

## **NBL Program Office**



U.S. Department of Energy

## Certificate of Analysis Certified Reference Material C113B Uranium Hexafluoride Assay and Isotopic Standard, 4.5% U-235

Uranium Assay (mass fraction):  $0.67579 \pm 0.00022 \text{ g/g}$ 

 $^{234}\text{U}/^{238}\text{U}$   $^{235}\text{U}/^{238}\text{U}$   $^{236}\text{U}/^{238}\text{U}$ 

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:**  $237.912937 \pm 0.000070$ 

Reported numerical uncertainties are expressed as expanded uncertainties (U) at the 95% level of confidence, where  $U = k \cdot u_c$  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 C113B consists of uranium (4.5% enriched) hexafluoride (UF<sub>6</sub>) 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 C113B was UF<sub>6</sub> 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<sub>6</sub> in the P-10 tubes was corrected for the effects of cover gas trapped over

the UF<sub>6</sub> in the tube and for air buoyancy as follows:

Corrected Sample Weight (g)= (1.00047) (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 112A, 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:

 $^{234}$ U - 234.0409456,  $^{235}$ U - 235.0439231,  $^{236}$ U - 236.0455619, and  $^{238}$ U - 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 <sup>235</sup>U/<sup>238</sup>U 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 <sup>235</sup>U/<sup>238</sup>U 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 <sup>233</sup>U was detected in C113B.

The expanded uncertainty (U) for a certified property of C113B 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 UF<sub>6</sub> 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 <sup>235</sup>U/<sup>238</sup>U 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 <sup>235</sup>U/<sup>238</sup>U 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. Orlowicz, 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 UF<sub>6</sub> 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 C113B 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. Orlowicz.

(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.