National Institute of Standards & Technology

Certificate

Standard Reference Material® 4334j

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 of SRM 4334j 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-242** massic activity, at a **Reference Time of 1200 EST, 9 August 2017**, is:

$$\mathbf{(26.08 \pm 0.13)\ Bq \bullet g^{-1}.}$$

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 4334j** 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 before the expiration of this certificate, 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. Photon-emitting-impurity analyses were provided by L. Pibida.

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: 30 May 2018

Steven J. Choquette, Director
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Table 1. Certified Massic Activity of SRM 4334j

Radionuclide	**Plutonium-242**
Reference time	**1200 EST, 09 August 2017**
Massic activity of the solution	**26.08 Bq•g^{-1}** [a]
Relative expanded uncertainty (*k* = 2)	**0.51 %** [b]

[a] Both SRM 4334j and SRM 4334i (a previous issue of ^{242}Pu) were derived from two independent gravimetric dilutions of the identical standard master solution. The massic activity of SRM 4334j is in agreement with the decay corrected massic activity of SRM 4334i to ± 0.12 %.

[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/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 4334j

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	3.1 mol•L^{-1} HNO_3
Solution density	(1.099 ± 0.002) g•mL^{-1} at 24.4 °C[(a)]
Solution mass	(5.499 ± 0.003) g[(a)]
Alpha-particle- emitting impurities	^{241}Am: (0.0021 ± 0.0003) Bq•g^{-1}[(a,b,c,d)]
Beta-particle- emitting impurities	^{241}Pu: (0.039 ± 0.009) Bq•g^{-1}[(a,d)]
Photon-emitting impurities	None detected [(e)], excepting ^{241}Am
Half-lifes used [6]	^{242}Pu: (3.73 ± 0.03) × 10^{5} a[(f)] ^{241}Pu: (14.33 ± 0.04) a ^{240}Pu: (6 561 ± 7) a ^{239}Pu: (24 100 ± 11) a ^{238}Pu: (87.74 ± 0.03) a ^{241}Am: (432.6 ± 0.6) a
Calibration methods (and instruments)	The certified massic activity for ^{242}Pu was obtained by 4πα liquid scintillation (LS) spectrometry with two commercial LS counters. Four separate measurement trials using nine LS cocktails prepared directly from the SRM solution and six cocktails prepared from a master stock solution with known gravimetric dilution factor.

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

(b) The ^{242}Pu was chemically purified 07 June 1994 at the Lawrence Livermore National Laboratory (LLNL). Americium-241, the daughter of ^{241}Pu, was removed but has been growing in since that time. Photonic emission measurements of the ^{241}Am ingrowth were made at NIST in 1998-1999 and 2017.

(c) 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^{-1}•g^{-1} for energies less than 3.1 MeV,
0.03 s^{-1}•g^{-1} for energies between 3.1 MeV and 4.4 MeV, and
0.003 s^{-1}•g^{-1} for energies greater than 5.0 MeV

(d) The ^{242}Pu was chemically purified 07 June 1994. The relative massic activities of radionuclidic impurities follow;

Radionuclide	Relative Activity at Purification Time (07 June 1994) As Measured By		
	LLNL in 1994	NIST in 1998-1999	NIST in 2017
^{242}Pu	1	1	1
^{241}Pu	--	(3.5 ± 0.4) × 10^{-3}(1,2)	(3.6 ± 1.1) × 10^{-3}(1,5)
^{240}Pu + ^{239}Pu	< 10^{-6} (3)	(2.0 ± 2.1) × 10^{-5}(1,4)	--
^{238}Pu + ^{241}Am	< 1.6 × 10^{-5} (3)	(9 ± 16) × 10^{-6}(1,4)	--
^{241}Am	--	assumed 0(2)	assumed 0 (5)

1) The stated uncertainty is the standard uncertainty.
2) The ^{241}Pu activity was calculated from a gamma-ray measurement of the ^{241}Am ingrowth as of 25 November 1998, assuming that ^{241}Am was completely removed at the time of chemical purification.
3) Using alpha-particle spectrometry. The value shown is an estimated upper limit based upon background and counting statistics. Measurements were made at LLNL in July of 1994.
4) Alpha-particle spectrometry measurements were made at the National Institute of Standards and Technology (NIST) in June and July 1999.
5) The ^{241}Pu activity was calculated from a gamma-ray measurement of the ^{241}Am ingrowth as of 01 September 2017, assuming that ^{241}Am was completely removed at the time of chemical purification.

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

1 × 10^{-3} s^{-1}•g^{-1} for energies between 20 keV and 35 keV,
6 × 10^{-4} s^{-1}•g^{-1} for energies between 40 keV and 50 keV,
5 × 10^{-4} s^{-1}•g^{-1} for energies between 55 keV and 95 keV,
4 × 10^{-4} s^{-1}•g^{-1} for energies between 100 keV and 600 keV
4 × 10^{-4} s^{-1}•g^{-1} for energies between 610 keV and 1440 keV
6 × 10^{-4} s^{-1}•g^{-1} for energies between 1450 keV and 1480 keV, and
3 × 10^{-4} s^{-1}•g^{-1} for energies between 1490 keV and 2000 keV,

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ^{242}Pu, ^{241}Pu, or ^{241}Am.

(f) The stated uncertainty is the standard uncertainty. See reference 6.

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

Uncertainty component		Assessment Type[a]	Relative standard uncertainty contribution on massic activity of ^{242}Pu (%)
1	LS measurement precision: Relative standard deviation of the mean on the great-grand mean for 4 LS measurement trials, considering all of the within-trial and between-trial components of variance. Each of the 4 grand mean values was based on 5 replicate measurements on each of either 6 or 9 LS counting sources. The typical within-trial relative standard deviation of the mean (considering the variations for the between 5 measurements and the between 6 to 9 sources) for each trial was 0.08 %. The between-trial relative standard deviation across the 4 trials was 0.18 %.	A	0.22
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	^{242}Pu decay corrections for half-life uncertainty of 0.22 %.	B	$< 10^{-10}$
8	Potential alpha- and photon-emitting impurities	B	0.1
Relative combined standard uncertainty			**0.26**
Relative expanded uncertainty (*k* = 2)			**0.51**

[a] Letter A denotes evaluation by statistical methods; B denotes evaluation by other methods.

INSTRUCTIONS FOR USE AND HANDLING

Storage: SRM 4334j 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 SDS for further information.

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 May 2018). 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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed May 2018).

[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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed May 2018).

[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 https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2018).

[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/nist-technical-note-1297 (accessed Apr 2018).

[6] Chechev, V.P.; *LNE-LNHB/CEA Table of Radionuclides*, *^{242}Pu*; (June 2009); available at http://www.nucleide.org/DDEP_WG/Nuclides/Pu-242_tables.pdf (accessed May 2018).

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 srmmsds@nist.gov*; or via the Internet at https://www.nist.gov/srm.*