

# National Institute of Standards & Technology

# Certificate

## Standard Reference Material® 4334I

## Plutonium-242 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive plutonium-242 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 solution, whose composition is specified in Table 1, contained in a flame-sealed 5 mL borosilicate-glass ampoule (see Note 1)\*.

The certified Plutonium-242 massic activity value, at a Reference Time of 1200 EST, 01 January 2010, is:

 $(26.77 \pm 0.18) \text{ Bq} \cdot \text{g}^{-1}$ .

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 Guide (see Note 2). Table 2 contains a specification of the components that comprise the uncertainty analyses.

**Expiration of Certification:** The certification of **SRM 4334I** is valid indefinitely provided 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 Storage and Handling"). 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.

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 L. Lucas of the NIST Radioactivity Group.

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

#### INSTRUCTIONS FOR STORAGE AND HANDLING

**Storage:** SRM 4334I 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: 30 June 2010

\* Notes and references may be found on pages 4 and 5. SRM 4334I

## Table 1. Properties of SRM 4334I

### **Certified values**

Radionuclide	Plutonium-242
Reference time	1200 EST, 1 January 2010 (See Note 3) *
Massic activity of the solution	26.77 Bq•g <sup>-1</sup>
Relative expanded uncertainty $(k = 2)$	<b>0.68</b> % (see Note 2)

### Uncertified information

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule (see Note 1)	
Solution composition	3.2 mol•L <sup>-1</sup> HNO <sub>3</sub>	
Solution density	$(1.105 \pm 0.002)$ g•mL <sup>-1</sup> at 22 °C (see Note 4)	
Solution mass	Approximately 5.5 g	
Alpha-particle- emitting impurities	<sup>241</sup> Am: (0.0016 ± 0.0003) Bq•g <sup>-1</sup> (See Note 3, 5 and 6)	
Beta-particle- emitting impurities	$^{241}$ Pu : (0.044 ± 0.009) Bq•g <sup>-1</sup> (see Note 4)	
Photon-emitting impurities	None detected (see Note 7)	
Half-lifes used	$^{242}$ Pu: (373 500 ± 1100) a (see Note 8) [1] $^{241}$ Pu: (14.290 ± 0.006) a $^{240}$ Pu: (6 561 ± 7) a $^{239}$ Pu: (24 110 ± 30) a $^{238}$ Pu: (87.7 ± 0.1) a $^{241}$ Am: (432.6 ± 0.6) a	
Calibration methods (and instruments)	I spectrometry using four commercial LN counters, a calibrated germanium detect	

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st Notes and references may be found on pages 4 and 5.

Table 2. Uncertainty evaluation for the massic activity of SRM 4334I

	Uncertainty component	Assessment Type †	Relative standard uncertainty contribution on massic activity of <sup>242</sup> Pu (%)
1	Massic alpha-particle emission rate, corrected for background and decay. Standard deviation of the mean for $180~4\pi\alpha$ liquid-scintillation measurements.	A	0.06
2	Half life of <sup>242</sup> Pu; standard uncertainty of the half life	В	$3 \times 10^{-6}$
3	Decay-scheme data; standard uncertainty of the probability of decay by alpha-particle emission	В	0.001
4	Extrapolation of the alpha-particle-count-rate-versus-energy to zero energy	В	0.25
5	Gravimetric measurements	В	0.10
6	Live time	В	0.10
7	Alpha-particle detection efficiency of scintillators	В	0.10
8	<sup>241</sup> Pu detection efficiency of scintillators	В	0.04
9	Alpha-particle emitting impurities; limit of detection	В	0.10
10	<sup>241</sup> Pu impurity	В	0.02
11	Photon emitting impurities; limit of detection	В	0.10
Rela	tive combined standard uncertainty	0.34	
Relative expanded uncertainty $(k = 2)$			0.68

 $<sup>^{\</sup>dagger}$  = (A) denotes evaluation by statistical methods; (B) denotes evaluation by other methods.

