National Institute of Standards & Technology

Certificate

Standard Reference Material 4338A
Plutonium-240 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive plutonium-240 nitrate and nitric acid dissolved in 5 mL of distilled water. The solution is contained in a flame-sealed NIST borosilicate-glass ampoule. The SRM is intended for the calibration of alpha-particle counting instruments and for the monitoring of radiochemical procedures.

Radiological Hazard: The SRM ampoule contains plutonium-240 with a total activity of approximately 230Bq. Plutonium-240 decays by alpha-particle emission. None of the alpha particles escape from the SRM ampoule. During the decay process X-rays and gamma rays, with energies from 45 keV to 913 keV are also emitted. Most of these photons escape from the SRM ampoule but their intensities are so small that they do not represent a radiation hazard. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]*. The SRM should be used only by persons qualified to handle radioactive material.

Chemical Hazard: The SRM ampoule contains nitric acid (HNO_3) with a concentration of 3 moles per liter of water. The solution is corrosive and represents a health hazard if it comes in contact with eyes or skin. If the ampoule is to be opened to transfer the solution, the recommended procedure is given on page 2. The ampoule should be opened only by persons qualified to handle both radioactive material and strong acid solution.

Storage and Handling: The SRM should be stored and used at a temperature between 5 and 65 °C. The solution in an unopened ampoule should remain stable and homogeneous for at least five (5) years after receipt. The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material. 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.

Preparation: This Standard Reference Material was prepared in the Physics Laboratory, Ionizing Radiation Division, Radioactivity Group, J.M.R. Hutchinson, Group Leader. The overall technical direction and physical measurements leading to certification were provided by L.L. Lucas, formerly of the Radioactivity Group. The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program.

Lisa R. Karam, Deputy Chief
Ionizing Radiation Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, Maryland 20899
August 1996 (Text only revised November 1997)
Text revised and expiration date extended March 2006

*Notes and references are on pages 5 to 7.

Recommended Procedure for Opening the SRM Ampoule

1) If the SRM solution is to be diluted, it is recommended that the diluting solution have a composition comparable to that of the SRM solution.

2) Wear eye protection, gloves, and protective clothing and work over a tray with absorbent paper in it. Work in a fume hood. In addition to the radioactive material, the solution contains strong acid and is corrosive.

3) Shake the ampoule to wet all of the inside surface of the ampoule. Return the ampoule to the upright position.

4) Check that all of the liquid has drained out of the neck of the ampoule. If necessary, gently tap the neck to speed the process.

5) Holding the ampoule upright, score the narrowest part of the neck around its entire circumference with a scribe or diamond pencil.

6) Lightly wet the scored line. This reduces the crack propagation velocity and makes for a cleaner break.

7) Hold the ampoule upright with a paper towel, a wiper, or a support jig. Using a paper towel or wiper to avoid contamination, snap off the top of the ampoule by pressing the narrowest part of the neck away from you while pulling the tip of the ampoule towards you.

8) Transfer the solution from the ampoule using a pycnometer or a pipet with dispenser handle. NEVER PIPETTE BY MOUTH.

9) Seal any unused SRM solution in a flame-sealed glass ampoule, if possible, to minimize the evaporation loss.

See also reference [4]*.

*Notes and references are on pages 5 to 7.

PROPERTIES OF SRM 4338A

Certified values

Radionuclide	**Plutonium-240**
Reference time	**1200 EST, 1 May 1996** [b]*
Massic activity of the solution [c]	**40.88 Bq •g^{-1}**
Relative expanded uncertainty (*k*=2)	**0.76%** [d][e]
Solution mass	**(5.471 ± 0.003) g** [b]

Uncertified values

Physical Properties:			
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule		
Ampoule specifications	Body outside diameter (16.5 ± 0.5) mm Wall thickness (0.60 ± 0.04) mm Barium content Less than 2.5% Lead-oxide content Less than 0.02% Other heavy elements Trace quantities		
Solution density	(1.091 ± 0.002) g•mL^{-1} at 20 °C [f]		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol•L^{-1})	Mass Fraction (g•g^{-1})
	H_2O HNO_3 HCl $^{240}Pu^{+4}$	51 2.8 0.085 2×10^{-8}	0.84 0.16 0.003 5×10^{-9}
Radiological Properties:			
Alpha-particle-emitting impurities (see table on page 5)	Plutonium-238: (0.38 ± 0.08) Bq •g^{-1} [f] [b] Americium-241: (0.01 ± 0.007) Bq •g^{-1} [f] [g]		
Photon-emitting impurities	None detected [h]		
Half lives used in the decay corrections	Plutonium-238: (87.7 ± 0.3) a [5][i] Plutonium-239: (24110 ± 30) a [5][i] Plutonium-240: (6564 ± 11) a [5][i] Plutonium-241: (14.35 ± 0.10) a [5][i] Plutonium-242: $(3.733 \pm 0.012) \times 10^5$ a [5][i] Plutonium-244: $(8.08 \pm 0.10) \times 10^7$ a [5][i] Americium-241: (432.2 ± 0.7) a [5][i]		
Calibration method and measuring instrument(s)	NIST "0.1π"α defined-solid-angle counter with scintillation detector, two 4πα liquid-scintillation counting systems, and a silicon surface-barrier alpha spectrometry system.		

