United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials NBS 22a and USGS78

(Hydrogen and Carbon Isotopes in Vacuum Pump Oils)

These reference materials (RMs) are intended for normalization of stable hydrogen (δ^2H) and carbon (δ^{13}C) measurements of unknown oils and similarly behaving hydrogen- and carbon-bearing substances. A unit consists of 1 mL in a sealed glass ampule. There is no limit on distribution. These RMs are also available in silver tubes. This RM was 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]. The stable hydrogen- and carbon-isotope delta values of NBS 22a and USGS78 vacuum pump oils with combined standard uncertainties are:

Reference	$\delta^2H_{VSMOW\text{-}SLAP}$	$\delta^{13}C_{VPDB\text{-}LSVEC}$	Data source
NBS 22a	–120.4 ± 1.0	–29.72 ± 0.04 ‰	[5]
USGS78	+397.0 ± 2.2	–29.72 ± 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).

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Expiration of Reference Value: The reference values for the isotopic compositions of NBS 22a and USGS78 are valid until December 31, 2036, provided the 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 by freezing or other means, contaminated, or otherwise modified.

Source of the RM: The following description is taken from Schimmelmann and others [5]. Nineteen liters of Fisher Scientific Maxima C Plus Vacuum Pump Oil, representing a triple-distilled hydrocarbon fraction that had been hydrogenated to reduce the abundance of aromatic components with exchangeable hydrogen and to increase chemical stability, was purchased. The vapor pressure at 25 °C of 0.133 mPa guarantees the absence of volatile components. A volume of 4.3 L (i.e. 3.35 kg) of vacuum oil was enriched in ^{2}H by heating it to 60 °C and dissolving 250 mg of perdeuterated *n*-tetracosane ($C_{24}{}^{2}H_{50}$, ^{2}H fraction = 99.1 %, MSD Isotopes) to yield a homogeneous solution. Users will receive aliquots of 1 mL sealed in glass ampules for use as RMs for $\delta^{2}H$ and $\delta^{13}C$ normalization.

Maintenance of RM Report of Stable 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 1 mL in a sealed glass ampule. These RMs are stable at normal room temperatures when stored under dry and dark conditions. These RMs are also available in crimp-sealed segments of silver tubes [6] for use in EA applications (see http://isotopes.usgs.gov/lab/referencematerials.html).

Instructions for use: NBS 22a and USGS78 can be interspersed among every 10–15 unknowns. They can be used with other hydrogen or nitrogen isotopic RMs. After opening a glass ampoule, the oil should be transferred into a suitable secondary glass container with a tight fitting cap or stopper. Do not use a rubber or cork stopper. The use of cap liners or septa in secondary glass containers should limit the contact of oils with polymers because monomers and oligomers can leach out of the polymer and contaminate the oils. Dark storage at ambient temperature is recommended.

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 [7].
- 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 [8].

REFERENCES

[1] Coplen, T. B., 2011, Guidelines and recommended terms for expression of stable-isotope-ratio and gas-ratio measurement results: Rapid Communications in Mass Spectrometry, v. 25(17), p. 2538–2560. https://doi.org/10.1002/rcm.5129

[2] Gonfiantini, R., 1978, Standards for stable isotope measurements in natural compounds: Nature, v. 271, p. 534–536. https://doi.org/10.1038/271534a0

[3] Coplen, T. B., 1994, Reporting of stable hydrogen, carbon, and oxygen isotopic abundances: Pure and Applied Chemistry, v. 66(2), p. 273–276. https://publications.iupac.org/pac/pdf/1994/pdf/6602x0273.pdf

[4] Coplen, T. B., Brand, W. A., Gehre, M., Gröning, M., Meijer, H. A. J., Toman, B., and Verkouteren, R. M., 2006, New guidelines for $\delta^{13}C$ measurements: Analytical Chemistry, v. 78(7), p. 2439–2441. https://doi.org/10.1021/ac052027c

[5] Schimmelmann, A., Qi, H., Coplen, T. B., Brand, W. A., Fong, J., Meier-Augenstein, W., Kemp, H. F., Toman, B., Ackermann, A., Assonov, S., Aerts-Bijma, A. T., Brejcha, R., Chikaraishi, Y., Darwish, T., Elsner, M., Gehre, M., Geilmann, H., Gröning, M., Hélie, J-F., Herrero-Martín, S., Meijer, H. A. J., Sauer, P. E., Sessions, A. L., and Werner, R. A., 2016, New organic reference materials for hydrogen, carbon, and nitrogen stable isotope-ratio measurements: caffeines, *n*-alkanes, fatty acid methyl esters, glycines, L-valines, polyethylenes, and oils, Analytical Chemistry, v. 88(8), p. 4294–4302. https://doi.org/10.1021/acs.analchem.5b04392

[6] Qi, H., Gröning, M., Coplen, T. B., Buck, B., Mroczkowski, S. J., Brand, W. A., Geilmann, H., and Gehre, M., 2010, Novel silver-tubing method for quantitative introduction of water into high-temperature conversion systems for stable hydrogen and oxygen isotopic measurements: Rapid Communications in Mass Spectrometry, v. 24(13), p. 1821–1827. https://doi.org/10.1002/rcm.4559

[7] Assonov, S., 2018, Summary and recommendations from the International Atomic Energy Agency Technical Meeting on the development of stable isotope reference products (21-25 November 2016): Rapid Communication in Mass Spectrometry, v. 32(10), p. 827−830. https://doi.org/10.1002/rcm.8102

[8] Coplen, T. B., 1995, Discontinuance of SMOW and PDB: Nature, v. 375, p. 285. https://doi.org/10.1038/375285a0