

MIT mixed U-Pb gravimetric solution

^{206}Pb - ^{235}U - ^{238}U solution derived from ultra-high-purity CRM-145 and SRM-991 materials mixed at MIT late summer/early fall 2004.

The Ingredients:

CRM 145 (natural U, solution)

SRM 991 (^{206}Pb , solution)

See below for certificates of analyses.

The mixture:

Aliquots of SRM 991 and CRM 145 were mixed.

CRM 145 – 3.3342 g

SRM 991 – 5.5668 g

The Dilution:

Step One. The CRM 145/SRM 991 mixture from above was diluted with 1000.11g 1N HNO_3 . Solution was left overnight to homogenize.

Step Two. An aliquot of the Step One dilute solution was taken (weighing 2.1015g and added to 1008.71g 1N HNO_3 . Solution was left overnight to homogenize.

The Resultant Solution:

After the two-stage dilution the resultant solution has the following concentrations.

^{206}Pb - 3.69967 E-6 micromoles/g

^{235}U – 2.10633 E-6 micromoles/g

^{238}U – 2.90421 E-4 micromoles/g



U.S. Department of Energy
New Brunswick Laboratory

New Brunswick Laboratory Certified Reference Material Certificate of Analysis

CRM 145

Uranium (Normal) Assay Solution Standard

Uranium Mass Fraction:	0.0101356 ± 0.0000011 kg U/kg solution (10.1356 ± 0.0011 mg U/g solution)
Relative Atomic Mass:	238.0289

This Certified Reference Material (CRM) is a uranium concentration solution standard intended for use in calibration of and/or quality control for uranium analysis methods. Each unit of CRM 145 consists of approximately 20-mL of a uranyl nitrate solution in 1 M HNO₃, contained in a sealed 20-mL glass ampule. **NOTE:** *The vial should be handled under proper radiologically-controlled conditions at all times.*

The certified value is based on the mass of high-purity metal dissolved and diluted to a known solution mass. The stated uranium concentration was calculated as the prepared value, and verified experimentally by the NBL-modified Davies and Gray titration. The uncertainty in the certified value is calculated as $2u_c$, where u_c is the combined uncertainty calculated according to the ISO Guide¹¹; the coverage factor of 2 is chosen to provide an approximate 95% level of confidence. The value of u_c is intended to represent, at the level of one standard deviation, the combined uncertainties due to weighing, purity and buoyancy factors. All contributors to the total uncertainty are Type B uncertainties. The value for the certified relative atomic mass is that given for CRM 112-A, Uranium Metal Assay Standard, which was used in the preparation of CRM 145. The value was verified by thermal ionization mass spectrometry.

Technical coordination for the preparation, certification, and issuance of NBL CRM 145 was provided by P.B. Mason. Preparation and packaging were performed by P.B. Mason, P.V. Croatto, I.W. Frank, and F.P. Orłowicz. Titrimetric assay measurements were performed by I.W. Frank and G.J. Orłowicz; isotopic abundance measurements were performed by A. J. Traina; statistical assessment of the data for certification was performed by M. D. Soriano. Technical guidance was provided by U. I. Narayanan and C. G. Gradle. Project supervision was provided by R. D. Oldham and W. G. Mitchell.

September 30, 1996
Argonne, Illinois

(over)

H. Rodney Martin
Acting Director



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 991

Lead-206 Assay and Isotopic Standard

This Standard Reference Material (SRM) is intended for use as an assay and isotopic standard. SRM 991 consists of a solution of lead nitrate sealed in quartz ampoules. Each ampoule contains a nominal 15 g of solution, 0.5N in nitric acid. The certified isotopic compositions are given below together with the atomic weight of lead.

Molality of Lead: 0.32261 mmol/kg \pm 0.00032 mmol/kg

Isotopic composition:

²⁰⁴ Pb, Atom Percent	< 0.0003
²⁰⁶ Pb, Atom Percent	99.979 \pm 0.002
²⁰⁷ Pb, Atom Percent	0.008 \pm 0.001
²⁰⁸ Pb, Atom Percent	0.013 \pm 0.001

Atomic Weight: 205.975

The concentration of lead in SRM 991 was determined by an isotope dilution mass spectrometry (IDMS) technique. A correction for isotopic fractionation was checked by analyzing SRM 981. The indicated uncertainty for the concentration is the 95 % tolerance limit [1,2] for coverage of at least 99 % of measured values of this lot of ampoules of SRM 991. The measured values should fall within the indicated tolerance limits with a confidence coefficient of 95 %.

The mass spectrometric measurements were made by L.J. Moore and J.W. Gramlich of the NIST Analytical Chemistry Division. The solutions measured were prepared by L.A. Machlan of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical measurements leading to certification were under the chairmanship of I.L. Barnes of the NIST Analytical Chemistry Division.

The technical and support aspects concerning the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by W.P. Reed. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

This Certificate of Analysis has undergone editorial revision to reflect program and editorial changes at NIST and the Department of Commerce.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 31 August 2004
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Acting Chief
Measurement Services Division