



Nuclear Reference Material Program

U.S. Department of Energy



Certificate of Analysis

Certified Reference Material 146

Uranium Isotopic Standard for Gamma Spectrometry

Description: Certified Reference Material (CRM) 146 consists of three sealed aluminum cylinders and one unsealed empty cylinder. Each sealed cylinder is filled with approximately 230 grams of U₃O₈ powder with nominal ²³⁵U isotope abundances of 20 %, 53 % and 93 %. The certified ²³⁵U isotope-amount and mass fraction values are reported in Table 1. Additional certified property values for the materials are provided in Table 2 for use with other nondestructive assay (NDA) techniques. Table 3 contains non-certified supplemental information values.

Table 1. ²³⁵U Certified Property Values and Uncertainties ^(a)

Cylinder Identification:	NBL	NBL	NBL
²³⁵ U Isotope-Amount	$n(^{235}\text{U})/n(\text{U})$	$n(^{235}\text{U})/n(\text{U})$	$n(^{235}\text{U})/n(\text{U})$
Fraction (•100):	20.311	52.800	93.2330
Uncertainty:	0.020	0.042	0.0053
²³⁵ U Isotope-Mass	$m(^{235}\text{U})/m(\text{U})$	$m(^{235}\text{U})/m(\text{U})$	$m(^{235}\text{U})/m(\text{U})$
Fraction (•100):	20.107	52.488	93.1703
Uncertainty:	0.020	0.042	0.0052

^(a) The last figure in the reported values and their uncertainties is provided for information purposes and is not intended to convey a significant degree of reliability.

Intended use: CRM 146 is primarily intended for use in the calibration and evaluation of gamma-ray counting procedures for the nondestructive determination of ²³⁵U isotopic abundance in bulk uranium material.

Storage: To maintain the integrity of the CRM cylinders, they should be stored in a dry, temperature-controlled location. The analysis window (on the cylinder bottom) should be protected from physical damage.

Period of validity: When stored in its original, unopened container, the certification of this material is valid indefinitely. The certification is nullified if the cylinder container is damaged or otherwise modified.

Minimum sample size: CRM 146 is intended for use as a non-destructive analysis (NDA) standard. Accordingly, a minimum samples size is not applicable.

Instructions for handling: The CRM 146 set should be handled under proper radiologically-controlled conditions at all times. This radioactive material should be handled only by qualified individuals. The cylinders should be handled with great care to avoid any damage or deformation to the bottoms of the cylinders, as this part of the cylinder was machined as a window for the emitted gamma radiation. Refer to the Safety Data Sheet for further information.

Traceability statement: The certified isotope amount ratios and isotope amount fractions are traceable to the SI unit mole. The uranium mass, uranium mass fractions, and certified isotope mass fractions are traceable to the SI unit kilogram. The certified uranium molar masses are traceable to the SI units mole and kilogram.

Table 2. Additional Certified Property Values and Uncertainties ^(a)

