National Institute of Standards & Technology

Certificate

Standard Reference Material® 4321d

Natural Uranium Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of standardized and certified massic activities of radioactive uranium-238, uranium-235, and uranium-234 in a suitably stable and homogeneous matrix. It is intended primarily for the calibration of instruments that are used to measure radioactivity and for the monitoring of radiochemical procedures. A unit of SRM 4321d consists of approximately 5 mL of a solution, whose composition is specified in Tables 1 and 2, contained in a flame-sealed borosilicate-glass ampoule [1].

The certified massic activities for the uranium isotopes at a **Reference Time of 1200 EST, 15 March 2017**, are:

Uranium-238: (228.93 ± 0.56) Bq•g^{-1}
Uranium-235: (10.540 ± 0.032) Bq•g^{-1}
Uranium-234: (220.16 ± 1.15) Bq•g^{-1}

A NIST certified value, as used within the context of this certificate, is a value for which NIST has the highest confidence in its uncertainty assessment. It is a "measurement result" [2] obtained directly or indirectly from a "primary reference measurement procedure" [3]. The certified value is traceable to the derived SI unit, becquerel (Bq).

Additional physical, chemical, and radiological properties for this SRM, as well as details on the standardization method, are given in Tables 1 and 2. Uncertainties for the certified quantities are expanded ($k = 2$). The uncertainties are calculated according to the ISO/JCGM and NIST Guides [4,5]. Table 3 contains a specification of the components that comprise the uncertainty analysis.

Expiration of Certification: The certification of **SRM 4321d** is valid indefinitely, within the measurement uncertainty specified, provided that the SRM is handled and stored properly and that no evaporation or change in composition has occurred. The solution matrix, in an unopened ampoule, is homogeneous and stable within its half-life-dependent useful lifetime provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling and Storage"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Radiological and chemical hazard: Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for radiological and chemical hazard information.

This SRM was prepared in the NIST Physical Measurement Laboratory, Radiation Physics Division, under the direction of M.P. Unterweger, Group Leader of the Radioactivity Group. Overall technical direction and physical measurement leading to certification were provided by R. Collé and L. Laureano-Perez of the NIST Radiation Physics Division, Radioactivity Group. Additional technical support and assistance was provided by J. LaRosa, S. Nour, L. Pibida, D. Bergeron, R. Fitzgerald and B. Zimmerman of the NIST Radiation Physics Division, Radioactivity Group.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Certificate Issue Date: 29 November 2017

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Table 1. Certified Massic Activity of SRM 4321d

Radionuclides	**Natural Uranium (Mixture of ^{238}U, ^{235}U, and ^{234}U)**
Reference time	**1200 EST, 15 March 2017**
Massic activities of the solution	**^{238}U: 228.93 Bq•g^{-1}** **^{235}U: 10.540 Bq•g^{-1}** **^{234}U: 220.16 Bq•g^{-1}**
Relative expanded uncertainties (k = 2)	**^{238}U: 0.25 %**[(a)] **^{235}U: 0.31 %**[(a)] **^{234}U: 0.52 %**[(a)]

[(a)] The uncertainties on certified values are expanded uncertainties, $U = ku_c$. The quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [4,5]. The combined standard uncertainty is multiplied by a coverage factor of k = 2 and was chosen to obtain an approximate 95 % level of confidence.

Table 2. Uncertified information of SRM 4321d

Source description	Liquid in flame-sealed, 5 mL borosilicate glass ampoule [1]
Solution composition	(1.06 ± 0.01) mol•L^{-1} HNO_3 with 21 mg UO_2^{+2} per gram of solution[(a)]
Solution density	(1.057 ± 0.001) g•mL^{-1} at 21.6 °C[(a)]
Solution mass	(5.284 ± 0.001) g[(a)]
Photon-emitting impurities	None detected [(b)]
Half-lives used	^{238}U : (4.468 ± 0.005) x 10^9 a[(c)] [6] ^{235}U : (7.04 ± 0.01) x 10^8 a[(c)] [7] ^{234}U : (2.455 ± 0.006) x 10^5 a[(c)] [8]
Other Uranium isotopes	^{233}U and ^{236}U were not detected[(d)]
Calibration method (and instruments)	Quantitative dissolution of a uranium (normal) metal assay and isotopic standard (CRM 112-A)[(e)] with conversion to massic activity values using assumed half-lifes of ^{234}U, ^{235}U and ^{238}U. The uranium assay for CRM 112-A was determined by coulometry. The certified isotopic atom fractions were determined by mass spectrometry. Solution homogeneity measurements were made with a NaI(Tl) well counter. Confirmatory measurements for total massic activity and ^{234}U and ^{238}U isotopic massic activity were performed by 4παβ liquid scintillation counting[(f)] and by isotope dilution alpha spectrometry (IDAS)[(g)], respectively.

[(a)] The stated uncertainty is two times the standard uncertainty. See reference 5.

[(b)] The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, as of April 2017, were:

1.4 s^{-1}•g^{-1} in the region 15 keV ≤ E ≤ 20 keV;
0.36 s^{-1}•g^{-1} in the region 25 keV ≤ E ≤ 105 keV;
0.25 s^{-1}•g^{-1} in the region 110 keV ≤ E ≤ 490 keV, and
0.32 s^{-1}•g^{-1} in the region 500 keV ≤ E ≤ 2000 keV;

[(c)] The stated uncertainty is the standard uncertainty. See reference 5.

