United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials USGS67, USGS68, and USGS69

(Hydrogen and Carbon Isotopes in *n*-Hexadecanes)

These reference materials (RMs) are intended for normalization of stable hydrogen (δ^2H) and carbon (δ^{13}C) measurements of unknown *n*-hexadecanes ($C_{16}H_{34}$) and similarly-behaving hydrogen- and carbon-bearing substances. A unit consists of 50 µL of the RM in a sealed glass tube. There is no limit on distribution. These RMs were prepared by A. Schimmelmann (Indiana University, Bloomington, Indiana).

Recommended values: Stable hydrogen isotopic compositions are expressed herein as delta values [1] relative to VSMOW (Vienna Standard Mean Ocean Water) on a scale normalized such that the δ^2H value of SLAP (Standard Light Antarctic Precipitation) is –428 ‰ [2,3]. Stable carbon isotopic compositions are expressed herein as delta values relative to VPDB (Vienna Peedee belemnite) on a scale normalized such that the δ^{13}C values of NBS 19 calcium carbonate and LSVEC lithium carbonate are +1.95 ‰ and –46.6 ‰, respectively [4]. Stable hydrogen- and carbon-isotope delta values of USGS67, USGS68, and USGS69 *n*-hexadecanes with combined standard uncertainties are:

Reference	$\delta^2H_{VSMOW\text{-}SLAP}$	$\delta^{13}C_{VPDB\text{-}LSVEC}$	Data source
USGS67	–166.2 ± 1.0 ‰	–34.50 ± 0.05 ‰	[5]
USGS68	–10.2 ± 0.9 ‰	–10.55 ± 0.04 ‰	[5]
USGS69	+381.4 ± 3.5 ‰	–0.57 ± 0.04 ‰	[5]

Technical coordination for this RM was provided by Arndt Schimmelmann of Indiana University and Haiping Qi of the U.S. Geological Survey Reston Stable Isotope Laboratory (RSIL).

Reston, Virginia 20192
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Tyler B. Coplen, Director
Reston Stable Isotope Laboratory

Expiration of Reference Value: The reference values for the isotopic compositions of USGS67, USGS68, and USGS69 are valid until December 31, 2036, provided these RMs are handled in accordance with the instructions given in this Report of Stable Isotopic Composition (see "Instructions for Use"). A reference value is nullified if the RM is damaged, contaminated, or otherwise modified.

Source of the RM: The following description is taken from Schimmelmann and others [5]. Two batches of 125 and 175 mL from Fluka Analytical (CAS # 544-76-3) with GC-based purities of at least 99.9 % and two isotopically enriched *n*-hexadecanes from Aldrich ($C_{16}{}^{2}HH_{33}$, ^{2}H fraction = 98 %; *n*-hexadecane-1,2-$^{13}C_2$, ^{13}C fraction = 99 %) were used in the preparation of 3 *n*-hexadecanes with different $\delta^{2}H$ and $\delta^{13}C$ values. Each stirred, homogeneous liquid was subdivided into glass containers and flame-sealed under argon. Users will receive aliquots of 50 µL sealed in glass tubes as RMs for $\delta^{2}H$ and $\delta^{13}C$ normalization.

Maintenance of RM Report of Isotopic Composition: The U.S. Geological Survey RSIL will monitor these RMs and will notify the purchaser if substantive technical changes occur that affect their isotopic compositions.

Distribution and Stability: A distribution unit is available in amounts of 50 µL sealed in glass tubes. USGS67, USGS68, and USGS69 are chemically stable at normal room temperatures.

Instructions for Use: Users may choose to mix a gravimetrically or volumetrically defined aliquot of the contents of a glass tube with a suitable pure organic solvent (e.g., dichloromethane or hexane) and use the stock solution for injections into gas chromatographic interfaces. Please note that *n*-hexadecane has a non-negligible vapor pressure at room temperature. Long-term exposure to air and partial evaporation may isotopically fractionate the RM. It is recommended that *n*-hexadecane and its stock solutions be stored in tightly closed glass containers in the dark in a refrigerator or freezer.

Reporting of stable-isotope-delta values: The following recommendations are provided for reporting stable hydrogen and carbon isotope-delta values. It is recommended that:

- The $\delta^{2}H$ values of all hydrogen-bearing substances be expressed relative to VSMOW-SLAP on a scale where the $\delta^{2}H$ of SLAP = –428 ‰ exactly [3].
- The $\delta^{13}C$ values of all carbon-bearing substances be expressed relative to VPDB-LSVEC on a scale such that the $\delta^{13}C$ values of NBS 19 calcium carbonate and LSVEC lithium carbonate are +1.95 ‰ and –46.6 ‰, respectively [4], even though LSVEC is no longer recommended as a RM for $\delta^{13}C$ measurement [6].
- Authors report delta values of international distributed (secondary) isotopic reference materials as though they had been interspersed among and used for normalization of unknowns, as appropriate for the measurement method. In this manner, measurement results can be adjusted in the future as analytical methods improve and consensus values of internationally distributed isotopic reference materials change.
- Reporting of delta values relative to SMOW and PDB (Peedee belemnite) be discontinued [7].

REFERENCES

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