

National Institute of Standards & Technology

Certificate

Standard Reference Material® 4226d

Nickel-63 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive nickel-63 in a suitably stable and homogeneous matrix. It is intended primarily for the calibration of instruments that are used to measure radioactivity and for the monitoring of radiochemical procedures. A unit consists of a solution, whose composition is specified in Tables 1 and 2, contained in a flame-sealed 5 mL borosilicate-glass ampoule [1].

The certified Nickel-63 massic activity value, at a Reference Time of 1200 EST, 11 November 2009, is:

 $(85.94 \pm 0.72) \text{ kBq} \cdot \text{g}^{-1}$

A NIST certified value, as used within the context of this certificate, is a value for which NIST has the highest confidence in its uncertainty assessment. It is a "measurement result" [2] obtained directly or indirectly from a "primary reference measurement procedure" [3]. The certified value is traceable to the derived SI unit, Becquerel (Bq).

Additional physical, chemical, and radiological properties for this SRM, as well as details on the standardization method, are given in Tables 1 and 2. Uncertainties for the certified quantities are expanded (k = 2). The uncertainties are calculated according to the ISO/JCGM and NIST Guides [4,5]. Table 3 contains a specification of the components that comprise the uncertainty analyses.

Expiration of Certification: The certification of **SRM 4226d** is valid indefinitely, within the measurement uncertainty specified, provided that the SRM is handled and stored properly and that no evaporation or change in composition has occurred. The solution matrix, in an unopened ampoule, is homogeneous and stable within its half-life-dependent useful lifetime provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Handling and Storage"). Periodic recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Radiological and chemical hazard: Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for radiological and chemical hazard information.

This Standard Reference Material was prepared in the NIST Physical Measurement Laboratory, Radiation Physics Division, Radioactivity Group, M.P. Unterweger, Group Leader. The overall technical direction and physical measurement leading to certification were provided by R. Collé and L. Laureano-Pérez of the NIST Radioactivity Group, with production assistance by D.B. Golas, Research Associate of the NRMAP, Inc., and photon-emitting impurity analyses by L. Pibida.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Gaithersburg, Maryland 20899 Certificate Issue Date: 11 January 2017 Certificate Revision History on Last Page

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Table 1. Certified Massic Activity of SRM 4226d

Radionuclide	Nickel-63
Reference time	1200 EST, 11 November 2009
Massic activity of the solution	85.94 kBq•g ⁻¹
Relative expanded uncertainty $(k = 2)$	0.84 % ^(a)

⁽a) The uncertainties on certified values are expanded uncertainties, $U = ku_c$. The quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM and NIST Guides [4,5]. The combined standard uncertainty is multiplied by a coverage factor of k = 2 and was chosen to obtain what is assumed to be an approximate 95 % level of confidence.

Table 2. Uncertified Information of SRM 4226d

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1].	
Solution composition	1.1 mol•L ⁻¹ HCl with 47 μg Ni ²⁺ per gram of solution	
Solution density	$(1.018 \pm 0.002) \text{ g} \cdot \text{mL}^{-1} \text{ at } 20.0 \text{ °C}^{(a)}$	
Solution mass	$(5.0899 \pm 0.0003) g^{(a)}$	
Photon-Emitting Impurities	None detected ^(b)	
Half-lives used	⁶³ Ni: $(101.1 \pm 1.4) a^{(c,d)}$ [6]	
Calibration methods (and instruments)	The certified massic activity for 63 Ni was obtained by $4\pi\beta$ liquid scintillation (LS) spectrometry with three commercial LS counters. The LS detection efficiency was calculated using the TRACER code [7] for the CIEMAT/NIST method with composition matched LS cocktails of a 3 H standard as the efficiency detection monitor. (e)	

⁽a) The stated uncertainty is two times the standard uncertainty. See reference 5.

- $1 \times 10^{-5} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 12 keV and 88 keV,
- $4 \times 10^{-6} \,\mathrm{s}^{-1} \,\mathrm{eg}^{-1}$ for energies between 96 keV and 507 keV,
- $2 \times 10^{-6} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 515 keV and 1456 keV, and
- $6 \times 10^{-7} \text{ s}^{-1} \cdot \text{g}^{-1}$ for energies between 1465 keV and 1900 keV.

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ⁶³Ni or progeny.

