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Reference Material 8542

IAEA-CH-6 Sucrose

(Carbon Isotopes in Sucrose)

REFERENCE MATERIAL INFORMATION SHEET

Purpose: This Reference Material (RM) is a secondary reference material with known stable isotope ratios for carbon (C) [1,2]. It is intended to be a control for working standards that have been calibrated to the VPDB (Vienna Pee Dee Belemnite) δ-scale. The equivalent name for this RM, as used by the International Atomic Energy Agency (IAEA) and the U.S. Geological Survey (USGS), is IAEA-CH-6.

Description: A unit of RM 8542 consists of one bottle containing approximately 1 g of sucrose.

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control [3,4]. The assigned values for this RM are at present the best estimate of the true values. RM 8542 is a secondary reference material with a known value for carbon stable isotope ratios. RM 8542 is traceable to NBS19 (RM 8544) and LSVEC (RM 8545) [5].

Table 1. Non-Certified Value and Uncertainties for Carbon Stable Isotopes of RM 8542

NIST RM Number	IAEA Name	Non-Certified Value $10^3\,\delta^{13}C_{VPDB}$	Combined Uncertainty $10^3\,\delta^{13}C_{VPDB}$ [(a)]	Expanded Uncertainty $10^3\,\delta^{13}C_{VPDB}$ [(a)]
8542	IAEA-CH-6	–10.45 ‰	0.03 ‰	0.07 ‰

[(a)] RM 8542 is given with a combined standard uncertainty in addition to an expanded uncertainty value, $k = 2$. The expanded uncertainty is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, as defined in the ISO/JCGM Guide [4]. The non-certified value and uncertainties are given in units of per mil (‰), which is equivalent to per thousand.

Period of Validity: The non-certified value is valid within the measurement uncertainty specified until **31 December 2031.** The value assignment is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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 Reference Material Information Sheet and notify registered users. RM 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 RM. 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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Storage: RM 8542 is stable at normal room temperatures. To minimize the potential for contamination, it is recommended that this RM be stored in the container in which it is supplied.

Additional Information: The distribution of RM 8542 is limited to one unit per three-year period. Users are encouraged to prepare their own standards for daily use and calibrate those standards against international reference materials. Preparation, analysis, and reporting information can be found in Appendix A.

REFERENCES

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Information Sheet Revision History: 06 May 2022 (Combined standard uncertainty added; change of period of validity; terminology updated; updated format; editorial changes); 19 May 2011 (Extension of the expiration date and minor editorial changes); 01 November 2007 (This technical revision reports a change in the reference value and associated uncertainty as well as an update of expiration date); 22 June 1992 (Original report date).

Certain commercial equipment, instruments, or materials may be identified in this Reference Material Information Sheet 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 RM should ensure that the Reference Material Information Sheet 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

PREPARATION AND ANALYSIS

Technical aspects involved in the issuance of this RM were coordinated through the NIST Chemical Sciences Division by R.A. Kraft.

Support aspects involved in the issuance of this RM were coordinated through the NIST Office of Reference Materials.

Sample Preparation: IAEA-CH-6 was produced by H. Polach, Australian National University, Canberra, Australia [6]. It was originally named ANU Sucrose and was collected to replace NBS oxalic acid as a reference material for ^{14}C measurements [7].

Analytical Methods: The $\delta^{13}C$ value and uncertainty were evaluated and updated in an interlaboratory comparison in 2006 [5]. Four laboratories participated in the interlaboratory comparison: Centrum voor Isotopen Onderzoek, Rijksuniversiteit Groningen, Groningen, Netherlands; Max-Planck-Institute for Biogeochemistry, Jena, Germany; UFZ [Umweltforschungszentrum] Leipzig-Halle GmbH, Leipzig, Germany; and United States Geological Survey (USGS), Reston, VA. The statistical analysis for mean values and uncertainties was performed at NIST. Continuous flow elemental analyzer isotope ratio mass spectrometry (EA-IRMS) methods were used for value and uncertainty determination.