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Note 1. Refer to http://physics.nist.gov/Divisions/Div846/srm.html (accessed Jun 2010) 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 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 <sup>242</sup>Pu was chemically purified 07 June 1994 at the Lawrence Livermore National Laboratory (LLNL). Americium-241, the daughter of <sup>241</sup>Pu, was removed but has been growing in since that time.

Note 4. The stated uncertainty is two times the standard uncertainty. See reference 3.

Note 5. The estimated limits of detection for alpha-particle-emitting impurities, expressed as massic alpha-particle emission rates (number of alpha-particles emission rates per second per gram), are:

 $0.003~s^{\text{-}1} \bullet g^{\text{-}1}$  for energies less than 3.1 MeV,

0.03 s<sup>-1</sup>•g<sup>-1</sup> for energies between 3.1 MeV and 4.4 keV, and

 $0.003~s^{\text{-}1} \bullet g^{\text{-}1}$  for energies greater than 5.0~MeV

Note 6. The <sup>242</sup>Pu was chemically purified 07 June 1994. The relative massic activities of radionuclidic impurities after purification follow;

Radionuclide	Relative Activity As Determined By	
	LLNL	NIST
<sup>242</sup> Pu	1	1
<sup>241</sup> Pu		$(3.5 \pm 0.4) \times 10^{-3}$ (a)
$^{240}$ Pu + $^{239}$ Pu	< 10 <sup>-6</sup> (b)	$(2.0 \pm 2.1) \times 10^{-5}$ (c)
$^{238}$ Pu + $^{241}$ Am	$< 1.6 \times 10^{-5}$ (b)	$(9 \pm 16) \times 10^{-6}$ (c)
<sup>241</sup> Am		assumed 0 (a)

- (a) The stated uncertainty is the standard uncertainty. The <sup>241</sup>Pu activity was calculated from a gamma-ray measurement of the <sup>241</sup>Am ingrowth as of 25 November 1998, assuming that <sup>241</sup>Am was completely removed at the time of chemical purification.
- (b) Using alpha-particle spectrometry. The value shown is an estimated upper limit based upon background and counting statistics. Measurements were made at the Lawrence Livermore National Laboratory (LLNL) in July of 1994.
- (c) Using alpha-particle spectrometry. The stated uncertainty is the standard uncertainty. Measurements were made at the National Institute of Standards and Technology (NIST) in June and July 1999.

Note 7. The estimated limits of detection for photon-emitting impurities, expressed as massic photon emission rates (numbers of photons per second per gram), are:

 $5 \times 10^{-5} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 19 keV and 39 keV,

 $7 \times 10^{-5} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 49 keV and 92 keV,

 $2 \times 10^{-5} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 106 keV and 507 keV,

 $1 \times 10^{-5} \, \text{s}^{-1} \, \text{eg}^{-1}$  for energies between 515 keV and 1456 keV, and

 $5 \times 10^{-6}$  s<sup>-1</sup>•g<sup>-1</sup> for energies between 1465 keV and 2750 keV.

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of <sup>242</sup>Pu, <sup>241</sup>Pu, or <sup>241</sup>Am.

Note 8. The stated uncertainty is the standard uncertainty. See reference 2.

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#### REFERENCES

- [1] Evaluated Nuclear Structure Data File (ENSDF), online database, National Nuclear Center, Brookhaven National Laboratory (Upton, NY), accessed November 2009. Refer to http://www.nndc.bnl.gov/nudat2/(accessed Jun 2010).
- [2] JCGM 100:2008; Evaluation of Measurement Data Guide to the Expression of Uncertainty in Measurement (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM\_100\_2008\_E.pdf (accessed May 2010); see also 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/physlab/pubs/index.cfm (accessed Jun 2010).
- [3] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: http://ts.nist.gov/WeightsAndMeasures/Metric/mpo\_pubs.cfm (accessed Jun 2010).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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