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⁽b) The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, in August 1995 are:

⁽c) The stated uncertainty is the standard uncertainty. See reference 5.

⁽d) See reference 6. This ⁶³Ni half-life value was re-confirmed in Reference 8. The current ⁶³Ni half-life value of 98.7 a tabulated in several compilations [9] is inconsistent with the most precise determinations.

⁽e) The certified 63 Ni massic activity of SRM 4226d is wholly consistent with the decay-corrected certified activity in SRM 4226C (issued in 1995), SRM 4226b (issued in 1984) and SRM 4226 (issued in 1969) using a half-life of (101.1 \pm 1.4) a. The present agreement with the previous issues are + 0.6 %, + 0.9 % and + 0.5 %, respectively.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4226d

	Uncertainty component	Assessment Type ^(a)	Relative standard uncertainty contribution on massic alphaparticle emission rate of ⁶³ Ni (%)
1	LS measurement precision; standard deviation for $n = 4$ sets of measurements obtained with 3 different LS counters using 6 different quenched sources, each measured 3 to 5 times in each counter. The typical internal relative standard deviation within a measurement data set is 0.17 % for $n = 6$ sources.	A	0.22
2	Background LS measurement variability; wholly embodied in component 1.	A	
3	Live time determinations for LS counting time intervals, includes uncorrected dead time effects; assumed from specified tolerance limits of counters' gated oscillators.	В	0.08
4	Gravimetric (mass) determinations for LS sources; estimated from calibration data and tests.	В	0.05
5	⁶³ Ni decay corrections for half-life uncertainty of 1.4 % [6].	В	8×10^{-4}
6	Computed detection efficiency for Ni-63 solution.	В	0.35
Relative combined standard uncertainty		0.42	
Relative expanded uncertainty $(k = 2)$		0.84	

^(a) Letter A, denotes evaluation by statistical methods; B denotes evaluation by other methods.

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INSTRUCTIONS FOR HANDLING AND STORAGE

Handling: 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. Only persons qualified to handle both radioactive material and alkaline and/or acidic solutions, should open the ampoule. To minimize personnel exposure, appropriate shielding and/or distance should be used. Refer to the SDS for further information.

Storage: SRM 4226d should be stored and used at a temperature between 5 °C and 65 °C. The ampoule (or any subsequent container) should always be clearly marked as containing radioactive material.

REFERENCES

- [1] NIST Physical Measurement Laboratory; *Storage and Handling of Radioactive Standard Reference Materials, Ampoule Specifications and Opening Procedure*, available at http://www.nist.gov/pml/div682/grp04/srm.cfm (accessed Jan 2017). Note: This SRM is contained in a generic borosilicate-glass ampoule and not in the standard NIST ampoule.
- [2] JCGM 200:2012; International Vocabulary of Metrology Basic and General Concepts and Associated Terms (VIM) (2008 version with Minor Corrections), 3rd edition; Joint Committee for Guides in Metrology (JCGM); p. 19 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Jan 2017).
- [3] JCGM 200:2012; International Vocabulary of Metrology Basic and General Concepts and Associated Terms (VIM) (2008 version with Minor Corrections), 3rd edition; JCGM; p. 18 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Jan 2017).
- [4] JCGM 100:2008; Evaluation of Measurement Data Guide to the Expression of Uncertainty in Measurement; (GUM 1995 with Minor Corrections), JCGM (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2017).
- [5] Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Jan 2017).
- [6] Collé, R. and Zimmerman, B.E.; ⁶³Ni Half Life: A New Experimental Determination and Critical Review; Appl. Radiat. Isot., Vol. 47, p. 677(1996).
- [7] Cassette P., (Laboratoire National Henri Becquerel, Gif-sur-Yvette, France), private communication on TRACER code (Jan. 2006).
- [8] Collé, R., Zimmerman, B.E., Cassette, P. and Laureano-Pérez, L.; ⁶³Ni, Its half-life and Standardization: Revisited; Appl Radiat Isot., Vol. 66, pp. 60-68 (2008).
- [9] Laboratoire National Henri Becquerel, *Recommended Data*, Gif-sur-Yvette, Cedex, France; available at http://www.nucleide.org/DDEP_WG/DDEPdata.htm (accessed Jan 2017).

Certificate Revision History: 11 January 2017 (Correction in solution composition and density, editorial changes); 22 March 2011 (Original certificate date).

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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