National Institute of Standards & Technology

Certificate

Standard Reference Material® 4340B
Plutonium-241 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive plutonium-241 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. The solution, whose composition is specified in Table 1, is contained in a flame-sealed, 5 mL borosilicate-glass ampoule (see Note 1)*.

The certified **Plutonium-241** massic activity value, at a **Reference Time of 1200 EST, 15 June 2007**, is:

$$\mathbf{(258.5 \pm 9.8)\ Bq{\bullet}g^{-1}}$$

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

Expiration of Certification: The certification of this SRM, within the measurement uncertainties specified, is valid for at least five (5) years after receipt. The solution matrix, in an unopened ampoule, is believed to be indefinitely homogeneous and stable within its half-life-dependent, useful lifetime.

Maintenance of Certification: NIST will monitor this material and will report any substantive changes in certification to the purchaser. Should any of the certified values change, purchasers of this SRM will be notified of the change by NIST.

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, Acting Group Leader. The overall technical direction and physical measurements leading to certification were provided by R. Collé, and L. Laureano-Pérez of the Radioactivity Group, with production assistance by D.B. Golas and O. Palabrica, Research Associates of the Nuclear Energy Institute, confirmatory measurement assistance by P. Cassette of Laboratoire National Henri Becquerel and B. Zimmerman, photonic emission impurity analyses by M. Hammond, and alpha spectrometric measurements by I. Outola. The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Measurement Services Division.

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*Notes and references are on page 5

Table 1. Properties of SRM 4340B

Certified values

Radionuclide	**Plutonium-241**
Reference time	**1200 EST, 15 June 2007**
Massic activity of the solution	**258.5 Bq•g^{-1}**
Relative expanded uncertainty (*k* = 2)	**3.8 %** (see Note 2)*

Uncertified information

Source description	Liquid in flame-sealed, 5 mL borosilicate-glass ampoule (see Note 1)
Solution composition	2.8 mol•L^{-1} HNO_3
Solution density	(1.087 ± 0.002) g•mL^{-1} at 22.1 °C (see Note 3)
Solution mass	(5.5050 ± 0.0003) g (see Note 3)
Alpha-particle-emitting impurities (see Note 4)	^{241}Am: 0.229 ± 0.006 Bq•g^{-1} ^{239,240}Pu: 0.108 ± 0.007 Bq•g^{-1} ^{238}Pu: 0.041 ± 0.002 Bq•g^{-1} ^{242}Pu: 0.002 ± 0.001 Bq•g^{-1}
Photon-emitting impurities	None detected above limits (see Note 5)
Half-lifes used [2] (See Note 6)	^{241}Pu : (14.33 ± 0.04) a ^{241}Am : (432.6 ± 0.6) a ^{239}Pu : (24110 ± 30) a ^{240}Pu : (6561 ± 7) a ^{238}Pu : (87.74 ± 0.03) a ^{242}Pu : (3.73 ± 0.03) × 10^{5} a ^{3}H : (12.32 ± 0.02) a
Nuclear data used in CN2003 [1] computations (beta-particle maximum energies; branching ratios; transitions) [2] (See Note 6)	^{3}H : (18.594 ± 0.008) keV; 1, allowed ^{241}Pu: (20.78 ± 0.13) keV; 1, first forbidden
Calibration method (and instruments)	The certified massic activity for ^{241}Pu was obtained by 4παβ liquid scintillation (LS) spectrometry with three commercial LS counters. The LS β detection efficiency was derived by the CIEMAT/NIST efficiency tracing (CNET) method using CN2003 code calculations with composition matched LS cocktails of a ^{3}H standard as the efficiency detection monitor (See Note 7). Confirmatory determinations, on linked ^{241}Pu solutions, were also performed by ^{241}Am ingrowth measurements and by LS counting using a triple-to-double coincidence ratio (TDCR) method. (See Note 8)

