National Institute of Standards & Technology

# Report of Investigation

## Reference Material 8543

### NBS 18
### (Carbon Isotopes in Carbonatite)

This Reference Material (RM) is intended for use in developing and validating methods for measuring relative differences in carbon (C) isotope-number ratios, $R(^{13}C/^{12}C)$ [1]. Even though the value for this RM is a reference value and not certified [2], its use will improve the comparability of data from different laboratories. The equivalent name for this RM as used by the International Atomic Energy Agency (IAEA) and the U.S. Geological Survey (USGS) is NBS 18. A unit of RM 8543 consists of one bottle containing approximately 0.4 g of igneous calcium carbonate.

Table 1. Reference Value[(a)] and Expanded Uncertainty
for the Relative C Isotope-Number Ratio Differences of RM 8543

| RM Number | Name | Reference Value $10^3\ \delta\ ^{13}C_{VPDB\text{-}LSVEC}$[(b)] | Expanded Uncertainty $10^3\ \delta\ ^{13}C_{VPDB\text{-}LSVEC}$[(b)] |
|---|---|---|---|
| 8543 | NBS 18 | –5.01 | ± 0.07 |

(a) A reference value is a non-certified value that is the best estimate of the true value; however, the value may reflect only the measurement precision and may not include all sources of uncertainty [2].

(b) The $\delta^{13}C$ value is expressed as a mean and an expanded uncertainty. An expanded uncertainty is equal to $U = ku_c$, where $u_c$ is the combined standard uncertainty as defined by the JCGM Guide [3] and $k$ is the coverage factor. The value of the consensus mean and the associated combined standard uncertainty were calculated using a multivariate Bayesian approach [4]. The combined standard uncertainty is intended to represent, at the level of one standard deviation, the combined effects of uncertainty sources evaluated by both Type A and B methods. Any uncertainty due to biases in the methods is not included in the expanded uncertainty. The coverage factor, $k = 2$, provides an expanded uncertainty interval that has about a 95 % probability of encompassing the consensus mean. The $\delta^{13}C$ value and expanded uncertainty are taken from Table S-4 (Supporting Info) [4]. (VPDB - Vienna Peedee belemnite; LSVEC - Li Svec (named for H. Svec, formerly of Ames Laboratory, Iowa)

**Expiration of Value Assignment: RM 8543** is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the RM is handled and stored in accordance with instructions given in this Report of Investigation (see "Instructions for Storage and Handling"). This report is nullified if the RM is damaged, contaminated, or otherwise modified.

**Maintenance of RM:** NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before the expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The technical aspects involved in the issuance of this RM were coordinated through the NIST Chemical Sciences Division by R.D. Vocke, Jr.

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

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Gaithersburg, MD 20899
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*Report Revision History on Last Page*

Robert L. Watters, Jr., Director
Office of Reference Materials

**Reference Difference in Isotope-Number Ratio Values:** The differences in measured isotope-number ratios of stable carbon isotopes in substance P, $R(^{13}C/^{12}C)_P = [N(^{13}C)_P / N(^{12}C)_P]$, are reported as $\delta^{13}C$ values [5]. The relative differences in isotope-number ratios for carbon are referenced to VPDB, where:

$$\delta^{13}C = [R(^{13}C/^{12}C)_{sample} / R(^{13}C/^{12}C)_{VPDB\text{-}LSVEC}] - 1 \quad (1)$$

VPDB-LSVEC refers to the Vienna PDB-LSVEC scale which is defined by assigning a $\delta^{13}C$ value of +1.95 ‰ to NBS 19 (RM 8544) and a consensus value of –46.6 ‰ to LSVEC (RM 8545) [6] for the purpose of normalizing stable carbon isotopic measurements (see *Normalization* [5,6]). The symbol ‰ is part per thousand and is equal to 0.001.

## INSTRUCTIONS FOR STORAGE AND HANDLING

**Storage and Handling:** RM 8543 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.

**Distribution:** The distribution of RM 8543 (NBS 18) is limited to one unit per customer per three-year period of time.

## PREPARATION AND ANALYSIS

**Sample Preparation:** RM 8543 (NBS 18) was prepared by H. Gerstenberger and M. Herrmann, Zentralinstitut für Isotopen und Strahlenforschung, Leipzig, Germany [7,8].

**Analytical Methods:** The $\delta^{13}C$ value and expanded uncertainty reported in Table 1 are taken from results of an inter-laboratory study involving a two point calibration [4]. Results from four expert laboratories (Centrum voor Isotopen Onderzoek, Rijksuniversiteit Groningen, Groningen, Netherlands; Max-Planck-Institute for Biogeochemistry, Jena, Germany; UFZ (Umweltforschungszentrum) Leipzig-Halle GmbH, Leipzig, Germany; U.S. Geological Survey, Reston, Virginia, USA) using continuous flow elemental-analyzer isotope-ratio mass spectrometry and following the general method of Qi *et al.* [9] were combined using a multivariate Bayesian approach for data reduction [4].

