National Institute of Standards & Technology

Certificate

Standard Reference Material® 4341a

Neptunium-237 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive neptunium-237 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 4341a consists of approximately 5 mL of a nitric acid solution, whose composition is specified in Table 1 and 2, contained in a flame-sealed borosilicate-glass ampoule [1].

The certified **neptunium-237** massic activity value, at a **Reference Time of 1200 EST, 01 September 2012**, is:

$$\mathbf{(152.3 \pm 1.4)\ Bq{\bullet}g^{-1}}$$

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 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 4341a** 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 NIST Physical Measurement Laboratory, Radiation and Biomolecular Physics Division, under the direction of M.P. Unterweger, Group Leader of the Radioactivity Group. The overall production, technical direction, and physical measurement leading to certification were provided by R. Collé and L. Laureano-Pérez of the NIST Radiation and Biomolecular Physics Division, Radioactivity Group. Independent confirmatory measurements of the massic activity were performed by R. Fitzgerald and photon-emitting impurity analyses were provided by L. Pibida of the NIST Radiation and Biomolecular Physics Division, Radioactivity Group.

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

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Table 1. Certified Massic Activity of SRM 4341a

Radionuclide	**Neptunium-237[(a)]**
Reference time	**1200 EST, 01 September 2012**
Massic activity of the solution	**152.3 Bq•g^{-1}[(b)]**
Relative expanded uncertainty (*k* = 2)	**0.94 %[(c)]**

[(a)] The ^{237}Np stock solution used to prepare this SRM was obtained from the National Physical Laboratory (NPL; Middlesex, UK) as part of the EUROMET action 416 (^{237}Np exercise) measurement comparison amongst national metrology institutes [6]. The stock solution was chemically purified on approximately 19-22 August 1997 by the Institute for Reference Materials and Measurements (IRMM; Geel, BE). Protactinium-233 is the daughter product that results from ^{237}Np decay and has been growing in since that time. Users should not assume that the ^{233}Pa daughter will remain in radioactive equilibrium with ^{237}Np in the SRM solution when aliquots are removed from the ampoule.

[(b)] The certified massic activity of SRM 4341a, as obtained from the 4παβ liquid scintillation based standardization, could be directly compared to the results obtained from the unweighted mean of 9 primary standardizations by 5 laboratories and performed in 1998-99 as part of the EUROMET ^{237}Np measurement comparison. NIST confirmatory standardizations of the ^{237}Np massic activity for SRM 4341a were performed by live-timed anticoincidence (LTAC) 4παβ(LS) - γ(NaI) measurements and by high-resolution HPGe gamma-ray spectrometry (γ-spec). A direct LS comparison of this SRMs standardization was also made with previous issue of ^{237}Np (SRM 4341) that was first disseminated in 1993. The results of these comparisons follow:

	Massic activity (Bq•g^{-1})	Relative Standard Uncertainty (%)	Difference (%)
SRM 4341a (LS)	152.3	0.46	---
LTAC	152.0	0.22	-0.20
γ-spec	158.0	6.5	+3.7
Relative to SRM 4341	152.5	0.46	+0.13
Relative to EUROMET	152.4	0.16	+0.07

[(c)] 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.

Table 2. Uncertified Information of SRM 4341a

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	2.0 mol•L^{-1} HNO_3
Solution density	(1.067 ± 0.002) g•mL^{-1} at 16.3 °C[(a)]
Solution mass	(5.320 ± 0.003) g[(a)]
Photon-Emitting Impurities	None detected[(b)]
Total alpha-emitting impurity activity ratio to ^{237}Np	0.0015 ± 0.0005 [6]
Half-lives used	^{237}Np: (2.144 ± 0.007) × 10^6 a [7][(c)] ^{233}Pa: 26.98 ± 0.02 d [8][(c)]
Calibration methods (and instruments)	The certified massic activity for ^{237}Np was obtained by 4παβ liquid scintillation (LS) spectrometry with three commercial LS counters. The LS detection efficiency was calculated using the CN2003 code [9] for the CIEMAT/NIST method with composition matched LS cocktails of a ^{3}H standard as the efficiency detection monitor. Confirmatory measurements were also performed by high-resolution HPGe gamma-ray spectrometry, and by 4παβ(LS) - γ(NaI) anticoincidence counting.

