New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 111-A
Uranium-233 in Nitrate Form
Uranium Spike Assay and Isotopic Solution Standard

Uranium Concentration : 2.06684 ± 0.00052 µmoles U/gram solution

	U-233	U-234	U-235	U-236	U-238
Atom Percent:	**99.4911**	**0.1847**	**0.0790**	**0.0166**	**0.2286**
Uncertainty:	**0.0006**	**0.0002**	**0.0002**	**0.0002**	**0.0004**

Relative Atomic Weight : 233.055

This Certified Reference Material (CRM) is intended for use as a spike in the analysis of uranium materials by isotope dilution mass spectrometry (IDMS). Each unit of CRM 111-A consists of approximately 5 milligrams of uranium dissolved in 10 grams of 0.8 N $HN0_3$. This solution is contained in a sealed glass ampoule.

NOTE: *The ampoule should be handled under proper radiologically-controlled conditions at all times.*

The indicated uncertainties for the certified values are 95% confidence intervals 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.

The uranium elemental concentration 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 NBL CRM 112-A (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 NBL CRM 111 (U-233 spike) to demonstrate compatibility. The uranium assay value obtained by chemical analysis was verified by IDMS using NBL CRM 115 (uranium (depleted) metal assay standard) and NBL CRM 135 (U-235 spike) as spikes. For both the isotopic abundance and the IDMS assay measurements, mass discrimination correction factors were established using NBL CRM U500 and were applied to the calculations. The total impurity content was less than 1600 µg/g U as determined by inductively-coupled plasma atomic emission spectrometry on a subsample of the master

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solution. Iron is the dominant impurity at 1330 μg/g U. Calcium, zinc, sodium, and magnesium were also found at 90, 50,50, and 10 μg/g U respectively. These impurity values are provided for information only; the impurity values are not certified.

The starting material from which this CRM was prepared was supplied by Argonne National Laboratory (ANL). Preparation and packaging of CRM 111-A were carried out by V. E. Connolly, M. I. Spaletto, F. P. Orlowicz, and F. E. Jones, NBL. Titrimetric assay measurements were performed by M. I. Spaletto, NBL; IDMS assay measurements were performed by V. E. Connolly, NBL; isotopic abundance measurements were performed by M. A. Legel and F. E. Jones, NBL; impurity measurements were performed by E. A. Huff and associates, ANL; statistical assessment of the data for certification was performed by M. D. Soriano, NBL. Project technical direction was provided by V. E. Connolly, NBL; overall technical direction and coordination of the preparation, certification, and issuance of this CRM was provided by N. M. Trahey, NBL.

RECOMMENDED PROCEDURE FOR USING CRM 111-A

The package is designed to prepare a solution having a known concentration of uranium on a weight basis. Once prepared, it is suggested that all 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 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.

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