National Institute of Standards and Technology
U.S. Department of Commerce

Date of Issue:
13 January 2023

Standard Reference Material® 4328d
Thorium-229 Radioactivity Standard
CERTIFICATE

Purpose: The certified value delivered by this Standard Reference Material (SRM) is intended primarily for the calibration of instruments that are used to measure radioactivity and for the monitoring of radiochemical procedures.

Description: A unit of SRM 4328d consists of approximately 5 mL of a solution of a standardized and certified quantity of radioactive thorium-229 in a suitably stable and homogeneous matrix, contained in a flame-sealed borosilicate-glass ampoule [1].

Certified Values: 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]. Uncertainties for the certified quantities are expanded ($k = 2$). The uncertainties are calculated according to the ISO/JCGM and NIST Guides [4,5]. The certified value is traceable to the derived SI unit, becquerel (Bq).

Table 1. Certified Massic Activity of SRM 4328d

Radionuclide	**Thorium-229**
Reference time	**1200 EST, 14 July 2022**
Massic activity of the solution	**41.19 Bq•g^{-1}**
Relative expanded uncertainty ($k = 2$) [(a)]	**1.0 %**

[(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 an approximate 95 % level of confidence.

Non-Certified Values: Non-certified values and additional information are provided in Appendix A.

Period of Validity: The certification of **SRM 4328d** 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 certification is nullified if the SRM is damaged, contaminated, or otherwise modified. Periodic recertification of this SRM is not required.

Maintenance of Certification: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

James M. Adams, Chief
Radiation Physics Division

Steven J. Choquette, Director
Office of Reference Materials

Table 2. Uncertainty Evaluation for the Massic Activity of SRM 4328d

	Uncertainty component	Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of ^{229}Th (%)
1	Liquid scintillation (LS) comparative measurement precision; relative standard deviation of the mean on the great grand mean for six LS measurement trials, considering all within-trial and between-trial components of variance. Each of the six grand mean values was based on five replicate measurements on each of three LS sources for SRM 4328c and six LS sources for 4328d. The sources were prepared with two different LS cocktail compositions and each source was measured in two different LS counters (on three separate measurement occasions). The typical within-trial relative standard deviation of the mean (considering the variations for the between five measurements and the between three or six sources) for each trial was 0.16 %. The between-trial relative standard deviation across the six trials was 0.26 %. The individual values for each data set fit Normal distributions. The relative standard deviation of the mean from the six trials was 0.11 % with a 95 % confidence interval about the mean of ±0.28 %.	A	0.31
2	Background; LS measurement variability, cocktail mismatch effects and cocktail composition stability; wholly embodied in component 1.	A	--
3	LS counters dependencies; wholly embodied in component 1.	A	--
4	Uncertainty on the standardization of SRM 4328c	A	0.30
5	Potential undetected alpha- and photon emitting impurity differences in SRM 4328c and 4328d; wholly embodied in component 1	B	--
6	Scintillator dependence (absolute difference of 0.49 %); partially embodied in component 1	B	0.24
7	Live time determinations for LS counting time intervals; includes uncorrected deadtime effects.	B	0.07
8	Aliquant mass determination by gravimetric measurements for preparation of LS counting sources; includes mass measurement precision partially embodied in component 1.	B	0.02
9	Decay correction for ^{229}Th for a half-life uncertainty of 0.30 %.	B	0.0004
Relative combined standard uncertainty			**0.50**
Relative expanded uncertainty (k = 2)			**1.0**

[(a)] Letter A denotes evaluation by statistical methods; Letter B denotes evaluation by other methods.

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

Storage and Handling: SRM 4328d 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. If the ampoule is transported, it should be packed, marked, labeled, and shipped in accordance with the applicable national, international, and carrier regulations. This is **not** a pre-scored ampoule; **the gold color band is only for identification**. The solution in the ampoule is a dangerous good (hazardous material) because of both the radioactivity and the strong acid. The ampoule should be opened only by persons qualified to handle both radioactive material and alkaline and/or acidic solutions. Appropriate shielding and/or distance should be used to minimize personnel exposure. Refer to the SDS for further information.

REFERENCES

[1] NIST Physical Measurement Laboratory; *Storage and Handling of Radioactive Standard Reference Materials, Ampoule Specifications and Opening Procedure*; available at https://www.nist.gov/pml/radiation-physics/ampoule-specifications-and-opening-procedure (accessed Nov 2022). **Note:** This SRM is contained in a generic borosilicate-glass ampoule (identified as NIST-3) and not in the standard NIST ampoule (identified as NIST-1) Refer to R.Collé, Ampoules for Radioactivity Standard Reference Materials, NIST Internal Report 8524 (2019) Available at http://www.nist.gov/publications/ampoules-radioactivity-standard-reference-materials (accessed Jan 2023). **This is not a pre-scored ampoule; the gold color band is only for identification.**

[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): BIPM, Sevres Cedex, France; p. 19 (2012); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jan 2023).

[3] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM)*; (2008 version with Minor Corrections), 3rd edition; JCGM: BIPM, Sevres Cedex, France; p. 18 (2012); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Nov 2022).

