# NBL Program Office
*U.S. Department of Energy*

# Certificate of Analysis
## Certified Reference Material C116A (1g)
## Uranium (Metal) Assay and Isotopic Standard, 93% U-235, 1 gram

**Certified Property Values**

| Amount Content | Value | Expanded[1] Uncertainty | Isotope-Amount Ratio | Value | Expanded[1] Uncertainty |
|---|---|---|---|---|---|
| g U•g$^{-1}$ metal | 0.99945 | 0.00014 | $n(^{233}U)/n(^{235}U)$ | 0.0000003863 | 0.0000000086 |
| | | | $n(^{234}U)/n(^{235}U)$ | 0.0115836 | 0.0000097 |
| Molar Mass | Value | Expanded[1] Uncertainty | $n(^{236}U)/n(^{235}U)$ | 0.0094713 | 0.0000077 |
| g•mol$^{-1}$ | 235.18572 | 0.00011 | $n(^{238}U)/n(^{235}U)$ | 0.051277 | 0.000041 |
| Isotope-Amount Fraction (•100) | Value | Expanded[1] Uncertainty | Isotope Mass Fraction (•100) | Value | Expanded[1] Uncertainty |
| $n(^{233}U)/n(U)$ | 0.00003603 | 0.00000080 | $m(^{233}U)/m(U)$ | 0.00003570 | 0.00000079 |
| $n(^{234}U)/n(U)$ | 1.08023 | 0.00089 | $m(^{234}U)/m(U)$ | 1.07497 | 0.00088 |
| $n(^{235}U)/n(U)$ | 93.2547 | 0.0038 | $m(^{235}U)/m(U)$ | 93.1985 | 0.0038 |
| $n(^{236}U)/n(U)$ | 0.88324 | 0.00071 | $m(^{236}U)/m(U)$ | 0.88647 | 0.00071 |
| $n(^{238}U)/n(U)$ | 4.7818 | 0.0036 | $m(^{238}U)/m(U)$ | 4.8401 | 0.0037 |

[1] Expanded uncertainties for certified property values have a coverage factor of approximately 2.0 with the exception of the amount content value which has a coverage factor of 2.4 and the $^{233}U$ values which have a coverage factor of 3.3 for isotope amount ratio, isotope-amount fraction, and isotope mass fraction.

Certified Reference Material C116A is a uranium amount content and isotope-amount ratio standard intended for use in calibration of and/or quality control for uranium analysis methods. Each unit of C116A consists of a metal piece with a mass of approximately 1.1 grams. This CRM is not characterized for total quantity of material which may be somewhat greater or less than the nominal mass (between 1.0 g and 1.2 g).

***C116A is a radioactive material and should be handled and stored under proper radiologically- controlled conditions at all times.***

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C116A units do not have an expiration date. To maintain the integrity of an unused unit, it should remain in the original packaging and should be stored in a dry, temperature-controlled location.

Measurements for uranium amount content and isotope-amount ratios were performed on metal samples with a mass of 1.1 gram or greater. The homogeneity of uranium amount content or isotopic composition has not been assessed for metal pieces smaller than 1.1 gram. Prior to use, surface oxide must be removed to ensure accurate uranium amount content values. A suggested procedure is provided below.

**Suggested Preparation Procedure for Achieving Accurate Mass and Amount Content Values**

1. Cover the uranium metal sample in 8 $mol \bullet L^{-1}$ nitric acid for 10-20 minutes to remove all visible surface oxides.
2. To minimize oxidation of the sample and ensure an accurate determination of uranium metal mass, the following steps should be performed immediately following Step 1.
   2.1 Thoroughly rinse the metal piece with distilled, deionized water.
   2.2 Remove excess water by thoroughly rinsing the metal piece with pure acetone.
   2.3 Allow the acetone to evaporate (30 – 60 seconds is typically sufficient).
   2.4 Perform a weighing of sufficient accuracy and precision for user's need.

