

## Standard Reference Material<sup>®</sup> 4324c Uranium-232 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 4324c consists of approximately 5 mL of a solution of a standardized and certified quantity of radioactive uranium-232 (in equilibrium with its progeny) 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" [2]. Uncertainties for the certified quantities are expanded (k = 2). The uncertainties are calculated according to the ISO/JCGM and NIST Guides [3,4]. The certified value is traceable to the derived International System of Units (SI) unit, becquerel (Bq).

Radionuclide	Uranium-232 (in equilibrium with its progeny)
Reference time	1200 EST, 31 October 2022
Massic activity of <sup>232</sup> U	26.30 Bq•g <sup>-1</sup>
<b>Relative expanded uncertainty</b> $(k = 2)^{(a)}$	0.86 %

Table 1. Certified <sup>232</sup>U Massic Activity of SRM 4324c

(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 [3,4]. 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 4324c** 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 at the time of purchase. 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 free of charge through the NIST SRM website.

Alan K. Thompson, Acting Chief Radiation Physics Division Steven J. Choquette, Director Office of Reference Materials

Uncertainty component		Assessment Type <sup>(a)</sup>	Relative standard uncertainty contribution on massic activity of <sup>232</sup> U (%)
1	Liquid scintillation (LS) measurement precision for the CIEMAT/NIST <sup>(b)</sup> efficiency tracing (CNET) method measurements; relative standard deviation of the mean for the grand mean of two measurement trials using two different LS counters, considering all within-trial and between-trial components of variance. Each trial was based on five measurements using five sources, quench matched with <sup>3</sup> H standards. The typical within-trial relative standard deviation (considering the variations for the between five measurements and the between five sources) for each trial was 0.08 %. The between-source relative standard deviation across the six trials was 0.06 %. The individual values for both data sets fit a normal distribution.	A	0.11
2	Background; LS measurement variability, cocktail mismatch effects and cocktail composition stability; wholly embodied in component 1.	А	
3	Live time determination for LS counting time intervals (includes uncorrected dead time effects)	В	0.07
4	Uncertainty on massic activity of <sup>3</sup> H standard used as efficiency detection monitor	В	0.0094
5	Uncertainty on <sup>3</sup> H dilution	В	0.02
6	Uncertainty on gravimetric (mass) measurements for preparation of <sup>3</sup> H LS sources	В	0.04
7	Uncertainty on gravimetric (mass) measurements for preparation of <sup>232</sup> U LS sources	В	0.003
8	Decay Correction for <sup>232</sup> U for half-life uncertainty of 1.56 %	В	0.0086
9	Decay Correction for <sup>3</sup> H for half-life uncertainty of 0.20 %	В	0.086
10	Computed $\beta$ detection efficiencies (Model dependencies and computed Beta spectra)^{(c)}	В	0.40
Relat	Relative combined standard uncertainty		0.43
Relat	ive expanded uncertainty ( <i>k</i> = 2)		0.86

<sup>(a)</sup> Letter A denotes evaluation by statistical methods; Letter B denotes evaluation by other methods.

(b) The acronym CIEMAT/NIST refers to the two laboratories that collaborated in developing the protocol for this LS efficiency tracing methodology, i.e., the Centro de Investigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT) and the National Institute of Standards and Technology (NIST).

<sup>(c)</sup> For <sup>212</sup>Pb, <sup>212</sup>Bi, and <sup>208</sup>Tl, the beta detection efficiencies were obtained using the MICELLE2 code and the simplified decay scheme from Napoli, et al (2020) [5]. All  $\alpha$  emitters in the <sup>232</sup>U decay chain were assumed to be detected with 100 % efficiency. The short-lived <sup>212</sup>Po ((300 ± 2) x 10<sup>-9</sup> s) daughter and its <sup>212</sup>Bi parent were assumed to be detected with a combined efficiency of 100 %.

