

Standard Reference Material® 4338b

Plutonium-240 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 4338b consists of approximately 5 mL of a solution of a standardized and certified quantity of radioactive Plutonium-240 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).

Table 1. Certified Massic Activity of SRM 4338b

| Radionuclide | Plutonium-240 |
|--|--------------------------|
| Reference time | 1200 EST, 27 August 2019 |
| Massic activity of the solution ^(a) | 40.81 Bq·g ⁻¹ |
| Relative expanded uncertainty ($k = 2$) ^(b) | 1.1 % |

^(a) This solution is gravimetrically linked to a ^{240}Pu stock solution obtained from Oak Ridge National Laboratory in February 1980 and was used to prepare the previous NIST ^{240}Pu standard SRM 4338a, which was certified using NIST “0.1 π ” α defined-solid-angle counter with scintillation detector and a reference time of 01 May 1996. A remeasurement of SRM 4338a at the time of this work in 2019 agreed with the decay-corrected 1996 certified value to within 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 [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 4338b** 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>).

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Table 2. Uncertainty Evaluation for the Massic Activity of SRM 4338b

| Uncertainty component | | Assessment Type ^(a) | Relative standard uncertainty contribution on massic activity of ^{240}Pu (%) |
|---|--|--------------------------------|--|
| 1 | Liquid scintillation (LS) measurement precision, relative standard deviation of the mean for six sets of measurements, considering all within- and between-source components of variance. This estimator was based on five replicate measurements on each of five sources prepared with one of two different LS cocktail compositions and measured in three different LS counters (on three separate measurement occasions). The typical standard deviation of the mean for the 25 measurements (5 replicates for 5 sources) within a data set was 0.13 %. | A | 0.54 |
| 2 | LS counters dependencies for an internal relative standard deviation of 0.031 % amongst three counters; wholly embodied in component 1. | A | -- |
| 3 | LS cocktail composition dependencies for an internal relative standard deviation of 0.031 %; wholly embodied in component 1. | A | -- |
| 4 | Gravimetric measurements for preparation of LS counting sources; partially embodied in component 1. | B | 0.02 |
| 5 | Live time determinations for LS counting time intervals; includes uncorrected deadtime effects. | B | 0.06 |
| 6 | Decay correction for ^{240}Pu for a half-life uncertainty of 0.11 %. | B | $<2 \times 10^{-7}$ |
| 7 | Nuclear decay scheme, data probabilities per decay for α -particle emissions and half-lives, including that used for impurity corrections. | B | 0.01 |
| 8 | Radionuclidic impurity corrections. | B | 0.16 |
| 9 | LS detection inefficiency including possible wall effects; partially embodied in component 1. | B | 0.01 |
| Relative combined standard uncertainty | | | 0.57 |
| Relative expanded uncertainty ($k = 2$) | | | 1.1 |

^(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 4338b 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 prescored ampoule; **the gold color band is only for identification** [1]. 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 Oct 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 IR 8524 (2019) available at <http://www.nist.gov/publications/ampoules-radioactivity-standard-reference-materials> (accessed Oct 2022). This is **not** a prescored 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) (2012); available at <https://www.bipm.org/fr/committees/jc/jcgm/publications> (accessed Oct 2022).
- [3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at <https://www.bipm.org/en/publications/guides> (accessed Oct 2022).
- [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 Oct 2022).
- [5] Chechnev, V.P.; *LNE-LNHB/CEA Table of Radionuclides, ²⁴⁰Pu*; (September 2009); available at http://www.lnhb.fr/nuclides/Pu-240_tables.pdf (accessed Oct 2022).
- [6] Chechnev, V.P.; Kuzmenko, N.K.; *LNE-LNHB/CEA Table of Radionuclides, ²⁴¹Pu*; (July 2009); available at http://www.lnhb.fr/nuclides/Pu-241_tables.pdf (accessed Oct 2022).
- [7] Chechnev, V.P.; Kuzmenko, N.K.; *LNE-LNHB/CEA Table of Radionuclides, ²⁴¹Am*; (July 2010); available at http://www.lnhb.fr/nuclides/Am-241_tables.pdf (accessed Oct 2022).
- [8] Chechnev, V.P.; *LNE-LNHB/CEA Table of Radionuclides, ²³⁸Pu*; (January 2010); available at http://www.lnhb.fr/nuclides/Pu-238_tables.pdf (accessed Oct 2022).

