NBL Program Office

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material CRM U630 (10mg) Uranium (U_3O_8) Isotopic Standard, 63% U-235, 10 mg U

Certified Values[1]:

	$n(^{234}U)/n(^{235}U)$	$n(^{236}U)/n(^{235}U)$	$n(^{238}U)/n(^{235}U)$	
Isotope-Amount Ratio:	0.0097698	0.0151895	0.55351	
Uncertainty:	0.0000062	0.0000097	0.00049	
	$n(^{234}U)/n(U)$	$n(^{235}U)/n(U)$	$n(^{236}U)/n(U)$	$n(^{238}U)/n(U)$
Isotope-Amount Fraction (•100):	0.61894	63.353	0.96230	35.066
Uncertainty:	0.00043	0.020	0.00067	0.020
	$m(^{234}U)/m(U)$	$m(^{235}U)/m(U)$	$m(^{236}U)/m(U)$	$m(^{238}U)/m(U)$
Isotope Mass Fraction (•100):	0.61354	63.069	0.96207	35.356
Uncertainty:	0.00043	0.020	0.00067	0.020
Molar Mass:	236.10175 $g•mol^{-1}$			
Uncertainty:	0.00061 $g•mol^{-1}$			

Reference Values[2]:

	$n(^{232}U)/n(^{235}U)$	$n(^{233}U)/n(^{235}U)$
Isotope-Amount Ratio:	$7.31•10^{-10}$	$2.372•10^{-6}$
Uncertainty:	$0.16•10^{-10}$	$0.055•10^{-6}$
Reference Date:	19 May 2014	
Uranium Amount Content	0.84661 $g•g^{-1}$	
Uncertainty:	0.00048 $g•g^{-1}$	

[1] An NBL PO Certified Value represents a result for which the NBL PO has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted and which has been determined using the modes of analysis that result in certified values.

[2] An NBL PO Reference Value represents the best estimate of the true value where all known or suspected sources of bias have not been fully investigated. An NBL PO Reference value is determined using the modes of analysis that result in reference values. A reference value represents the best information available for the quantity value and uncertainty of a measurand, but may still reflect unevaluated sources of error and uncertainty.

Notes: ***CRM U630 is a radioactive material and should be handled and stored under proper radiologically-controlled conditions at all times.***

Certified Reference Material (CRM) U630 is a uranium isotope-amount ratio standard intended for use in measurement calibration and/or quality control for analysis of high enriched uranium oxide. Each unit of CRM U630 consists of a 20 mL glass scintillation vial containing approximately 13 milligrams of uranium oxide (U_3O_8).

Reported numerical uncertainties for Certified Values are expressed as expanded uncertainties ($U = k \bullet u_c$) 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). 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. To assure proper uncertainty propagation, it is recommended that isotope-amount ratios and associated uncertainties be used for calculations incorporating U630 values.

U630 units do not have an expiration date. 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.

Certification and/or verification measurements for isotope-amount ratios were performed on aliquots as small as 10 mg of U_3O_8. Accordingly, the material homogeneity for the attribute values is not certified for samples smaller than 10 mg. Note that U630 has also been characterized for model purification date but certification analyses for this attribute were performed on 100 mg oxide samples. Therefore, 10 mg U630 units are only intended as uranium isotope-amount ratio standards. 1 g size U630 units are certified for both isotope amount ratios and model purification age.

Description:

In 2001 to 2002, a detailed thermal ionization mass spectrometry measurement (TIMS) campaign was performed on the U630 to determine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample-turret basis using multiple measurements of NBL U500 and U750 Uranium Isotopic Standards. Metrological traceability to the SI unit of mass for all ratios and abundances was established through the use and incorporation of uncertainties associated with CRM U500 and CRM U750.

Measurement Uncertainty:

Uncertainties were determined according to the protocols outlined in JCGM 100:2008 *Guide to the Expression of Uncertainty in Measurement*. The combined standard uncertainties for certified values consist of Type A and Type B components. The Type A components for isotope-amount ratios are derived from standard deviations associated with isotopic ratios measured for the samples and the $n(^{235}U)/n(^{238}U)$ ratio of NBL CRM U500 and U750. Type B components are based on the combined standard uncertainties for the $n(^{235}U)/n(^{238}U)$ ratio of CRM U500 and U750. Isotope mass fractions incorporate an additional Type B component associated with the uncertainty of the atomic mass for the U isotopes. The coverage factor (k) for each expanded uncertainty is the Student's t-factor necessary to provide a 95% level of confidence ($k \approx 2$ for all values cited in this certificate). A more detailed explanation of measurement uncertainty can be obtained upon request from NBL.

The $n(^{232}U)/n(^{235}U)$ ratio, $n(^{233}U)/n(^{235}U)$ ratio, and amount content values for this material are provided as Reference Values determined in separate studies performed from 2011 to 2013. An NBL PO Reference Value represents the best estimate of the true value where all known or suspected sources of bias have not been fully investigated. An NBL PO Reference value is determined using the modes of analysis that result in reference values. A reference value represents the best information available for the quantity value and uncertainty of a measurand, but may still reflect unevaluated sources of error and uncertainty.

The $n(^{232}U)/n(^{235}U)$ isotope amount ratio was measured by α-spectrometry with ^{232}U and ^{234}U converted from α counts to atoms using half-lives provided in the NNDC Nuclear Wallet Card Database (June 1, 2012). The resulting ratio of isotopes was normalized to ^{235}U using the previously certified CRM U630 $n(^{234}U)/n(^{235}U)$ isotope amount ratio. Measurement of the $n(^{233}U)/n(^{235}U)$ ratio was by TIMS using a combined secondary electron multiplier - Faraday Cup method with mass discrimination internally corrected using the previously certified $n(^{238}U)/n(^{235}U)$ ratio. The uranium amount content value was determined by NBL-Modified Davies & Gray Titrimetric analyses performed on oxide that was calcined at 900° C for 3 hours. This value is traceable to the CRM 112A metal standard (formerly National Bureau of Standards SRM 960) which was used to normalize the SRM 136e Potassium Dichromate solutions used for titrations. The amount-content value was verified by ceric titrations perform at Los Alamos National Laboratory.

Project Support:

Characterization analyses for U amount content, $n(^{232}U)/n(^{235}U)$ isotope-amount ratio, and $n(^{233}U)/n(^{235}U)$ isotope-amount ratio measurements were funded by the Department of Homeland Security, National Technical Nuclear Forensic Center (NTNFC) under Inter-Agency Agreement HSHQDC-10-X-00135.