



National Institute of Standards & Technology

Certificate

Standard Reference Material 4233D Cesium-137 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive cesium-137 chloride, non-radioactive cesium 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 ionization chambers and solid-state gamma-ray spectrometry systems.

Radiological Hazard

The SRM ampoule contains cesium-137 with a total activity of approximately 3.2 MBq. Cesium-137 decays by beta-particle emission to barium-137m which decays by internal conversion. During the decay process X-rays and gamma rays with energies from 4 to 662 keV are emitted. Most of these photons escape from the SRM ampoule and can represent a radiation hazard. Approximate unshielded dose rates at several distances (as of the reference time) are given in note [a]*. Appropriate shielding and/or distance should be used to minimize personnel exposure. The SRM should be used only by persons qualified to handle radioactive material.

Chemical Hazard

The SRM ampoule contains hydrochloric acid (HCl) with a concentration of 1 mole 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 July 2005.

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 and E.L. Garner of the Inorganic Analytical Research Division.

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
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Thomas E. Gills, Chief
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CAUTION! Inconsistent Data

SRM 4322D is calibrated in terms of both massic activity and massic number of cesium-137 atoms. Each calibration was done independently and a best estimate was made of the uncertainty associated with each reported value. In addition, the half life of cesium-137 has been measured and is in good agreement with other reported values. Since the three quantities (activity, A , number of cesium-137 atoms, N , and half-life, $t_{1/2}$) are related by the equation $A = -(\partial N/\partial t) = N \cdot \lambda = N \cdot (\ln 2/t_{1/2})$, it is possible to take any two of the three values and calculate the third. When this is done, it is observed that the calculated third value differs from the measured value by approximately 1.6 percent. This difference is significantly greater than the combined stated uncertainties.

This inconsistency has been observed for almost 20 years, but the uncertainties associated with the massic activity and the half-life measurements were large enough that it was not clear whether the inconsistency was statistically significant. The uncertainties associated with the current measurements of each of the three values is such that there is little question that the inconsistency is real. The cause of the inconsistency is being investigated. At present we have no justification for changing any of the reported values or their estimated uncertainties. We consider the massic activity value the most likely source of the inconsistency, based upon evidence from gamma-ray-emission measurements. Additional measurements will be necessary to resolve this inconsistency and you will be notified when it is resolved. If appropriate you will receive a revised certificate.

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 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 4233D
(Certified values are shown in bold type)

Source identification number	NIST SRM 4233D		
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	(1.015 ± 0.002) g·mL⁻¹ at 20 °C [b]*		
Solution mass	(5.014 ± 0.003) g [b]		
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol·L ⁻¹)	Mass Fraction (g·g ⁻¹)
	H ₂ O	54	0.96
	HCl	1.0	0.04
	CsCl	1.1 × 10 ⁻⁴	1.9 × 10 ⁻⁵
	¹³⁷ CsCl	1.5 × 10 ⁻⁶	2.5 × 10 ⁻⁷
Radiological Properties:			
Radionuclide	Cesium-137		
Reference time	1200 EST, 1 July 1995		
Massic activity of the solution [c]	639.0 kBq·g⁻¹		
Relative expanded uncertainty (<i>k</i> =2) of the massic activity	0.68% [d] [e]		
Massic number of Cs-137 atoms [c]	8.635 × 10¹⁴ g⁻¹		
Relative expanded uncertainty (<i>k</i> =2) of the massic number of Cs-137 atoms	0.54% [d] [e]		
Photon-emitting impurities	None detected [f]		
Half lives used	Cesium-137: (11015 ± 20) d [g] [5] Radium-226: (1600 ± 7) a [g] [6]		
Measuring instruments	Pressurized "4π"γ ionization chamber A calibrated using a cesium-137 solution whose activity was determined by 4π(e+X)-γ-anticoincidence counting. Pressurized "4π"γ ionization chamber A calibrated using a cesium-137 solution whose number of cesium-137 atoms was determined by isotope-dilution mass spectrometry.		

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

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, $u(x_i)/x_i$, (%) [h]	Relative Sensitivity Factor, $ \partial y/\partial x_i \cdot$ (x_i/y) [i]	Relative Uncertainty Of Output Quantity, $u_c(y)/y$, (%) [j]
PIC A net response per gram of SRM 4233D, measured relative to RRS20 [k]	Standard deviation of the mean for 20 repeated measurements on each of 6 samples (A)	0.04	1.0	0.04
PIC A net response per Bq of cesium-137 in solution, measured relative to RRS20	Standard deviation of the mean for 120 repeated measurements on 1 sample (A)	0.05	1.0	0.05
Activity used to calibrate PIC A net response per Bq of cesium-137 in solution	Standard uncertainty of the activity determined by $4\pi(e+X)\gamma$ -anticoincidence counting (B)	0.31	1.0	0.31
Decay correction for cesium-137	Standard uncertainty of the half life (A)	0.18 [m]	0.001 [n]	0.0002
Decay correction for radium-226	Standard uncertainty of the half life (A)	0.44 [m]	0.006 [n]	0.003
Gravimetric measurements	Estimated (B)	0.02	1.0	0.02
Live time [p]	Estimated (B)	0.05	1.0	0.05
PIC A charge collection	Estimated (B)	0.05	1.0	0.05
Source Positioning	Estimated (B)	0.05	1.0	0.05
Photon-emitting impurities	Limit of detection (B) [q]	100.	0.0005	0.05
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

