



Report of Investigation

Reference Material 8540

IAEA-CH-7

(Carbon and Hydrogen Isotopes in Polyethylene Foil)

This Reference Material (RM) is intended for use in developing and validating methods for measuring relative differences in carbon (C) isotope-number ratios, $R(^{13}\text{C}/^{12}\text{C})$, and hydrogen (H) isotope-number ratios, $R(^2\text{H}/^1\text{H})$ [1]. Even though the values for this RM are reference values 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 IAEA-CH-7. This RM was formerly also called PEF-1 (PEF – Polyethylene Foil). A unit of RM 8540 consists of approximately 3.5 g of polyethylene foil.

Table 1. Reference Values^(a) and Expanded Uncertainties for the Relative C and H Isotope-Number Ratio Differences of RM 8540

RM Number	Name ^(b)	Reference Value	Expanded Uncertainty	Reference Value	Expanded Uncertainty
		$10^3 \delta^{13}\text{C}_{\text{VPDB-LSVEC}}^{(c)}$	$10^3 \delta^{13}\text{C}_{\text{VPDB-LSVEC}}^{(c)}$	$10^3 \delta^2\text{H}_{\text{VSMOW-SLAP}}^{(d)}$	$10^3 \delta^2\text{H}_{\text{VSMOW-SLAP}}^{(d)}$
8540	IAEA-CH-7	-32.15	±0.10	-100.3	±2.2

^(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) IAEA-CH-7 is also formerly known as PEF-1.

^(c) The $\delta^{13}\text{C}$ 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 ISO 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. Uncertainty in the bias of the methods is not included. The coverage factor, $k = 2$, provides an expanded uncertainty interval that has about a 95 % probability of encompassing the consensus mean. The $\delta^{13}\text{C}$ value and expanded uncertainty are taken from Table S-4 (Supporting Info) [4]. (VPDB – Vienna Pee Dee belemnite; LSVEC – Li Svec [named for H. Svec, formerly of Ames Laboratory, Iowa])

^(d) The $\delta^2\text{H}$ value is expressed as a mean and an expanded uncertainty. The expanded uncertainty is equal to $U = ku_c$, where u_c is the combined standard uncertainty as defined by the ISO Guide [3] and k is the coverage factor. The combined standard uncertainty is intended to represent, at the level of one standard deviation, the effect of random errors on the reference value that were evaluated by statistical means (Type A). Any uncertainty due to biases in the methods is not included in the expanded uncertainty. The coverage factor for the $\delta^2\text{H}$ value, $k = 2.571$ ($n=6$), provides an expanded uncertainty interval that has about a 95 % probability of encompassing the mean. The $\delta^2\text{H}$ value and uncertainty are taken from data in reference 5 (VSMOW – Vienna Standard Mean Ocean Water; SLAP-Standard Light Antarctic Precipitation).

Expiration of Reference Values: RM 8540 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 Handling and Use). 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.

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Support aspects involved in the issuance of this RM were coordinated through the NIST 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, $R(^{13}\text{C}/^{12}\text{C})_{\text{P}} = [N(^{13}\text{C})_{\text{P}} / N(^{12}\text{C})_{\text{P}}]$, are reported as $\delta^{13}\text{C}$ values [6]. The differences in measured isotope-number ratios of stable hydrogen isotopes in substance P, $R(^2\text{H}/^1\text{H})_{\text{P}} = [N(^2\text{H})_{\text{P}} / N(^1\text{H})_{\text{P}}]$, are reported as $\delta^2\text{H}$ values [6]. The relative differences in isotope-number ratios for carbon are referenced to VPDB while those for hydrogen are referenced to VSMOW, where:

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

$$\delta^2\text{H} = [R(^2\text{H}/^1\text{H})_{\text{sample}} / R(^2\text{H}/^1\text{H})_{\text{VSMOW-SLAP}}] - 1$$

VPDB-LSVEC refers to the Vienna PDB-LSVEC scale which is defined by assigning a $\delta^{13}\text{C}$ value of +1.95 ‰ to NBS 19 (RM 8544) and a consensus value of -46.6 ‰ to LSVEC (RM 8545) [7] for the purpose of normalizing stable carbon isotopic measurements (see *Normalization* [6,7]). The symbol ‰ is part per thousand and is equal to 0.001.

VSMOW-SLAP refers to the Vienna SMOW-SLAP scale which is defined by assigning a $\delta^2\text{H}$ value of 0 to VSMOW2 (RM 8535a) and a value of -428 ‰ to measurements of SLAP (RM 8537) or -427.5 ‰ to measurements of SLAP2 (RM 8537a) [9] for the purpose of normalizing stable hydrogen isotope measurements (see *Normalization* [8,9]). The symbol ‰ is part per thousand and is equal to 0.001.

INSTRUCTIONS FOR HANDLING AND STORAGE

Handling and Storage: RM 8540 (IAEA-CH-7) is stable at normal room temperatures.