*Notes and references are on pages 5 to 7.

EVALUATION OF THE UNCERTAINTY OF THE MASSIC ACTIVITY [d][e]*

Input Quantity x_i, the source of uncertainty (and individual uncertainty components where appropriate)	Method Used To Evaluate $u(x_i)$, the standard uncertainty of x_i (A) denotes evaluation by statistical methods (B) denotes evaluation by other methods	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$, (%) [j]	Relative Sensitivity Factor, $\|\partial y/\partial x_i\| \cdot (x_i/y)$ [k]	Relative Uncertainty Of Output Quantity, $u_i(y)/y$, (%) [m]
Massic alpha-particle-emission rate, corrected for background and decay	Standard deviation of the mean for 5 sets of "0.1π"α measurements and four sets of 4πα liquid scintillation measurements. (A)	0.11	1.0	0.11
Half-life of Pu-240	Standard uncertainty of the half life (A)	0.17 [n]	0.002 [p]	0.0003
Decay-scheme data	Standard uncertainty of the probability of decay by alpha-particle emission (A)	0.01	1.0	0.01
Extrapolation of alpha-particle-count-rate-versus-energy to zero energy	Estimated (B)	0.25	1.0	0.25
Gravimetric measurements	Estimated (B)	0.10	1.0	0.10
Live time [q]	Estimated (B)	0.10	1.0	0.10
Alpha-particle detection efficiency of scintillators	Estimated (B)	0.10	1.0	0.10
Geometry of "0.1π"α counter	Estimated (B)	0.10	0.6	0.06
Alpha-particle-emitting impurities	Estimated (B) [r] Limit of detection (B) [s]	10 100	0.01 0.001	0.10 0.10
Photon-emitting impurities	Limit of detection (B) [s]	100	0.001	0.10

Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$, (%)	0.37
Coverage Factor, k	x 2
Relative Expanded Uncertainty of the Output Quantity, U/y, (%)	0.74

*Notes and references are on pages 5 to 7.

ATOM FRACTIONS AND RELATIVE ACTIVITIES OF RADIONUCLIDIC IMPURITIES

Radionuclide	Half Life (years) [i]	Atom Fraction 24 July 1979	Relative Activity 1 May 1996
Pu-238 (α)	87.7 ± 0.3	0.00014	0.009
Pu-239 (α)	24110 ± 30	0.00023	0.00006
Pu-240 (α)	6564 ± 11	0.99930	1.000000
Pu-241 (α + β)	14.35 ± 0.10	0.00003	(0.006)
Pu-241 (α only)	14.35 ± 0.10	--	0.000000
Pu-242 (α)	$(3.733 \pm 0.012) \times 10^5$	0.00029	0.000005
Pu-244 (α)	$(8.08 \pm 0.10) \times 10^7$	0.00001	0.000000
Am-241 (α)	432.2 ± 0.7	=0 [b]	0.0003
Total Alpha	--	--	1.0096

NOTES

[a] The Sievert is the SI unit for dose equivalent. See reference [1]. One µSv is equal to 0.1 mrem.

Distance from Ampoule (cm):	1	30	100
Approximate Dose Rate (µSv/h):	< 0.1	-	-

[b] The Plutonium-240 master solution was chemically purified on approximately 10 December 1979. The daughter radionuclides have been growing in since that time.

[c] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].

[d] The reported value, y, of massic alpha-particle emission rate (alpha-particle emission rate per unit mass) at the reference time was not measured directly but was derived from measurements and calculations of other quantities. This can be expressed as $y = f(x_1, x_2, x_3, \ldots x_n)$, where f is a mathematical function derived from the assumed model of the measurement process. The value, x_i, used for each input quantity i has a **standard uncertainty**, $u(x_i)$, that generates a corresponding uncertainty in y, $u_i(y) \equiv |\partial y/\partial x_i| \cdot u(x_i)$, called a **component of combined standard uncertainty** of y. The **combined standard uncertainty** of y, $u_c(y)$, is the positive square root of the sum of the squares of the components of combined standard uncertainty. The combined standard uncertainty is multiplied by a **coverage factor** of $k = 2$ to obtain U, the **expanded uncertainty** of y.

Since it can be assumed that the possible estimated values of the massic alpha-particle emission rate are approximately normally distributed with approximate standard deviation $u_c(y)$, the unknown value of the massic activity is believed to lie in the interval $y \pm U$ with a level of confidence of approximately 95 percent.

