

# National Institute of Standards & Technology **Certificate**

# Standard Reference Material 4326 Polonium-209 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive Polonium-209 chloride and hydrochloric 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 Polonium-209 with a total activity of approximately 500Bq. Polonium-209 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 approximately 10 keV to 900 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 hydrochloric acid with a concentration of 2 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. Refer to reference [5] for details on the long-term stability of polonium solution standards. 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 R. Collé of the Radioactivity Group, and Z. Lin, Guest Researcher. 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

Gaithersburg, Maryland 20899 January 1995 (Text only revised November 1997) Text revised and expiration date extended December 2005

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

#### **Liquid-Scintillation Counting Warning**

Polonium-209 decays primarily by alpha-particle emission. One of the principal alpha-particle transition feeds a low-energy, **delayed**, isomeric state in the lead-205 daughter. Liquid scintillation measurements of polonium-209 activity can include some of the activity of this isomeric state. Refer to reference [6] for further information about the effect of this isomeric transition on routine liquid-scintillation counting of polonium-209.

#### **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.
- 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]\*.

## PROPERTIES OF SRM 4326

## **Certified values**

Radionuclide	Polonium-209
Reference time	1200 EST, 15 March 1994
Massic alpha-particle-emission rate of the solution [b]*	<b>85.42</b> s <sup>-1</sup> •g <sup>-1</sup> (Polonium-209 only) [c]
Relative expanded uncertainty (k=2)	<b>0.42%</b> [d]
Solution mass	$(5.160 \pm 0.003) g [e]$
Solution density	$(1.031 \pm 0.004) \text{ g·mL}^{-1} \text{ at } 22 \text{ °C } [e]$

# Uncertified values

Physical Properties:						
Source description	Liquid in flame-sealed NIST borosilicate-glass ampoule					
Ampoule specifications	$ \begin{array}{llllllllllllllllllllllllllllllllllll$					
Chemical Properties:						
Solution composition	Chemical Formula	Concentration (mol·L <sup>-1</sup> )	Mass Fraction (g•g <sup>-1</sup> )			
	H <sub>2</sub> O HCl HNO <sub>3</sub> PoCl <sub>4</sub>	$ \begin{array}{c} 53 \\ 2.0 \\ < 3 \times 10^{-3} \\ 7 \times 10^{-10} \end{array} $	$0.93 \\ 0.07 \\ < 2 \times 10^{-4} \\ 2 \times 10^{-10}$			
Radiological Properties:						
Alpha-particle-emitting impurities (massic alpha-particle emission rate)	Polonium-208: $(0.106 \pm 0.017) \text{ s}^{-1} \cdot \text{g}^{-1} \text{ [e, f]}$					
Photon-emitting impurities	None detected [g]					
Half lives used	Polonium-209: (102 ± 5) a [8][h] Polonium-208: (2.898 ± 0.002) a [8][h]					
Calibration method and measuring instrument(s)	Two $4\pi\alpha$ liquid-scintillation counting systems, $2\pi\alpha$ gas-flow proportional counter, and silicon surface-barrier detector.					

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

Imput Overtites	Mothod Hand To Fredricks ( )	Dolotica	Daletina	Dolotica	
Input Quantity $x_i$ , the source of uncertainty	Method Used To Evaluate $u(x_i)$ , the standard uncertainty of $x_i$ (A) denotes evaluation by	Relative Uncertainty Of Input	Relative Sensitivity Factor,	Relative Uncertainty Of Output	
(and individual uncertainty components where appropriate)	statistical methods (B) denotes evaluation by other methods	Quantity, $u(x_i)/x_i$ , (%) [i]	$  \partial y/\partial x_i  \cdot \\ (x_i/y) \\ [j] $	Quantity, $u_i(y)/y$ , (%) [k]	
Massic liquid scintillation count rate, corrected for background and decay	Standard deviation for repeated measurements. Six degrees of freedom. (A)	0.06	1.0	0.06	
Background variability	Multiple comparisons (A) [7]	0.20	0.02 [m]	0.004	
Liquid-scintillator quench corrections	Multiple comparisons (A) [7]	0.12	1.0	0.12	
Liquid-scintillation- cocktail stability	Multiple comparisons (A) [7]	0.7	0.007	0.005	
Gravimetric measurements	Estimated (B)	0.05	1.0	0.05	
Half-life of Po-208 Half-life of Po-209	Standard uncertainty of the half life (A)	0.07 [n] 4.9 [n]	0.0003 [p] 0.004 [p]	0.00002 0.02	
Extrapolation of alpha- particle-count-rate- versus-energy to zero energy	Estimated (B) [7]	0.06	1.0	0.06	
Live time [q]	Estimated (B) [7]	0.04	1.0	0.04	
Alpha-particle detection efficiency of scintillator	Estimated (B) [7]	0.10	1.0	0.10	
Correction for non- alpha-particle-decay modes	Estimated (B) [7]	0.06	1.0	0.06	
Alpha-particle-emitting impurities	Estimated (B) [r] Limit of detection [s]	8.1 100	0.001 0.0006	0.01 0.06	
Photon-emitting impurities	Limit of detection (B) [s]	100	0.0002	0.02	
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)					
Coverage Factor, k					
Relative Expanded Uncertainty of the Output Quantity, <i>Uly</i> , (%)					

