New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM U630
Uranium (U_3O_8) Isotopic and Radiochronometric Standard

	$n(^{234}U)/n(^{235}U)$	$n(^{236}U)/n(^{235}U)$	$n(^{238}U)/n(^{235}U)$	
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}**	**Model Purification Date:**	**June 6, 1989**
Uncertainty:	**0.00061**	**g•mol^{-1}**	**Uncertainty:**	**190 days**

Notes:

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

Certified Reference Material U630 (CRM U630) is a uranium isotope-amount ratio and radiochromometric 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 1.2 grams of uranium oxide (U_3O_8).

Reported numerical uncertainties for certified 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). 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. The isotope-amount and 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 U630 values.

CRM 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 uranium isotope-amount ratios were performed on aliquots as small as 10 mg of U_3O_8 and model purification dates were performed on 100 mg oxide samples.

Accordingly, the material homogeneity for the attribute values is not certified for samples smaller than 10 mg for isotopic analysis or 100 mg for purification age analyses.

Amount content value for this material (0.84661±0.00048 g U•g^{-1} oxide) is provided for user information. This value was determined by NBL-Modified Davies & Gray Titrimetric analyses performed on oxide that was calcined at 900° C for 3 hours. The amount content value was verified by ceric titrations perform at Los Alamos National Laboratory.

Description:

In 2001 to 2002, a detailed thermal ionization mass spectrometry measurement campaign was performed on the CRM U630 to determine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample-turret basis using multiple measurements of NBL CRM U500 and CRM U750 Uranium Isotopic Standards. Traceability of the isotope-amount ratio measurements for CRM U630 is primarily established by calibration of the mass spectrometer using measurements of CRMs U500 and U750 Uranium Isotopic Standards that were originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM U500 and SRM U750 Uranium Isotopic Standards.

In 2011 to 2012, a detailed study was performed to determine the model U purification date for the CRM U630 oxide. The certified purification date is a derived value based on the ^{234}U-^{230}Th isotope parent daughter system (Equation 1) and is consistent with independent measurements using the ^{235}U-^{231}Pa isotope parent daughter system. The certified value represents results for combined measurements from 3 laboratories: Argonne National Laboratory facility (Bldg 203), the ACL lab at Argonne National Laboratory (Bldg 205), and New Brunswick Laboratory. The variables necessary for the derived value include ^{234}U content (N_{234U}), ^{230}Th content (N_{230Th}), and the decay constants of both isotopes (λ_{234U}, λ_{230Th}). The ^{234}U content was derived from the certified amount content and isotope mass fractions. The ^{230}Th content was determined by isotope dilution α spectrometry using ^{229}Th Radioactivity Standard, SRM 4328C, provided by the National Institute of Standards and Technology. It is the direct comparison of measured activity between ^{230}Th in the sample and the SRM 4328C ^{229}Th added to the sample that provides traceability for the measurements used to determine the certified value. The model purification date, based on the combined measurements, is provided as a fixed certified value for this material because the ^{230}Th content of CRM U630 changes continuously due to in-growth. For information purposes, the composite value for ^{230}Th content, as of January 2012 is 1.7 x 10^{-9} mols•g^{-1} U. The decay constants used for the purification date calculations are λ_{230Th} = (9.193x10^{-6} ± 0.073x10^{-6})•$year^{-1}$ and λ_{234U} = (2.823x10^{-6} ± 0.014x10^{-6})•$year^{-1}$ (calculated from half-lives provided in the NNDC Nuclear Wallet Card Database June 1, 2012).

Equation 1: $$Model\ Purification\ Date = t_{separation} - \frac{\ln\left[1-\left(\frac{\lambda_{230Th}-\lambda_{234U}}{\lambda_{234U}}\right)\times\left(\frac{N_{230Th}}{N_{234U}}\right)\right]}{(\lambda_{234U}-\lambda_{230Th})}$$

$t_{separation}$ is the date of Th separation for the analysis samples.

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 Type A uncertainty component for the Model Purification Date is derived from the standard deviation of the replicate age determinations. Type B Components include SRM 4328C certificate uncertainty, U630 amount content and isotope mass fraction uncertainties, U and Th half-life uncertainties, data correction factors, and a lab-to-lab bias factor. 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.

Project Support:

Characterization analyses for revised isotope-amount ratio measurements and the model purification date determination were funded by the Department of Homeland Security, National Technical Nuclear Forensic Center (NTNFC) under Inter-Agency Agreement HSHQDC-10-X-00135.