**Description:**

The C116A metal pieces are machined metal cylinders. The stock material for the CRM was obtained from a single casting of a HEU right-annular cylinder of metal. Several wedges of material were cut from the annular cylinder and machined into rods which were stamped into narrow-diameter rods. The rods were then machined to shape and cut into the individual 1.1-gram metal cylinders that comprise each C116A unit.

Uranium amount content for C116A was determined 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.
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. CRM 99 was calibrated against C112A which, in turn, was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM 960.

A detailed thermal ionization mass spectrometry measurement campaign was performed on C116A to determine uranium isotope-amount ratios and uncertainties. Mass discrimination calibrations were performed on a sample turret basis using multiple measurements of NBL Uranium Isotopic Standards U900 and U930D. Analyses of U970 Uranium Isotopic Standard were performed to verify that mass spectrometric measurements were in control. Traceability of the isotope-amount ratio measurements for C116A was established by calibration of the mass spectrometers using combined measurements of CRMs U900 and U930D Uranium Isotopic Standards. CRM 900 was originally provided by the National Bureau of Standards (now known as the National Institute of Standards and Technology) as SRM U900. U930D is directly traceable to National Bureau of Standards SRM U930 Uranium Isotopic Standard.

**Measurement Uncertainty:**

Reported numerical uncertainties for values are expressed as expanded uncertainties ($U = k \bullet u_c$) 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 reported values and uncertainties is provided for information purposes and is not intended to convey a significant degree of reliability. The isotope- amount and weight 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 C116A values.

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 attribute values consist of Type A and Type B components. The Type A uncertainty components for amount content is derived from the standard

deviation of high precision titrations performed on 1.1 g U metal samples and the standard uncertainty for the primary analytical amount content measurements, which utilized 3-g U metal samples. 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 ratio measurements of the samples and the $n(^{238}U)/n(^{235}U)$ ratio of NBL CRMs U900 and U930D. Type B components are based on the combined standard uncertainties for the $n(^{238}U)/n(^{235}U)$ ratios of CRMs U900 and U930D 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 coverage factor (k) for each expanded uncertainty is based on the effective degrees of freedom for that quantity and is the Student's t-factor necessary to provide a 95% level of confidence ($k \approx 2.0$ for the values cited in this certificate except for the amount content value with $k = 2.4$ and the $^{233}U$ isotope amount ratio, amount fraction, and mass fraction which have coverage factors of $k = 3.3$). A more detailed explanation of measurement uncertainty can be obtained upon request from NBL.

**Expiration of Certificate:** The NBL Program Office has produced and evaluated uranium metal 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:** This material should be stored in its original packaging under normal laboratory environmental conditions. Presence of water vapor may speed surface oxidation. The material as sold is packaged in a PFA snap-cap plastic container, which is sealed in an aluminized pouch which serves as a vapor barrier. The pouch is inside a cardboard slip-top mailer tube.

**References:**

Bureau International des Poids et Measures (BIPM), Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement, JCGM 100:2008

**Information Values**

The information below is provided on the basis that it will be of interest and use to the user, but insufficient information is available to assess the accuracy and uncertainty associated with the values. Per the NBL Program Office Quality Management System, values below have been derived from data generated by a collaborating laboratory but lack sufficient information to assess the uncertainty.

The data presented below is from samples taken of the actual casting used to produce CRM 116A. Prior to production of CRM 116A, a homogeneity study of a different casting was performed using samples taken from more than 40 locations on the casting to ensure the casting process yielded sufficient homogeneity to meet the CRM 116A production requirements.

The analysis data below is taken from four samples of the metal casting that was machined to produce CRM 116A. The first three columns represent the physical location of the samples taken from the casting. The fourth column is a sample taken at a different time, with no physical location information available.