[4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), JCGM: BIPM, Sevres Cedex, France (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Jan 2023).

[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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Jan 2023).

[6] Goldstein SJ; Murrell MT; Williams RW; *Half-Life of ^{229}Th*; Phys Rev C Nucl Phys., Vol. 40, p. 2793 (1989).

[7] Kikunaga H; Suzuki T; Nomura M; Mitsugashira T; Shinohara A; *Determination of the Half-Life of the Ground State of ^{229}Th by Using ^{232}U and ^{233}U Decay Series*; Phys. Rev. C., Vol. 84, pp. 1-6 (2011).

[8] Varga Z.; Nicholl A.; Maye K.; *Determination of the ^{229}Th Half-Life*; Phys. Rev. C., Vol. 89, pp. 1-6 (2014).

[9] Essex, R.M.; Mann, J.L.; Colle, R; Laureano-Perez, L.; Bennett, M.E; Dion, H.; Fitzgerald, R.; Gaffney, A.M.; Gourgiotis, A.; Hubert, A.; Inn, K.G.W.; Kinman, W.S.; Lamont, S.P.; Steiner, R.; William, R.W.; *A New Determination of the ^{229}Th Half-Life*; J. Radioanal. Nucl. Chem., Vol. 318, pp. 515-525 (2018).

[10] Nichols, A.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2008); available at http://www.lnhb.fr/nuclides/Th-228_tables.pdf (accessed Jan 2023).

[11] Fitzgerald R.; Collé R.; Laureano-Pérez L.; Pibida L.; Hammond MM.; Nour S.; Zimmerman BE.; *A New Primary Standardization of ^{229}Th*; Appl. Radiat. Isot., Vol. 68, pp. 1303-1308 (2010).

Certain commercial equipment, instruments, or materials may be identified in this Certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * * * * * End of Certificate * * * * * * * * * * *

APPENDIX A

This SRM was prepared by the NIST Physical Measurement Laboratory, Radiation Physics Division, under the direction of B.E. Zimmerman, Group Leader of the Radioactivity Group. Overall technical direction and physical measurement leading to certification were provided by R. Collé and L. Laureano-Pérez of the NIST Radiation Physics Division, Radioactivity Group. Photon-emitting-impurity analyses were provided by L. Pibida of the NIST Radiation Physics Division, Radioactivity Group. Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher-order reference system. Non-certified values are provided in Table A1.

Table A1. Non-Certified Values for SRM 4328d[(a)]

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	(1.00 ± 0.03) mol•L^{-1} HNO_3[(b)]
Solution density	(1.032 ± 0.001) g•mL^{-1} at 20.0 °C[(b)]
Solution mass	(5.149 ± 0.002) g[(b)]
Alpha-particle-emitting impurities	^{228}Th: (0.097 ± 0.011) Bq•g^{-1}[(b, c)]
Photon-emitting impurities	None detected [(d)]
Half-lives used	^{229}Th: (7889 ± 24) a[(e)] ^{228}Th: (1.9126 ± 0.0009) a[(f)]
Calibration methods (and instruments)	The certified massic activity for ^{229}Th was obtained by $4\pi\alpha\beta$ LS counting by comparative measurements against SRM 4328c[(g)] using two different LS measurement systems (with varying operating systems) and two different LS cocktail compositions. Solution homogeneity measurements were made with a NaI(Tl) well counter.

[(a)] References on page 3.

[(b)] The stated uncertainty is two times the standard uncertainty [5].

[(c)] The ^{228}Th impurity was based on 2π alpha-emission rate measurements with Si surface barrier detectors following chemical separations, and by photon-emission rate measurements by HPGe detectors. The former was performed on 17 March 2008 and the latter on 01 March 2008. The ^{228}Th / ^{229}Th activity ratio, decay-corrected to 31 December 2007, as reported by the source supplier (Oak Ridge National Laboratory), based on γ-ray spectrometry, was 0.014.

[(d)] The estimated limits of detection for photon-emitting impurities as of August 2022, expressed as massic photon emission rates (numbers of photons per second per gram), are:

0.041 s^{-1}•g^{-1} for energies between 15 keV and 100 keV,
0.036 s^{-1}•g^{-1} for energies between 105 keV and 430 keV,
0.039 s^{-1}•g^{-1} for energies between 450 keV and 1450 keV, and
0.036 s^{-1}•g^{-1} for energies between 1470 keV and 2000 keV,

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay chain of ^{229}Th.

[(e)] The ^{229}Th half-life was based on the unweighted average of the four determinations made by Goldstein, et al. (1989), Kikunaga, et al. (2011), Varga, et al. (2014), and Essex, et al. (2018). A weighted average was not used because of the inconsistent and unequal uncertainty treatments for the determinations. The cited uncertainty is the standard deviation of the mean for 3 degrees of freedom expanded by k = 3.179. See references 6-9.

[(f)] The stated uncertainty is the standard uncertainty. See reference 10.

[(g)] SRM 4328c was standardized in 2007 by live-timed anticoincidence counting (a primary standardization method), which is traceable to the derived SI unit, Becquerel (Bq) [11].

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

* * * * * * * * * * * End of Appendix A * * * * * * * * * * *