 *Notes and references are on page 5

Table 2. Uncertainty evaluation for the massic activity of SRM 4340B

Uncertainty component		Assessment Type †	Relative standard uncertainty contribution on massic activity of ^{241}Pu (%)
1	LS measurement precision; reproducibility in massic activity for 3 cocktail compositions, each with 5 to 6 samples, measured in 3 counters on 1 or 2 measurement occasions; standard deviation of the mean for n = 12 data sets (ν = 312 effective degrees freedom), normally distributed. The LS within-measurement precision for a given data set, in terms of the standard deviation of the mean for 5 to 6 samples measured for 2 to 10 cycles on three measurement occasions, ranged from 0.43 % to 1.46 %	A	1.7
2	Background; wholly embodied in 1	A	--
3	Cocktail composition dependencies (for an internal relative standard deviation of 0.06 % for 3 compositions); wholly embodied in component 1	A	--
4	LS counters dependencies (for an internal relative standard deviation of 0.09 % for 3 counters on one or two occasions); wholly embodied in component 1	A	--
5	Gravimetric (mass) measurements for LS sources and for ^{3}H standard dilution	B	0.15
6	Live time determinations for LS counting time intervals, includes uncorrected dead time effects	B	0.06
7	Massic activity of ^{3}H (for uncertainty in standard of 0.36 %)	B	0.32
8	^{3}H decay corrections for half life uncertainty of 0.16 % [2]	B	0.0009
9	^{241}Pu decay corrections for half-life uncertainty of 0.28 % [2]	B	0.0013
10	Correction for ^{241}Am ingrowth and alpha-emitting impurities	B	0.006
11	Limit for photon-emitting impurities	B	0.09
12	Computed β detection efficiencies (model dependencies and computed β spectra)	B	0.8
Relative combined standard uncertainty			**1.9**
Relative expanded uncertainty (*k* = 2)			**3.8**

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

NOTES

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 in 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 (see references [3] and [4]). 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 [4]

Note 4: The estimated limits of detection for alpha-emitting-impurities, expressed as a massic alpha emission rate, were:

0.0003 $s^{-1}\bullet g^{-1}$ for energies between 3.0 MeV and 4.75 MeV, and
0.0003 $s^{-1}\bullet g^{-1}$ for energies between 5.6 MeV end 12 MeV.

Note 5. The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rates, on 9 May 2007 were:

0.15 $s^{-1}\bullet g^{-1}$ for energies between 30 keV and 450 keV, and
0.24 $s^{-1}\bullet g^{-1}$ for energies between 451 keV and 1800 keV, provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ^{241}Am and progeny.

Note 6. The stated uncertainty is the standard uncertainty. See reference [4].

Note 7. The computed β detection efficiencies were invariant of the CNET model's ionization quench parameter *kB* over a range of 0.009 $cm\cdot MeV^{-1}$ to 0.015 $cm\cdot MeV^{-1}$. The efficiencies from the CN2003 tracing code [1] agreed with those from the TRACER code [5] to about 0.04 % for $kB = 0.012$ $cm\cdot MeV^{-1}$ at a ^{3}H efficiency of 30 %. The tracing results using efficiencies from the EFFY4 code [6] would lower the ^{241}Pu massic activity by 0.7 % as a result of differences in the ionization quenching functions used in the codes..

Note 8. The certified ^{241}Pu massic activity is wholly consistent with the certified activity in SRM 4340A issued in 1996. The certified massic activity in this SRM (SRM 4340B) was based on LS CNET measurements, but was independently confirmed by agreement with ^{241}Am ingrowth measurements that have been performed over a period of 31 years on a linked ^{241}Pu solution. Two independent determinations by NIST and the Laboratoire National Henri Becquerel on gravimetrically linked solutions, based on LS measurements using the TDCR method, are in excellent agreement with each other but are discrepant with the certified activity given here.

REFERENCES

[1] Gunther, E., Physikalisch-Technische Bundesanstalt (Braunschweig, Germany). Private communication, 2003.

[2] Laboratoire National Henri Becquerel, C.E.A. Saclay - Gif-sur-Yvette Cedex - France. *Table of Radionuclides, Recommended Data (on-line),* updated February 28, 2008. http://www.nucleide.org/DDEP_WG/DDEPdata.htm (accessed February 29, 2008)

[3] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993 (corrected and reprinted, 1995). Ordering and purchasing information available at http://physics.nist.gov/cuu/Uncertainty/isoorder.html

[4] 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.physics.nist.gov/Pubs/guidelines/contents.html

[5] P. Cassette, Laboratoire National Henri Becquerel (Gif-sur-Yvette, France). Private communication, September 2007.

[6] García-Toraño, E., Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT), private

communication on *EFFY4*, 1993; García-Toraño, E., Grau Malonda, A., Effy, A New Program to Compute the Counting Efficiency of Beta Particles in Liquid Scintillators, *Comput. Phys. Commun.*, **36**, 307-312, 1985.

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.