National Institute of Standards & Technology

Certificate

Standard Reference Material® 4330C

Plutonium-239 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive plutonium-239 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 consists of a nominal 3 g of solution, whose composition is specified in Table 1, contained in a flame-sealed 5 mL borosilicate-glass ampoule (see Note 1)*.

The certified **Plutonium-239** massic activity value, at a **Reference Time of 1200 EST, 1 May 2009**, is:

$$\mathbf{(38.41 \pm 0.18)\ Bq \cdot g^{-1}}$$

Additional physical, chemical, and radiological properties for this SRM, as well as details on the standardization method, are given in Table 1. Uncertainties for the certified quantities are expanded ($k = 2$). The uncertainties are calculated according to the ISO and NIST Guide (see Note 2). Table 2 contains a specification of the components that comprise the uncertainty analyses.

Expiration of Certification: The certification of **SRM 4330C** is valid indefinitely 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 Use"). 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

This SRM may represent a radiological hazard and a chemical hazard. Consult the Material Safety Data Sheet (MSDS), enclosed with the SRM shipment, for details (see Note 1).

This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, M.P. Unterweger, Group Leader. The overall technical direction and physical measurement leading to certification were provided by R. Collé and L. Laureano-Pérez of the NIST Radioactivity Group, with production assistance by D.B. Golas, Research Associate of the NRMAP, Inc., and photon-emitting impurity analyses by L. Pibida.

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

INSTRUCTIONS FOR USE

Storage: SRM 4330C 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.

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 MSDS for further information.

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Certificate Issue Date: 16 April 2010
See Certificate Revision History on Last Page

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* Notes and references may be found on page 4.

Table 1. Properties of SRM 4330C

Certified values

Radionuclide	**Plutonium-239**
Reference time	**1200 EST, 1 May 2009**
Massic activity of the solution	**38.41 Bq•g^{-1}**
Relative expanded uncertainty (k = 2)	**0.46 %** (see Note 2)*****

Uncertified information

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule (see Note 1)
Solution composition	3.4 mol•L^{-1} HNO_3
Solution density	(1.1082 ± 0.002) g•mL^{-1} at 23.9 °C (see Note 3)
Solution mass	(2.7707 ± 0.0003) g (see Note 3)
Photon-emitting impurities	None detected (see Note 4)
Half-lifes used	^{239}Pu : (24100 ± 11) a (see Note 5) [1]
Calibration methods (and instruments)	The certified massic activity for ^{239}Pu was obtained by 4πα liquid scintillation (LS) spectrometry with three commercial LS counters.

***** Notes and references may be found on page 4.

Table 2. Uncertainty evaluation for the massic activity of SRM 4330C

	Uncertainty component	Assessment Type †	Relative standard uncertainty contribution on massic activity of ^{239}Pu (%)
1	LS measurement precision; standard deviation for n = 9 mean determinations as obtained with 3 different cocktail compositions (5 sources of each composition) measured 3 times in 3 different LS counters (135 determinations in all). (see Note 6)*. Data passes normality test at 95 % and 99 %. The typical internal relative standard deviation of the mean (n = 15 for the 5 sources measured 3 times) for each of the 9 determinations ranged from 0.06 % to 0.10 %.	A	0.17
2	Background LS measurement variability; wholly embodied in component 1	A	---
3	Gravimetric (mass) determinations for LS sources; estimated from calibration data and tests.	B	0.07
4	Decay correction due to the decay time interval and 0.046 % uncertainty in half-life.	B	5×10^{-8}
5	Live time determinations for LS counting time intervals, includes uncorrected dead time effects; assumed from specified tolerance limits of counters' gated oscillators	B	0.1
6	LS detection efficiency, including wall effects loss and extrapolation to zero energy; estimated from nuclear data and tests.	B	0.07
7	Alpha decay probability; assumed from nuclear decay data based on % of spontaneous fission	B	3×10^{-10}
8	LS non-detection of 26 min $^{235}U^{m}$; estimated from nuclear data and tests of LS counter detection threshold	B	< 0.001
9	Correction for ingrowth of ^{235}U; estimated from nuclear data	B	3×10^{-8}
10	Alpha-particle emitting impurities; based on nuclear data and mass spectrometric measurements of supplier (see Note 7).	B	0.06
11	Photon-emitting impurities (non-detected) (see Note 4).	B	---
Relative combined standard uncertainty			**0.23**
Relative expanded uncertainty (k = 2)			**0.46**

† =

(A) denotes evaluation by statistical methods; (B) denotes evaluation by other methods.

* Notes and references may be found on page 4.

Note 1. Refer to http://physics.nist.gov/Divisions/Div846/srm.html for assistance and instructions on how to properly open an ampoule. Information on additional storage and handling requirements is also included on the website. This SRM is contained in a generic borosilicate-glass ampoule and not in the standard NIST ampoule.

Note 2. 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 [2-3]. The combined standard uncertainty is multiplied by a coverage factor of $k = 2$ and was chosen to obtain an approximate 95 % level of confidence.

Note 3. The stated uncertainty is two times the standard uncertainty. See reference [3].

Note 4. The estimated lower limit of detection for photon-emitting impurities, expressed as massic photon emission rate, on 1 May 2009 is:

$0.64\ s^{-1} \bullet g^{-1}$ for energies between 40 keV and 50 keV,
$0.17\ s^{-1} \bullet g^{-1}$ for energies between 50 keV and 250 keV, and
$0.13\ s^{-1} \bullet g^{-1}$ for energies between 250 keV and 2600 keV.

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ^{239}Pu or progeny.

Note 5. The stated uncertainty is the standard uncertainty. See reference [3].

Note 6. The mean was found to be invariant of: (i) cocktail composition based on scintillation fluid used and the aqueous fraction, (ii) aliquant mass in cocktails, (iii) sample quenching; and (iv) instrument (with different detection thresholds).

Note 7. From mass spectrometric measurements performed by the supplier, the non-certified massic activities of other detected radionuclides (in $Bq \bullet g^{-1}$ as of 1200 EST, 15 November 1999) are: $^{240}Pu = 0.002$, $^{241}Pu = 0.02$, $^{241}Am = 0.001$. Solution was purified 10 December 1979.

REFERENCES

[1] E. Browne, *Nuclear Data Sheets* 98, 665 (2003), Evaluated Nuclear Structure Data File (ENSDF), online database, National Nuclear Center, Brookhaven National Laboratory (Upton, NY). Refer to http://www.nndc.bnl.gov/ensdf/ (accessed Apr 2010).

[2] 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 Apr 2010).

[3] B.N. Taylor and C.E. Kuyatt; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1994. Available at http://www.nist.gov/physlab/pubs/tn1297/index.cfm (accessed Apr 2010).

Certificate Revision History: 16 April 2010 (The unit content description was included.); 29 December 2009 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.