Cylinder Identification:	NBL	NBL	NBL
Isotope-Amount Ratio:	$n(^{234}\text{U})/n(^{238}\text{U})$ 0.0019002	$n(^{234}\text{U})/n(^{238}\text{U})$ 0.008067	$n(^{234}\text{U})/n(^{238}\text{U})$ 0.17942
Uncertainty:	0.0000050	0.000028	0.00058
²³⁴ U Isotope-Amount Fraction (•100):	$n(^{234}\text{U})/n(\text{U})$ 0.15076	$n(^{234}\text{U})/n(\text{U})$ 0.3756	$n(^{234}\text{U})/n(\text{U})$ 0.9849
Uncertainty:	0.00037	0.0011	0.0029
²³⁴ U Isotope-Mass Fraction (•100):	$m(^{234}\text{U})/m(\text{U})$ 0.14861	$m(^{234}\text{U})/m(\text{U})$ 0.3718	$m(^{234}\text{U})/m(\text{U})$ 0.9800
Uncertainty:	0.00037	0.0010	0.0029
Isotope-Amount Ratio:	$n(^{236}\text{U})/n(^{238}\text{U})$ 0.002501	$n(^{236}\text{U})/n(^{238}\text{U})$ 0.005691	$n(^{236}\text{U})/n(^{238}\text{U})$ 0.05332
Uncertainty:	0.000016	0.000013	0.00038
²³⁶ U Isotope-Amount Fraction (•100):	$n(^{236}\text{U})/n(\text{U})$ 0.1985	$n(^{236}\text{U})/n(\text{U})$ 0.26495	$n(^{236}\text{U})/n(\text{U})$ 0.2927
Uncertainty:	0.0013	0.00060	0.0022
²³⁶ U Isotope-Mass Fraction (•100):	$m(^{236}\text{U})/m(\text{U})$ 0.1973	$m(^{236}\text{U})/m(\text{U})$ 0.26451	$m(^{236}\text{U})/m(\text{U})$ 0.2937
Uncertainty:	0.0013	0.00060	0.0022
Isotope-Amount Ratio:	$n(^{235}\text{U})/n(^{238}\text{U})$ 0.25601	$n(^{235}\text{U})/n(^{238}\text{U})$ 1.1340	$n(^{235}\text{U})/n(^{238}\text{U})$ 16.984
Uncertainty:	0.00031	0.0020	0.017
²³⁸ U Isotope-Amount Fraction (•100):	$n(^{238}\text{U})/n(\text{U})$ 79.339	$n(^{238}\text{U})/n(\text{U})$ 46.560	$n(^{238}\text{U})/n(\text{U})$ 5.4895
Uncertainty:	0.020	0.043	0.0053
²³⁸ U Isotope-Mass Fraction (•100):	$m(^{238}\text{U})/m(\text{U})$ 79.547	$m(^{238}\text{U})/m(\text{U})$ 46.876	$m(^{238}\text{U})/m(\text{U})$ 5.5559
Uncertainty:	0.020	0.043	0.0053
Uranium Molar Mass:	g mol^{-1} 237.43002	g mol^{-1} 236.4428	g mol^{-1} 235.20204
Uncertainty:	0.00060	0.0012	0.00017
Uranium Mass Fraction:	g g^{-1} 0.84553	g g^{-1} 0.84286	g g^{-1} 0.84519
Uncertainty:	0.00019	0.00030	0.00074
U ₃ O ₈ Mass (total):	g	g	g
Uncertainty:			
²³⁵ U Mass (total):	g	g	g
Uncertainty:			

^(a) The last figure in the reported values and their uncertainties is provided for information purposes and is not intended to convey a significant degree of reliability.

Additional information: The aluminum cylinders were manufactured to specific dimensions and each cylinder has a unique engraved identification number and a paper label indicating the nominal ²³⁵U isotope abundance. New Brunswick Laboratory (NBL) prepared CRM 146 as a set to permit measurement of uranium-bearing materials by using the theoretically expected linear relationship between ²³⁵U isotopic abundance and the counting rate of the 185.7 keV gamma-ray from ²³⁵U. Appropriate correction factors must be applied for calibration of assay systems used to measure other types of uranium samples and containers.

CRM 146 can be used with CRM 969 (formerly NBS SRM 969/EC 171) to extend the calibration range for uranium isotope enrichment measurements from depleted (0.32 % ^{235}U) to highly enriched uranium (93 % ^{235}U). The National Bureau of Standards (NBS; now the National Institute of Standards and Technology) issued NBS Standard Reference Material (SRM) 969 in June 1985. NBS SRM 969 was produced in cooperation with the Commission of the European Communities Central Bureau for Nuclear Measurement (now Joint Research Center (JRC) Geel), Geel, Belgium and the U.S. Department of Energy New Brunswick Laboratory, Argonne, Illinois (see Reference).

The Y-12 National Security Complex, Oak Ridge, Tennessee, provided the source materials for the preparation of CRM 146. NBL accepted the materials based on available analytical data and process knowledge from Y-12. The materials were packaged into aluminum cans at NBL. The CRM 146 cans were packed to a sufficient density to assure that they would meet the “infinitely thick” criteria. NDA tests performed on each set showed a linear correlation coefficient greater than 0.999, indicating that the criterion was met.

