New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 130
Plutonium-242 Spike Assay and Isotopic Standard in Nitrate Form

Plutonium-242 Content*: ± **µmoles**

Serial Number (Teflon bottle #):

	^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu	^{244}Pu
Atom Percent*:	**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**

Relative Atomic Weight*: 242.0578

***All values are as of January 1, 1987**

This Certified Reference Material (CRM) is an assay and isotopic standard for use as a spike in the analysis of plutonium materials by isotope dilution mass spectrometry (IDMS). 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.

NOTE: *The bottle and its outer plastic containment should be handled under proper radiologically-controlled conditions at all times.*

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

The master solution, from which CRM 130 was produced, was chemically purified before being apportioned and dried into units. The plutonium content was determined by the NBL controlled-potential coulometric method verified with NBS SRM 949f. The plutonium isotopic 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

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SRM 949f (^{239}Pu), respectively. The ^{242}Pu abundance was then calculated by difference. NBL 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. Total elemental impurity content was determined by spark source mass spectrometry on selected subsamples and is estimated to be 230 μg/g plutonium. The calculated americium ingrowth from the decay of ^{241}Pu present in small amounts in the CRM is 19 μg/g plutonium as of January 1, 1987, and will increase at a rate of approximately 5% of the total ^{241}Pu per year. Further information on impurity analysis of the material and preparation of the master solution may be found in NBL's Research and Development Report No. 316.

CRM 130 had a radioactivity of 1.1 x l0^6 Bq (30 μCi) per unit as of January 1, 1987, which is dominated by 241Pu.

The plutonium material used to produce this CRM was obtained from the Oak Ridge National Laboratory (ORNL) Isotope Sales Group with the approval of the DOE Research Materialsl/Transplutonium Program Committee chaired by J. L. Burnett. Preparation of the material was performed by C. G. Cacic, NBL; assay measurements were performed by M. I. Spaletto, NBL; isotopic measurements were performed by M. A. Legel, NBL; impurity measurements were performed by J. A. Carter and associates, ORNL. Statistical evaluation of the data was performed by M. D. Soriano, NBL. Overall direction and coordination of the preparation, certification and issuance of the CRM were provided by N. M. Trahey, NBL.

RECOMMENDED PROCEDURE FOR USING CRM 130

The package is designed to prepare a solution having a known concentration of plutonium on a weight 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 prior to use is essential for high accuracy, since these daughters contain isotopes which 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 8M HNO_3 and carefully warm the bottle to insure total dissolution.

Do **not** heat the bottle above 150°C because bottle deformation 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 amcentration by:

$$^{242}\text{Pu } \mu\text{moles/g} = \frac{(\text{Certified content of } ^{242}\text{Pu, } \mu\text{moles})}{(\text{wt. of bottle and solution[g]}) - (\text{tare of bottle,[g]} - 0.0020)}$$

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, transfer the solution quantitatively to a larger tared container, weigh, mix vigorously, and distribute all the solution as weighed portions.

Reference: 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.

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