The $\delta^{13}C$ value and expanded uncertainty reported in Table 1 for RM 8543 (NBS 18) is the value accepted by the Commission on Isotopic Abundances and Atomic Weights (CIAAW) of the International Union of Pure and Applied Chemistry (IUPAC) (http://ciaaw.org/Carbon.htm) for this RM as of the date of this report.

**Isotopic Homogeneity**: Data from the inter-laboratory comparisons of NBS 18 suggest that there may be carbon isotopic heterogeneity at the grain to grain level.

**Normalization:** The $\delta^{13}C$ values in samples should be normalized to the VPTB-LSVEC $\delta$-scale by calibrating the measurement with respect to the $\delta$-value for NBS 19 (RM 8544) and the $\delta$-value for LSVEC (RM 8545), the $^{13}C$-depleted anchor RM [5,6]. A general formula for normalizing measured carbon isotope number ratios using two laboratory standards LS1 (NBS 19) and LS2 (LSVEC) can be expressed as:

$$\delta^{13}C_{sample,cal} = \delta^{13}C_{LS1,cal} + \left(\delta^{13}C_{sample,WS} - \delta^{13}C_{LS1,WS}\right) \times f \quad (2)$$

where the normalization factor $f$ is:

$$f = \left(\delta^{13}C_{LS2,cal} - \delta^{13}C_{LS1,cal}\right) \Big/ \left(\delta^{13}C_{LS2,WS} - \delta^{13}C_{LS1,WS}\right) \quad (3)$$

**Note**: In the above formulas, 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 NBS 19 and LSVEC. WS denotes measurements made versus a transfer gas (working standard), $\delta^{13}C_{LS1,WS}$, and $\delta^{13}C_{LS2,WS}$ are the $\delta^{13}C$ values for calibrated laboratory working standards.

**Reporting of Stable Carbon Isotope $\delta$ values:** The following recommendations from IUPAC are provided for reporting $\delta^{13}C$ values [5,6,10]. It is recommended that:

- $\delta^{13}C$ values of all carbon-bearing substances be measured and expressed relative to VPDB on a normalized scale where LSVEC has a consensus value of –46.6 ‰ and NBS 19 has a value of +1.95 ‰;
- Authors should clearly state that their data have been normalized.

In addition, researchers are encouraged to report the isotopic compositions of RM 8543 (NBS 18) and other internationally distributed carbon isotopic reference materials [11] in their publications, as appropriate to the method, as though they have been interspersed among unknowns.

Current Reports of Investigation (ROI) for all light stable isotopic Reference Materials mentioned in this report are available on the NIST Standard Reference Materials web site [12].

## REFERENCES


[1] Coplen, T.B.; *Guidelines and Recommended Terms for Expression of Stable-Isotope-Ratio and Gas-Ratio Measurement Results*, Rapid Commun. Mass Spectrom., Vol. 25, pp. 2538–2560 (2011); available at http://onlinelibrary.wiley.com/doi/10.1002/rcm.5129/pdf (accessed Mar 2013).

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[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Mar 2013); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Mar 2013).

[4] Coplen, T.B.; Brand, W.A.; Gehre, M.; Groening, M.; Meijer, H.A.J.; Toman, B.; Verkouteren, R.M.; *New Guidelines for $\delta^{13}C$ Measurements*; Analytical Chemistry, Vol. 78, pp. 2439-2441 (2006).

[5] Coplen, T.B.; *Reporting of Stable Hydrogen, Carbon and Oxygen Isotopic Abundances*; Pure & Applied Chemistry, Vol. 66, No. (2), pp. 273–276 (1994).

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[7] Gerstenberger, H.; Hermann, M.; *Report on the Intercomparison for the Isotope Standard Limestone LH2 and Polyethylene Foil PEF-1.* ZFI Mitteilungen, Vol. 66, pp. 67–83 (1983).

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[11] Coplen, T.B.; Hopple, J.A.; Böhlke, J.K.; Peiser, H.S.; Rieder, S.E.; Krouse, H.R.; Rosman, K.J.R.; Ding, T.; Vocke, Jr., R.D.; Révész, K.M.; Lamberty, A.; Taylor, P.; De Bièvre, P; *Compilation of Minimum and Maximum Isotope Ratios of Selected Elements in Naturally Occurring Terrestrial Materials and Reagents*; U.S. Geological Survey Water-Resources Investigations Report 01-4222, p. 98 (2001); available at http://pubs.usgs.gov/wri/wri014222/pdf/wri01-4222.pdf (accessed Mar 2013).

[12] *Light Stable Isotopic Materials (gas, liquid and solid forms)*; NIST SRM Order Request System; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD 20899; available at https://www-s.nist.gov/srmors/viewTableV.cfm?tableid=42 (accessed Mar 2013).


**Report Revision History:** 09 April 2013 (Reference value updated and uncertainty changed to expanded uncertainty for $\delta^{13}C_{VPDB\text{-}LSVEC}$; expiration date assigned; editorial changes); 22 June 1992 (Original report date).

*Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Group: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.*