



NBL Program Office
U.S. Department of Energy



Certificate of Analysis
Certified Reference Material C126 (1g)
Plutonium (Metal) Assay and Isotopic Standard

Plutonium Concentration (Mass Fraction)0.99960 ± 0.00026 kg Pu/kg

<u>Isotopic Ratios</u>	<u>$\frac{^{238}\text{Pu}}{^{239}\text{Pu}}$</u>	<u>$\frac{^{240}\text{Pu}}{^{239}\text{Pu}}$</u>	<u>$\frac{^{241}\text{Pu}}{^{239}\text{Pu}}$</u>	<u>$\frac{^{242}\text{Pu}}{^{239}\text{Pu}}$</u>	
Atom Ratios	0.00013022 ± 0.00000030	0.062744 ± 0.000016	0.00157886 ± 0.00000076	0.00038465 ± 0.00000025	
<u>Isotopic Abundance</u>	<u>^{238}Pu</u>	<u>^{239}Pu</u>	<u>^{240}Pu</u>	<u>^{241}Pu</u>	<u>^{242}Pu</u>
Atom Fraction (x 100)	0.012229 ± 0.000028	93.9110 ± 0.0015	5.8923 ± 0.0015	0.148272 ± 0.000072	0.036123 ± 0.000024
Mass Fraction (x 100)	0.012175 ± 0.000028	93.8863 ± 0.0015	5.9155 ± 0.0015	0.149476 ± 0.000072	0.036568 ± 0.000024

Relative Atomic Mass of Plutonium239.115113 ± 0.000015

Certified values above are valid for July 30, 2003

The expanded uncertainties (U) are expressed as 95% confidence interval for the values. U is defined as $k \cdot u_c$ where k is the coverage factor and u_c is the combined standard uncertainty. The last figure in the reported values and their uncertainties is provided to reduce errors in rounding numbers and is not intended to convey reliability at that level.

This Certified Reference Material (CRM) is an assay (elemental concentration) and isotopic standard for use in plutonium determinations. Each unit of C126A contains a single approximately 1 gram piece of plutonium metal sealed in a glass tube under reduced pressure argon atmosphere. Before use, follow a recommended procedure given on this certificate for removing the surface oxide layer.

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

The preparation and packaging of C126A was completed at the Los Alamos National Laboratory (LANL). The source material for C126A was double electrocleaned, the cleaned metal was cast into rods, the rods were extruded into wires, the wires were cut into 1-gram pieces, and the pieces were individually sealed in Pyrex glass tubes under a reduced pressure argon atmosphere.

Samples of C126A for certification measurements were selected according to a statistical plan. The selected units were electrolytically cleaned, weighed, dissolved in acid, subsampled and purified for plutonium concentration and isotopic analyses. The plutonium concentration was determined by the Controlled Potential Coulometric Method by two analysts using two different coulometers. Samples of CRM 126, Plutonium Metal Assay and Isotopic Standard, were analyzed along with CRM 126-A samples. The CRM 126 samples served as controls to verify performance of the measurement systems. The plutonium concentration determined at NBL was verified at LANL by the Actinide Analytical Chemistry (C-AAC) Group using the coulometric and the 100% minus impurities methods.

The plutonium isotopic composition and the relative atomic mass of plutonium were determined by thermal ionization mass spectrometry (TIMS) by one analyst using two different mass spectrometers. The total evaporation method was used in the determination of the $^{240}\text{Pu}/^{239}\text{Pu}$, $^{241}\text{Pu}/^{239}\text{Pu}$ and $^{242}\text{Pu}/^{239}\text{Pu}$ ratios. Measurement system accuracy was assessed by analyses of NBL CRM 128, Plutonium Isotopic Standard ($^{242}\text{Pu}/^{239}\text{Pu}$ equal atom standard) run along with C126A samples. NBL CRM 122, Plutonium Isotopic Standard, was used as an additional control to verify performance of the measurement systems. The $^{238}\text{Pu}/^{239}\text{Pu}$ ratio was measured using a fractionation correction method with ion counting. No values are reported for ^{244}Pu . Under the measurement conditions used, ion counts at the ^{244}Pu mass were indistinguishable from background. The plutonium isotopic values determined at NBL were verified at LANL by the C-AAC Group using the total evaporation method.

