



Nuclear Reference Material Program

U.S. Department of Energy



Certificate of Analysis

Certified Reference Material 128

Plutonium-239/Plutonium-242, 1:1 Atom Ratio

Isotopic Standard in Nitrate Form

Description: Certified Reference Material (CRM) 128 is a plutonium isotope-amount ratio standard. Each unit of CRM 128 consists of approximately 1 mg of a nominal 1:1 mixture of ^{239}Pu and ^{242}Pu , as evaporated plutonium nitrate contained in a 30 mL FEP Teflon bottle. The certified isotope-amount ratio property value is 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)

Isotope-Amount Ratio:	$n(^{239}\text{Pu})/n(^{242}\text{Pu})$
Ratio:	0.99937
Uncertainty:	0.00026

^(a) **Certified value is provided for a reference date of October 1, 1984. The certified isotope amount ratio value must be decay-corrected to the date of use.**

^(b) The statistical uncertainty assigned to the certified ratio value is the 95 % confidence interval for the unweighted mean of the ratio calculated from assay and mass measurements of the separated isotopes and the ratio determined by mass spectrometric measurements of the CRM. The uncertainty is propagated from all known non-negligible sources of random and systematic variations associated with the measurement methods used.

Intended use: CRM 128 is primarily intended for the calibration of mass spectrometers used to perform plutonium isotope-amount ratio measurements. The specific purpose of this isotope standard is for the determination of a mass discrimination factor which will place measured plutonium isotopic ratios on an absolute basis.

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 National Nuclear Security Administration's (NNSA) Nuclear Reference Material Program (NRMP) will notify customers should degradation be detected.

Minimum sample size: Once the entire unit is dissolved, as described below, no minimum sample size is recommended for the resulting homogeneous solution.

Instructions for handling: The reference material in the unit is radioactive. This radioactive material should be handled only by qualified individuals. To minimize personnel exposure, appropriate facilities and personal protective equipment should be used. Refer to the Safety Data Sheet for further information.

Each CRM unit contains 1.00 ± 0.03 mg of plutonium and is designated for in-situ dissolution. When converted to solution form, a unit may be used as is. No additional purification of the CRM is required. Wipe the Teflon bottle with a chamois or damp cloth to dissipate any static charge which may cause expulsion of the material upon opening. Unscrew the cap, add sufficient $1 \text{ mol L}^{-1} \text{ HNO}_3$ to the CRM bottle to yield the mass fraction of plutonium desired, and carefully warm the bottle to ensure total dissolution. Do not heat the bottle above $150 \text{ }^\circ\text{C}$ because bottle

(Revision of Certificate Dated December 2020)
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January 2026

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deformation will occur. Replace and tighten the cap, then allow the bottle to cool before shaking to homogenize contents. Wipe cap and bottle threads each time a portion of the CRM solution is removed from the bottle.

Traceability statement: The certified property value is traceable to the SI unit of mole.

Additional information: The ^{239}Pu and ^{242}Pu separated isotopes (>99.9 % isotopic purity) comprising CRM 128 were separately dissolved, chemically purified, and assayed by controlled potential coulometry before being combined by mass [1]. The assay characterization measurements were used to calculate a precise gravimetric $^{239}\text{Pu}/^{242}\text{Pu}$ value for the CRM. The CRM was then isotopically equilibrated, apportioned, and dried into units. Isotopic certification measurements were performed on CRM units randomly selected according to a statistical sampling plan. The $^{239}\text{Pu}/^{242}\text{Pu}$ ratio data obtained for the CRM were corrected for mass discrimination effects by concurrent analysis of five $^{239}\text{Pu}/^{242}\text{Pu}$ calibration mixtures, prepared by weight to closely bracket the isotopic ratio of the CRM.

The plutonium materials used to produce this CRM were obtained from the ORNL Isotope Sales Group with the approval of the DOE Research Materials/Transplutonium Program Committee.

Table 2. Non-Certified Property Values

	Estimated Massic Activity ^(a)	2.7 x 10 ⁶	Bq per unit		
	Mass Fraction ^{241}Am ^(a)	21	$\mu\text{g g}^{-1}$ Pu		
	Mass Fraction Total Impurity ^(b)	300	$\mu\text{g g}^{-1}$ Pu		
Isotope-Amount	$n(^{238}\text{Pu})/n(\text{Pu})$	$n(^{239}\text{Pu})/n(\text{Pu})$	$n(^{240}\text{Pu})/n(\text{Pu})$	$n(^{241}\text{Pu})/n(\text{Pu})$	$n(^{242}\text{Pu})/n(\text{Pu})$
Fraction ($\bullet 100$) ^(c) :	0.004	49.947	0.035	0.036	49.978

^(a) Value is for a reference date of July 1, 1984.

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

^(c) Values are for a reference date of October 1, 1984. The $n(^{244}\text{Pu})/n(\text{Pu})$ ($\bullet 100$) value is estimated as <0.001.

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 NBL Program Office.