



# Nuclear Reference Material Program

## U.S. Department of Energy



# Certificate of Analysis

## Certified Reference Material 126A (1 g)

### Plutonium (Metal) Assay and Isotopic Standard

**Description:** Certified Reference Material (CRM) 126A is a plutonium mass fraction and isotope-amount ratio standard. Each unit of CRM 126A contains a single approximately 1.0 g ± 0.2 g piece of plutonium metal sealed in a glass tube under reduced pressure argon atmosphere (mass of the metal piece is not certified). Certified isotope composition and mass fraction values are reported in Table 1. Supplemental non-certified property values for the sample material are provided in Tables 2 and 3.

**Table 1. Certified Property Values and Uncertainties** <sup>(a) (b)</sup>

Plutonium	<b>g g<sup>-1</sup></b>			<b>g mol<sup>-1</sup></b>	
Mass Fraction:	0.99960	Plutonium Molar Mass:			239.115113
Uncertainty:	0.00026	Uncertainty:			0.000015
Isotope-Amount Ratio:	$n(^{238}\text{Pu})/n(^{239}\text{Pu})$	$n(^{240}\text{Pu})/n(^{239}\text{Pu})$	$n(^{241}\text{Pu})/n(^{239}\text{Pu})$	$n(^{242}\text{Pu})/n(^{239}\text{Pu})$	
Ratio:	0.00013022	0.062744	0.00157886	0.00038465	
Uncertainty:	0.00000030	0.000016	0.00000076	0.00000025	
Isotope-Amount Fraction (•100):	$n(^{238}\text{Pu})/n(\text{Pu})$	$n(^{239}\text{Pu})/n(\text{Pu})$	$n(^{240}\text{Pu})/n(\text{Pu})$	$n(^{241}\text{Pu})/n(\text{Pu})$	$n(^{242}\text{Pu})/n(\text{Pu})$
Fraction (•100):	0.012229	93.9110	5.8923	0.148272	0.036123
Uncertainty:	0.000028	0.0015	0.0015	0.000072	0.000024
Isotope-Mass Fraction (•100):	$m(^{238}\text{Pu})/m(\text{Pu})$	$m(^{239}\text{Pu})/m(\text{Pu})$	$m(^{240}\text{Pu})/m(\text{Pu})$	$m(^{241}\text{Pu})/m(\text{Pu})$	$m(^{242}\text{Pu})/m(\text{Pu})$
Fraction (•100):	0.012175	93.8863	5.9155	0.149476	0.036568
Uncertainty:	0.000028	0.0015	0.0015	0.000072	0.000024

<sup>(a)</sup> Certified values are provided for a reference date of July 30, 2003. Certified plutonium mass fraction and isotope values must be decay-corrected to the date of use.

<sup>(b)</sup> 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.

**Intended use:** CRM 126A is a plutonium mass fraction (assay) and isotope standard for use in plutonium determinations.

**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:** Certification and/or verification measurements for uranium mass fraction and isotope-amount ratios were performed on a random sampling of CRM 126A units. The homogeneity of plutonium mass fraction or isotopic composition is not certified for subsamples of the undissolved metal comprising a unit.

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**Instructions for handling:** The reference material in the unit is radioactive. The primary glass tube container and its outer plastic containment should be handled under proper radiologically controlled conditions at all times. This radioactive material should be handled only by qualified individuals. Refer to the Safety Data Sheet for further information.

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.

#### **Methods Used for Accurate Weighing and Mass Fraction Values for CRM 126A**

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 a brushing method. The procedure is as follows: use a clean file or metallic brush to remove oxide from the surface of the metal piece.

**Traceability statement:** The certified isotope amount fraction and isotope amount ratio values are traceable to the SI unit of mole. The certified isotope mass fraction and plutonium mass fraction values are traceable to the SI unit kilogram. The certified plutonium molar mass is traceable to the SI units mole and kilogram.

**Measurement uncertainty:** The expanded uncertainty (U) for a certified property of CRM 126A defines an interval around the value of the property and was obtained by multiplying the combined standard uncertainty ( $u_c$ ) 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 ( $u_c$ ) for plutonium concentration consists of the standard deviation of sample measurements and the measurement precision and bias of CRM 126 standards applied as Type A evaluated components. The uncertainty of the CRM 126 plutonium mass fraction certified value is applied as a Type B evaluated component. Traceability for the CRM 126A plutonium concentration value is established through the use and incorporation of uncertainties associated with CRM 126. The combined standard uncertainties ( $u_c$ ) for plutonium isotopic parameters consist of the standard deviation of the isotopic ratio measurements of samples, uncertainties associated with detector efficiency corrections, instrument performance factors applied as Type A evaluated components. The uncertainty associated with the NBL CRM 128 certified value was applied as a Type B evaluated component. Additionally, the  $^{238}\text{Pu}/^{239}\text{Pu}$  ratio uncertainty incorporates the standard uncertainty of the  $^{240}\text{Pu}/^{239}\text{Pu}$  ratio for CRM 126A and the uncertainty associated with detector linearity applied as Type B evaluated components. Traceability for all ratios and abundances is established through the use and incorporation of uncertainties associated with CRM 128.

**Additional information:** The preparation and packaging of CRM 126A was completed at the Los Alamos National Laboratory (LANL). The source material for CRM 126A was double electro cleaned, the cleaned metal was cast into rods, the rods were extruded into wires, the wires were cut into approximately 1-gram pieces, and the pieces were individually sealed in Pyrex® glass tubes under a reduced pressure argon atmosphere.

