



New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis

CRM 113-B

Uranium Hexafluoride Assay and Isotopic Standard

Uranium Assay (mass fraction): 0.67579 ± 0.00022 g/g

| | $^{234}\text{U}/^{238}\text{U}$ | $^{235}\text{U}/^{238}\text{U}$ | $^{236}\text{U}/^{238}\text{U}$ |
|---------------------|---------------------------------|---------------------------------|---------------------------------|
| Atom Ratio: | 0.00040579 | 0.047310 | 0.00028983 |
| Uncertainty: | 0.00000031 | 0.000025 | 0.00000027 |

| | ^{234}U | ^{235}U | ^{236}U | ^{238}U |
|------------------------------|------------------|------------------|------------------|------------------|
| Atom Fraction (x100): | 0.038720 | 4.5143 | 0.027655 | 95.4193 |
| Uncertainty: | 0.000029 | 0.0023 | 0.000025 | 0.0023 |
| Weight Fraction: | 0.038090 | 4.4599 | 0.027438 | 95.4746 |
| Uncertainty: | 0.000029 | 0.0023 | 0.000025 | 0.0023 |

Relative Atomic Mass of Uranium: 237.912937 ± 0.000070

Reported numerical uncertainties are expressed as expanded uncertainties (U) at the 95% level of confidence, where $U = k u_c$ and 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 the analysis of uranium feed and product materials associated with the uranium enrichment process. Each unit of CRM 113-B consists of uranium (4.5% enriched) hexafluoride (UF_6) contained in a 2S cylinder. For use as a concentration standard, material is normally transferred from the individual 2S cylinders into approved sample tubes.

NOTE: *The 2S cylinders should be handled under proper radiologically-controlled conditions at all times.*

The source material for CRM 113-B was UF_6 contained in a single 30B cylinder. Material for the CRM was transferred into fifteen new 2S cylinders by the Portsmouth Gaseous Diffusion Plant, Piketon, Ohio. Samples for certification measurements were taken from all of the cylinders and placed into P-10 tubes. The weight of the UF_6 in the P-10 tubes was corrected for the effects of cover gas trapped over

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the UF₆ in the tube and for air buoyancy as follows:

$$\text{Corrected Sample Weight (g)} = (1.00047) (\text{Observed Sample Weight, g}) - 0.0058.$$

This equation is applicable for sample weights in the range of 7 to 13 g (1). The uranium assay was determined by the NBL high precision titration using NBL CRM 99, Potassium Dichromate Oxidimetric Standard, as the titrant. NBL CRM 112-A, Uranium Metal Assay Standard, was used as a control to verify proper performance of the measurement systems; uranium hexafluoride samples from the NBL Safeguards Measurement Evaluation Program were also used to verify proper sample handling and analysis. Uranium assay measurements were performed by two analysts each using independent titration systems. The uranium isotopic composition and the relative atomic mass were determined by thermal ionization mass spectrometry. The following relative atomic masses were used in calculations: ²³⁴U - 234.0409456, ²³⁵U - 235.0439231, ²³⁶U - 236.0455619, and ²³⁸U - 238.0507826. Uranium isotopic ratio measurements were performed by two analysts each using two different mass spectrometers. The first instrument, utilizing a Total Evaporation procedure, was used to generate values used for the certification of the ²³⁵U/²³⁸U ratio only. A second instrument, utilizing the NBL-Modified Total Evaporation procedure, was used to generate values for all certified ratios. The minor ratios were corrected internally using the ²³⁵U/²³⁸U ratio of the same sample. Mass discrimination correction factors applied to measured CRM 113-B isotopic ratios were determined from multiple analyses of NBL CRM U030-A, Uranium Isotopic Standard (3% enriched), run sequentially with CRM 113-B. Measurements of NBL CRM U050, Uranium Isotopic Standard (5% enriched) and NBL CRM U500, Uranium Isotopic Standard (50% enriched), were used as controls to verify the measurement system. No measurable ²³³U was detected in CRM 113-B.

The expanded uncertainty (U) for a certified property of CRM 113-B defines an interval around the value of the property. The magnitude of this interval is obtained by multiplying the combined standard uncertainty by a coverage factor. The coverage factor (k), is the Student's t factor based on effective degrees of freedom to provide a 95% level of confidence. The combined standard uncertainty for uranium assay consists of Type A components derived from standard deviations associated with cylinder-to-cylinder differences, tube-to-tube differences, sample preparation and titration measurements; and of Type B components which are based on the standard uncertainties taken from the NBL CRM 99 certificate, and cover gas and air buoyancy corrections for UF₆ samples in the P-10 tubes. The combined standard uncertainties 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 ²³⁵U/²³⁸U ratio of NBL CRM U030-A, and Type B components which are based on the standard uncertainties derived from the NBL CRM U030-A certified value for the ²³⁵U/²³⁸U ratio.

Project coordination was provided by A.M. Voeks and A.J. Traina. Sample preparation and titrimetric assay measurements were performed by A.M. Voeks, G.J. Orłowicz, K.S. Scheidleman, A.J. Traina and M.I. Spaletto. Isotopic abundance measurements were performed by S. Richter and R.M. Essex; verification measurements on the UF₆ gas mass spectrometer were made by P.V. Croatto; experimental design for the isotopic certification and measurement of isotopic data were provided by S.A. Goldberg. The statistical plan of analysis for assay certification was prepared by M.M. Smith, and assessment of the data was performed by M.D. Soriano, M.M. Smith and D.T. Baran. Technical guidance for CRM 113-B packaging, certification, and issuance was provided by U.I. Narayanan, M.A. Legel and M.I. Spaletto. Project supervision was provided by R.D. Oldham, D.T. Baran, W.G. Mitchell and J.W. Neuhoff. Health physics support was provided by F.P. Orłowicz.

(1) C 761, "Standard Test Methods for Chemical, Mass Spectrometric, Nuclear, and Radiochemical Analysis of Uranium Hexafluoride", Annual Book of ASTM Standards, Vol. 12.01.

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