EVALUATION OF THE UNCERTAINTY OF THE MASSIC NUMBER OF Cs-137 ATOMS [d]*

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, $u(x_i)/x_i$, (%) [h]	Relative Sensitivity Factor, $ \partial y/\partial x_i \cdot$ (x_i/y) [i]	Relative Uncertainty Of Output Quantity, $u_i(y)/y$, (%) [j]
PIC A net response per gram of SRM 4233D, measured relative to RRS20 [k]	Standard deviation of the mean for 20 repeated measurements on each of 6 samples (A)	0.04	1.0	0.04
PIC A net response for RRS50, measured relative to RRS20	Standard deviation of the mean for > 100 repeated measurements (A)	0.05	1.0	0.05
PIC A net response per atom of cesium-137 in solution, measured relative to RRS50.	Standard deviation of the mean for 20 repeated measurements on each of 9 samples (A)	0.01	1.0	0.01
Number of cesium-137 atoms used to calibrate PIC A net response per atom of cesium-137 in solution	Standard uncertainty of the number of cesium- 137 atoms determined by isotope-dilution mass spectrometry. (B)	0.24	1.0	0.24
Decay correction for cesium-137	Standard uncertainty of the half life (A)	0.18 [m]	0.001 [n]	0.0002
Decay correction for radium-226	Standard uncertainty of the half life (A)	0.44 [m]	0.006 [n]	0.003
Gravimetric measurements	Estimated (B)	0.02	1.0	0.02
Live time [p]	Estimated (B)	0.05	1.0	0.05
PIC A charge collection	Estimated (B)	0.05	1.0	0.05
Source Positioning	Estimated (B)	0.05	1.0	0.05
Photon-emitting impurities	Limit of detection (B) [q]	100.	0.0005	0.05
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$, (%)				
Coverage Factor, k	x 2			
Relative Expanded Uncertainty of the Output Quantity, U/y , (%)	0.54			

NOTES

- [a] The Sievert is the SI unit for dose equivalent. See reference [1]. One μSv is equal to 0.1 mrem.
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|---|-----|----|-----|
| Distance from Ampoule (cm): | 1 | 30 | 100 |
| Approximate Dose Rate ($\mu\text{Sv/h}$): | 300 | 4 | 0.3 |
- [b] The stated uncertainty is two times the standard uncertainty.
- [c] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. **Massic number of atoms** is the preferred name for the quantity number of atoms divided by the total mass of the sample. See reference [1].
- [d] The reported value, y , of massic activity or massic number of Cs-137 atoms 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 , $u_i(y) \equiv |\partial y / \partial x_i| \cdot u(x_i)$, 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 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] The value of each standard uncertainty component, 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] Estimated limits of detection for photon-emitting impurities are:
600 $\gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$ for energies between 90 and 657 keV, and
60 $\gamma \cdot \text{s}^{-1} \cdot \text{g}^{-1}$ for energies between 666 and 1900 keV.
- The detection limit for cesium-134 is 0.6 $\text{Bq} \cdot \text{g}^{-1}$.
- [g] 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 $u_i(y)/y \equiv |\partial y/\partial x_i| \cdot u(x_i)/y = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$. 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.
- [k] The response of pressurized ionization chamber A (PIC A) is determined from measurement of the time required to collect a given amount of charge on a stable fixed capacitor. All of the response measurements in the NIST pressurized ionization chambers are made relative to the response of one or more artifact standards. These artifact standards consist of microgram quantities of aged radium-226 in small welded stainless-steel capsules. These capsules are encapsulated in plastic rods whose dimensions are similar to those of the standard NIST ampoule. The artifact standards are called **Radium Reference Sources** and are designated as RRS x , where x is the nominal mass (in micrograms) of radium-226 in the capsule.
- [m] The relative standard uncertainty of $\lambda \cdot t$ is determined by the relative standard uncertainty of λ (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. $u(x_i)/x_i = 100\%$. $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of Cs-137})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of Cs-137})\}$. 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, 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] M.P. Unterweger, D.D. Hoppes, and F.J. Schima, *New and Revised Half-Life Measurements Results, Nuclear Instruments and Methods in Physical Research A312* (1992) 349.
- [6] Evaluated Nuclear Structure Data File (ENSDF), May 1996.