As part of the interlaboratory comparison data each laboratory submitted data on multiple reference materials. The mean value for each measurement was deconvoluted into two components, u_i and α_{ij}. A distribution for the α_{ij} values was calculated with a mean (λ_i) and variance (τ_j). The λ_i value for each laboratory represents a systematic effect assumed to be present across all reference materials for that laboratory. An estimated u_i for each reference material is used as a consensus mean value. Estimation of the consensus means and uncertainties was done using Win-BUGS [8,9], which employs Markov Chain Monte Carlo computation with a Bayesian hierarchical model that has noninformative priors on all of the hyperparameters [10].

Homogeneity: While no homogeneity analysis is available from the original introduction of the material [6], data from the interlaboratory comparison suggests that there is no evidence of carbon isotopic heterogeneity [5].

REPORTING

Terminology: The terminology used here is based on the guidance given by IUPAC for isotope terminology, where stable isotope-number ratio refers to the number of atoms of one isotope relative to the number of atoms of a second isotope in the same system [2]. This is often abbreviated to stable isotope ratio. Isotope-delta value refers to the stable isotope-number ratio of a measured sample relative to the stable isotope-number ratio of a reference material (see example below). Isotope-amount ratio is numerically the same as isotope-number ratio but refers specifically to the amount (moles) of an isotope relative to the amount (moles) of another isotope in the same system [11].

Isotope-delta Values: The carbon stable isotope-delta values of a measured sample reported on the VPDB scale are defined as the difference in measured isotope-number ratio of carbon in a sample relative to the isotope-number ratio of carbon in VPDB:

$$\delta^{13}C = \frac{\left[\frac{N_{sample}(^{13}C)}{N_{sample}(^{12}C)}\right] - \left[\frac{N_{VPDB}(^{13}C)}{N_{VPDB}(^{12}C)}\right]}{\left[\frac{N_{VPDB}(^{13}C)}{N_{VPDB}(^{12}C)}\right]}$$

Normalization: IAEA-603 has been proposed as a replacement for RM 8544 (NBS19) the zero point of the carbon stable isotope δ-scale. In 2006 LSVEC was proposed as a second ^{13}C anchor for a two-point normalization of the $\delta^{13}C$ scale with a value of –46.60 ‰ with no uncertainty, as a scale defining material [5]. In 2016 measurements at the IAEA [12] and USGS [13] clearly established that LSVEC shows at least a 0.2 ‰ range in $\delta^{13}C$ values and therefore is no longer considered to be isotopically stable or homogeneous. At this time, no second anchor has been accepted for the VPDB scale, however, USGS44 has been proposed as a second anchor, and has a $\delta^{13}C$ value of –42.21 ‰. While the isotope metrology community deliberates, USGS44 is available as a second anchor calibration.

A formula for normalizing carbon isotope measurement results using two laboratory standards LS1 (IAEA-603) and LS2 (USGS44) can be expressed as:

$$\delta^{13}C_{sample,cal} = \delta^{13}C_{LS1,cal} + \left(\delta^{13}C_{sample,WS} - \delta^{13}C_{LS1,WS}\right) \times f$$

where the normalization factor f is:

$$f = \frac{\left(\delta^{13}C_{LS2,cal} - \delta^{13}C_{LS1,cal}\right)}{\left(\delta^{13}C_{LS2,WS} - \delta^{13}C_{LS1,WS}\right)}$$

where *WS* denotes measurements made versus a transfer gas (working standard),
cal denotes calibrated measurements made versus the VPDB scale, and
$\delta^{13}C_{LS1,cal}$ and $\delta^{13}C_{LS2,cal}$ are the conventionally fixed $\delta^{13}C$ values for IAEA-603 and USGS44 or those of calibrated laboratory working standards.

The δ-definition above assumes $f = 1$, and does not account for scale compression.

Please note that the reporting scales for $\delta^{13}C$ is still referred to as the VPDB scale despite the exhaustion of the original supply of RM 8544 (NBS19).

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