraction and isotope analyses [1-3]. The plutonium content of each subsample was determined by the NBL coulometric method verified with NBS SRM 949f; plutonium isotopic composition for ^{238}Pu through ^{242}Pu 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 % isotope-amount fraction.

Table 2. Non-Certified Property Values

Impurity	Mass Fraction [(a)] (ug g^{-1} PuO_2)	Analysis Method
Americium	2719	Radiochemical Analysis
Uranium	231	Isotope Dilution Mass Spectrometry
Other Combined Metallic	524	Emission Spectrography & Spectrophotometry

[(a)] Reported values are for a reference date January 1, 1985.

In 2016, the New Brunswick Laboratory facility was transitioned to a program office within the Department of Energy and is now operating within the National Nuclear Security Administration (NNSA) as the Nuclear Reference Material Program (NRMP).

[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).