Certificate of Analysis

Certified Reference Material 129A (25 g)

Uranium (U_3O_8) Assay and Isotopic Standard, 0.7 % U-235, 25 grams

Description: Certified Reference Material (CRM) 129A is a uranium mass fraction and isotope-amount ratio standard. Each unit of CRM 129A consists of approximately 26 grams of uranium (nominally normal isotope composition) as oxide powder (U_3O_8) contained in a glass jar. Certified isotope composition and mass fraction values are reported in Table 1.

Table 1. Certified Property Values and Uncertainties [(a)]

	g g^{-1}			**g mol^{-1}**
Uranium Mass Fraction:	0.847698	Uranium Molar Mass:		238.028894
Uncertainty:	0.000090	Uncertainty:		0.000012
	$n(^{234}U)/n(^{238}U)$	$n(^{235}U)/n(^{238}U)$	$n(^{236}U)/n(^{238}U)$	
Isotope-Amount Ratio:	0.000053350	0.0072614	0.000000097	
Uncertainty:	0.000000039	0.0000039	0.000000012	
	$n(^{234}U)/n(U)$	$n(^{235}U)/n(U)$	$n(^{236}U)/n(U)$	$n(^{238}U)/n(U)$
Isotope-Amount Fraction (•100):	0.0052962	0.72087	0.0000097	99.27382
Uncertainty:	0.0000038	0.00039	0.0000012	0.00039
	$m(^{234}U)/m(U)$	$m(^{235}U)/m(U)$	$m(^{236}U)/m(U)$	$m(^{238}U)/m(U)$
Isotope-Mass Fraction (•100):	0.0052075	0.71183	0.0000096	99.28295
Uncertainty:	0.0000038	0.00039	0.0000012	0.00039

[(a)] Reported numerical uncertainties are expressed as expanded uncertainties (U) at the 95 % level of confidence, where $U = k \bullet u_c$, 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.

Intended use: CRM 129A is a mass fraction (assay) and isotope standard primarily for use in uranium determinations. Before using, follow the recommended procedure for ignition of material.

Storage: 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.

Period of validity: When stored in its original, unopened container, the certification of this material is valid indefinitely. The certification is nullified if the material or container is damaged, contaminated or otherwise modified. The NRMP will notify customers should degradation be detected.

Minimum sample size: The NRMP does not guarantee uranium elemental assay for oxide samples smaller than 1 g or uranium isotopic homogeneity for oxide samples smaller than 10 mg.

(Revision of Certificate Dated November 2020)
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January 2026

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Instructions for handling: The material in the unit is radioactive. The material should be handled under proper radiologically-controlled conditions at all times. This radioactive material should be handled only by qualified individuals. To minimize personnel exposure, appropriate facilities and personal protective equipment should be used. Refer to the Safety Data Sheet for further information.

Required Procedure for Ignition of Material

To ensure accurate measurement results for uranium mass fraction determination, CRM 129A 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.

Traceability statement: The certified isotope amount ratios and isotope amount fractions are traceable to the SI unit mole. The certified isotope mass fractions and uranium mass fraction are traceable to the SI unit kilogram. The certified uranium molar mass is traceable to the SI units mole and kilogram.

Measurement uncertainty: The expanded uncertainty (U) for a certified property values of CRM 129A 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 the standard deviations associated with analyst-to-analyst differences and titration measurements applied as Type A evaluated components; and the standard uncertainty taken from the NIST SRM 136e certificate applied as a Type B evaluated component. The combined standard uncertainties (u_c) for uranium isotopic parameters consist of the standard deviations associated with isotope amount ratio measurements of the samples and the measurements of the $^{235}U/^{238}U$ ratio of CRM U030A, and estimates of isotopic inhomogeneity of the samples, all applied as Type A evaluated components; and on the standard uncertainty derived from the uncertainties associated with the CRM U030A certified value for the $^{235}U/^{238}U$ ratio applied as a Type B evaluated component.

Additional information: The source material for CRM 129A 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.

A random sample of the units was taken for uranium mass fraction (assay) and isotope-amount ratio 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. CRM 112A, Uranium Metal Assay Standard, and CRM 129, Uranium Oxide (U_3O_8) Assay Standards were used as controls to verify proper performance of the measurement systems. Uranium mass fraction measurements were performed by two analysts, each using independent titration systems. Prior to preparation for titration, CRM 129A analysis samples were ignited at 800 °C to constant weight.

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 isotope-amount ratio measurements were performed by two analysts, each using a different mass spectrometer. One TIMS instrument, utilized the Total Evaporation procedure to measure values for the certification of only the $^{235}U/^{238}U$ ratio. A second TIMS instrument, used the NBL-Modified Total Evaporation procedure to measure 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 129A $^{235}U/^{238}U$ isotopic ratios were determined from multiple analyses of CRM U030A, Uranium Isotope Standard (3 % enriched), run sequentially with CRM 129A. Measurements of 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 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.

In 2016, the New Brunswick Laboratory facility was transitioned to a program office within the Department of Energy and is now operating within the National Nuclear Security Administration (NNSA) as the Nuclear Reference Material Program (NRMP).

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