



*New Brunswick Laboratory*  
*U.S. Department of Energy*

## Certificate of Analysis

### CRM 128

### 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 CRM No. 128 consists of approximately 1 mg of a nominal 1:1 mixture of  $^{239}\text{Pu}$  and  $^{242}\text{Pu}$ , 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  $^{239}\text{Pu}$  and  $^{242}\text{Pu}$  separated isotopes (>99.9% isotopic purity) comprising CRM 128 were separately dissolved, chemically purified, and assayed by controlled potential coulometry before being combined by 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  $^{241}\text{Pu}$  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  $^{241}\text{Pu}$  per year.

CRM 128 had a radioactivity of  $2.7 \times 10^6$  Bq (73  $\mu\text{Ci}$ ) per unit as of July 1, 1984, which is dominated by  $^{239}\text{Pu}$  and  $^{241}\text{Pu}$ .

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(Editorial revision of Certificate dated October 1, 1984)

## RECOMMENDED PROCEDURE FOR USING CRM 128

Each CRM unit contains  $1 \pm 0.03$  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 HNO<sub>3</sub> 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.

### CRM 128 Isotopic Distribution (as of October 1, 1984)

	<sup>238</sup> Pu	<sup>239</sup> Pu	<sup>240</sup> Pu	<sup>241</sup> Pu	<sup>242</sup> Pu	<sup>244</sup> Pu
<b>Atom Percent:</b>	<b>0.004</b>	<b>49.947</b>	<b>0.035</b>	<b>0.036</b>	<b>49.978</b>	<b>&lt;0.001</b>

The half-life values that were used to calculate the abundances above, expressed in years: <sup>238</sup>Pu - 87.74; <sup>239</sup>Pu - 24,119; <sup>240</sup>Pu - 6,562; <sup>241</sup>Pu - 14.35; <sup>242</sup>Pu - 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 chaired by J. L. Burnett. Preparation and assay measurements of the CRM and calibration mixtures were performed by C.G. Cacic, NBL; isotopic measurements were performed by D. W. Crawford, NBL; impurity measurements were performed by J. A. Carter and associates, ORNL. Technical assistance was provided by L. A. Machlan, NBS. Isotopic verification measurements were performed by E.L. Callis, ANL. Statistical assessment of the data for certification was performed by M. D. Soriano, NBL. Initial project technical direction was provided by E.L. Garner, NBS; overall direction and coordination of the preparation, certification and issuance of this CRM was provided by N. M. Trahey, NBL.

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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