Impurities in the metal were measured by the C-AAC Group at LANL. Impurity content (excluding radionuclides) was determined to be 175 $\mu\text{g/g}$ metal. On July 30, 2003, the determined radionuclide content was: 71 $\mu\text{g U/g}$ metal, 23 $\mu\text{g }^{237}\text{Np/g}$ metal, and 183 $\mu\text{g }^{241}\text{Am/g}$ metal. These impurity values are not certified.

The radioactive decay rate of C126A was 8.4×10^9 Bq per gram metal, as of July 30, 2003.

The expanded uncertainty (U) for a certified property of C126A defines an interval around the value of the property and was obtained by multiplying the combined standard uncertainty (uc) by a coverage factor (k). The coverage factor, k, is the Student's t factor based on the effective degrees of freedom to provide a 95% level of confidence. The combined standard uncertainty (uc) for plutonium concentration consists of Type A components associated with measurement precision of samples, and measurement precision and bias of CRM 126 standards, and a Type B component associated with the uncertainty of the CRM 126 plutonium concentration certified value. Traceability for the C126A plutonium concentration value is established through the use and incorporation of uncertainties associated with CRM 126. The combined standard uncertainties (uc) for plutonium isotopic parameters consist of Type A components derived from precision of the isotopic ratio measurements of samples, uncertainties associated with detector efficiency corrections, instrument performance factors, and a Type B component based on the uncertainty associated with the NBL CRM 128 certified value. Additionally, the $^{238}\text{Pu}/^{239}\text{Pu}$ ratio uncertainty incorporates Type B components based on the standard uncertainty of the $^{240}\text{Pu}/^{239}\text{Pu}$ ratio for C126A and the uncertainty associated with detector linearity. Traceability for all ratios and abundances is established through the use and incorporation of uncertainties associated with CRM 128.

The Los Alamos National Laboratory fabricated and packaged CRM 126-A standards, and performed verification analyses.

RECOMMENDED PROCEDURE FOR USE

Each CRM package unit contains 1 ± 0.2 g of plutonium metal. The mass of the metal piece is not certified. Prior to use, the surface oxide layer must be removed by the electrolytic or the brushing/filing method. The cleaned metal must be immediately weighed, and the measured mass must be corrected for buoyancy.

NBL cleaned the samples for certification analysis by the electrolytic method. The procedure is as follows: place the metal into a glass beaker containing 20% potassium carbonate solution. Apply an electrical potential to the system to remove the oxide coating from the metal surface. Rinse the clean metal with distilled water and acetone and dry thoroughly before weighing.

LANL cleaned the samples for verification analysis by the brushing method. The procedure is as follows: use a clean file or metallic brush to remove oxide from the surface of the metal piece.

The following relative atomic masses and half-lives (years) were used in calculations:

	²³⁸ Pu	²³⁹ Pu	²⁴⁰ Pu	²⁴¹ Pu	²⁴² Pu
Atomic Mass	238.0495534	239.0521565	240.0538075	241.0568453	242.0587368
Uncertainty	0.0000021	0.0000021	0.0000021	0.0000021	0.0000021
Half-Life (years)	87.7	24,110	6564	14.290	373,300
Uncertainty	0.3	30	11	0.006	1,200

The values used for half-lives were taken from the National Nuclear Data Center's January 2000 Sixth Edition Nuclear Wallet Card. The values used for atomic masses were taken from "The 1995 Update to the Atomic Mass Evaluation" by Audi and Wapstra, published in Nuclear Physics A, 595 (1995) 409-480. Uncertainties for the atomic masses are expressed as one standard deviation errors.

Certified plutonium concentration and isotopic values must be decay-corrected to the date of use.