



# Nuclear Reference Material Program

## U.S. Department of Energy



# Certificate of Analysis

## Certified Reference Material 112A (4 g)

### Uranium (Metal) Assay and Isotopic Standard, 0.7 % U-235, 4 grams

**Description:** Certified Reference Material (CRM) 112A is a uranium mass fraction and isotope-amount ratio standard. Each unit of CRM 112A consists of single uranium metal piece with a mass of approximately 4 grams. Certified isotope composition and mass fraction values are reported in Table 1.

**Table 1. Certified Property Values and Uncertainties** <sup>(a) (b)</sup>

	$\text{g g}^{-1}$		$\text{g mol}^{-1}$
Uranium Mass Fraction:	0.99975	Uranium Molar Mass:	238.028918
Uncertainty:	0.00006	Uncertainty:	0.000012
	$n(^{234}\text{U})/n(^{238}\text{U})$		$n(^{235}\text{U})/n(^{238}\text{U})$
Isotope-Amount Ratio:	0.000052841		0.0072543
Uncertainty:	0.000000082		0.0000040
	$n(^{234}\text{U})/n(\text{U})$		$n(^{235}\text{U})/n(\text{U})$
Isotope-Amount Fraction ( $\cdot 100$ ):	0.0052458		$n(^{238}\text{U})/n(\text{U})$
Uncertainty:	0.0000081		99.27458
	$m(^{234}\text{U})/m(\text{U})$		0.00039
Isotope-Mass Fraction ( $\cdot 100$ ):	0.0051579		0.00039
Uncertainty:	0.0000080		99.28370
			$m(^{238}\text{U})/m(\text{U})$
			0.00038

<sup>(a)</sup> The uncertainty assigned to the certified assay value is the 95 % confidence limit for the mean. This limit includes components due to both random analytical variability and allowances for all known and quantified sources of systematic uncertainties. 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 ( $k$ ).

<sup>(b)</sup>  $^{233}\text{U}$  and  $^{236}\text{U}$  were not detected. The limit of detection of uranium ratios for the technique used is  $5 \times 10^{-9}$ .

**Intended use:** Certified Reference Material (CRM) 112A is a uranium metal standard intended for use in calibration of and/or quality control for uranium analysis methods. The isotope-amount fraction and isotope-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 certified values.

**Storage:** This material should be stored in its original packaging under normal laboratory environmental conditions. Presence of water vapor may speed surface oxidation.

**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 National Nuclear Security Administration's (NNSA) Nuclear Reference Material Program (NRMP) will notify customers should degradation be detected.

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**Minimum sample size:** Uranium isotopic certification measurements were performed on a random sampling of metal pieces of one (1) gram. NRMP does not guarantee uranium isotopic homogeneity for metal pieces smaller than one gram.

**Instructions for handling:** The material in the unit is radioactive. 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 SDS for further information.

Prior to use, surface oxide must be removed to ensure accurate uranium assay values. A suggested procedure is given below.

**Suggested Procedure for Achieving Accurate Weighing and Assay Values**

1. Soak the uranium metal sample in 8 M nitric acid for 10-20 minutes to remove all visible surface oxides and impurities.
2. To ensure an accurate uranium metal weight, the following steps should be performed rapidly to minimize oxidation of the sample.
3. Thoroughly rinse the metal piece with distilled, deionized water.
4. Remove excess water by thoroughly rinsing the metal piece with pure acetone.
5. Allow the acetone to evaporate (30 – 60 seconds is typically sufficient).
6. Perform a weighing of sufficient accuracy for user's need.

**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 uncertainties for the uranium isotopic and molar mass values are expressed as expanded uncertainties (U) as  $U = k \cdot u_c$  where  $u_c$  is the combined standard uncertainty and k is the coverage factor ( $k = 2$ ). Uncertainties were determined according to the JCGM 100:2008 Guide to the Expression of Uncertainty in Measurement. The coverage factor of 2 was chosen to provide an approximate 95 % level of confidence. The input quantities associated with the uranium isotopic composition included uncertainties from the certified value for CRM U030A, measurement precision, and background corrections associated with the analytical techniques.

**Additional information:** CRM 112A was originally issued in 1972 by the National Bureau of Standards (now the National Institute of Standards and Technology (NIST)) as Standard Reference Material (SRM) 960. The material was produced and shipped to NBL, at the request of the NBL, by the Mallinckrodt Chemical Works (MCW) factory in St. Louis, Missouri. The MCW factory used the 'direct- ingot' or 'dingot' technique which produces high purity massive uranium metals with low carbon content. The dingot was shipped to NBL in 1959 and subsequently cut and processed at National Lead of Ohio prior to certification by NIST. Details of the production of the material may be found in NRMP report NBL-RM-2010-CRM 112A.

The measurements leading to the certification of the uranium assay were performed at NIST. The uranium assay was determined using a constant-current coulometric reduction of uranyl ions with electrogenerated titanous ions in dilute sulfuric acid. A correction was made for iron and vanadium content of the material. The total estimated impurities in the CRM ( $223 \mu\text{g g}^{-1}$ ) yield a calculated uranium assay value of  $0.99978 \text{ g g}^{-1}$ . In 1998, the standard material was repackaged and verified for uranium assay and atomic mass. In 2010, the material was again repackaged, and assay verification measurements were performed at the New Brunswick Laboratory. The certified isotope-amount ratio property values were determined in a measurements project performed at NBL in 2010. Eight (8) samples of CRM 112A were analyzed by thermal ionization mass spectrometry using three measurement techniques; total evaporation, modified total evaporation, and static analysis with internal normalization. CRM U030A was used as a calibration material for all analyses and CRM 129A was measured for quality control. In 2021 the U mass fraction of CRM 112A was verified at Oak Ridge National Laboratory (ORNL) by high precision titration and in 2023 another batch of 26 g CRM 112A units repackaged at ORNL under the direction of NRMP.

In 1987, the technical and administrative transfer of Special Nuclear SRMs to NBL was completed. In 2016 NBL 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).