For further information on the expression of uncertainties, see references [2] and [3].

[e] The value of each component of combined standard uncertainty, and hence the value of the expanded uncertainty itself, is a best estimate based upon all available information, but is only approximately known. That is to say, the "uncertainty of the uncertainty" is large and not well known. This is true for uncertainties evaluated by statistical methods (e.g., the relative standard deviation of the standard deviation of the mean for the massic response is approximately 50%) and for uncertainties evaluated by other methods (which could easily be over estimated or under estimated by substantial amounts). The unknown value of the expanded uncertainty is believed to lie in the interval $U/2$ to $2U$ (i.e., within a factor of 2 of the estimated value).

[f] The stated uncertainty is two times the standard uncertainty.

[g] Estimated limits of detection for alpha-particle-emitting impurities, expressed as massic alpha-particle emission rates, are:
0.04 $s^{-1}\bullet g^{-1}$ for energies less than 5.0 MeV,
0.04 $s^{-1}\bullet g^{-1}$ for energies between 5.25 and 5.35 MeV, and
0.04 $s^{-1}\bullet g^{-1}$ for energies greater than 5.6 MeV.

[h] Estimated limits of detection for photon-emitting impurities, expressed as massic photon emission rates, are:
0.00015 $s^{-1}\bullet g^{-1}$ for energies between 12 keV and 41 keV,
0.00008 $s^{-1}\bullet g^{-1}$ for energies between 49 keV and 100 keV,
0.00005 $s^{-1}\bullet g^{-1}$ for energies between 108 keV and 156 keV,
0.00001 $s^{-1}\bullet g^{-1}$ for energies between 164 keV and 507 keV,
0.000004 $s^{-1}\bullet g^{-1}$ for energies between 515 keV and 1456 keV,and
0.000002 $s^{-1}\bullet g^{-1}$ for energies between 1464 keV and 2750 keV, provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of plutonium-240.

[i] The stated uncertainty is the standard uncertainty.

[j] Relative standard uncertainty of the input quantity x_i.

[k] The relative change in the output quantity y divided by the relative change in the input quantity x_i. If $|\partial y/\partial x_i|\bullet(x_i/y) = 1.0$, then a 1% change in x_i results in a 1% change in y. If $|\partial y/\partial x_i|\bullet(x_i/y) = 0.05$, then a 1% change in x_i results in a 0.05% change in y.

[m] Relative component of combined standard uncertainty of output quantity y, rounded to two significant figures or less. The relative component of combined standard uncertainty of y is given by $u_i(y)/y \equiv |\partial y/\partial x_i|\bullet u(x_i)/y = |\partial y/\partial x_i|\bullet(x_i/y) \bullet u(x_i)/x_i$. The numerical values of $u(x_i)/x_i$, $|\partial y/\partial x_i|\bullet(x_i/y)$, and $u_i(y)/y$, all dimensionless quantities, are listed in columns 3, 4, and 5, respectively. Thus, the value in column 5 is equal to the value in column 4 multiplied by the value in column 3. The input quantities are independent, or very nearly so. Hence the covariances are zero or negligible.

[n] The relative standard uncertainty of $\lambda\bullet t$ is determined by the relative standard uncertainty of λ (i.e., of the half life). The relative standard uncertainty of t is negligible.

[p] $|\partial y/\partial x_i|\bullet(x_i/y) = |\lambda\bullet t|$

[q] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.

[r] The standard uncertainty given is for the detected impurities. $|\partial y/\partial x_i|\bullet(x_i/y)$ = {(response per Bq of impurity)/(response per Bq of plutonium-240)}•{(Bq of impurity)/(Bq of plutonium-240)}.

[s] The standard uncertainty for each undetected impurity that might reasonably be expected to be present is estimated to be equal to the estimated limit of detection for that impurity, i.e. $u(x_i)/x_i = 100\%$. $|\partial y/\partial x_i| \cdot (x_i/y)$ = {(response per Bq of impurity)/(response per Bq of plutonium-240)}$\cdot${(Bq of impurity)/(Bq of plutonium-240)}. Thus, $u_i(y)/y$ is the relative change in y if the impurity were present with a massic activity equal to the estimated limit of detection.

REFERENCES

[1] International Organization for Standardization (ISO), *ISO Standards Handbook - Quantities and Units*, 1993. Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.

[2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993 (corrected and reprinted, 1995). Available from Global Engineering Documents, 12 Inverness Way East, Englewood, CO 80112, U.S.A. Telephone 1-800-854-7179.

[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 from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20407, U.S.A.

[4] National Council on Radiation Protection and Measurements Report No. 58, *A Handbook of Radioactivity Measurements Procedures*, Second Edition, 1985. Available from the National Council on Radiation Protection and Measurements, 7910 Woodmont Avenue, Bethesda, MD 20814 U.S.A.

[5] Evaluated Nuclear Structure Data File (ENSDF), February 1996.