Certificate

Standard Reference Material® 4328c

Thorium-229 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive thorium-229 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 of SRM 4328c consists of approximately 5 mL of a solution, whose composition is specified in Tables 1 and 2, contained in a flame-sealed borosilicate-glass ampoule [1].

The certified **thorium-229** massic activity value, at a **Reference Time of 1200 EST, 31 December 2007**, is:

$$\mathbf{(35.29 \pm 0.21)\ Bq{\bullet}g^{-1}}$$

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 4328c** 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 SRM 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) 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 SRM was prepared in the 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, R. Fitzgerald and L. Laureano-Pérez of the NIST Radioactivity Group, with production assistance by D.B. Golas and O. Palabrica, Research Associates of the Nuclear Energy Institute, with confirmatory measurement assistance by B.E. Zimmerman and L. King and with impurity analyses by M. Hammond and S. Nour.

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

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Certificate Revision History on Last Page

Table 1. Certified Massic Activity of SRM 4328c

Radionuclide	**Thorium-229**
Reference time	**1200 EST, 31 December 2007**
Massic activity of the solution	**35.29 Bq•g^{-1}**
Relative expanded uncertainty (*k* = 2)	**0.60 %[(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 4328c

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	1.13 mol•L^{-1} HNO_3
Solution density	(1.036 ± 0.002) g•mL^{-1} at 21 °C[(a)]
Solution mass	(5.1791 ± 0.0003) g[(a)]
Alpha-particle-emitting impurities	^{228}Th: 0.60 ± 0.07 Bq•g^{-1}[(b)]
Photon-emitting impurities	None detected[(c)]
Half-lifes used	^{229}Th : (7340 ± 160) a[(d)] [6] ^{228}Th : (698.60 ± 0.23) d[(d)] [7]
Calibration methods (and instruments)	The certified massic activity for ^{229}Th was obtained by 4παβ(LS) - γ(NaI) live-timed anti-coincidence counting. Confirmatory measurements were performed by five other methods: (i) 4παβ liquid scintillation (LS) spectrometry (with ^{3}H standard efficiency tracing for β efficiencies); (ii) an LS-based 4παβ triple-to-double coincidence ratio (TDCR) method; (iii) 2πα proportional counting; (iv) 2πα spectrometry using Si surface barrier detector, following chemical separation, with a ^{230}Th standard tracer; and (v) HPGe γ-ray spectrometry.[(e)]

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

[(b)] 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 decay-corrected ^{228}Th / ^{229}Th activity ratio as reported by the source supplier (Oak Ridge National Laboratory), based on γ-ray spectrometry, was 0.014.

[(c)] The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, on 01 March 2008 were:

0.053 s^{-1}•g^{-1} for energies between 30 keV and 90 keV and,
0.008 s^{-1}•g^{-1} for energies between 91 keV and 260 keV and,
0.003 s^{-1}•g^{-1} for energies between 261 keV and 2600 keV,

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

[(d)] The stated uncertainty is the standard uncertainty. See reference 5.

[(e)] The expanded ($k = 2$) uncertainties for the five confirmatory methods were: (i) 1.2 %; (ii) 1.0 %; (iii) 2 %; (iv) 2 %; and (v) 3 %, respectively. All of the confirmatory measurements agreed with the certified value within their respective measurement uncertainties, except that for the TDCR result (–1.7 % difference). The results for methods (i) and (iv) agreed with the certified anti-coincidence value to better than 0.1 %.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4328c

	Uncertainty component	Assessment Type[a]	Relative standard uncertainty contribution on massic activity of ^{229}Th (%)
1	Measurement repeatability; standard deviation of the mean for n = 8 activity determinations using 4 sources and 2 blanks (accept normal distribution assumption at 95 % level)	A	0.04
2	Background; uncertainty due to the variation in background throughout measurement not encompassed by measurement repeatability	B	0.015
3	Standard uncertainty of the extrapolation of the LS rate to 100 % efficiency	B	0.17
4	Live-time determinations for counting time intervals	B	0.1
5	Gravimetric (mass) measurements for preparation of sources and for dilution of the ^{229}Th master solution	B	0.1
6	Decay probabilities (branching ratios) and half-lifes of ^{229}Th progeny	B	0.017
7	^{229}Th decay corrections for half-life uncertainty of 2.2 %	B	4×10^{-7}
8	^{228}Th decay corrections for half-life uncertainty of 0.03 %	B	1×10^{-6}
9	Correction of ^{228}Th impurity for a 12 % standard uncertainty in the ^{228}Th/^{229}Th ratio	B	0.2
10	Limit for other alpha-emitting impurities[b]	B	0.001
11	Limit for photon-emitting impurities	B	0.02
Relative combined standard uncertainty			**0.30**
Relative expanded uncertainty (*k* = 2)			**0.60**

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

[b] The ^{229}Th source was obtained from Oak Ridge National Laboratory (ORNL) and was separated in October 2004. Other radionuclidic impurities, as obtained by mass spectrometry and as reported by ORNL, in terms of activity ratios at the reference time, were:

^{230}Th / ^{229}Th = 8.42×10^{-6}

^{232}Th / ^{229}Th = 1.61×10^{-10}

^{233}U / ^{229}Th = 1.44×10^{-6}

The isotopes ^{239}Pu, ^{240}Pu, ^{242}Pu, ^{234}U, ^{235}U, ^{236}U and ^{238}U were not detected. Each of these had lower limits of detection of $< 2 \times 10^{-5}$ in terms of the mass ratio to ^{229}Th.

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. 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 Safety Data Sheet for further information.

Storage: SRM 4328c 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 https://www.nist.gov/pml/radiation-physics/ampoule-specifications-and-opening-procedure (accessed Aug 2019). 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: BIPM, Sevres Cedex, France; p. 19 (2012); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Aug 2019).

[3] 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: BIPM, Sevres Cedex, France; p. 18 (2012); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Aug 2019).

[4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement;* (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2019).

[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 Aug 2019).

[6] Akovali, Y.A.; *Nuclear Data Sheets for A = 229*, Nucl. Data Sheets, Vol. 58, pp 555-588(1989), Evaluated Nuclear Structure Data File (ENSDF) online database, National Nuclear Center, Brookhaven National Laboratory; available at https://www.nndc.bnl.gov/ensdf/ (accessed Aug 2019)

[7] Nichols, A.; *Table of Radionuclides, Recommended Data;* Laboratoire National Henri Becquerel (2008); available at http://www.nucleide.org/DDEP_WG/DDEPdata.htm (accessed Aug 2019).

Certificate Revision History: 05 September 2019 (Updated acid concentration in Table 2; editorial changes); 20 December 2013 (Editorial changes); 30 June 2008 (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 https://www.nist.gov/srm.