NBL Program Office

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material CRM U045 (5mg)
Uranyl Nitrate Isotopic Standard, 4.5% U-235, 5 mg U in Solution Form

Certified Values[1]:

Isotopic Ratios	$^{234}U/^{238}U$	$^{235}U/^{238}U$	$^{236}U/^{238}U$
Atom Ratios	0.00040579 ± 0.00000031	0.047310 ± 0.000025	0.00028983 ± 0.00000027

Isotopic Abundance	^{234}U	^{235}U	^{236}U	^{238}U
Atom Fraction (x 100)	0.038720 ± 0.000029	4.5143 ± 0.0023	0.027655 ± 0.000025	95.4193 ± 0.0023
Mass Fraction (x 100)	0.038090 ± 0.000029	4.4599 ± 0.0023	0.027438 ± 0.000025	95.4746 ± 0.0023

Relative Atomic Mass of Uranium ……237.912937 ± 0.000070

Reference Values[2]:

Isotopic Ratios:	$^{232}U/^{238}U$	$^{233}U/^{238}U$
Atom Ratios:	$8.6 \cdot 10^{-12}$	$2.438 \cdot 10^{-7}$
	$\pm 1.3 \cdot 10^{-12}$	$\pm 0.032 \cdot 10^{-7}$
Reference Date:	19 May 2014	

Reported numerical uncertainties are expressed as expanded uncertainties (U) at the 95% level of confidence, where $U = k \cdot u_c$, 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 for information purposes only and is not intended to convey a significant degree of reliability.

[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.

This Certified Reference Material (CRM) is an isotopic standard primarily for use in the mass spectrometric analysis of uranium. Each unit of U045 contains approximately 5 mg of uranium dissolved in approximately 5 mL of a 1 M nitric acid solution. The solution is contained in a sealed 5-mL glass ampoule.

NOTE: *The ampoule should be handled under proper radiologically-controlled conditions at all times. Care should be taken when scoring and breaking the ampoule to avoid injury and possible contamination.*

CRM U045 was prepared from CRM 113-B, Uranium Hexafluoride Assay and Isotopic Standard. The solid uranium hexafluoride was hydrolyzed, converted to U_3O_8 by ignition, dissolved in nitric acid, and ampoulated. A number of filled and sealed ampules were selected according to a statistical sampling plan for certification of the uranium isotopic composition.

The uranium isotopic composition and the relative atomic mass of uranium were determined by thermal ionization mass spectrometry. Uranium isotopic ratio measurements were performed by two analysts each using a different mass spectrometer. The first instrument utilized the total evaporation method to generate values used for the certification of the $^{235}U/^{238}U$ ratio. The second instrument utilized the NBL modified total evaporation procedure to generate values for all certified ratios. CRMs U030-A, U050 and U500 were used for mass fractionation corrections and quality control. 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 U030-A. The isotopic analysis results for CRM U045 and CRM 113-B were in very good statistical agreement, with the exception of a small but statistically significant difference in the $^{236}U/^{238}U$ ratios and ^{236}U abundances. The results from the two materials were combined to yield the final certified values for isotopic composition and their uncertainties. The following relative atomic masses were used in relative atomic mass calculations: ^{234}U: 234.0409456, ^{235}U: 235.0439231, ^{236}U: 236.0455619, and ^{238}U: 238.0507826.

The expanded uncertainty (U) for a certified property of CRM U045 defines an interval around the value of the property. The magnitude of this interval is 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 uncertainties (u_c) for uranium isotopic parameters consist of Type A components derived from standard deviations associated with isotopic ratio measurements of the samples and the measurements of the $^{235}U/^{238}U$ ratio of NBL CRM U030-A, and Type B components which are based on the standard uncertainties derived from the NBL CRM U030-A certified value for the $^{235}U/^{238}U$ ratio.

The $^{232}U/^{238}U$ ratio and $^{233}U/^{238}U$ ratio values for this material are also provided as Reference Values, determined in separate studies performed from 2012 to 2013. The $^{232}U/^{238}U$ 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 ^{238}U using the previously certified CRM U045 $^{234}U/^{238}U$ isotope amount ratio. Measurement of the $^{233}U/^{238}U$ ratio was by TIMS using a combined secondary electron multiplier - Faraday Cup method with mass discrimination internally corrected using the previously certified $^{235}U/^{238}U$ ratio. This value is reported as a Reference Value and is not certified. 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.

Expiration of Certificate: When stored in its original, unopened container, the certification of this material is valid indefinitely. The NBL PO will periodically monitor the materials in inventory and notify customers should degradation be detected.

Stability and Storage: This material should be stored in its original packaging under normal laboratory environmental conditions.

Minimum Sample Size: The material is considered a pure solution, and thus no minimum sample size is declared.

Project Support: Characterization analyses for $^{232}U/^{238}U$ isotope-amount ratio and $^{233}U/^{238}U$ isotope ratio measurements were funded by the Department of Homeland Security, National Technical Nuclear Forensic Center (NTNFC) under Inter-Agency Agreement HSHQDC-10-X-00135.