Certified Reference Material

National Research Council Canada | Conseil national de recherches Canada

SELM-1

Selenium Enriched Yeast Certified Reference Material

The following table shows those analytes for which certified values have been established for this selenium enriched yeast reference material (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 $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO 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]. A coverage factor of 2 was applied. The table below lists measurands certified in SELM-1.

CERTIFIED VALUES
(milligram/kilogram)

Total Selenium	2059	± 64
Selenomethionine	3431	± 157
Methionine	5758	± 277

A comprehensive certification report is available upon request.

The analytical methods used / developed for the measurement of SeMet and Met in yeast matrix are documented in the peer-reviewed literature [4-8].

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Intended Use

This certified reference material is intended for the calibration of instruments and evaluation of methods for the determination of SeMet, Met, and total selenium in yeast or materials of a similar matrix. The material is not intended for nutritional, medical or diagnostic use as its safety has not been established.

Storage and Sampling

To ensure the stability of the SeMet and Met 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.

Preparation of SELM-1

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 8g nominal weight. 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 at the Canadian Irradiation Centre, Laval, Québec.

Instructions for Drying

A separate aliquot of the sample should be used to obtain a dry weight correction factor. Drying for 4 days in a freeze-dryer has proved to be a relatively simple method to achieve constant weight.

Uncertainty

New guidelines for CRM producers suggest all sources relevant to the user of the material should contribute to the uncertainty of the certified value [2,3]. Included in the overall uncertainty estimate are uncertainties associated with the batch characterisation (u_{char}), uncertainties related to possible between-bottle variation (u_{hom}) as well as instability derived from effects relating to long-term storage and transport (u_{stab}). Expressed as standard uncertainties these components can be combined as:

$$u^2_{c(CRM)} = u^2_{char} + u^2_{hom} + u^2_{stab} \qquad (1)$$

Results for the various statistics used to calculate the certified values are shown in Table 2.

Characterization

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-OES for total Se. All measurements contributed to this certificate were conducted by Chemical Metrology personnel at INMS NRC Ottawa.

The characterisation uncertainties (u_{char}) were calculated in accordance with equation 2,

$$u_{char} = \frac{s}{\sqrt{p}} \qquad (2)$$

where s is the standard deviation of the means and p is the number of mean results included in the calculation. The calculated uncertainty components related to the characterization of SELM-1 are reported in Table 2.

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 contribution to uncertainty due to inhomogeneity, u_{hom}, was equated to the experimentally determined between-unit standard deviation ($s_{between}$) as the best estimate of the uncertainty due to between-unit heterogeneity. However, for Se, the situation depicted by equation 3 occurred:

$$s^2_{between} < \frac{s^2_{meas}}{n} \qquad (3)$$

where s_{meas} is the repeatability standard deviation for the method used in the homogeneity assessment and n is the number of replicates per unit. For this case, u_{hom} was calculated according to 4:

$$u_{hom} = \sqrt{\frac{MS_{within}}{n}} \sqrt[4]{\frac{2}{v_{MS_{within}}}} \qquad (4)$$

where MS_{within} represents the mean squares within groups and $v_{MS_{within}}$ is the number of degrees of freedom.

The homogeneity is warranted for sub-samples of 250 mg or greater.

Stability

Based on a half-year study of the stability of Met and SeMet in SELM-1, an uncertainty component was assigned. Measurements were conducted on samples stored for one month at -20°C, +4°C, +22°C and +44°C.

An uncertainty component associated with the short term stability (transport) was evaluated but considered insignificant for Met and total Se. However, some loss of SeMet was observed and a short term stability uncertainty component was thus assigned.

Expiration of Certification

The certified values are valid until November 2006 within the measurement uncertainty specified, provided the CRM is handled and stored in accordance with instructions herein. The stability of this CRM will continue to be monitored. Our web site should be consulted for any new information.

Updates

It is anticipated that as more data become available, the established values may be updated and reliable values assigned to more elements.

Our web site at http://inms-ienm.nrc-cnrc.gc.ca/calserv/chemical_metrology_e.html will contain any new information.

Table 2. Uncertainty component for SELM-1.

	Se, mg/kg	SeMet, mg/kg	Met, mg/kg
u_{char}	28	29	54
u_{hom}	15	20	31
u_{stab}	-	70	124
u_c	32	78	138
U_{CRM} (k=2)	64	157	277

References

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The results listed in this certificate are traceable to the SI through gravimetrically prepared standards of established purity and international measurement intercomparisons. As such, they serve as suitable reference materials for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

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