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Certificate of Analysis

Certified Reference Material

SELM-1

Selenium enriched yeast Certified Reference Material

SELM-1 is a selenium enriched yeast Certified Reference Material (CRM) from the National Research Council Canada (NRC) with information on total selenium, selenomethionine and methionine content. A unit of SELM-1 consists of approximately 8 grams of selenium enriched yeast in an amber glass vial.

Table 1 shows those analytes for which certified values have been established for SELM-1. Certified values are based on unweighted mean results from data generated by three independent sets of measurements in the case of selenomethionine (SeMet) and methionine (Met) and two independent measurements for total selenium. The expanded uncertainty (U_{CRM}) in the certified value is equal to $U_{CRM} = ku_c$ where u_c is the combined standard uncertainty calculated according to the JCGM Guide [1] and k is the coverage factor. The value of u_c is determined from the combined uncertainties of the various analytical methods (u_{char}) as well as uncertainties associated with homogeneity (u_{hom}) and stability (u_{stab}). It is intended that U_{CRM} encompasses every aspect that reasonably contributes to the uncertainty of the measurand [2,3]. A coverage factor of two ($k = 2$) was applied.

Table 1: Certified quantity values and expanded uncertainties ($k = 2$) for SELM-1

Measurand	Mass fraction, mg/kg
total selenium	2031 ± 70
selenomethionine	3190 ± 260
methionine	5790 ± 100

The analytical methods used/developed for the measurement of SeMet and Met in yeast matrix are documented in the peer-reviewed literature [4-9]. Property values were determined by isotope dilution GC-MS and isotope dilution LC-MS (SeMet and Met) and by isotope dilution ICP-MS and ICP-AES for total Se.

Apart from SeMet, a number Se species have been detected in yeast and specifically in the NRC SELM-1 CRM. Please see ref. [10] and the associated database, available through NRC's Digital Repository [11]. In addition to Se molecular species, Se nanoparticles have also been detected in SELM-1 CRM [12].

Certified values

Certified values are considered to be those for which the NRC has the highest confidence in accuracy and that all known and suspected sources of bias have been taken into account and

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are reflected in the stated expanded uncertainties. Certified values are the best estimate of the true value and uncertainty.

Intended use

This certified reference material is intended for the calibration of instruments and evaluation of methods for the determination of selenomethionine, methionine, and total selenium in yeast or materials of a similar matrix. The material is not intended for nutritional, medical or diagnostic use. A minimum sample mass of 60 mg is recommended.

Storage and sampling

To ensure the stability of the selenomethionine and methionine it is necessary to store this material at a temperature of –20 °C. Prior to use, the bottle should be rotated and shaken to ensure the contents are well mixed. The bottle should be tightly closed thereafter and returned to –20 °C storage.

Instructions for drying

A separate sample aliquot should be dried to a constant mass to obtain moisture content. Drying for 4 days in a freeze-dryer has proved to be a relatively simple method to achieve constant mass. The moisture content in SELM-1 is approx. 0.043 g/g.

Preparation of material

A dry commercial selenized yeast sample (yeast grown in Se rich media) was used for the preparation of this CRM. No additional screening or blending was used. The material was bottled ‘as is’. The material was bottled in cleaned amber glass bottles at an 8 g nominal mass. During bottling argon gas was used to flush the headspace of the vials to displace air. After bottling the material was sterilized by subjecting it to a minimum dose of 25 kGy gamma irradiation.

Stability

Long- and short-term stability (transport) of SELM-1 were evaluated and considered satisfactory for total Se. However, some minor losses of Met and SeMet were observed and a stability uncertainty component was thus assigned.

Homogeneity

This material was tested for homogeneity using ANOVA based on results from randomly selected bottles. Results from different bottles, as determined by ID-GC-MS, resulted in uncertainty components reported in Table 2. The homogeneity is warranted for sub-samples of 60 mg or greater.

Uncertainty

Included in the combined uncertainty estimate (u_c) are uncertainties in the batch characterization (u_{char}), uncertainties related to possible between-bottle variation (u_{hom}), uncertainties related to long-term storage and transportation ($u_{stabity}$), and uncertainties related to inconsistency between the various measurement methods (u_{method}). 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 [13-14]. Expressed as standard uncertainties, these components are listed in Table 2.

Table 2: Uncertainty Components for SELM-1

Substance	u_c, mg/kg	u_{char}, mg/kg	u_{hom}, mg/kg	$u_{stability}$, mg/kg	u_{method}, mg/kg
total selenium	35	11	15	–	30
selenomethionine	130	30	20	99	80
methionine	50	20	30	32	10

Metrological traceability

Results presented in this certificate are traceable to the International System of Units (SI) through gravimetrically prepared standards established purity. As such, they serve as suitable reference materials 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

For updates please refer to doi.org/10.4224/crm.2010.selm-1.

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Cited by

A list of scientific publications citing SELM-1 can be found at doi.org/10.4224/crm.2010.selm-1.

Authorship

Z. Mester[1], C. Brophy[1], M. McCooeye[1], V.P. Clancy[1], P. Maxwell[1], S. McSheehy[1], R.E. Sturgeon[1], S.N. Willie[1], L. Yang[1] and K. LeBlanc[1]

[1] National Research Council Canada, 1200 Montreal Rd, Ottawa, ON, K1A 0R6, Canada

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SELM-1

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Approved by:

Zoltan Mester, Ph. D.
Team Leader, Inorganic Chemical Metrology
NRC Metrology

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

National Research Council Canada
Metrology
1200 Montreal Road
Building M36, Room 1029
Ottawa, Ontario K1A 0R6

Telephone: 613-993-2359
Fax: 613-993-8915
Email: CRM-MRCOttawa@nrc-cnrc.gc.ca

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