Nuclear Reference Material Program
U.S. Department of Energy

Certificate of Analysis

Certified Reference Material 130

Plutonium-242 Assay and Isotopic Standard in Nitrate Form

Description: Certified Reference Material (CRM) 130 is a plutonium amount and isotope-amount ratio standard. Each unit of CRM 130 consists of approximately 1 mg of ^{242}Pu as evaporated plutonium nitrate in a 30 mL Teflon bottle. Each bottle contains a unique quantity of plutonium and is assigned a serial number for identification and reference. The certified plutonium amount and isotope-amount ratio property values are provided in Table 1. Supplemental non-certified property values for the material are provided in Table 2.

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

Bottle Number		Plutonium-242 Amount:	**µmols**	Plutonium Molar Mass:	**g mol^{-1}**
		Uncertainty:	0.0030	Uncertainty:	0.0021
	Isotope-Amount Fraction (•100):				
	***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)**	***n*(^{244}Pu)/*n*(Pu)**
Value:	0.00419	0.00478	0.01974	0.02466	99.94623	0.00040
Uncertainty:	0.00026	0.00012	0.00038	0.00034	0.00065	0.00010

[a] **Certified values are provided for a reference date of January 1, 1987. Certified plutonium amount and isotope values must be decay-corrected to the date of use.**

[b] The indicated uncertainties for the above property values are 95 % confidence intervals for the mean. The uncertainty for the plutonium amount includes components due to analytical variation and weighing uncertainties of individual units. In addition to random measurement variations, the uncertainties assigned to the isotope values, which were determined by isotope dilution mass spectrometry (IDMS), include a component due to the uncertainties associated with the SRM 996 and SRM 949f materials used as spikes.

Intended use: CRM 130 is a plutonium amount and isotope standard for use as a spike in the analysis of plutonium materials by IDMS.

Storage: To maintain the integrity of an unused CRM unit, it should remain in the original packaging and should be stored in a dry, temperature-controlled location.

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

Minimum sample size: Certification and/or verification measurements for uranium mass fraction and isotope-amount ratios were performed on a random sampling of whole CRM 130 units. The homogeneity of plutonium mass fraction or isotopic composition is not certified for subsamples of the undissolved material comprising a unit.

Instructions for handling: The reference material in the unit is radioactive. The bottle and its outer plastic containment should be handled under proper radiologically-controlled conditions at all times. This radioactive material should be handled only by qualified individuals. Refer to the Safety Data Sheet for further information.

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Recommended procedure for using CRM 130

The CRM unit is designed to prepare a solution having a known concentration of plutonium on a mass basis. Once prepared, it is suggested that all the solution be immediately distributed as subportions for later use as individual spikes. Chemical separation of the plutonium from its uranium and americium daughters after spiking but prior to analysis is essential for high accuracy, since these daughters contain isotopes that are isobaric with plutonium isotopes.

Wipe the Teflon bottle with a chamois or damp cloth to dissipate any static charge which may cause expulsion of the material upon opening. Weigh the bottle then unscrew the cap, add a desired quantity of 8 mol L^{-1} HNO_3 and carefully warm the bottle to ensure total dissolution.

NOTE: Do not heat the bottle above 150 °C because bottle deformation or melting will occur. Replace and tighten cap, then allow the bottle to cool to ambient temperature. Loosen the cap to equalize air pressure, retighten, wipe the bottle with a chamois or damp cloth, and weigh. Shake vigorously to homogenize the contents and distribute all the solution as weighed portions into suitable containers for use as spikes. Calculate the plutonium concentration by:

$$^{242}Pu\ \mu moles/g \;=\; \frac{certified\ amount\ of\ ^{242}Pu\ (\mu moles)}{[wt.of\ bottle\ \&\ solution\ (g)] - [tare\ of\ bottle\ (g) - 0.0020\ g]}$$

in which 0.0020 grams is the nominal weight of evaporated plutonium nitrate residue. If a more dilute solution is desired, dissolve the residue as above, quantitatively transfer the solution to a larger tared container, weigh, mix vigorously, and distribute all the solution as weighed portions.

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

Additional information: The plutonium material used to produce this CRM 130 was obtained from the Oak Ridge National Laboratory (ORNL) Isotope Sales Group with the approval of the DOE Research Materials/Transplutonium Program Committee. The master solution, from which the CRM was produced, was chemically purified before being apportioned into units and dried [1]. The plutonium content was determined by the NBL controlled-potential coulometric method verified with NBS SRM 949f. The plutonium isotope distribution was obtained by thermal ionization mass spectrometry using an isotope dilution technique. The ^{238}Pu through ^{241}Pu values, and the ^{244}Pu value were determined by spiking CRM 130 with NBS SRM 996 (^{244}Pu) and NBS SRM 949f (^{239}Pu), respectively. The ^{242}Pu abundance was then calculated by difference. CRM 128 was used to monitor instrument performance. The mass discrimination effects did not significantly impact on the isotopic abundance calculations since the minor isotopes are present in very small amounts. Thus, no corrections for these effects were necessary.

Table 2. Non-Certified Property Values

Estimated Activity [(a)]	1.1 x 10^6	Bq per unit
Mass Fraction ^{241}Am [(a)]	19	μg g^{-1} Pu
Mass Fraction Total Impurities [(b)]	230	μg g^{-1} Pu

[(a)] **Value is for a reference date of January 1, 1987.**

[(b)] Total element impurity content was determined by spark source mass spectrometry on selected subsamples.

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] Crawford, D., Cacic, C., and Soriano, M., "The Production and Certification of a Plutonium Equal-Atom Reference Material – NBL CRM 128," USDOE Report NBL-316, July 1987. Copies available upon request to the NRMP.