National Research Council Canada | Conseil national de recherches Canada

Certificate of Analysis

Certified Reference Material

BEET-1

Isotopic Certified Reference Material of beet sugar

BEET-1 is a beet sugar Certified Reference Material (CRM) that is certified for carbon isotope delta values. BEET-1 is part of a suite of sugar based isotopic CRMs from the National Research Council of Canada (NRC). Each unit of BEET-1 contains approximately 1 g of beet sugar. In conjunction with FRUT-1 and GALT-1, this CRM is intended for the calibration of procedures and development of methods for normalizing carbon isotope delta measurements of sugar-based or other carbon-bearing materials to the Vienna Peedee Belemnite (VPDB) scale.

The following table shows the certified carbon isotope delta value that has been established for this beet sugar isotopic CRM. The combined 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. A coverage factor of two (2) was applied. It is intended that U_{CRM} accounts for every aspect that reasonably contributes to the uncertainty of the measurement. The certified carbon isotope delta value and its expanded uncertainty are expressed on the VPDB scale with a value of –46.6 ‰ assigned to LSVEC and +1.95 ‰ to NBS19 [2].

Table 1: Certified quantity value and uncertainty for BEET-1

Material	Isotope delta, $\delta_{VPDB}(^{13}C)$, ‰	Combined standard uncertainty, ‰	Combined expanded uncertainty, ‰
BEET-1	–26.01	0.04	0.07

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

The certified value for carbon isotope delta in BEET-1 was determined by combining elemental analysis with isotope ratio mass spectrometry (EA-IRMS) measurement results from seven participating expert laboratories, including the NRC, using a random laboratory effects statistical model.

Canada

Intended use

This certified reference material is primarily intended to be used with at least one other carbon reference material [3], preferably as a set of three with FRUT-1 [4] and GALT-1 [5] CRMs, for normalizing isotope delta measurements of sugar-based or other carbon-bearing materials to the VPDB scale. A minimum sample mass of 550 µg is recommended.

Storage and sampling

Unopened ampules of BEET-1 may be stored at room temperature in a clean location out of direct sunlight. Once the ampule is opened, it is recommended that all contents be immediately transferred to a clean, dry vial with a tight-fitting closure. The vial should be stored at room temperature in a dry box or desiccator out of direct sunlight when not in use.

Preparation of material

BEET-1 was prepared from commercially available beet sugar. Beet sugar was sieved in small aliquots using a 0.3 mm mesh sieve. All beet sugar crystals larger than 0.3 mm were transferred into a clean plastic bag, flushed with argon, sealed, and then tumbled for further homogenization. A unit of BEET-1 comprises a sealed, argon-flushed, ampule containing approximately 1 g of beet sugar [6].

Stability

BEET-1 is isotopically stable when stored under recommended conditions and typical transportation temperatures.

The transportation and long-term stability were assessed at the NRC using EA-IRMS. The transportation stability was carried out via an isochronous approach, over a period of two weeks, using unopened ampoules at temperatures ranging from –20 °C to +37 °C. The long-term stability was assessed at room temperature for a period of 5 years.

Homogeneity

The homogeneity of the carbon isotope delta values in BEET-1 was assessed by analyzing 33 units of BEET-1. These results were evaluated using Bayesian random effects model and were included in the calculation of the certified value.

Uncertainty

Included in the combined standard uncertainty estimate (u_c) are the uncertainties from characterization by the NRC and six external laboratories (u_{char}), and the uncertainty related to between-bottle variation (u_{hom}). Expressed as standard uncertainties, these components are listed in Table 2.

Table 2: Uncertainty components for BEET-1

Material	u_c, ‰	u_{char}, ‰	u_{hom}, ‰
BEET-1	0.04	0.021	0.028

The uncertainty due to characterization (u_{char}) includes the uncertainty due to measurement of the sugar samples and the international reference materials, uncertainty due to possible

inconsistencies between the laboratory results, and uncertainty assigned to the values of the international reference materials.

Metrological traceability

The *Comité international des poids et mesures* has noted that isotope delta measurements that cannot presently be made traceable to the International System of Units (SI) should be made traceable to materials recognized as international standards by the IUPAC Commission on Isotopic Abundances and Atomic Weights [7]. Since measurements used to determine the certified carbon isotope delta value in beet sugar are traceable to internationally recognized standards [3], BEET-1 serves as a suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

Traceability of carbon isotope delta values in BEET-1 was established using internationally recognized reference materials. The standard uncertainties for IAEA-CH-6 [8], USGS62 [9], and USGS40 [10] were revised to incorporate the uncertainty associated with the coherence between the reference materials (u = 0.029 ‰) [6], resulting in the following values:

IAEA-CH-6: –10.450(49) ‰ USGS62: –14.790(49) ‰ USGS40: –26.390(49) ‰

Additionally, IAEA-603, IAEA-610, IAEA-611, and IAEA-612 were used as calibrators. However, the isotope delta values for these materials are certified relative to the VPDB with no reference to LSVEC [11,12]. Consequently, these values were converted to the VPDB scale as defined by IUPAC using both NBS19 and LSVEC [13, 14]. Thus, the following isotope delta values were used for these reference materials:

IAEA-603: +2.474(23) ‰	IAEA-610: –9.145(19) ‰
IAEA-611: –30.925(21) ‰	IAEA-612: –36.878(26) ‰

The reassessment of IAEA-603, IAEA-610, IAEA-611, and IAEA-612 did not include the aforementioned additional uncertainty due to lack of coherence [6] because this suite of materials was calibrated independent of the other internationally recognized reference materials.

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. For updates, please refer to doi.org/10.4224/crm.2018.beet-1.

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Cited by

A list of scientific publications citing BEET-1 can be found at doi.org/10.4224/crm.2018.beet-1

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BEET-1

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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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