NBL Program Office

U.S. Department of Energy

Certificate of Analysis
Certified Reference Material C125A (5g)
Uranium (UO_2) Assay, Isotopic, and Radiochronometric Standard, 4.0% U-235
5.4 gram Pellet

Amount Content :	**0.88129**	**g U•g^{-1} pellet**		
Uncertainty:	**0.00014**	**g U•g^{-1} pellet**		
	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$	
Isotope-Amount Ratio:	**0.00039130**	**0.042301**	**0.0000040754**	
Uncertainty:	**0.00000038**	**0.000025**	**0.0000000047**	
	$n(^{234}U)/n(U)$	$n(^{235}U)/n(U)$	$n(^{236}U)/n(U)$	$n(^{238}U)/n(U)$
Isotope-Amount Fraction (•100):	**0.037528**	**4.0569**	**0.00039085**	**95.9052**
Uncertainty:	**0.000037**	**0.0023**	**0.00000045**	**0.0023**
	$m(^{234}U)/m(U)$	$m(^{235}U)/m(U)$	$m(^{236}U)/m(U)$	$m(^{238}U)/m(U)$
Isotope Mass Fraction (•100):	**0.036915**	**4.0077**	**0.00038776**	**95.9550**
Uncertainty:	**0.000036**	**0.0023**	**0.00000045**	**0.0023**

Molar Mass:	**237.927291**	**g•mol^{-1}**	**Model Purification Date:**	**August 18, 1994**
Uncertainty:	**0.000071**	**g•mol^{-1}**	**Uncertainty:**	**116 days**

CRM C125A is a radioactive material and should be handled and stored under proper radiologically-controlled conditions at all times.

Certified Reference Material C125A is a uranium amount content, isotope-amount ratio, and radiochromometric standard intended for use in calibration of and/or quality control for analysis of uranium in fabricated fuel form. Each unit of C125A consists of one enriched uranium dioxide (UO_2) pellet with a mass of approximately 5.4 grams.

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 C125A values.

Expiration of Certificate: C125A units do not have an expiration date.

Storage and Stability: 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 amount content, isotope-amount ratios, and model purification date were performed on a random sampling of UO_2 pellets. All analyses were performed on pellets that were dissolved in their entirety. Accordingly, the material homogeneity for the attribute values is not certified for samples smaller than a single UO_2 pellet.

Note that small quantities of refractory particulates have been observed following nitric acid-only sample dissolution but do not have a discernible effect on the measurement of values cited in this certificate. The total metal impurity content is estimated to be less than 80 µg/g pellet as determined by optical emission spectrometry. This impurity content value is provided for information only and is not certified. Therefore, if this material is analyzed by gravimetry, it is the responsibility of the user to determine and subtract impurity content, as necessary.

Description:

In 1994, the NBL procured UO_2 fuel pellets to serve as CRM 125A from Westinghouse Commercial Nuclear Fuels (CNFD), Analytical Services Laboratories, Columbia, SC. The NBL specified all pellets to be procured from a single production lot, from a single source lot of UF_6, with no scrap recycle. Analytical data regarding source material purity are given in the Table of Impurities below.

Uranium amount content for C125A was originally determined in 1997 by the NBL High Precision Titrimetric method using CRM 99 Potassium Dichromate Oxidimetric Standard as the titrant. The C112A Uranium Metal Assay and Isotopic Standard was used as a control to verify performance of the measurement system. In addition, gravimetric analyses were performed to verify that pellet-to-pellet inhomogeneity was negligible. 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.

In 2011 to 2012, a detailed thermal ionization mass spectrometry measurement campaign was performed on the C125A to refine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample-turret basis using multiple measurements of NBL U030A Uranium Isotopic Standard. Analyses of U045 Uranium Isotopic Standard were performed to verify that mass spectrometric measurements were in control. Traceability of the isotope-amount ratio measurements for C125A is primarily established by calibration of the mass spectrometer using measurements of U030A Uranium Isotopic Standard that was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM U030a Uranium Isotopic Standard.

In 2011 to 2012, a detailed study was performed to determine the model U purification date for the C125A pellets. 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 C125A changes continuously due to in-growth. For information purposes, the composite value for ^{230}Th content, as of January 2012, is 7.0 x 10^{-11} mols/g pellet. The decay constants used for the purification date calculations are λ_{230Th} = (9.193x10^{-6} ± 0.073x10^{-6})/year and λ_{234U} = (2.823x10^{-6} ± 0.014x10^{-6})/year (calculated from half-lives provided in the NNDC Nuclear Wallet Card Database June 1, 2012).

Equation 1: Model Purification Date:

$$t_{separation} - \frac{\ln\left[1 - \left(\frac{\lambda 230Th - \lambda 234U}{\lambda 234U}\right) \times \left(\frac{N230Th}{N234U}\right)\right]}{(\lambda 234U - \lambda 230Th)}$$

$t_{separation}$ 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 uncertainty component for amount content is derived from the standard deviation of the titrations. The Type B component is the combined standard uncertainty of the CRM 99 oxidimetric standard. 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 U030A. Type B components are based on the combined standard uncertainties for the certificate-derived $n(^{235}U)/n(^{238}U)$ ratio of U030A and components to account for additional sources of uncertainty associated with background corrections and analytical biases. 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, C125A U amount content and isotope mass fraction uncertainties, U and Th half-life uncertainties, and Th activity data correction factors. The coverage factor (k) for each expanded uncertainty is the Student's t-factor necessary to provide a 95% level of confidence (k≈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.

Table of Impurities: The table of impurities below was received from Westinghouse Commercial Nuclear Fuels (CNFD), Analytical Services Laboratories, and is believed to have been performed on samples of the source UO_2 powder prior to production of the pellets. Limited information on analytical methods was provided, though a note was included mentioning carrier distillation optical emission spectrometry. The data was reported on August 8, 1994.

Table 1. Trace Impurities Analysis Data, in ppm

Element	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Al	<10.0	<10.0	13.0	<10.0	12.0
Bi	<1.0	<1.0	<1.0	<1.0	<1.0
B	0.3	<0.3	<0.3	<0.3	<0.3
Cd	<0.3	<0.3	<0.3	<0.3	<0.3
Ca	<10	<10.0	<10.0	<10.0	<10.0
Cr	1.7	1.8	1.9	1.4	1.0
Co	<0.5	0.6	0.7	0.9	0.8
Cu	<1.0	<1.0	<1.0	<1.0	<1.0
In	<0.5	<0.5	<0.5	<0.5	<0.5
Fe	<10	<10	<10	<10	<10
Pb	<0.5	<0.5	<0.5	<0.5	<0.5
Mg	<0.5	<0.5	0.8	<0.5	<0.5
Mn	<1.0	1.3	1.4	1.1	1.0
Mo	0.9	6.4	0.9	0.8	0.7
Ni	<5.0	<5.0	<5.0	<5.0	<5.0
Si	<10	<10.0	<10.0	<10.0	<10.0
Sn	<0.5	<0.5	<0.5	<0.5	<0.5
Ti	<1.0	<1.0	<1.0	<1.0	<1.0
w	<10	<10.0	<10.0	<10.0	<10.0
V	<0.5	<0.5	<0.5	<0.5	<0.5
Zn	<1.0	<1.0	<1.0	<1.0	<1.0
Ba	<10	<10.0	<10.0	<10.0	<10.0
Na	<4.0	<4.0	<4.0	<4.0	<4.0
Zr	<50	<50	<50	<50	<50
EBC*	0.455	0.459	0.459	0.459	0.457

*EBC = "equivalent boron content" - refers to apparent cross-section