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United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials USGS84, USGS85, USGS86, and USGS87

(Hydrogen, Carbon, and Oxygen Isotopes in Vegetable Oils)

These reference materials (RMs) are 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. A unit consists of 1 mL in a 2-mL glass ampule that is flame-sealed under argon. There is no limit on distribution. These RMs were prepared by A. Schimmelmann (Indiana University, Bloomington, Indiana). These RMs are not safe for human consumption and are strictly intended for laboratory use only.

Recommended values: Stable hydrogen and oxygen isotopic compositions are expressed herein as delta values [1] 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 [2,3,4]. 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 [5]. 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 through USGS87 vegetable oils with combined expanded uncertainties are given below. The hydrogen- and oxygen-isotope data were obtained by measurements of USGS84, USGS85, USGS86, and USGS87 sealed in silver tubes [6,7].

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 ‰	[7]
USGS85 Peruvian olive oil	–158.6 ± 2.7 ‰	–29.74 ± 0.08 ‰	+22.00 ± 0.60 ‰	[7]
USGS86 Tropical Vietnamese peanut oil	–207.4 ± 4.5 ‰	–30.63 ± 0.09 ‰	+18.76 ± 1.03 ‰	[7]
USGS87 United States corn oil	–168.1 ± 2.7 ‰	–15.51 ± 0.09 ‰	+20.11 ± 0.85 ‰	[7]

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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, USGS85, USGS86, and USGS87 sealed in silver tubes [6,7]. Uncertainties are standard deviations.

Reference	Element	Mass fraction	Data source
USGS84 Sicilian olive oil	hydrogen	0.1178 ± 0.0023 (n = 6)	[7]
	carbon	0.8024 ± 0.0117 (n = 9)	[7]
	oxygen	0.1093 ± 0.0031 (n = 15)	[7]
USGS85 Peruvian olive oil	hydrogen	0.1184 ± 0.0004 (n = 6)	[7]
	carbon	0.7949 ± 0.0117 (n = 10)	[7]
	oxygen	0.1098 ± 0.0033 (n = 14)	[7]
USGS86 Tropical Vietnamese peanut oil	hydrogen	0.1178 ± 0.0008 (n = 6)	[7]
	carbon	0.7916 ± 0.0081 (n = 10)	[7]
	oxygen	0.1130 ± 0.0046 (n = 10)	[7]
USGS87 United States corn oil	hydrogen	0.1152 ± 0.0016 (n = 5)	[7]
	carbon	0.7990 ± 0.0075 (n = 10)	[7]
	oxygen	0.1119 ± 0.0059 (n = 10)	[7]

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).

Expiration of Reference Value: The reference values for the isotopic compositions of USGS84,USGS85, USGS86, and USGS87 are valid until December 31, 2026, provided these RMs are stored in a freezer upon receipt and 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: This information is taken from Schimmelmann and others [7]. Olive oil (USGS84) produced from C3 plants in Sicily, Italy (37°45'N, 14°15'E), was obtained from the 2018 harvest. In February 2019, it was filtered through Whatman #1 cellulose filters at room temperature and then stored in a refrigerator until it was sealed in glass bulbs under vacuum in May and June 2019. Temporary storage in a refrigerator did not result in partial solidification and loss of homogeneity.

Olive oil from Peru (USGS85) originated from the March 2018 harvest on the family-operated *Chacra Blanca* farm on the desert coast of the Chilca Valley, 60 km south of Lima (12°30′S 76°45′W; http://www.olivico.com/ingles/aboutUs.html). Irrigation water for the farm relies on artesian wells tapping an aquifer that is replenished by precipitation in the high Andes. The 5 L of "Olivico" oil were filtered through Whatman #1 cellulose filters at room temperature in January 2019, followed by sealing in glass bulbs under vacuum.

Peanut oil (C3 plant; USGS86) from the Quảng Bình Province in central Vietnam (17°30′N 106°20′E) was freshly squeezed from the 2018 autumn harvest and was supplied in October 2018 by Mrs. Nguyen Thi Hong Thinh at the Institute for Nuclear Science and Technology, Hanoi. After being expedited to Indiana University, it was filtered through Whatman #1 cellulose filters at room temperature in November 2018, followed by sealing in glass bulbs under vacuum.

Corn oil (C4 plant; USGS87) was obtained from the germ of *Zea mays*. The commercially distributed oil was purchased in Bloomington (Indiana, USA) as 5.6 L of "*carlini pure corn oil*" from a retail outlet and filtered through Whatman #1 cellulose filters at room temperature in December 2018, followed by sealing in glass bulbs under vacuum.

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 1 mL in a 2-mL glass ampule that has been flame-sealed under argon. The shelf life of unopened glass ampules of USGS84, USGS85, USGS86, and USGS87 stored in a freezer is five years. After opening of an ampule and exposing the vegetable oil to atmospheric oxygen and moisture, the remaining shelf life is two years as long as the vegetable oil is shielded from prolonged exchange with atmospheric moisture and kept in a freezer.

Instructions for Use: USGS84, USGS85, USGS86, and USGS87 can be interspersed among every 10–15 unknowns. After opening of any container of food matrix RMs, the remaining shelf life greatly depends on the handling and further storage of the material. For example, after opening a glass ampoule it is necessary to expeditiously transfer the liquid RM into another suitable container that can be closed tightly and stored at low temperature. In any case, exposure to atmospheric oxygen decreases the shelf life of RMs.

Vegetable oils sealed in silver tubing are also available from the USGS, and they offer several advantages because (i) silver metal has natural antimicrobial properties, (ii) samples are shielded from light and air, and (iii) uncertainties in weighing errors and potential contamination and fractionation by the end user are eliminated. Each crimp-sealed segment of silver tube contains vegetable oil with a bulk amount of hydrogen equivalent to the amount of hydrogen in 0.15 µL or 0.25 µL of reference water (available from the USGS).

Reporting of Stable-isotope-delta Values: The following recommendations are provided for reporting stable hydrogen-, carbon-, and oxygen-isotope data. It is recommended that:

- The δ^2H and δ^{18}O values of vegetable oils and similar hydrogen- and oxygen-bearing materials be expressed relative to VSMOW-SLAP on a scale where δ^2H_{SLAP} = –428 ‰ exactly or δ^2H_{SLAP2} = –427.5 ‰ [3,8].
- 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 [3,5], 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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