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>.

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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 SI or other higher-order reference system. Non-certified values are provided in Table A1.

Table A1. Non-Certified Values for SRM 4338b^(a)

| | |
|---|--|
| Source description | Liquid in a flame-sealed 5 mL borosilicate-glass ampoule [1] |
| Solution composition | $(3.08 \pm 0.03) \text{ mol}\cdot\text{L}^{-1} \text{ HNO}_3$ ^(b) |
| Solution density | $(1.099 \pm 0.001) \text{ g}\cdot\text{mL}^{-1}$ at 22.7°C ^(b) |
| Solution mass | $(5.49 \pm 0.01) \text{ g}$ ^(b) |
| Alpha-particle-emitting impurities ^(b,c) | $^{238}\text{Pu}: (0.31 \pm 0.06) \text{ Bq}\cdot\text{g}^{-1}$ ^(d) $^{241}\text{Am}: (0.015 \pm 0.003) \text{ Bq}\cdot\text{g}^{-1}$ ^(e) |
| Beta-particle-emitting impurities | $^{241}\text{Pu}: (0.08 \pm 0.02) \text{ Bq}\cdot\text{g}^{-1}$ ^(b,d) |
| Photon-emitting impurities | None detected ^(f) |
| Half-lives used ^(g) | $^{240}\text{Pu}: (6561 \pm 7) \text{ a}$ $^{241}\text{Pu}: (14.33 \pm 0.04) \text{ a}$ $^{241}\text{Am}: (432.6 \pm 0.6) \text{ a}$ $^{238}\text{Pu}: (87.74 \pm 0.03) \text{ a}$ |
| Calibration methods (and instruments) | The certified massic activity for ^{240}Pu was obtained by $4\pi\alpha$ liquid scintillation (LS) counting using three different LS measurement systems (with widely varying operating systems). For the determination two different LS cocktails compositions (with 5 counting sources each) were measured 5 times on each of the three instruments. |

^(a) References on Page 3

^(b) The stated uncertainty is two times the standard uncertainty [4].

^(c) The estimated limits of detection for alpha-emitting impurities were:

- 0.04 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies less than 5.0 MeV,
- 0.04 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 5.25 MeV and 5.35 MeV, and
- 0.04 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies greater than 5.6 MeV.

^(d) Based on mass spectrometry on 24 July 1979 by Oak Ridge National Laboratory. Massic activities of the impurities as of reference time 27 August 2019. The total massic activity of other alpha-emitting impurities; $(^{239}\text{Pu} + ^{242}\text{Pu} + ^{241}\text{Pu} [\alpha \text{ only}] + ^{244}\text{Pu}) < 0.003 \text{ Bq}\cdot\text{g}^{-1}$.

^(e) The ^{240}Pu master solution was chemically purified on approximately 10 December 1979, and the daughter radionuclides have been growing in since that time.

^(f) The estimated limits of detection for photon-emitting impurities as of May 1996, expressed as massic photon emission rates (numbers of photons per second per gram), are:

- 0.00015 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 12 keV and 41 keV,
- 0.00008 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 49 keV and 100 keV,
- 0.00005 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 108 keV and 156 keV,
- 0.00001 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 164 keV and 507 keV,
- 0.000004 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 515 keV and 1456 keV, and
- 0.000002 $\text{s}^{-1}\cdot\text{g}^{-1}$ for energies between 1464 keV and 2750 keV,

provided that the photons are separated in energy by 4 keV or more from photons emitted in the decay of ^{240}Pu .

^(g) The stated uncertainty is the standard uncertainty. See references 5-8.

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>).