Nuclear Reference Material Program
U.S. Department of Energy

Certificate of Analysis

Certified Reference Material 116A (1 g)

Uranium (Metal) Assay and Isotopic Standard, 93 % U-235, 1 gram

Description: Certified Reference Material (CRM) 116A is a uranium mass fraction and isotope-amount ratio standard. Each unit of CRM 116A consists of single uranium metal piece with a mass of approximately 1.1 gram. This CRM is not certified for absolute quantity of material which may be somewhat greater or less than the nominal mass (between 1.0 g and 1.2 g). Certified isotope composition and mass fraction values are reported in Table 1. Non-certified supplemental values are provided in an appendix.

Table 1. Certified Property Values and Uncertainties [(a)]

		$g\ g^{-1}$		**$g\ mol^{-1}$**	
Uranium Mass Fraction:		0.99945	Uranium Molar Mass:	235.18572	
Uncertainty:		0.00014	Uncertainty:	0.00011	
Uranium Isotope-Amount Ratio:	**$n(^{233}U)/n(^{235}U)$**	**$n(^{234}U)/n(^{235}U)$**	**$n(^{236}U)/n(^{235}U)$**	**$n(^{238}U)/n(^{235}U)$**	
	0.0000003863	0.0115836	0.0094713	0.051277	
Uncertainty:	0.0000000086	0.0000097	0.0000077	0.000041	
Isotope-Amount Fraction (•100):	**$n(^{233}U)/n(U)$**	**$n(^{234}U)/n(U)$**	**$n(^{235}U)/n(U)$**	**$n(^{236}U)/n(U)$**	**$n(^{238}U)/n(U)$**
	0.00003603	1.08023	93.2547	0.88324	4.7818
Uncertainty:	0.00000080	0.00089	0.0038	0.00071	0.0036
Isotope-Mass Fraction (•100):	**$m(^{233}U)/m(U)$**	**$m(^{234}U)/m(U)$**	**$m(^{235}U)/m(U)$**	**$m(^{236}U)/m(U)$**	**$m(^{238}U)/m(U)$**
	0.00003570	1.07497	93.1985	0.88647	4.8401
Uncertainty:	0.00000079	0.00088	0.0038	0.00071	0.0037

[(a)] Reported numerical uncertainties for values are expressed as expanded uncertainties ($U = k \cdot u_c$) at the 95 % level of confidence, where the expanded uncertainty (U) is the product of the combined standard uncertainty (u_c) and a coverage factor (k). Certified property values have a coverage factor of approximately 2.0 except for the mass fraction value, which has a coverage factor of 2.4 and the ^{233}U values, which have coverage factors of 3.3 for isotope-amount ratio, isotope-amount fraction, and isotope-mass fraction.

Intended use: CRM 116A is a uranium metal standard intended for use in calibration of and/or quality control for uranium analysis methods. The isotope-amount fraction and isotope-mass fraction values and uncertainties are provided primarily for information purposes. To assure proper uncertainty propagation, it is recommended that isotope-amount ratios and associated uncertainties be used for calculations incorporating CRM 116A values.

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.

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Richard Essex, Director
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Nuclear Reference Material Program
Germantown, Maryland
NRMPsales@nnsa.doe.gov

Minimum sample size: Certification/verification measurements for uranium mass fraction and isotope-amount ratios were performed on metal samples with a mass of approximately 1 gram. The homogeneity of uranium mass fraction or isotopic composition is not certified for metal pieces smaller than approximately 1 gram.

Instructions for handling: The material in the unit is radioactive. This radioactive material should be handled only by qualified individuals. To minimize personnel exposure, appropriate facilities and personal protective equipment should be used. Refer to the Safety Data Sheet for further information.
Prior to use, surface oxide must be removed to ensure accurate uranium mass fraction values. A suggested procedure is given below.

Suggested Procedure for Achieving Accurate Weighing and Mass Fraction Values

1. Soak the uranium metal sample in 8 mol L^{-1} nitric acid for 10-20 minutes to remove all visible surface oxides and impurities.
2. To ensure an accurate uranium metal weight, the following steps should be performed rapidly to minimize oxidation of the sample.
3. Thoroughly rinse the metal piece with distilled, deionized water.
4. Remove excess water by thoroughly rinsing the metal piece with pure acetone.
5. Allow the acetone to evaporate (30 – 60 seconds is typically sufficient).
6. Perform a weighing of sufficient accuracy for users need.

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

Measurement uncertainty: Uncertainties were determined according to the protocols outlined in JCGM 100:2008 Guide to the Expression of Uncertainty in Measurement [1]. The combined standard uncertainties for attribute values consist of both Type A and Type B evaluated components. For amount content, the standard deviation of high precision titrations performed on 1.1 g U metal samples and the standard uncertainty for the primary analytical amount content measurements, which utilized 3 g size U metal samples, are applied as Type A evaluated components. The combined standard uncertainty of the NBL CRM 99 oxidimetric standard is applied as a Type B evaluated component of uncertainty. For isotope-amount ratios the standard deviations associated with isotope ratio measurements of the samples and the $n(^{238}U)/n(^{235}U)$ ratio of CRMs U900 and U930D are applied as Type B evaluated components. The combined standard uncertainties for the $n(^{238}U)/n(^{235}U)$ ratios of CRMs U900 and U930D and components to account for additional sources of uncertainty associated with background corrections and analytical biases are applied as Type B evaluated components. Isotope mass fractions incorporate an additional component associated with the uncertainty of the atomic mass for the U isotopes. These are applied as Type B evaluated components. A more detailed explanation of measurement uncertainty can be obtained upon request from NRMP.

