



# National Institute of Standards & Technology Certificate

## Standard Reference Material 4226C Nickel-63 Radioactivity Standard

This Standard Reference Material (SRM) consists of radioactive nickel-63 chloride, non-radioactive nickel 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 beta-particle counting instruments and for the monitoring of radiochemical procedures.

**Radiological Hazard:** The SRM ampoule contains nickel-63 with a total activity of approximately 240 kBq. Nickel-63 decays by beta-particle emission. None of the beta particles escape from the SRM ampoule. During the decay process no photons are emitted. 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 (HCl) with a concentration of 0.9 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 December 2015. The long-term stability of these solutions is discussed in references [9] and [10]. 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, M.P. Unterweger, Acting Group Leader. The overall technical direction and physical measurements leading to certification were provided by R. Collé, and B.E. Zimmerman 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.

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### **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]\*.

PROPERTIES OF SRM 4226C

Certified values

<b>Radionuclide</b>	<b>Nickel-63</b>
<b>Reference time</b>	<b>1200 EST, 15 November 2005</b>
<b>Massic activity of the solution [b]*</b>	<b>47.10 kBq·g<sup>-1</sup></b>
<b>Relative expanded uncertainty (k=2)</b>	<b>0.94% [c][d]</b>
<b>Solution mass</b>	<b>(5.080 ± 0.002) g [e]</b>
<b>Solution density</b>	<b>(1.014 ± 0.004) g·mL<sup>-1</sup> at 22 °C [e]</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	
Chemical Properties:			
Solution composition	Chemical Formula	Concentration (mol·L <sup>-1</sup> )	Mass Fraction (g·g <sup>-1</sup> )
	H <sub>2</sub> O	54	0.96
	HCl	0.9	0.04
	NiCl <sub>2</sub>	0.0017	0.00022
	<sup>63</sup> NiCl <sub>2</sub>	4 × 10 <sup>-7</sup>	5 × 10 <sup>-8</sup>
Radiological Properties:			
Photon-emitting impurities	None detected [f]		
Half lives used	Hydrogen-3: (12.32 ±0.02) a [g], [5] Nickel-63: (101.1 ± 1.4) a [g], [h]		
Beta-particle maximum energies used in the EFFY4 computations	Hydrogen-3: (18.594 ± 0.008) keV [g], [5] Nickel-63: (66.945 ± 0.004) keV [g],[5]		
Calibration method and measuring instrument(s)	Two 4πβ liquid-scintillation counters. The Ni-63 detection efficiency was calculated using the CIEMAT/NIST method with H-3 as the detection-efficiency monitor. [i]		

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, $u(x_i)/x_i$ , (%) [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 liquid- scintillation count rate of the Ni-63 solution, corrected for background and decay, divided by the computed detection efficiency [n]	Typical standard deviation of the mean for 10 repeated measurements on a single sample (A)	0.06	1.0	0.06
	Typical standard deviation for measurements on 4 differently quenched samples (A)	0.15	1.0	0.15
	Typical standard deviation for 18 computed detection efficiency curves (A)	0.18	1.0	0.18
Quench-indicating- parameter (QIP) measurements	Typical standard deviation of the mean for 10 repeated measurements on a single sample (A)	0.48	0.27	0.13
Live time measurements [p]	Estimated (B)	0.08	1.0	0.08
Gravimetric measurements	Estimated (B)	0.05	1.0	0.05
Photon-emitting impurities	Limit of detection (B) [q]	100	0.00004	0.004
Decay corrections for H-3 for Ni-63	Standard uncertainty of the half life (A)	0.49 [r] 1.4 [r]	0.006 [s] 0.069 [s]	0.003 0.096
LS cocktail effects	Estimated (B)	0.08	1.0	0.08
Computed detection efficiency for Ni-63 solution	Estimated (B) [i]	0.35	1.0	0.35
Relative Combined Standard Uncertainty of the Output Quantity, $u_c(y)/y$ , (%)				0.47
Coverage Factor, $k$				<u>x 2</u>
Relative Expanded Uncertainty of the Output Quantity, $U/y$ , (%)				0.94

## 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 ( $\mu\text{Sv/h}$ ):   < 0.1       -       -
- [b] **Massic activity** is the preferred name for the quantity activity divided by the total mass of the sample. See reference [1].
- [c] 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  $y$ ,  $u_i(y) \equiv |\partial y / \partial x_i| \cdot u(x_i)$ , 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].
- [d] 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).
- [e] The stated uncertainty is two times the standard uncertainty.
- [f] Estimated limits of detection for photon-emitting impurities, as of August 1995, expressed as massic photon emission rates, are:  
 $5 \times 10^{-6} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 12 keV and 88 keV,  
 $2 \times 10^{-6} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 96 keV and 507 keV,  
 $8 \times 10^{-7} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 515 keV and 1456 keV, and  
 $3 \times 10^{-7} \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 1465 keV and 1900 keV.
- [g] The stated uncertainty is the standard uncertainty.
- [h] The half-life of nickel-63 was reevaluated as part of the calibration of this SRM. See reference [10]. The current ENSDF value,  $(100.0 \pm 2.0) \text{ a}$ , see reference [5], from a 2001 review, neglects to correct the tabulated 1971 calorimetry-based half life value by the more recently revised average energy per decay for nickel-63. See also footnote [n].

- [i] The relationship between the detection efficiency for Ni-63 and the detection efficiency for H-3 was computed using the CIEMAT/NIST method as embodied in the computer program EFFY4. See references [6,7,8]. The program computes the detection efficiency for each radionuclide based upon an assumed model. No estimate is made of the uncertainty associated with this model.
- [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  $|\partial y/\partial x_i| \cdot (x_i/y) = 1.0$ , 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  $y$ .
- [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  $u_i(y)/y \equiv |\partial y/\partial x_i| \cdot u(x_i)/x_i = |\partial y/\partial x_i| \cdot (x_i/y) \cdot u(x_i)/x_i$ . 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.
- [n] The Ni-63 massic activity of SRM 4226C is gravimetrically related to the massic activity of SRM 4226 (issued in 1969) and SRM 4226B (issued in 1984). The calibrations of SRM 4226 and SRM 4226B were based upon microcalorimetric measurements made in 1968. The calibration of SRM 4226C differs by less than 0.2 percent from the previous calibrations, after revision of the calorimetry data by a factor of 17.23/17.426 to account for a change in the assumed mean beta-particle energy of Ni-63 and after correction for decay using a half life of 101.1 years. See references [9],[10],and [11].
- [p] The live time is determined by counting the pulses from a gated crystal-controlled 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.  $u(x_i)/x_i = 100\%$ .  $|\partial y/\partial x_i| \cdot (x_i/y) = \{(\text{response per Bq of impurity})/(\text{response per Bq of Nickel-63})\} \cdot \{(\text{Bq of impurity})/(\text{Bq of Nickel-63})\}$ . 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.
- [r] 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.
- [s]  $|\partial y/\partial x_i| \cdot (x_i/y) = |\lambda \cdot t|$

## REFERENCES

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