Samples of CRM 126A 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 mass fraction and isotopic analyses. The plutonium mass fraction 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 126A samples as controls to verify performance of the measurement systems. The plutonium mass fraction determined at NBL was verified at LANL by the Actinide Analytical Chemistry (C-AAC) Group using the coulometric method and the 100 % minus impurities method.

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 CRM 128, Plutonium Isotopic Standard ( $^{242}\text{Pu}/^{239}\text{Pu}$  equal atom standard) measured along with CRM 126A samples. 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 an internal fractionation correction method with ion counting. Under the measurement conditions used, ion counts at the  $^{244}\text{Pu}$  mass were indistinguishable from background. Accordingly, no values are reported for  $^{244}\text{Pu}$ . Plutonium isotope values determined at NBL were verified at LANL by the C-AAC Group using the total evaporation method.

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).

**Table 2. Non-Certified Property Values for Radioactivity & Actinide Impurities** <sup>(a) (b)</sup>

Estimated Massic Activity	8.4 x 10 <sup>9</sup>	Bq g <sup>-1</sup> metal
Mass Fraction <sup>241</sup> Am	183	µg g <sup>-1</sup> metal
Mass Fraction <sup>237</sup> Np	23	µg g <sup>-1</sup> metal
Mass Fraction Thorium	<0.1	µg g <sup>-1</sup> metal
Mass Fraction Uranium	71	µg g <sup>-1</sup> metal

<sup>(a)</sup> Values are for a reference date of July 30, 2003.

<sup>(b)</sup> Impurities in the metal were measured by the C-AAC Group at LANL.

**Table 3. Non-Certified Property Values for Stable Impurities** <sup>(a)</sup>

Analyte	Average (µg g <sup>-1</sup> )	Std Dev (µg g <sup>-1</sup> )	Relative Std Dev (%)	Number of Samples	Replicates per Sample
Aluminum	2.6	0.9	33	3	3
Antimony	0.15	0.08	51	3	3
Arsenic	5	0.6	12	3	3
Barium	<0.3			3	3
Beryllium	<0.2			3	3
Bismuth	0.16	0.02	14	3	3
Boron	<2			3	3
Cadmium	0.12	0	0	3	3
Calcium	<1			3	3
Carbon	0.00001 <sup>(b)</sup>			3	2
Cerium	0.26	0.05	21	3	3
Cesium	1.24	0.38	30	3	3
Chloride	<20			3	1
Chromium	1.41	0.9	64	3	3
Cobalt	0.32	0.25	80	3	3
Copper	0.5 <sup>(b)</sup>			3	3
Dysprosium	<0.1			3	3
Erbium	<0.1			3	3
Europium	0.05	0	0	3	3
Fluoride	<20			3	1
Gadolinium	<0.1			3	3
Gallium <sup>(c)</sup>	1.5	0.4	28	3	2
Gallium	1.29	0.32	25	3	3
Germanium	0.31	<sup>(a)</sup>		3	3
Gold	0.52	0.28	54	3	3
Hafnium	<1			3	3
Holmium	0.05	0.01	28	3	3
Hydrogen	<0.00004			3	2
Indium	0.08	0.03	42	3	3
Iridium	<0.5			3	3
Iron <sup>(c)</sup>	20	5	25	6	4
Iron	5.2 <sup>(b)</sup>			3	3
Lanthanum	0.12	0.02	17	3	3
Lead	0.37	0.01	1.9	3	3
Lithium	<2			3	3
Lutetium	0.04	0.01	31	3	3
Magnesium	<0.4			3	3
Manganese	<0.1			3	3
Molybdenum	0.29	0.12	41	3	3
Neodymium	<0.2			3	3
Nickel	<2			3	3
Niobium	0.45	0.21	45	3	3
Nitrogen	<10			3	1
Oxygen	0.00025	0.00008	33	3	2
Palladium	0.16	0.01	8.8	3	3

**Table 3. Continued**

Analyte	Average ( $\mu\text{g g}^{-1}$ )	Std Dev ( $\mu\text{g g}^{-1}$ )	% Relative Std Dev ( $\mu\text{g g}^{-1}$ )	Number of Samples	Replicates per Sample
Platinum	3.49	3.98	114	3	3
Praseodymium	0.05	(a)		3	3
Rhenium	1.4	0.1	7.6	3	3
Rubidium	2.62	0.3	12	3	3
Ruthenium	<0.1			3	3
Samarium	<0.2			3	3
Selenium	2.12 <sup>(b)</sup>			3	3
Silicon	<16			3	3
Silver	0.9 <sup>(b)</sup>			3	3
Sodium	<29			3	3
Strontium	<0.4			3	3
Sulfate	<20			3	1
Tantalum	1.2	0.1	6.8	3	3
Tellurium	<0.6			3	3
Terbium	0.05	0.01	26	3	3
Thallium	<0.2			3	3
Tin	0.17 <sup>(b)</sup>			3	3
Titanium	4.6	0.9	20	3	3
Tungsten	62.4	1.8	2.9	3	3
Vanadium	<6			3	3
Ytterbium	<0.1			3	3
Yttrium	<0.7			3	3
Zinc	<1			3	3
Zirconium	3.20 <sup>(b)</sup>				
<b>Total Stable Impurity (<math>\mu\text{g g}^{-1}</math> metal)</b>	<b>175</b>				

<sup>(a)</sup> Impurities in the metal were measured by the C-AAC Group at LANL.

<sup>(b)</sup> Elements for which no values are reported for standard deviation (Std Dev) or percent relative standard deviation (% Relative Std Dev) had some or all measurement replicates that were below the detection limit.

<sup>(c)</sup> Duplicate entries for an element indicate independent measurement by different methods.