[(d)] From the CRM 112-A Certificate of Analysis [9] the limit of detection of uranium isotope ratios for the technique used is 5 x 10^{-9}.

[(e)] This CRM was originally issued in 1972 by the National Bureau of Standards (NBS) (now National Institute of Standards and Technology, NIST) as SRM 960. The assay measurements leading to the certification of the uranium concentration were performed by NBS. In 1987, the technical and administrative aspects regarding this material were transferred to the U.S. Department of Energy, New Brunswick Laboratory (NBL). In 1998, the standard material was repackaged and verified for uranium assay and atomic mass by NBL. In 2010, the material was again repackaged and isotopic certification and assay verification measurements were performed at NBL.

[(f)] The total massic activity in SRM 4321d was confirmed by consistency with measured 4παβ liquid scintillation counting rates, taken in combination with known alpha detection efficiency and with calculated beta detection efficiencies for the ^{231}Th, ^{234}Th and ^{234m}Pa daughter radionuclides as obtained from the MICELLE2 code for ^{234}U, ^{235}U and ^{238}U using ^{3}H as an efficiency tracing monitor. The relative agreement was within 0.8 % of the certified massic activity, well within the uncertainty of the efficiency calculations.

[(g)] Aliquots of a gravimetric dilution of SRM 4321d were taken and spiked with known activities of ^{232}U from SRM 4324b to radiochemically prepare thin electrodeposited sources for alpha-particle spectrometry. The results for the ^{234}U and ^{238}U massic activities in SRM 4321d from this isotope dilution analysis were within -0.12 % and +0.25 % respectively, of the certified values, which is well within the uncertainty of the massic activity of the SRM 4324b ^{234}U tracer.

Table 3. Uncertainty evaluation for the massic activity for SRM 4321d

Uncertainty component		Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of Natural Uranium (%)		
			^{238}U	^{235}U	^{234}U
1	Uranium mass fraction in CRM 112-A; from CRM 112-A certificate [9]	B	0.003	0.003	0.003
2	Isotopic uranium atom fraction in CRM 112-A; from CRM 112-A certificate [9]	B	0.00020	0.0271	0.077
3	Half-life; standard uncertainty of the half-life [6,7,8]	B	0.112	0.142	0.244
4	Mass of uranium metal	B	0.05	0.05	0.05
5	Quantitative dissolution of metal	B	0.05	0.05	0.05
6	Mass of SRM master solution	B	0.005	0.005	0.005
Relative combined standard uncertainty			**0.123**	**0.153**	**0.261**
Relative expanded uncertainty (k = 2)			**0.25**	**0.31**	**0.52**

[(a)] B denotes evaluation by other methods.

INSTRUCTIONS FOR HANDLING AND STORAGE

Handling: If the ampoule is transported, it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. The solution in the ampoule is a dangerous good (hazardous material) because of the radioactivity. Only persons qualified to handle both radioactive material and alkaline and/or acidic solutions, should open the ampoule. To minimize personnel exposure, appropriate shielding and/or distance should be used. Refer to the SDS for further information.

Storage: SRM 4321d should be stored and used at a temperature between 5 °C and 65 °C. The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material.

REFERENCES

[1] NIST Physical Measurement Laboratory; *Storage and Handling of Radioactive Standard Reference Materials, Ampoule Specifications and Opening Procedure*, available at http://www.nist.gov/pml/div682/grp04/srm.cfm (accessed Nov 2017). Note: This SRM is contained in a generic borosilicate-glass ampoule and not in the standard NIST ampoule.

[2] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)* (2008 version with Minor Corrections), 3rd edition; Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France; p. 19 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2017).

[3] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)* (2008 version with Minor Corrections), 3rd edition; Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France; p. 18 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2017).

[4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2017).

[5] Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Nov 2017).

[6] Chisté, V. and M.M. Bé; *LNE-LNHB/CEA Table of Radionuclides, ^{238}U*; (April 2006); available at http://www.nucleide.org/DDEP_WG/Nuclides/U-238_tables.pdf (accessed Nov 2017).

[7] Huang Xiaolong and Wang Baosong; *LNE-LNHB/CEA Table of Radionuclides, ^{235}U*; (February 2014); available at http://www.nucleide.org/DDEP_WG/Nuclides/U-235_tables.pdf (accessed Nov 2017).
[8] Chisté, V. and M.M. Bé; *LNE-LNHB/CEA Table of Radionuclides, ^{234}U*; (September 2006); available at http://www.nucleide.org/DDEP_WG/Nuclides/U-234_tables.pdf (accessed Nov 2017).
[9] Certificate of Analysis CRM 112-A, Uranium (Normal) Metal Assay and Isotopic Standard, New Brunswick Laboratory, U.S. Department of Energy, (September, 2010); available at https://science.energy.gov/~/media/nbl/pdf/price-lists/certificates/CRM_112A_Uranium_Metal_Sept_2010.pdf (accessed Nov 2017).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.