New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 129-A
Uranium Oxide (U_3O_8) Assay and Isotopic Standard

Uranium Assay (Mass Fraction) ……..0.847698 ± 0.000090 kg U/kg

Isotopic Ratios	$^{234}U/^{238}U$	$^{235}U/^{238}U$	$^{236}U/^{238}U$
Atom Ratios	0.000053350 ± 0.000000039	0.0072614 ± 0.0000039	0.000000097 ± 0.000000012

Isotopic Abundance	^{234}U	^{235}U	^{236}U	^{238}U
Atom Fraction (x 100)	0.0052962 ± 0.0000038	0.72087 ± 0.00039	0.0000097 ± 0.0000012	99.27382 ± 0.00039
Mass Fraction (x 100)	0.0052075 ± 0.0000038	0.71183 ± 0.00039	0.0000096 ± 0.0000012	99.28295 ± 0.00039

Relative Atomic Mass of Uranium ……238.028894 ± 0.000012

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.

This Certified Reference Material (CRM) is an assay (elemental concentration) and isotopic standard primarily for use in uranium determinations. Each unit of CRM 129-A contains approximately 25 grams of uranium (nominally normal) oxide (U_3O_8) contained in a glass jar. Before use, follow the recommended procedure for ignition of material.

NOTE: *The material should be handled under proper radiologically-controlled conditions at all times.*

RECOMMENDED PROCEDURE FOR IGNITION OF MATERIAL

To ensure accurate measurement results for uranium determination, CRM 129-A must be ignited in an open dish or crucible in a muffle furnace at 800°C for one hour and cooled in a desiccator prior to use. The ignition temperature, 800°C, was determined to provide the greatest weight loss stability for this specific lot of material.

The source material for CRM 129-A was prepared in 1984, at NLO, Inc., Cincinnati, OH, from a supply of highly pure UO_2 pellets. The pellets were crushed, dissolved in nitric acid, the solution precipitated with hydrogen peroxide, then filtered, dried, calcined at 900°C, milled, and screened. The final product was blended and shipped to New Brunswick Laboratory.

March 1, 2008
Argonne, Illinois

Jon Neuhoff, Director
New Brunswick Laboratory

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A random sample of the units was taken for uranium assay (elemental concentration) and isotopic abundance analyses. The uranium assay was determined by the NBL High Precision Titrimetric Method using National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 136e, Potassium Dichromate ($K_2Cr_2O_7$) Oxidimetric Standard, as the titrant. NBL CRM 112-A, Uranium Metal Assay Standard, and NBL CRM 129, Uranium Oxide (U_3O_8) Assay Standard were used as controls to verify proper performance of the measurement systems. Uranium assay measurements were performed by two analysts each using independent titration systems. Prior to titration preparation, CRM 129-A analysis samples were ignited at 800°C to constant weight to determine the recommended procedure for ignition.

The uranium isotopic composition and the relative atomic mass of uranium were determined by thermal ionization mass spectrometry (TIMS). The following relative atomic masses were used in calculations: ^{234}U - 234.0409456, ^{235}U - 235.0439231, ^{236}U - 236.0455619, and ^{238}U - 238.0507826. Uranium isotopic ratio measurements were performed by two analysts each using a different mass spectrometer. One TIMS instrument, utilizing the Total Evaporation procedure, was used to generate values for the certification of only the ^{235}U/^{238}U ratio. A second TIMS instrument, utilizing the NBL-Modified Total Evaporation procedure, was used to generate values for the ^{235}U/^{238}U ratio. The ^{234}U/^{238}U and ^{236}U/^{238}U ratios were also measured on this instrument using an energy/direction filter lens assembly and a high signal-intensity static multi-collector method with the ^{236}U measured using a secondary electron multiplier. The minor ratios were corrected internally using the ^{235}U/^{238}U ratio determined by the Total Evaporation and NBL Modified Total Evaporation methods. Mass discrimination correction factors applied to measured CRM 129-A ^{235}U/^{238}U isotopic ratios were determined from multiple analyses of NBL CRM U030-A, Uranium Isotope Standard (3% enriched), run sequentially with CRM 129-A. Measurements of NBL CRM U500, Uranium Isotopic Standard (50% enriched), were used as a control to verify proper performance of the measurement system for the ^{235}U/^{238}U measurements. Measurements of NBL CRM U010, Uranium Isotopic Standard (1.0% enriched), were used as a control to verify proper performance of the measurement system for the ^{234}U/^{238}U and ^{236}U/^{238}U measurements. Mass spectrometric measurements indicate that there is no detectable ^{233}U and no significant heterogeneity in the isotopic abundances of ^{234}U, ^{235}U, and ^{238}U within and between units. Isotopic heterogeneity was, however, observed in the abundance of ^{236}U. The uncertainties calculated for the ^{236}U isotopic abundance and ^{236}U/^{238}U isotopic ratio incorporate the observed variability.

The expanded uncertainty (U) for a certified property of CRM 129-A defines an interval around the value of the property and is calculated according to the ANSI/NCSL Guide[1]. 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 uncertainty (u_c) for uranium assay consists of Type A components derived from standard deviations associated with analyst-to-analyst differences and titration measurements; and a Type B component based on the standard uncertainty taken from the NIST SRM 136e certificate. 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 estimates of isotopic inhomogeneity of the samples; and a Type B component based on the standard uncertainty derived from the uncertainties associated with the NBL CRM U030-A certified value for the ^{235}U/^{238}U ratio.

Project coordination was provided by A. M. Voeks. High precision titrimetric assay measurements were performed by G. J. Orlowicz and A. M. Voeks. Isotopic abundance measurements were performed by R. M. Essex and S. Richter. Health physics support was provided by H. S. Gruhn. The statistical plan of analysis for assay measurements was prepared by M. D. Soriano and for isotopic measurements by R. M. Essex and S. A. Goldberg. The statistical evaluation of data was performed by M. D. Soriano and reviewed by W. C. Losinger. Technical guidance for CRM 129-A certification and issuance was provided by M. A. Legel and U. I. Narayanan. Project supervision was provided by S. A. Goldberg, U. I. Narayanan and J. W. Neuhoff.

[1] American National Standard for Calibration - U.S. Guide to the Expression of Uncertainty in Measurement [GUM], ANSI/NCSL Z540-2-1997.

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