



# National Institute of Standards & Technology

## Certificate

### Standard Reference Material 4323A Plutonium-238 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive plutonium-238 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-238 with a total activity of approximately 150 Bq. Plutonium-238 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 10 keV to 1 MeV 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 ( $\text{HNO}_3$ ) 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 until at least February 2004.

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) both because of the radioactivity and because of 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 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 by N.M. Trahey.

Gaithersburg, Maryland 20899  
February 1995

Thomas E. Gills, Chief  
Standard Reference Materials Program

### Recommended Procedure for Opening the SRM Ampoule

- 1) If the SRM solution is to be diluted, it is recommended that the diluting solution have an acid concentration 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 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. Position the scored line away from you. 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]\*.

PROPERTIES OF SRM 4323A  
(Certified values are shown in bold type)

Source identification number	NIST SRM 4323A		
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	<b>(1.103 ± 0.002) g·mL<sup>-1</sup> at 22.6 °C [b]*</b>		
Solution mass	Approximately 5.5 g		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol·L <sup>-1</sup> )	Mass Fraction (g·g <sup>-1</sup> )
	H <sub>2</sub> O	50	0.82
	HNO <sub>3</sub>	3.2	0.18
	HCl	<0.02	<0.0005
	Pu(NO <sub>3</sub> ) <sub>6</sub>	2 × 10 <sup>-10</sup>	1 × 10 <sup>-10</sup>
Radiological Properties:			
Radionuclide	Plutonium-238		
Reference time	1200 EST, 19 February 1994		
Massic activity of the solution [c]	<b>30.08 Bq·g<sup>-1</sup></b>		
Relative expanded uncertainty (k=2)	<b>0.68% [d]</b>		
Alpha-particle-emitting impurities	None detected [e]		
Photon-emitting impurities	None detected [f]		
Half lives used	Plutonium-238: (87.74 ± 0.04) a [g]		
Measuring instruments	NIST "0.1π"α defined-solid-angle counter with scintillation detector and two 4πα liquid-scintillation counting systems		

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

Input Quantity $x_i$ , the source of uncertainty	Method Used To Evaluate $u(x_i)$ , the standard uncertainty of $x_i$	Relative Uncertainty Of Input Quantity, $u(x_i)/x_i$ , (%) [h]	Relative Sensitivity Factor, $ \partial y/\partial x_i  \cdot$ $(x_i/y)$ [i]	Relative Uncertainty Of Output Quantity, $u_i(y)/y$ , (%) [j]
Uncertainties Evaluated By Statistical Methods				
Massic alpha-particle emission rate	Standard deviation of the mean for nine "0.1 $\pi$ " $\alpha$ measurements and thirty-one 4 $\pi$ liquid-scintillation measurements	0.07	1.0	0.07
Background	Standard deviation of the mean for 40 repeated measurements	1.43	[k] 0.08	0.11
Uncertainties Evaluated By Other Means				
Mass calibration of the balance	Estimated from manufacturer's data	0.05	1.0	0.05
Decay correction for plutonium-238	Standard uncertainty of the half life	0.05 [m]	0.02 [n]	0.001
Decay-scheme data	Standard uncertainty of the probability of decay by alpha-particle emission	0.01	1.0	0.01
Extrapolation of alpha-particle-count-rate-versus-energy to zero energy	Estimated	0.25	1.0	0.25
Live-time [p]	Estimated	0.10	1.0	0.10
Alpha-particle detection efficiency of scintillators	Estimated	0.10	1.0	0.10
Geometry of "0.1 $\pi$ " $\alpha$ counter	Estimated	0.25	0.2	0.05
Alpha-particle-emitting impurities	Limit of detection [q]	100.	0.001	0.10
Photon-emitting impurities	Limit of detection [q]	100.	0.0001	0.01
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)				
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)		0.34		
Coverage Factor, $k$		$\times 2$		
Relative Expanded Uncertainty of the Output Quantity, $U(y)$ , (%)		0.68		

## NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One  $\mu\text{Sv}$  is equal to 0.1 mrem.  
 Distance from Ampoule (cm):           1       30       100  
 Approximate Dose Rate ( $\mu\text{Sv}/\text{h}$ ) 0.1       -       -

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

[c] **Massic activity** is the preferred name for the quantity activity per unit mass. See reference [1].

[d] The reported value,  $y$ , of massic activity (activity 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  $y = f(x_1, x_2, x_3, \dots, x_n)$ , 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  $y$ ,  $u_i(y) = |\partial y / \partial x_i| \cdot u(x_i)$ , 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 activity 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] Estimated limits of detection for alpha-particle-emitting impurities are:  
 0.03  $\alpha \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies less than 5.25 MeV and  
 0.001  $\alpha \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies greater than 5.5 MeV.

A (Pu-239/Pu-238) atom ratio of 0.00107 was measured by mass spectrometry in 1981. As of the reference time, the massic activity of plutonium-239 is well below the detection limits and is calculated to be approximately 0.0001  $\text{Bq} \cdot \text{g}^{-1}$ .

- [f] Estimated limits of detection for photon-emitting impurities are:  
 $3 \times 10^{-4} \gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies between 6 and 39 keV,  
 $1 \times 10^{-4} \gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies between 48 and 95 keV,  
 $5 \times 10^{-5} \gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies between 103 and 147 keV,  
 $1 \times 10^{-5} \gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies between 157 and 762 keV, and  
 $2 \times 10^{-6} \gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$  for energies between 770 and 1900 keV,  
 provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of plutonium-238.

[g] Evaluated Nuclear Structure Data File (ENSDF), January 1995. The stated uncertainty is the standard uncertainty.

[h] Relative standard uncertainty of the input quantity  $x_i$ .

- [i] The relative change in the output quantity  $y$  divided by the relative change in the input quantity  $x_i$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 1.0$ , then a 1% change in  $x_i$  results in a 1% change in  $y$ . If  $|\partial y/\partial x_i| \cdot (x_i/y) = 0.05$ , then a 1% change in  $x_i$  results in a 0.05% change in  $y$ .
- [j] 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  $u_i(y)/y \approx |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$ . The numerical values of  $u(x_i)/x_i$ ,  $|\partial y/\partial x_i| \cdot (x_i/y)$ , 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.
- [k]  $|\partial y/\partial x_i| \cdot (x_i/y) = (\text{average background count rate})/(\text{average net sample count rate})$ .
- [m] The relative standard uncertainty of  $\lambda \cdot t$  is determined by the relative standard uncertainty of  $\lambda$  (i.e., of the half life). The relative standard uncertainty of  $t$  is negligible.
- [n]  $|\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$
- [p] The live time is determined by counting the pulses from a gated oscillator.
- [q] 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.  $u(x_i)/x_i = 100\%$ .  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of Pu-238})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of Pu-238})\}$ . 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 the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900.
- [2] International Organization for Standardization (ISO), *Guide to the Expression of Uncertainty in Measurement*, 1993. Available from the American National Standards Institute, 11 West 42nd Street, New York, NY 10036, U.S.A. 1-212-642-4900. (Listed under ISO miscellaneous publications as "ISO Guide to the Expression 1993".)
- [3] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, 1993. 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.