(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 October 2012 are:

200 s^{-1}•g^{-1} for energies between 30 keV and 115 keV,
100 s^{-1}•g^{-1} for energies between 120 keV and 290 keV,
250 s^{-1}•g^{-1} for energies between 295 keV and 320 keV,
100 s^{-1}•g^{-1} for energies between 330 keV and 360 keV,
100 s^{-1}•g^{-1} for energies between 370 keV and 430 keV, and
20 s^{-1}•g^{-1} for energies between 440 keV and 2000 keV.

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ^{237}Np or progeny.

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

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4341a

	Uncertainty component	Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of ^{237}Np (%)
1	LS measurement precision; standard deviation of the mean for 4 sets of measurements obtained with 3 different LS counters; each set of 6 LS sources was measured 3 to 5 times in each counter on 1 or 2 occasions. The typical internal relative standard deviation of the mean within a measurement data set was typically 0.03 % for $n = 18$ to $n = 30$ measurements with 6 LS sources.	A	0.12
2	Background LS measurement variability and cocktail stability; wholly embodied in component 1	B	---
3	Live time determinations for LS counting time intervals, includes uncorrected dead time effects; assumed from specified tolerance limits of counters' gated oscillators	B	0.10
4	LS α-detection inefficiency for ^{237}Np	B	<0.01
5	Gravimetric (mass) determinations for LS sources, dilution factors and counting source preparations	B	0.17
6	Decay corrections for ^{237}Np and ^{233}Pa; half-life uncertainties of 0.07 % and 0.33 %, respectively [6]	B	2×10^{-7}
7	Assumed radioactive equilibrium between ^{237}Np and ^{233}Pa in the LS sources after 33 days of decay; wholly embodied in component 1	B	---
8	Uncertainty in massic activity for the ^{3}H efficiency monitor; includes that for the ^{3}H standard of 0.36 % and decay corrections for ^{3}H half-life uncertainty of 0.16 % [6]	B	0.06
9	Calculated beta efficiency for ^{233}Pa, including uncertainties in decay scheme data	B	0.4
10	Impurities, report of alpha impurity activity ratio to ^{237}Np of 0.0015 (5) from the 1997 EUROMET measurement comparison [6] of the master solution. No photon-emitting impurities were found. No ^{241}Am was found, indicating that beta-emitting ^{241}Pu was not present.	B	0.05
Relative combined standard uncertainty			**0.47**
Relative expanded uncertainty (*k* = 2)			**0.94**

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

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. Only persons qualified to handle both radioactive material and alkaline and/or acidic solutions, 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 4341a 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 Mar 2013). 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 Mar 2013).

[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 Mar 2013).

[4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (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 Mar 2013).

[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://www.nist.gov/pml/pubs/index.cfm (accessed Mar 2013).

[6] Smith D., Woods M.J., Woods D.H.; *Results from the ^{237}Np Exercise EUROMET Action 416*, NPL Report CIRM 43, p 76 (2001); available at http://publications.npl.co.uk/npl_web/pdf/CIRM43.pdf (accessed Mar 2013)

[7] Chechev, V.P.; Kuzmenko N.K.; *July 2010, ^{237}Np*; LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Np-237_tables.pdf (accessed Mar 2013).

[8] Chechev, V.P.; Kuzmenko N.K.; *July 2010, ^{233}Pa*; LNE-LNHB/CEA Table of Radionuclides, available at http://www.nucleide.org/DDEP_WG/Nuclides/Pa-233_tables.pdf (accessed Mar 2013).

[9] Gunther, E.; Physikalisch-Technische Bundesanstalt (Braunschweig, Germany), personal communication (2003).

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.