#### **NOTES**

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One  $\mu Sv$  is equal to 0.1 mrem. Distance from Ampoule (cm): 1 30 100 Approximate Dose Rate ( $\mu Sv/h$ ): < 0.1 -
- [b] Massic alpha-particle-emission rate is the preferred name for the quantity alpha-particle-emission rate divided by the total mass of the sample. Massic activity is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [c] The polonium-209 massic activity of the solution is 85.83 Bq·g<sup>-1</sup>, assuming an alpha-particle branching ratio of  $(0.9952 \pm 0.0004)$  s<sup>-1</sup> •Bq<sup>-1</sup> [h].
- In 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  $y = f(x_1, x_2, x_3, \dots x_n)$ , 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 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 stated uncertainty is two times the standard uncertainty.
- [f] Estimated limits of detection for alpha-particle-emitting impurities, expressed as massic alpha-particle emission rate, are:

```
0.002 s<sup>-1</sup>·g<sup>-1</sup> for energies less than 3.5 MeV,
```

 $0.05 ext{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 3.5 and 4.2 MeV, and

0.0002 s<sup>-1</sup>·g<sup>-1</sup> for energies greater than 5.18 MeV.

- [g] Estimated limits of detection for photon-emitting impurities, as of March 1994, expressed as massic photon emission rates, are:
  - $2 \times 10^4$  s<sup>-1</sup>·g<sup>-1</sup> for energies between 15 keV and 68 keV,
  - 2×10<sup>4</sup> s<sup>-1</sup>•g<sup>-1</sup> for energies between 81 keV and 256 keV,
  - 6×10<sup>-5</sup> s<sup>-1</sup>·g<sup>-1</sup> for energies between 266 keV and 892 keV, and
  - 4×10<sup>-6</sup> s<sup>-1</sup>•g<sup>-1</sup> for energies between 900 keV and 3300 keV, provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of polonium-209 See reference [7] for further information about the impurity analyses.
- [h] The stated uncertainty is the standard uncertainty.
- [i] Relative standard uncertainty of the input quantity  $x_i$ .
- 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.

- [k] 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  $u_i(y)/y \equiv |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$ . 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.
- [m]  $|\partial y/\partial x_i| \cdot (x/y) = \text{(average background count rate)}/\text{(average net sample count rate)}$
- [n] 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.
- $[p] \qquad |\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$
- [q] The live time is determined by counting the pulses from a gated crystal-controlled oscillator.
- [r] The standard uncertainty the detected impurity.  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/((\text{Rq of Polonium-209}))\}$ .
- [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.  $u(x_i)/x_i = 100\%$ .  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of Polonium-209})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of Polonium-209})\}$ . 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] R. Collé, Long-term Stability of a Carrier-Free Polonium Solution Standards, *Radioactivity and Radiochemistry*, **4** (1993) 18.
- [6] R. Collé, Z. Lin, J.M.R. Hutchinson and F.J. Schima, A Delayed Isomeric State in <sup>205</sup>Pb and Its Implications for 4πα Liquid-Scintillation Spectrometry of <sup>209</sup>Po, *Appl. Radiat. Isot.*, **45** (1994) 1165.
- [7] R. Collé, Z. Lin, F.J. Schima, P.A. Hodge, J.W.L. Thomas, J.M.R. Hutchinson, and B.M. Coursey, Preparation and Calibration of Carrier Free <sup>209</sup>Po Solution Standards, *J. Res. NIST*, **100** (1995)1.
- [8] Evaluated Nuclear Structure Data File (ENSDF), July 1994.