National Institute of Standards & Technology

Certificate

Standard Reference Material® 4222d

Carbon-14-n-hexadecane Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of a standardized and certified quantity of radioactive carbon-14-n-hexadecane 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 4222d 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 **carbon-14** massic activity, at a **Reference Time of 1200 EST, 10 September 2014**, is:

$$(54.01 \pm 0.52)\ \mathrm{kBq \cdot g^{-1}}$$

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 and NIST Guides [4,5]. Table 3 contains a specification of the components that comprise the uncertainty analysis.

Expiration of Certification: The certification of **SRM 4222d** 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 SRM was prepared in the NIST Physical Measurement Laboratory, Radiation Physics Division, under the direction of M.P. Unterweger, Group Leader of the Radioactivity Group. Overall technical direction and physical measurement leading to recertification were provided by D. Bergeron and L. Laureano-Perez of the NIST Radiation Physics Division, Radioactivity Group. Photon-emitting impurity analyses were provided by D. Hoppes formerly 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.

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Certificate Issue Date: 01 November 2017

Steven J. Choquette, Director
Office of Reference Materials

Table 1. Certified Massic Activity of SRM 4222d

Radionuclide	**Carbon-14-n-hexadecane**
Reference time	**1200 EST, 10 September 2014**
Massic activity of the solution	**54.01 kBq•g^{-1}[(a)]**
Relative expanded uncertainty (*k* = 2)	**0.96 %[(b)]**

[(a)] The certified value was based on Triple-to-Double Coincidence Ratio (TDCR) measurements with the beta spectrum calculated using the shape factor limit given by Alimonti et al., 1998, the shape factor coefficient a = - 0.37 (in natural units, m_e) was used for final analysis [6]. The difference in this result from that obtained on assuming an allowed transition is +0.13 %. The confirmatory measurements by CIEMAT/NIST efficiency tracing (CNET) were in agreement to -0.75 % and -1.1 % under the same two assumptions [7]. The certified value for the massic activity for SRM 4222d is consistent with the decay-corrected value for SRM 4222C [8] to +0.26 %.

[(b)]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.

Table 2. Uncertified Information of SRM 4222d

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	^{14}C-labeled n-hexadecane[(a)]
Solution density	(0.771 ± 0.001) g•mL^{-1} at 25.0 °C[(b)]
Solution mass	Approximately 4 g
Photon-Emitting Impurities	None detected[(c)]
Half-lives used	^{14}C: (5700 ± 30) a [(d)] [9]
Calibration methods (and instruments)	The certified value was based on Triple-to-Double Coincidence Ratio (TDCR) measurements with the beta spectrum calculated using the shape factor limit given by Alimonti et al., 1998 [6]. The liquid scintillation (LS) detection efficiency was calculated using the MICELLE2 code [10]. Confirmatory measurements were done by 4παβ LS spectrometry with two commercial LS counters. The LS detection efficiency was also calculated using the MICELLE2 code for the CIEMAT/NIST method with composition-matched LS cocktails of a ^{3}H standard as the efficiency detection monitor.

[(a)] Solution was prepared and originally calibrated in 1990 in the Radioactivity Group [8].
[(b)] The stated uncertainty is two times the standard uncertainty. See reference 5.
[(c)] The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, on 1990, was: 0.2 s^{-1}•g^{-1} in the region 100 keV ≤ E ≤ 1900 keV.
[(d)]The stated uncertainty is the standard uncertainty. See reference 5.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4222d

Uncertainty component		Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of ^{14}C (%)
1	Measurement precision; standard deviation of the mean on the grand mean for 5 counting sources at 3 different detection efficiencies (varied with grey filters) and 2 different scintillation fluids, considering both the between-mean and the typical within-mean variations. The mean for each source is based on 3 replicate measurements at each grey-filter efficiency on 2 separate measurement occasions. The relative standard deviation for the between-mean ($n = 5$) standard deviation is 0.09 %. The average relative standard deviation of the mean for the typical within-mean variation is 0.19 %, as obtained from the between-measurement occasion ($n = 2$) variation (0.09 % average relative standard deviation), the between-filter ($n = 3$) variation (0.16 % average relative standard deviation), and the within-measurement ($n = 3$) standard deviation of the mean (0.07 %).	A	0.21
2	Data collection methodology; estimated as the standard deviation on the massic activity determined from data collected with the MAC3 and FPGA systems	A	0.20
3	Counting source aliquot mass determinations includes mass measurement precision	B	0.05
4	Effect of kB on the massic activity, using $kB = (0.0075 \pm 0.0025)$ $cm \cdot MeV^{-1}$	B	0.35
5	Standard error on the fit of the MICELLE2 code data used to calculate efficiencies	B	0.01
6	Decay corrections of counting sources for ^{14}C half-life uncertainty of 0.52 %. [9]	B	0.000018
7	Beta endpoint energy for ^{14}C for an uncertainty of 0.02 % [9]	B	0.00014
8	Beta spectrum shape; estimated as the standard deviation on the massic activity recovered with several different shape factor coefficients.	B	0.13
Relative combined standard uncertainty			**0.48**
Relative expanded uncertainty ($k = 2$)			**0.96**

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

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 the radioactivity. 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 4222d 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 Nov 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: BIPM, Sevres Cedex, France; p. 19 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2017).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2017).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 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 Nov 2017).

[6] Alimonti, G., Angloher, G., Arpesella, C., et al.; *Measurement of the ^{14}C Abundance in a Low-Background Liquid Scintillator*; Phys. Lett. B; Vol. 422, pp. 349-358 (1998).

[7] Bergeron, D.E.; Galea, R.; Laureano-Pérez, L.; Zimmerman, B.E.; *Comparison of ^{14}C Liquid Scintillation Counting at NIST and NRC Canada*, Appl. Radiat. Isotop.*;* Vol 109; pp. 30-35 (2016).

[8] National Institute of Standards and Technology (NIST); *Certificate for Standard Reference Material 4222C, Radioactivity Standard for Liquid Scintillation Counting, Carbon-14-n-Hexadecane*; NIST Gaithersburg, MD (1991).

[9] Chechev, V.P.; Bé, M.M; October 2012; *^{14}C LNE-LNHB/CEA Table of Radionuclides*, available at http://www.nucleide.org/DDEP_WG/Nuclides/C-14_tables.pdf (accessed Nov 2017).

[10] Kossert, K., Grau Carles, A.; *Improved Method for The Calculation of The Counting Efficiency of Electron-Capture Nuclides in Liquid Scintillation Samples*; *Appl. Radiat. Isotop.;* Vol. 68, pp. 1482-1488 (2010).

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.