National Institute of Standards & Technology

Certificate

Standard Reference Material® 4323c

Plutonium-238 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive plutonium-238 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 4323c 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 **plutonium-238** massic activity, at a **Reference Time of 1200 EST, 11 October 2016**, is:

$$\mathbf{(22.73 \pm 0.11)\ Bq \cdot g^{-1}}$$

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 4323c** 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. All additional technical support was provided by members of the NIST Radiation Physics Division, Radioactivity Group. Confirmatory measurements by J. LaRosa and S. Nour were performed by isotope dilution analysis with alpha-particle spectrometry. Homogeneity evaluations were assisted by D. Bergeron. Alpha-emitting-impurity and photon-emitting-impurity analyses were provided by J. LaRosa and S. Nour, and L. Pibida, respectively.

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

Michael G. Mitch, Acting Chief
Radiation Physics Division

Gaithersburg, Maryland 20899
Certificate Issue Date: 03 November 2017

Steven J. Choquette, Director
Office of Reference Materials

Table 1. Certified Massic Activity of SRM 4323c

Radionuclide	**Plutonium-238**
Reference time	**1200 EST, 11 October 2016**
Massic activity of the solution	**22.73 Bq•g^{-1}**
Relative expanded uncertainty (k = 2)	**0.50 %**[(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 4323c

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	(3.16 ± 0.05) mol•L^{-1} HNO_3[(a)]
Solution density	(1.102 ± 0.001) g•mL^{-1} at 22.3 °C[(a)]
Solution mass	(5.511 ± 0.003) g[(a)]
Photon-Emitting Impurities	None detected[(b)]
Alpha-emitting Impurities	None detected[(c)]
Half-lives used	^{238}Pu: (87.74 ± 0.03) a [(d)] [6]
Calibration methods (and instruments)	The certified value was determined by liquid scintillation (LS) spectrometry with two LS commercial counters using six cocktail compositions (each with three counting sources) in sixteen separate measurement trials. Confirmatory measurements were performed by isotope dilution analysis with alpha-particle spectrometry.[(e)]

[(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, in May 2016, were:

0.24 s^{-1}•g^{-1} in the region	15 keV ≤ E ≤ 30 keV;
0.04 s^{-1}•g^{-1} in the region	35 keV ≤ E ≤ 1430 keV;
0.05 s^{-1}•g^{-1} in the region	1440 keV ≤ E ≤ 1480 keV, and
0.03 s^{-1}•g^{-1} in the region	1490 keV ≤ E ≤ 2000 keV;

[(c)] The massic activity of ^{239}Pu at the Reference Time is estimated to be 0.00012 Bq•g^{-1}, based on a ^{239}Pu/^{238}Pu atom ratio of 0.00107 as obtained from mass spectrometry by Oak Ridge National Laboratory in 1981 for the stock material used to prepare SRM 4323c. The estimated lower limits of detection for alpha -emitting impurities, expressed as massic alpha emission rate, in June 2016, were:

0.003 s^{-1}•g^{-1} in the region	$E \leq$ 4580 keV;
0.007 s^{-1}•g^{-1} in the region	5020 keV ≤ E ≤ 5280 keV; and
0.0001 s^{-1}•g^{-1} in the region	$E \geq$ 5630 keV.

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

[e)] Aliquants of SRM 4323c were taken and spiked with known activities of ^{242}Pu from SRM 4334h to radiochemically prepare thin counting sources for the alpha spectrometry measurements. The result for the ^{238}Pu massic activity in SRM 4323c from this isotope dilution analysis was (22.71 ± 0.27) Bq•g^{-1} at k = 2, and agreed with the LS-based certified value to within 0.06 %.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4323c

Uncertainty component		Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of ^{238}Pu (%)
1	LS measurement precision: Relative standard deviation of the mean on the great-grand mean for 16 LS measurement trails, considering all of the within-trial and between-trial components of variance. Each of the 16 grand mean values was based on 5 replicate measurements on each of 3 LS counting sources. The typical within-trial relative standard deviation of the mean (considering the variations for the between 5 measurements and the between 3 sources) for each trial was 0.12 %. The between-trial relative standard deviation across the 16 trials was 0.17 %. The 16 data values fit a Normal distribution.	A	0.21
2	Background; LS measurement variability and cocktail composition stability effects; wholly embodied in component 1.	A	--
3	LS counters dependencies; wholly embodied in components 1 & 2	A	--
4	Live time determinations for LS counting time intervals, includes uncorrected dead time effects	B	0.07
5	Aliquant mass determinations by gravimetric measurements for preparation of counting sources; includes mass measurement precision partially embodied in component 1.	B	0.05
6	LS detection inefficiency, includes wall effect; partially embodied in component 1.	B	0.01
7	^{238}Pu decay corrections for half-life uncertainty of 0.034 %.	B	< 0.0002
8	Potential alpha- and photon-emitting impurities	B	0.1
Relative combined standard uncertainty			**0.25**
Relative expanded uncertainty (k = 2)			**0.50**

[(a)] Letter A, denotes evaluation by statistical methods; B denotes evaluation by other methods [4,5].

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 radioactive material 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 4323c 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 https://www.nist.gov/pml/radiation-physics/ampoule-specifications-and-opening-procedure (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 (JCGM): 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; JCGM: 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), JCGM: 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 https://www.nist.gov/pml/productsservices/special-publications-tutorials (accessed Nov 2017).

[6] Chechev, V.P.; *LNE-LNHB/CEA Table of Radionuclides*, ^{238}Pu; (June 2009); available at http://www.nucleide.org/DDEP_WG/Nuclides/Pu-238_com.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.