National Institute of Standards & Technology

Certificate

Standard Reference Material® 4927g

Hydrogen-3 Radioactivity Standard

This Standard Reference Material (SRM) consists of a solution of tritiated water (^{3}H labeled oxidane) having a standardized and certified quantity of radioactive hydrogen-3 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 4927g 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 **hydrogen-3** massic activity, at a **Reference Time of 1200 EST, 1 May 2015**, is:

(544.2 ± 5.2) kBq•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, the 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 analysis.

Expiration of Certification: The certification of **SRM 4927g** 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. The overall technical direction and physical measurement leading to recertification were provided by R. Collé and L. Laureano-Perez of the NIST Radiation Physics Division, Radioactivity Group. Confirmatory measurements were performed by D. Bergeron 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.

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Table 1. Certified Massic Activity of SRM 4927g

Radionuclide	**Hydrogen-3**
Reference time	**1200 EST, 1 May 2015**
Massic activity of the solution	**544.2 kBq•g^{-1}**[a]
Relative expanded uncertainty (k = 2)	**0.96 %**[b]

[a] Tracing of 4927g against SRM 4926E [6] was in agreement with that obtained by tracing with SRM 4927F [7] to –0.07 %. SRM 4927g is also in agreement to –0.27 % with a Key Comparison Reference Value (KCRV) for a tritiated water solution that was measured by 19 laboratories as part of an international measurement comparison organized by the Bureau Internationale du Poids et Measures (BIPM) in 2009 [8]. Independent confirmatory measurements by triple-to-double coincidence ratio (TDCR) on SRM 4927F and 4927g were in agreement to –0.32 % and –0.16 %, respectively.

[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 4927g

Source description	Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1]
Solution composition	Distilled water as H_2O and ^3HHO
Solution density	(0.998 ± 0.001) g•mL^{-1} at 21.6 °C[a]
Solution mass	(4.99 ± 0.01) g[a]
Photon-Emitting Impurities	None detected[b]
Half-lives used	^{3}H: (12.312 ± 0.025) a [c] [9]
Calibration methods (and instruments)	The certified value was determined by relative liquid scintillation (LS) counting with two LS commercial counters and five cocktail compositions using quench-varied efficiency tracing with SRM 4927F (originally calibrated in 1998 by length-compensated internal gas proportional counting). Confirmatory measurements of both SRM 4927F and SRM 4927g were performed by triple-to-double coincidence ratio (TDCR) measurements.

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

[b] The estimated lower limits of detection for photon-emitting impurities, expressed as massic photon emission rate, in June 2015, were:

0.13 s^{-1}•g^{-1} in the region 25 keV ≤ E ≤ 45 keV;
0.06 s^{-1}•g^{-1} in the region 50 keV ≤ E ≤ 1430 keV;
0.09 s^{-1}•g^{-1} in the region 1440 keV ≤ E ≤ 1480 keV, and
0.05 s^{-1}•g^{-1} in the region 1490 keV ≤ E ≤ 2000 keV;

[c] The stated uncertainty is the standard uncertainty. See reference 5.

Table 3. Uncertainty Evaluation for the Massic Activity of SRM 4927g

Uncertainty component		Assessment Type[(a)]	Relative standard uncertainty contribution on massic activity of ^{3}H (%)
1	LS measurement precision; standard deviation of the great-grand mean from 14 grand mean determinations, considering both the between-mean (0.19 %) and the typical within-mean (0.12 %) variations. Each grand mean is based on 5 or 6 sources and 5 replicate measurements per source. The within-mean variation on each of the 14 grand means includes the between variation across the 5 or 6 sources (typically 0.12 %) and the within variation for 5 measurements on each source (typically in the range of 0.2 % to 0.4 %).	A	0.23
2	Background; LS measurement variability and cocktail composition stability effects; wholly embodied in component 1.	A	--
3	Quench indicating parameter measurements; wholly embodied in component 1.	A	--
4	Fitting of quench curves (^{3}H efficiency versus quench indicating parameter), includes precision partially embodied in component 1.	A	0.08
5	Counting source aliquot mass determinations includes mass measurement precision partially embodied in component 1.	B	0.05
6	Certified massic activity of SRM 4927F (1998 calibration by gas counting).	B	0.36
7	Decay correction of SRM 4927F to reference time for ^{3}H half-life uncertainty of 0.20 %.	B	0.19
8	Gravimetric dilution of SRM 4927F.	B	0.07
9	Gravimetric dilutions of 3 sampled ampoules of SRM 4927g.	B	0.04
10	Decay corrections of counting sources for ^{3}H half-life uncertainty of 0.20 %.	B	0.006
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 radioactive material 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 4927g 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 2015). 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): BIPM, Sevres Cedex, France; p. 19 (2012); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2015).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_200_2012.pdf (accessed Nov 2015).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2015).

[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 2015).

[6] SRM 4926E; *Hydrogen-3 Radioactivity Standard*; U.S. Department of Commerce, NIST: Gaithersburg, MD (07 February 2011); available at https://www-s.nist.gov/srmors/view_detail.cfm?srm=4926E (accessed Nov 2015).

[7] SRM 4927F; *Hydrogen-3 Radioactivity Standard*; U.S. Department of Commerce, NIST: Gaithersburg, MD (May 2008); available at https://www-s.nist.gov/srmors/view_detail.cfm?srm=4927F (accessed Nov 2015).

[8] Ratel, G.; *International Comparison of Activity Measurement of a Solution of Tritiated Water*; Consultative Committee for Ionizing Radiation (CCRI), CCRI(II)-K2.H-3, Draft B (2015).

[9] Chechev, V.P.; *LNE-LNHB/CEA Table of Radionuclides*, ^{3}H; (June 2006); available at http://www.nucleide.org/DDEP_WG/Nuclides/H-3_tables.pdf (accessed Nov 2015).

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.