

National Institute of Standards & Technology

Certificate

Standard Reference Material® 4320b

Curium-244 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive curium-244 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 4320b consists of approximately 5 mL of a solution, whose composition is specified in Table 1 and 2, contained in a flame-sealed borosilicate-glass ampoule [1].

The certified curium-244 massic activity value, at a Reference Time of 1200 EST, 01 September 2011, is:

 $(35.47 \pm 0.50) \text{ Bq} \cdot \text{g}^{-1}$

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 Table 1 and 2. Uncertainties for the certified quantities are expanded (k = 2). The uncertainties are calculated according to the ISO and NIST Guides [4,5]. Table 3 contains a specification of the components that comprise the uncertainty analyses.

Expiration of Certification: The certification of **SRM 4320b** 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) 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 Physical Measurement Laboratory, Radiation and Biomolecular Physics Division, Radioactivity Group, M.P. Unterweger, Group Leader. The overall production, technical direction and physical measurement leading to certification were provided by R. Collé and L. Laureano-Pérez and photon-emitting impurity analyses were provided by L. Pibida of the NIST Radioactivity Group.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Lisa R. Karam, Chief Radiation and Biomolecular Physics Division

Gaithersburg, Maryland 20899 Robert L. Watters, Jr., Chief Certificate Issue Date: 17 August 2012 Measurement Services Division

SRM 4320b Page 1 of 4

Table 1. Certified Massic Activity of SRM 4320b

Radionuclide	Curium-244 ^(a)	
Reference time	1200 EST, 01 September 2011	
Massic activity of the solution	35.47 Bq•g ⁻¹	
Relative expanded uncertainty $(k = 2)$	1.4 % ^(b)	

⁽a) The ²⁴⁴Cm master solution was chemically purified on approximately 01 January 1966. Plutonium-240 is the daughter of ²⁴⁴Cm and has been growing in since that time. The certified ²⁴⁴Cm massic activity of SRM 4320b is in agreement with the previous certified value.

Table 2. Uncertified Information of SRM 4320b

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]	
Solution composition	1.0 mol•L ⁻¹ HNO ₃	
Solution density	$(1.033 \pm 0.002) \text{ g} \cdot \text{mL}^{-1} \text{ at } 20 {}^{\circ}\text{C}^{(a)}$	
Solution mass	$(5.170 \pm 0.003) \text{ g}^{(a)}$	
Impurities	²⁴⁰ Pu: (0.47 ± 0.05) Bq•g ⁻¹ (Ingrowth daughter) ²⁴³ Cm: (0.009 ± 0.002) Bq•g ^{-1(b)} ²⁴⁵ Cm: (0.003 ± 0.002) Bq•g ^{-1(b)}	
Half-lives used	²⁴⁴ Cm: 18.11 a [6] \pm 1.2 % ^(c,d) ²⁴⁰ Pu: 6561 a [7] \pm 0.4 % ^(e) ²⁴³ Cm: (1600 \pm 7) a [8] ²⁴⁵ Cm: (8250 \pm 70) a [9]	
Calibration methods (and instruments)	The certified massic activity for 244 Cm was obtained by $4\pi\alpha$ liquid scintillation (LS) spectrometry with three commercial LS counters. Confirmatory measurements were performed by high-resolution HPGe gamma-ray spectrometry.	

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

- $1 \times 10^{1} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 15 keV and 25 keV,
- $3 \times 10^{0} \,\mathrm{s^{-1} \cdot g^{-1}}$ for energies between 30 keV and 35 keV,
- $4 \times 10^{-1} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 40 keV and 55 keV,
- $3 \times 10^{-1} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 60 keV and 300 keV,
- $3 \times 10^{-1} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 310 keV and 1530 keV, and
- $2 \times 10^{-1} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 1540 keV and 2000 keV.

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ²⁴⁴Cm or progeny.

SRM 4320b Page 2 of 4

⁽b) 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 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.