Additional information: The CRM 116A metal pieces are machined metal cylinders. The stock material for the CRM was obtained from a single casting of a HEU right-annular cylinder of metal. Several wedges of material were cut from the annular cylinder and machined into rods which were stamped into narrow-diameter rods. The rods were then machined to shape and cut into individual 1.1 g metal cylinders that comprise each CRM 116A unit.

Uranium amount content for CRM 116A was determined by the NBL High Precision Titrimetric method using CRM 99 Potassium Dichromate Oxidimetric Standard as the titrant. The CRM 112A Uranium Metal Assay and Isotopic Standard was used as a control to verify performance of the measurement system. Traceability of the measurements is primarily established by direct determination of uranium amount content based on the titration of uranium using CRM 99 Potassium Dichromate Oxidimetric Standard. CRM 99 was calibrated against CRM 112A which, in turn, was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM 960.

A detailed thermal ionization mass spectrometry measurement campaign was performed on CRM 116A to determine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample turret basis using multiple measurements of CRMs U900 and U930D. Analyses of CRM U970 Uranium Isotopic Standard were performed to verify that mass spectrometric measurements were in control. Traceability of the isotope-amount ratio measurements for CRM 116A was established by calibration of the mass spectrometers using combined measurements of CRMs U900 and U930D Uranium Isotopic Standards. CRM 900 was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM U900. U930D is directly traceable to National Bureau of Standards SRM U930 Uranium Isotopic Standard.

In 2016 NBL 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).

[1] Bureau International des Poids et Measures (BIPM), Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008

Appendix: Non-certified values presented below are from four samples of the metal casting that was machined to produce CRM 116A. Per the NRMP Quality Management System, insufficient information is available to assess the accuracy and uncertainty associated with the values.

Table A1: Mass Fraction of Impurity Element in CRM 116A Metal

Element	Top [(a)] (µg g^{-1})	Middle (µg g^{-1})	Bottom (µg g^{-1})	Unknown (µg g^{-1})	Analysis Method
Aluminum	12	12	14	13	ICP-OES
Carbon	109	108	108	115	LECO
Copper	5.3	5.1	6	5.2	ICP-OES
Chromium	25	26	27	26	ICP-OES
Iron	71	57	69	57	ICP-OES
Molybdenum	43	45	45	44	ICP-MS
Manganese	4.3	4.3	4.5	4.3	ICP-OES
Nickel	63	69	65	65	ICP-OES
Nitrogen	2.46	0.96	0.78	--	LECO
Oxygen	111	44	10	--	LECO
Silicon	57	56	54	63	ICP-OES
Tungsten	37	38	37	41	ICP-MS
Zirconium	62	69	66	73	ICP-MS

ICP-OES is inductively coupled plasma optical emission spectroscopy. ICP-MS is inductively coupled plasma mass spectrometry. LECO is a combustion-based method for measuring C, O, N, and S as gas or gaseous compounds.
(a) Top, Middle, and Bottom refer to the physical location of the samples taken from the casting. The fourth column is a sample taken at a different time, with no physical location information specified.

Table A2: Impurity Elements not detected in CRM 116A Metal

Element	MDL (µg g^{-1})	Analysis Method	Element	MDL (µg g^{-1})	Analysis Method
Arsenic	<1	ICP-MS	Niobium	<1	ICP-MS
Boron	<0.5	ICP-MS	Neodymium	<1	ICP-MS
Barium	<1	ICP-MS	Osmium	<1	ICP-MS
Beryllium	<0.2	ICP-OES	Palladium	<1	ICP-MS
Bismuth	<1	ICP-MS	Phosphorus	<20	ICP-MS
Cadmium	<0.3	ICP-MS	Platinum	<1	ICP-MS
Calcium	<5	ICP-OES	Praesidium	<1	ICP-MS
Cerium	<1	ICP-MS	Rubidium	<1	ICP-MS
Cobalt	<1	ICP-OES	Rhenium	<1	ICP-MS
Cesium	<1	ICP-MS	Rhodium	<1	ICP-MS
Dysprosium	<0.3	ICP-MS	Ruthenium	<1	ICP-MS
Erbium	<2	ICP-MS	Samarium	<0.3	ICP-MS
Europium	<0.3	ICP-MS	Scandium	<2	ICP-MS
Gallium	<1	ICP-MS	Selenium	<1	ICP-MS
Gadolinium	<0.3	ICP-MS	Silver	<1	ICP-MS
Germanium	<1	ICP-MS	Strontium	<1	ICP-MS
Hafnium	<1	ICP-MS	Tantalum	<1	ICP-MS
Holmium	<1	ICP-MS	Tellurium	<1	ICP-MS
Indium	<1	ICP-MS	Terbium	<2	ICP-OES
Iridium	<1	ICP-MS	Tin	<1	ICP-MS
Potassium	<15	ICP-MS	Titanium	<2	ICP-OES
Lanthanum	<1	ICP-MS	Thallium	<2	ICP-MS
Lead	<1	ICP-MS	Thorium	<2	ICP-MS
Lithium	<1	ICP-MS	Thulium	<1	ICP-MS
Lutecium	<1	ICP-MS	Vanadium	<1	ICP-OES
Magnesium	<1	ICP-OES	Yttrium	<5	ICP-OES
Mercury	<1	ICP-MS	Ytterbium	<1	ICP-MS
Sodium	<5	ICP-OES	Zinc	<2	ICP-OES
Sulfur	<10	LECO			

MDL is minimum detection level, defined by the equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water. ICP-OES is inductively couple plasma optical emission spectroscopy. ICP-MS is inductively coupled plasma mass spectrometry. LECO is a combustion-based method for measuring C, O, N, and S as gas or gaseous compounds.