NBL Program Office

U.S. Department of Energy

Certificate of Analysis

Certified Reference Material C129A (1g)
Uranium Oxide (U_3O_8) Assay and Isotopic Standard, 0.7% U-235, 1 gram

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 C129A contains approximately 1 gram 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.*

REQUIRED PROCEDURE FOR IGNITION OF MATERIAL

To ensure accurate measurement results for uranium determination, C129A 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 C129A 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 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 C112A, Uranium Metal Assay Standard, and NBL C129, 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, C129A 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 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 C129A $^{235}U/^{238}U$ isotopic ratios were determined from multiple analyses of NBL U030A, Uranium Isotope Standard (3% enriched), run sequentially with C129A. Measurements of NBL 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 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 C129A 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 U030A, 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 U030A certified value for the $^{235}U/^{238}U$ ratio.

Expiration of Certificate: The NBL Program Office has produced and evaluated uranium oxide reference materials stored for extended periods, exceeding 40 years. 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 NBL PO will periodically monitor the materials in inventory and notify customers should degradation be detected.

Stability and 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.

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