



Nuclear Reference Material Program

U.S. Department of Energy



Certificate of Analysis

Certified Reference Material 111A

Uranium (U-233) in Nitrate Form Spike Assay and Isotopic Solution Standard

Description: Certified Reference Material (CRM) 111A consists of approximately 5 milligrams of uranium dissolved in 10 grams of 0.8 mol L⁻¹ HNO₃ contained in a sealed glass ampoule. Certified property values for the standard are provided in Table 1. Supplemental non-certified property values are provided in Table 2.

Table 1. Certified Property Values and Uncertainties ^(a)

Uranium Molality:	$\mu\text{mol g}^{-1}$					Uranium Molar Mass:	g mol^{-1}
Uncertainty:	2.06684						233.0550
	0.00052						0.0019
Isotope-Amount Fraction ($\bullet 100$):	$n(^{233}\text{U})/n(\text{U})$	$n(^{234}\text{U})/n(\text{U})$	$n(^{235}\text{U})/n(\text{U})$	$n(^{236}\text{U})/n(\text{U})$	$n(^{238}\text{U})/n(\text{U})$		
Uncertainty:	99.4911	0.1847	0.0790	0.0166	0.2286		
	0.0006	0.0002	0.0002	0.0002	0.0004		

^(a) The uncertainty assigned to the certified assay value is the 95 % confidence limit for the means. The uncertainty assigned to the uranium assay includes components due to analytical variation. The uncertainties assigned to the isotopic abundance values include random measurement variations and a component based on the uncertainties associated with the determination of the mass discrimination correction factors.

Intended use: This Certified Reference Material (CRM) is intended for use as a spike in the analysis of uranium materials by isotope dilution mass spectrometry (IDMS).

Storage: This material should be stored in its original packaging under normal laboratory environmental conditions.

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

Minimum sample size: CRM 111A is considered a pure solution, and thus no minimum sample size is declared.

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

Recommended Procedure for Using CRM 111A

The reference material is designed to prepare a solution having a known molality of uranium on a mass basis. Once prepared, it is suggested that all of the solution be immediately distributed as subportions for later use as individual spikes. After the spike has been added to a sample and isotopic equilibrium has been assured, a chemical separation should be performed to remove isobaric isotopes before mass spectrometric analysis.

Shake the ampoule vigorously before opening to homogenize contents. Secure in an upright position and allow solution trapped in the ampoule tip to drain back into the reservoir. Wrap the ampoule in a small cloth or towel and

(Revision of Certificate Dated December 2020)
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January 2026

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break off the tip at the pre-scored line. Discard the tip, transfer the solution to a tared container, and proceed to distribute it as weighed portions into suitable containers for use as spikes.

If a more dilute solution is desired, transfer the solution to a larger tared container, weigh, dilute and weigh again, mix vigorously, and distribute all the solution as weighed portions.

Traceability statement: The certified isotope-amount fractions are traceable to the SI unit of mole. The certified uranium molality is traceable to the SI units mole and kilogram.

Additional information: The uranium elemental molality was determined by the NBL-modified Davies-Gray titrimetric method [Reference: A. R. Eberle, M. W. Lerner, C. G. Goldbeck, and C. J. Rodden, NBL-252 (1970), pp.1-25] using CRM 112A (uranium (normal) metal assay standard) as a control to verify the measurement system. The uranium isotopic composition was determined by thermal ionization mass spectrometry using CRM 111 (U-233 spike) to demonstrate compatibility. The uranium assay value obtained by chemical analysis was verified by IDMS using CRM 115 (uranium (depleted) metal assay standard) and CRM 135 (U-235 spike) as spikes. For both the isotopic abundance and the IDMS assay measurements, mass discrimination correction factors were established using CRM U500 and were applied to the calculations. The total impurity content was less than 1600 $\mu\text{g g}^{-1}$ U as determined by inductively coupled plasma atomic emission spectrometry on a subsample of the master solution (Table 2).

Table 2. Non-Certified Property Values

Impurity	Mass Fraction ($\mu\text{g g}^{-1}$)
Calcium	90
Iron	1330
Magnesium	50
Sodium	50
Zinc	10

In 2016 the New Brunswick Laboratory 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).