The uranium isotope compositions and their molar masses were determined by thermal ionization mass spectrometry. Uranium isotope-amount ratio measurements were performed by two analysts, each using a different mass spectrometer. Mass discrimination correction factors applied to measured CRM 146 isotope ratios were determined from multiple analyses of matching Uranium Isotope Standards (CRM U200, CRM U500 or CRM U930D), run sequentially with CRM 146 materials. CRM U500, Uranium Isotopic Standard (50 % enriched), was used as a control to verify proper performance of the measurement system. No measurable ^{233}U was detected in CRM 146 materials. The following nuclide masses were used in calculations: ^{234}U – 234.0409447, ^{235}U – 235.0439222, ^{236}U – 236.0455610, and ^{238}U – 238.0507835. The uranium mass fraction was determined by the NBL Titrimetric Method (Modified Davies and Gray Titrimetric Method) using potassium dichromate as the titrant. The uranium equivalency of the potassium dichromate titrant was determined by titrating against CRM 112A, Uranium Metal Assay Standard. CRM 112A was also used to verify proper performance of the measurement systems. Uranium assay measurements were performed by two analysts each using independent titration systems. The U_3O_8 mass in each can of CRM 146 was determined using a two-place balance. The balance was monitored through the laboratory balance control program and checked during the packaging of the cans for accuracy and linearity by using calibrated and traceable mass standards.

Table 3. Non-Certified Property Values and Uncertainties ^(a)

Cylinder Identification:	NBL	NBL	NBL
Aluminum Window Thickness (mm):	1.9940	1.9940	1.9940
Uncertainty:	0.0052	0.0052	0.0052
Cylinder Inner Diameter (mm):	70.00	70.00	70.00
Tolerance (mm):	0.01	0.01	0.01
Material fill height (mm):	18.8	18.8	18.8
U_3O_8 Surface Density (g cm^{-3}):	5.98	5.98	5.98
U_3O_8 Density (g cm^{-3}):	3.18	3.18	3.18
Moisture Content (U_3O_8):	Insignificant	< 0.05 %	Insignificant
Volatile Substances in (U_3O_8):	< 0.2 %	< 0.3 %	Insignificant
Element Impurities ($\mu\text{g g}^{-1}$):	456	143	310
Chemical Separation Date	1990	1990	1990

In 1987, the technical and administrative transfer of Special Nuclear SRMs to the NBL reference material program was completed. 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).

Measurement uncertainty: The expanded uncertainty (U) for a certified property of CRM 146 defines a confidence interval around the value of the property. The magnitude of U is obtained by multiplying the combined standard uncertainty, u_c , by a coverage factor, k. The coverage factor is the Student’s t factor based on the effective degrees of freedom to provide a 95 % level of confidence. The combined standard uncertainties for uranium isotopic abundances consist of the standard deviations associated with isotopic ratio measurements applied as Type A evaluated components and the standard uncertainties taken from the certified values of the NBL Uranium Isotopic Standards applied as Type B evaluated components. Uncertainties for the minor isotopic abundances (^{234}U and ^{236}U) and ratios ($^{234}\text{U}/^{238}\text{U}$ and $^{236}\text{U}/^{238}\text{U}$) are augmented to accommodate possible bias in the measurement of small signal intensities by including the repeatability of the NBL Uranium Isotopic Standard measurements corrected for mass discrimination

and the associated uncertainties of these standards. The combined standard uncertainty for the uranium mass fraction consists of standard deviations associated with sample and standard measurements applied as Type A evaluated components. The combined standard uncertainty for the U_3O_8 mass in each can consists of standard deviations associated with repeated weighings of traceable mass standards and components due to the difference in the measured and reference values taken from the certificate of calibration for the mass standards, each applied as a Type A evaluated component.

Reference: NBS Special Publication 260-96, "Standard Reference Materials: Uranium-235 Isotope Abundance Standard Reference Materials for Gamma Spectrometry Measurements," by B.S. Carpenter et.al., September 1986.