Table 1: Elements with detectable measurements, mass fraction as micrograms per gram.

| Element | Location in Casting | | | | Method |
|---|---|---|---|---|---|
| | Top | Middle | Bottom | Unknown | |
| Mo | 43 | 45 | 45 | 44 | ICP-MS |
| W | 37 | 38 | 37 | 41 | ICP-MS |
| Zr | 62 | 69 | 66 | 73 | ICP-MS |
| Al | 12 | 12 | 14 | 13 | ICP-OES |
| Cr | 25 | 26 | 27 | 26 | ICP-OES |
| Cu | 5.3 | 5.1 | 6 | 5.2 | ICP-OES |
| Fe | 71 | 57 | 69 | 57 | ICP-OES |
| Mn | 4.3 | 4.3 | 4.5 | 4.3 | ICP-OES |
| Ni | 63 | 69 | 65 | 65 | ICP-OES |
| Si | 57 | 56 | 54 | 63 | ICP-OES |
| **Total Measured:** | 380 | 381 | 388 | 392 | |

Table 2: Non-metals, mass fraction as micrograms per gram

| Element | Location in Casting | | | | Method |
|---|---|---|---|---|---|
| | Top | Middle | Bottom | Unknown | |
| C | 107 | 110 | 108 | 111 | LECO |
| | 111 | 105 | 108 | 118 | |
| N | 2.72 | 1.46 | 1.72 | - | LECO |
| | 2.19 | 0.76 | 0 | - | |
| | - | 0.68 | 0.63 | - | |
| O | 112 | 61.49 | 17.5 | - | LECO |
| | 110.3 | 29.38 | 3.54 | - | |
| | - | 41.98 | 10 | - | |
| S | - | - | - | <10 | LECO |

Table 3: Elements measured but not detectable, with the MDL defined by the concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

| Element | MDL | Method | Element | MDL | Method |
|---|---|---|---|---|---|
| Ag | <1 | ICP-MS | Nb | <1 | ICP-MS |
| As | <1 | ICP-MS | Nd | <1 | ICP-MS |
| B | <0.5 | ICP-MS | Os | <1 | ICP-MS |
| Ba | <1 | ICP-MS | P | <20 | ICP-MS |
| Be | <0.2 | ICP-OES | Pb | <1 | ICP-MS |
| Bi | <1 | ICP-MS | Pd | <1 | ICP-MS |
| Ca | <5 | ICP-OES | Pr | <1 | ICP-MS |
| Cd | <0.3 | ICP-MS | Pt | <1 | ICP-MS |
| Ce | <1 | ICP-MS | Rb | <1 | ICP-MS |
| Co | <1 | ICP-OES | Re | <1 | ICP-MS |
| Cs | <1 | ICP-MS | Rh | <1 | ICP-MS |
| Dy | <0.3 | ICP-MS | Ru | <1 | ICP-MS |
| Er | <2 | ICP-MS | Sc | <2 | ICP-MS |
| Eu | <0.3 | ICP-MS | Se | <1 | ICP-MS |
| Ga | <1 | ICP-MS | Sm | <0.3 | ICP-MS |
| Gd | <0.3 | ICP-MS | Sn | <1 | ICP-MS |
| Ge | <1 | ICP-MS | Sr | <1 | ICP-MS |
| Hf | <1 | ICP-MS | Ta | <1 | ICP-MS |
| Hg | <1 | ICP-MS | Tb | <2 | ICP-OES |
| Ho | <1 | ICP-MS | Te | <1 | ICP-MS |
| In | <1 | ICP-MS | Th | <2 | ICP-MS |
| Ir | <1 | ICP-MS | Ti | <2 | ICP-OES |
| K | <15 | ICP-MS | Tl | <2 | ICP-MS |
| La | <1 | ICP-MS | Tm | <1 | ICP-MS |
| Li | <1 | ICP-MS | V | <1 | ICP-OES |
| Lu | <1 | ICP-MS | Y | <5 | ICP-OES |
| Mg | <1 | ICP-OES | Yb | <1 | ICP-MS |
| Na | <5 | ICP-OES | Zn | <2 | ICP-OES |