⁽b) The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, in December 2011 are:

⁽c) The uncertainty given here in the ²⁴⁴Cm half-life was the standard deviation of the mean for the unweighted average of the six values cited in reference 6. The 0.2 % uncertainty given in reference 6 was considered to be unrealistic, being based on a weighted mean and after rejecting two values on the basis of them being outliers rather than for experimental reasons.

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

⁽e) The uncertainty given here in the ²⁴⁰Pu half-life was the standard deviation of the mean for the unweighted average of the 11 values cited in reference 7. The 0.1 % uncertainty given in reference 7 was considered to be unrealistic, being based on many re-estimates and the between-determination dispersion in the values.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4320b

	Uncertainty component	Assessment Type ^(a)	Relative standard uncertainty contribution on massic activity of ²⁴⁴ Cm (%)
1	LS measurement precision; standard deviation of the mean for $n = 13$ sets of measurements obtained with 3 different LS counters using 3 different cocktail compositions with aliquots from 4 different ampoules, each measured 3 to 5 times in each counter on 1 or 2 occasions. The typical internal relative standard deviation within a measurement data set is 0.57 % for $n = 12$ sources.	A	0.70
2	Background LS measurement variability and cocktail stability; wholly embodied in component 1	В	
3	Live time determinations for LS counting time intervals, includes uncorrected dead time effects; assumed from specified tolerance limits of counters' gated oscillators	В	0.06
4	LS detection inefficiency for alpha particles	В	0.01
5	Gravimetric (mass) determinations for LS sources; estimated from calibration data and tests. Mass determinations for dilution factors and counting source preparations	В	0.1
6	Decay corrections for ²⁴⁴ Cm half-life uncertainty of 1.2 % ^(b) [6]	В	0.03
7	Correction for 240 Pu daughter ingrowth for half-life uncertainty of 0.4 $\%^{(c)}$ [7]	В	0.02
8	Correction for ²⁴⁰ Pu daughter ingrowth	В	0.1
9	Impurity corrections (excluding ²⁴⁰ Pu ingrowth)	В	0.05
Relative combined standard uncertainty			0.72
Relative expanded uncertainty $(k = 2)$			1.4

SRM 4320b Page 3 of 4

 $^{^{(}a)}$ Letter A, denotes evaluation by statistical methods; B denotes evaluation by other methods. $^{(b)}$ The uncertainty given here in the 244 Cm half-life was the standard deviation of the mean for the unweighted average of the six values cited in reference 6. The 0.2 % uncertainty given in reference 6 was considered to be unrealistic, being based on a weighted

mean and after rejecting two values on the basis of them being outliers rather than for experimental reasons.

(c) The uncertainty given here in the ²⁴⁰Pu half-life was the standard deviation of the mean for the unweighted average of the eleven values cited in reference 7. The 0.1 % uncertainty given in reference 7 was considered to be unrealistic, being based on many re-estimates and the between-determination dispersion in the values.

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 both the radioactivity and the strong acid. The ampoule should be opened only by persons qualified to handle both radioactive material and alkaline and/or acidic solutions. Appropriate shielding and/or distance should be used to minimize personnel exposure. Refer to SDS for further information.

Storage: SRM 4320b 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 Aug 2012). 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 Aug 2012).
- [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 Aug 2012).
- [4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (ISO 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 Aug 2012).
- [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://physics.nist.gov/Pubs/ (accessed Aug 2012).
- [6] Chechev, V.P., May 2010. ²⁴⁴Cm. LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Cm-244_tables.pdf (accessed Aug 2012).
- [7] Chechev, V.P., September 2009, ²⁴⁰Pu. LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Pu-240_tables.pdf (accessed Aug 2012).
- [8] Chechev, V.P., January 2012. ²⁴³Cm. LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Cm-243_tables.pdf (accessed Aug 2012).
- [9] Chechev, V.P., December 2010 ²⁴⁵Cm. LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Cm-245_tables.pdf (accessed Aug 2012).

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.

SRM 4320b Page 4 of 4