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Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials USGS84-0.15 µL and USGS84-0.25 µL

(Hydrogen, Carbon, and Oxygen Isotopes in Sicilian Olive Oil that is Crimp-sealed in Silver Tube Segments)

This reference material (RM) is intended for normalization of stable hydrogen (δ^2H), carbon (δ^{13}C), and oxygen (δ^{18}O) isotope measurements of unknown vegetable oil and similarly-behaving hydrogen-, carbon-, and oxygen-bearing substances. These RMs consist of 0.15 µL or 0.25 µL of USGS84 [1] sealed in a silver tube [2]. These RMs are issued in quantities of 50 sealed silver tubes per bottle. There is no limit on distribution. USGS84 is also available from the Reston Stable Isotope Laboratory in units of 1 mL in a 2-mL glass ampule that is flame-sealed under argon [1]. This RM is not safe for human consumption and is strictly intended for laboratory use only.

Recommended Values: Stable hydrogen and oxygen isotopic compositions are expressed herein as delta values [3] relative to VSMOW (Vienna Standard Mean Ocean Water) on scales normalized such that the δ^2H and δ^{18}O values of SLAP (Standard Light Antarctic Precipitation) are –428 ‰ and –55.5 ‰, respectively [4,5,6]. 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 [7]. Expanded measurement uncertainties at the 95 % confidence level are provided, and the coverage factors, k, used were (i) $k = 4$ for $\delta^{13}C_{VPDB\text{-}LSVEC}$ measurements, (ii) $k = 6$ for $\delta^{18}O_{VSMOW\text{-}SLAP}$ measurements, and (iii) $k = 9$ for $\delta^{2}H_{VSMOW\text{-}SLAP}$ measurements. Stable hydrogen-, carbon-, and oxygen-isotope delta values of USGS84 vegetable oil with combined expanded uncertainties are given below. The hydrogen- and oxygen-isotope data were obtained by measurements of USGS84 sealed in silver tubes [1,2].

Reference	$\delta^{2}H_{VSMOW\text{-}SLAP}$	$\delta^{13}C_{VPDB\text{-}LSVEC}$	$\delta^{18}O_{VSMOW\text{-}SLAP}$	Data source
USGS84 Sicilian olive oil	–140.4 ± 3.1 ‰	–28.80 ± 0.09 ‰	+26.36 ± 0.50 ‰	[1]

Technical coordination for these RMs 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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Information Values: Hydrogen-, carbon-, and oxygen-mass fractions are provided as information values. These values were obtained by measurements of USGS84 sealed in silver tubes [1,2]. Uncertainties are standard deviations.

Reference	Element	Mass fraction	Data source
USGS84 Sicilian olive oil	hydrogen	0.1178 ± 0.0023 (n = 6)	[1]
	carbon	0.8024 ± 0.0117 (n = 9)	[1]
	oxygen	0.1093 ± 0.0031 (n = 15)	[1]

Nominal Volume of Vegetable Oil: 0.15 µL or 0.25 µL (Although the RSIL attempts to ensure that each silver tube has the same volume of vegetable oil, slight differences are observed owing to variations of the inside diameter of the silver tubing provided by the manufacturer. The typical relative variation in volume among 50 tubes is ±5 %, but this cannot be guaranteed.)

Expiration of Reference Values: The reference values for the isotopic compositions of USGS84-0.15 µL and USGS84-0.25 µL are valid for a period of two years from receipt of the RM as long as the RM is stored at 4 °C in a refrigerator and provided the RM is 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.

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

Distribution and Stability: Crimp-sealed silver tube segments must not be frozen to protect the integrity of the crimp-seal, but they can be stored at 4 °C in a refrigerator to maximize their lifespan. We estimate that the shelf life of liquid food matrix RMs in silver tubes in refrigerators above freezing is two years. To minimize the potential for contamination, it is recommended that this RM be stored in the container in which it is supplied. The RM container should be sealed well after use to minimize tarnishing of the silver tubes.

Instructions for Use: USGS84 can be used at the beginning, the middle, and the end of the analysis sequence to enable satisfactory correction of drift with time. The amount of hydrogen, carbon or oxygen in references and unknowns should be the same or similar to minimize bias in measurement results. Two or three silver tubes containing USGS84 can be combined in a single port of a TC/EA carousel to increase the size of the sample. Although the RSIL attempts to ensure that each silver tube has the same volume of vegetable oil, slight differences are observed owing to variations of the inside diameter of the silver tubing provided by the manufacturer. Therefore, the volume of 0.15 µL or 0.25 µL should not be used for quantifying the elemental mass amount in unknown samples. For precise elemental mass amount quantification, analyzing reference material with precise weighing is necessary.

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 δ^2H and δ^{18}O values of vegetable oil and similar hydrogen- and oxygen-bearing materials be expressed relative to VSMOW-SLAP on a scale where $\delta^2H_{SLAP} = -428$ ‰ exactly or $\delta^2H_{SLAP2} = -427.5$ ‰ [5,8].
- The δ^{13}C values of all carbon-bearing substances be expressed relative to VPDB-LSVEC on a scale such that the δ^{13}C values of NBS 19 calcium carbonate and LSVEC lithium carbonate are +1.95 ‰ and –46.6 ‰, respectively [5,7], even though LSVEC is no longer recommended as a RM for δ^{13}C measurement [9].
- Authors of new publications 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 PDB (Peedee belemnite) be discontinued [10].

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