

Certificate of Analysis

Certified Reference Material

SENS-1

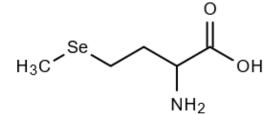
Certified Reference Material of natural selenomethionine

SENS-1 is a primary standard Certified Reference Material (CRM) of natural selenomethionine. A unit of SENS-1 consists of an approximately 25 mg of selenomethionine powder. This material is intended for the calibration of instruments and evaluation of analysis methods for the determination of selenomethionine.

The following table shows those constituents for which certified and reference values have been established for this primary standard CRM. The expanded uncertainty (U_{CRM}) in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor. A coverage factor of two (2) was applied which corresponds to approximately 95 % confidence. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement.

Element/substance	Mass Fraction, g/g	Type of value
selenomethionine (a,b,c)	0.961 ± 0.007	certified
selenomethionine, as Se (a,b,c)	0.387 ± 0.003	certified
total selenium (d)	0.398 ± 0.009	reference

Table 1: Quantity values and expanded uncertainties (k = 2) for SENS-1



selenomethionine

CAS registry number: 1464-42-2 (DL enantiomer) InChI Key: RJFAYQIBOAGBLC-UHFFFAOYSA-N

Molecular formula: $C_5H_{11}NO_2Se$ Molar Mass: 196.117 ± 0.012 g/mol



Coding

The coding refers to the instrumental method of analyte determination.

- **a** Proton nuclear magnetic resonance spectroscopy (¹H-qNMR)
- **b** High performance liquid chromatography inductively-coupled plasma mass spectrometry (HPLC-ICP-MS)
- c High performance liquid chromatography electrospray tandem mass spectrometry (HPLC-ES-MS/MS)
- **d** Isotope dilution inductively-coupled plasma mass spectrometry (ID-ICP-MS)

Certified values

Certified values are considered to be those for which the National Research Council Canada (NRC) has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and are reflected in the stated expanded uncertainties. Certified values are the best estimate of the true value and uncertainty.

Reference values

Reference values are those for which insufficient data are available to provide a comprehensive estimate of uncertainty.

Intended use

This primary standard certified reference material is primarily intended for use in the calibration of procedures and the development of methods used for the quantitative analysis of selenomethionine. A minimum sample size of 10 mg is recommended.

Storage and sampling

It is recommended that the material is stored at nominal temperature of -20 °C under typical freezer conditions. The bottle should be opened only in a clean area with precautions taken against contamination during sampling.

Preparation of material

L-(+)selenomethionine powder was acquired from a commercial supplier and was used as received. The material was bottled into 25 mg units.

Stability

The purity of selenomethionine has not changed over a 12 month period at nominal temperature of–20 °C. Uncertainty components for long and short-term stability were considered negligible and are thus not included in the uncertainty budget.

Homogeneity

The material was tested for homogeneity at the NRC using ¹H-qNMR. Results from a representative number of bottles across the fill series were evaluated using DerSimonian-Laird random effects model [2] and included in the calculation of the quantity values.



Uncertainty

Included in the overall combined uncertainty estimate are uncertainties associated with the two methods of analysis, uncertainties related to possible between-bottle variation, and uncertainties related to inconsistency between the various measurement methods. The latter is estimated as the heterogeneity in the random effects model fitted to the results of individual methods, also known as the dark uncertainty [3,4]. The overall combined uncertainty associated with the selenomethionine content of SENS-1 is largely driven by the uncertainties associated with the measurement methods and inconsistencies between these methods.

Metrological traceability

Results presented in this certificate are traceable to the SI through gravimetrically prepared standards of NIST SRM 84L (potassium hydrogen phthalate, KHP), which was employed as an internal standard for ¹H-qNMR. As such, SENS-1 serves as suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Quality Management System (ISO 17034, ISO/IEC 17025)

This material was produced in compliance with the NRC Metrology Quality Management System, which conforms to the requirements of ISO 17034 and ISO/IEC 17025. The Metrology Quality Management System supporting NRC Calibration and Measurement Capabilities, as listed in the *Bureau international des poids et mesures* (BIPM) Key Comparison Database (kcdb.bipm.org/), has been reviewed and approved under the authority of the Inter-American Metrology System (SIM) and found to be in compliance with the expectations of the *Comité international des poids et mesures* (CIPM) Mutual Recognition Arrangement. The SIM approval is available upon request.

Updates

Users should ensure that the certificate they have is current. Our website at <u>www.nrc.gc.ca/crm</u> will contain any new information.

References

- 1. Evaluation of measurement data: Guide to the expression of uncertainty in measurement JCGM100:2008. https://www.bipm.org/en/publications/guides/gum.html
- 2. DerSimonian R, Laird N (1986) Meta-analysis in clinical trials. *Controlled Clinical Trials*, 7: 177-188. doi.org/10.1016/0197-2456(86)90046-2
- 3. Possolo A, Toman B (2007) Assessment of measurement uncertainty via observation equations. *Metrologia*, 44: 464-475. <u>doi.org/10.1088/0026-1394/44/6/005</u>
- 4. Thompson M, Ellison SLR (2011) Dark uncertainty. *Accreditation and Quality Assurance*, 16: 483-487. doi.org/10.1007/s00769-011-0803-0

Cited by

A list of scientific publications citing SENS-1 CRM can be found at doi.org/10.4224/crm.2020.sens-1.

Authorship

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Acknowledgements

The following NRC staff members are gratefully acknowledged: Enea Pagliano, Indumathi Pihillagawa Gedara, Garnet McRae, Ovidiu Mihai, and Kenny Nadeau.

Citation

LeBlanc K, Le PM, Meija J, Grinberg P, Melanson J, Mester Z. SENS-1: Certified Reference Material of natural selenomethionine. Ottawa: National Research Council Canada; 2020.

Available from: doi.org/10.4224/crm.2020.sens-1



SENS-1

Date of issue: September 2020 Date of expiry: September 2028 Revised: July 2022 (date of expiry extended)

Approved by:

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This Certificate is only valid if the corresponding material was obtained directly from the NRC or an Authorized Reseller.

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