Distribution: The distribution of RM 8540 (IAEA-CH-7) is limited to one unit per customer per three-year period of time.

PREPARATION AND ANALYSIS

Sample Preparation: RM 8540 (IAEA-CH-7) was prepared by H. Gerstenberger and M. Herrmann, Zentralinstitut für Isotopen und Strahlenforschung, Leipzig, Germany [10,11].

Analytical Methods: The $\delta^{13}\text{C}$ 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.* [12] were combined using a multivariate Bayesian approach for data reduction [4].

The $\delta^2\text{H}$ values were measured by six different laboratories, normalized to yield a value of -428 ‰ for RM 8537 (SLAP) and reported at the 1993 IAEA Consultants' Group Meeting on Stable Isotope Reference Samples [5].

The $\delta^{13}\text{C}$ and $\delta^2\text{H}$ values and expanded uncertainties reported in Table 1 for RM 8540 (IAEA-CH-7) are the values accepted by the Commission on Isotopic Abundances and Atomic Weights of the International Union of Pure and Applied Chemistry (IUPAC) (<http://ciaaw.org/Carbon.htm> and <http://ciaaw.org/Hydrogen.htm>, respectively) for this RM as of the date of this report.

Isotopic Homogeneity: Data from the inter-laboratory comparisons of IAEA-CH-7 suggest that there is no evidence of carbon or hydrogen isotopic heterogeneity.

Normalization: The $\delta^{13}\text{C}$ values in samples should be normalized to the VPTB-LSVEC δ -scale by calibrating the measurement with respect to the δ -value for NBS 19 (RM 8544) and the δ -value for LSVEC (RM 8545), the ^{13}C -depleted anchor RM [6,7]. A general formula for normalizing measured carbon isotope-number ratios using two laboratory standards LS1 (NBS 19) and LS2 (LSVEC) can be expressed as:

$$\delta^{13}\text{C}_{\text{sample, cal}} = \delta^{13}\text{C}_{\text{LS1, cal}} + (\delta^{13}\text{C}_{\text{sample, WS}} - \delta^{13}\text{C}_{\text{LS1, WS}}) \times f \quad (1)$$

where the normalization factor f is:

$$f = \frac{(\delta^{13}\text{C}_{\text{LS2, cal}} - \delta^{13}\text{C}_{\text{LS1, cal}})}{(\delta^{13}\text{C}_{\text{LS2, WS}} - \delta^{13}\text{C}_{\text{LS1, WS}})} \quad (2)$$

Note: In the formulae above, cal denotes calibrated measurements made versus the VPDB scale, and $\delta^{13}\text{C}_{\text{LS1, cal}}$ and $\delta^{13}\text{C}_{\text{LS2, cal}}$ are the conventionally fixed $\delta^{13}\text{C}$ values for NBS 19 and LSVEC. WS denotes measurements made versus a transfer gas (working standard), and $\delta^{13}\text{C}_{\text{LS1, WS}}$ and $\delta^{13}\text{C}_{\text{LS2, WS}}$ are the $\delta^{13}\text{C}$ values for calibrated laboratory working standards.

Similar formulae are used for normalizing $\delta^2\text{H}$ values in samples to the VSMOW-SLAP scale where LS1 is VSMOW2 and LS2 is SLAP or SLAP2 [8,9].

Please note that the reporting scale for $\delta^2\text{H}$ is still denoted and referred to as the VSMOW-SLAP scale in spite of the exhaustion of the original supply of VSMOW and SLAP [13]. Of course, the combined standard uncertainties of VSMOW2 and SLAP2 isotopic values have to be included in any uncertainty budget.

Reporting of Stable Isotope δ values: The following recommendations from IUPAC are provided for reporting $\delta^{13}\text{C}$ and $\delta^2\text{H}$ values [6-9]. It is recommended that:

- $\delta^{13}\text{C}$ 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 $+1.95\text{‰}$;
- $\delta^2\text{H}$ values of all hydrogen-bearing substances be expressed relative to VSMOW-SLAP on a scale where SLAP or SLAP2 has a consensus value of -428‰ or -427.5‰ , respectively and VSMOW2 has a value of 0;
- Authors should clearly state that their data have been normalized.

In addition, researchers are encouraged to report the isotopic compositions of RM 8539 (NBS22) and other internationally distributed carbon or hydrogen isotopic reference materials [14] 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 RMs mentioned in this report are available on the SRM web site [15].

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<p>Report Revision History: 06 February 2013 (Reference values revised and uncertainties changed to expanded uncertainties for $\delta^{13}\text{C}_{\text{VPDB-LSVEC}}$ and $\delta^2\text{H}_{\text{VSMOW-SLAP}}$; expiration date assigned; editorial changes); 22 June 1992 (Original report issue date).</p>
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Users of this RM should ensure that the Report of Investigation in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.