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

**Storage and Handling:** SRM 4324c 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/radiationphysics/ampoule-specifications-and-opening-procedure (accessed Nov 2023). 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 https://www.nist.gov/publications/ampoules-radioactivity-standard-reference-materials (accessed Nov 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 Nov 2023).
- [3] 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 Nov 2023).
- [4] 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 Nov 2023).
- [5] Napoli, E.; Cessna, J.T; Fitzgerald, R.; Pibida, L.; Collé, R.; Laureano-Pérez, L.; Zimmerman, B.E.; Bergeron, D.E.; *Primary Standardization of <sup>224</sup>Ra Activity by Liquid Scintillation Counting*, Appl. Radiat. Isotop., Vol. 155, pp. 108933 (2020).
- [6] Pearce, A.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2008); available at http://www.lnhb.fr/nuclides/U-232\_tables.pdf (accessed Nov 2023).
- [7] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2013); available at http://www.lnhb.fr/nuclides/Th-228\_tables.pdf (accessed Nov 2023).
- [8] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Ra-224\_tables.pdf (accessed Nov 2023).
- [9] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Rn-220\_tables.pdf (accessed Nov 2023).
- [10] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Po-216\_tables.pdf (accessed Nov 2023).
- [11] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Pb-212\_tables.pdf (accessed Nov 2023).
- [12] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Bi-212\_tables.pdf (accessed Nov 2023).
- [13] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2011); available at http://www.lnhb.fr/nuclides/Po-212\_tables.pdf (accessed Nov 2023).
- [14] Nichols, A.L.; *Table of Radionuclides, Recommended Data*; Laboratoire National Henri Becquerel (2016); available at http://www.lnhb.fr/nuclides/Tl-208\_tables.pdf (accessed Nov 2023).

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, L. Laureano-Pérez and M. DiGiorgio 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 SI or other higher-order reference system. Non-certified values are provided in Table A1.

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]	
	Elquid in a name-scaled 5 mE borosmeate-glass ampoure [1]	
Solution composition	$(2.00 \pm 0.03) \text{ mol} \cdot \text{L}^{-1} \text{ HNO}_3^{(b)}$	
Solution density	$(1.064 \pm 0.001)$ g•mL <sup>-1</sup> at 23.9 °C <sup>(b)</sup>	
Solution mass	$(5.313 \pm 0.003)$ g <sup>(b)</sup>	
Photon-emitting impurities	None detected <sup>(c)</sup>	
Half-lives used <sup>(d)</sup>	<sup>232</sup> U: $(70.6 \pm 1.1)$ a <sup>228</sup> Th: $(1.9126 \pm 0.0009)$ a <sup>224</sup> Ra: $(3.631 \pm 0.002)$ d <sup>220</sup> Rn: $(55.8 \pm 0.3)$ s <sup>216</sup> Po: $(0.148 \pm 0.004)$ s <sup>212</sup> Pb: $(10.64 \pm 0.01)$ h <sup>212</sup> Bi: $(60.54 \pm 0.06)$ m <sup>212</sup> Po: $(300 \pm 2)$ x $10^{-9}$ s <sup>208</sup> Tl: $(3.058 \pm 0.006)$ m	
Calibration methods (and instruments)	ibration methods (and The certified massic activity for $^{232}$ U was obtained by $4\pi\alpha\beta$ LS spectromet ibration methods (and The certified massic activity for $^{232}$ U was obtained by $4\pi\alpha\beta$ LS spectromet daughters were calculated using the MICELLE2 code for the CIEMAT/NIST efficiency tracing (CNET) method with composition matched LS cocktails of 3H standard as the efficiency detection monitor. Confirmatory measurement	

<sup>(a)</sup> References on page 3.

<sup>(b)</sup> The stated uncertainty is two times the standard uncertainty [4].

<sup>(c)</sup> The estimated limits of detection for photon-emitting impurities as of November 2022, expressed as massic photon emission rates (numbers of photons per second per gram), are:

0.12 s<sup>-1</sup>•g<sup>-1</sup> for energies between 40 keV and 75 keV,

 $0.097 \text{ s}^{-1} \cdot \text{g}^{-1}$  for energies between 80 keV and 1450 keV (skipping over main gamma-ray energies from the <sup>232</sup>U decay), and  $0.05 \text{ s}^{-1} \cdot \text{g}^{-1}$  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 <sup>232</sup>U.

<sup>(d)</sup> The stated uncertainty is the standard uncertainty. See references 6–14.

(e) The acronym CIEMAT/NIST refers to the two laboratories that collaborated in developing the protocol for this LS efficiency tracing methodology, i.e., the Centro de Invenstigaciones Energéticas, Medioambientales y Tecnológicas (CIEMAT) and the National Institute of Standards and Technology (NIST).

<sup>(f)</sup> SRM 4324b was standardized in 2002 by live-timed anticoincidence counting. A re-standardization of 4324b by CNET as part of this work was in agreement with the decay corrected certified value by 0.66 %.

<sup>(g)</sup> Confirmatory values by LS comparative measurements and gamma-ray spectrometry were in agreement with the CNET certified value by 0.30 % and 0.84 %, respectively.

**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 Appendix 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).

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