



NBL Program Office
U.S. Department of Energy



Certificate of Analysis
Certified Reference Material C128
Plutonium-239/Plutonium-242, 1:1 Atom Ratio Plutonium
Isotopic Standard in Nitrate Form

$^{239}\text{Pu}/^{242}\text{Pu}$ ratio: $0.9993_7 \pm 0.0002_6$ Atom Ratio*

*Ratio valid as of October 1, 1984

This Certified Reference Material (CRM) is primarily intended for the calibration of mass spectrometers used to perform plutonium isotopic 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. Each unit of C128 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 Teflon bottle.

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

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.

The ^{239}Pu and ^{242}Pu separated isotopes (>99.9% isotopic purity) comprising C128 were separately dissolved, chemically purified, and assayed by controlled potential coulometry before being combined by weight. 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. Total element impurity content was determined by spark source mass spectrometry on selected subsamples and is estimated to be 300 $\mu\text{g/g}$ plutonium. Although the CRM was americium-free at the time of preparation, the calculated americium ingrowth from the decay of ^{241}Pu present in small amounts in the CRM is 21 $\mu\text{g/g}$ plutonium as of October 1, 1984, and will increase at a rate of approximately 5 percent of the total ^{241}Pu per year.

C128 had a radioactivity of 2.7×10^6 Bq (73 μCi) per unit as of July 1, 1984, which is dominated by ^{239}Pu and ^{241}Pu .

RECOMMENDED PROCEDURE FOR USING C128

Each CRM unit contains 1 ± 0.03 mg of plutonium and is designated for in-situ dissolution. When converted to solution form, a unit can 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 1M HNO₃ to the CRM bottle to yield the concentration desired, 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 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.

C128 Isotopic Distribution (as of October 1, 1984)

	²³⁸Pu	²³⁹Pu	²⁴⁰Pu	²⁴¹Pu	²⁴²Pu	²⁴⁴Pu
Atom Percent:	0.004	49.947	0.035	0.036	49.978	<0.001

The half-life values that were used to calculate the abundances above, expressed in years: ²³⁸Pu - 87.74; ²³⁹Pu - 24,119; ²⁴⁰Pu- 6,562; ²⁴¹Pu - 14.35; ²⁴²Pu - 376,300.

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.

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. Copies available upon request to the NBL Program Office.