United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials GFLES-1, GFLES-2, GFLES-3, and GFLES-4

(Hydrogen and Oxygen Isotopes in Water)

These reference materials (RMs) are intended for normalization of stable hydrogen-isotope (δ^2H) and oxygen-isotope (δ^{18}O and δ^{17}O) measurements of waters with an isotope-ratio mass spectrometer or a laser absorption spectrometer [1]. A unit of these RMs consists of one autoclaved glass ampoule containing 5 mL of water. A set of one each of GFLES-1, GFLES-2, GFLES-3, and GFLES-4 reference waters, for a total of four glass ampoules, is also available (20 mL total).

Recommended Values: Stable hydrogen and oxygen isotopic compositions are expressed herein as delta values [2] relative to VSMOW (Standard Mean Ocean Water) on scales normalized such that the δ^2H, δ^{18}O, and δ^{17}O values of SLAP (Standard Light Antarctic Precipitation) are –428 ‰, –55.5 ‰, and –29.70 ‰, respectively [3–6]. Each stable isotopic composition is given as a reference isotope-delta value with an estimated combined uncertainty (1 σ) about the reference value that provides an interval that has about a 68-percent probability of encompassing the true value.

Name	$\delta^2H_{VSMOW\text{-}SLAP}$	$\delta^{18}O_{VSMOW\text{-}SLAP}$	$\delta^{17}O_{VSMOW\text{-}SLAP}$
GFLES-1	+80.1 ± 0.4 ‰	–6.25 ± 0.04 ‰	–3.32 ± 0.40 ‰
GFLES-2	+159.9 ± 0.4 ‰	–6.21 ± 0.04 ‰	–3.30 ± 0.40 ‰
GFLES-3	+280.2 ± 0.4 ‰	–6.14 ± 0.04 ‰	–3.28 ± 0.40 ‰
GFLES-4	+399.8 ± 0.4 ‰	–6.08 ± 0.04 ‰	–3.25 ± 0.40 ‰

Technical coordination for this RM was provided by Haiping Qi of the Reston Stable Isotope Laboratory (RSIL).

Reston, Virginia 20192
May 20, 2020

Tyler B. Coplen, Director
Reston Stable Isotope Laboratory

Expiration of Reference Value: The reference values for the isotopic compositions of GFLES-1, GFLES-2, GFLES-3, and GFLES-4 are valid until December 31, 2029, 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 glass ampoule containing the RM is inadvertently broken.

Source of the RM: These four reference waters were prepared by gravimetrical mixing of International Atomic Energy Agency (IAEA) reference water IAEA-606 (δ^2H = 15993.6 ± 1.0 ‰) with a well-characterised natural water (δ^2H = –42.7 ± 0.4 ‰) by B. Verstappen-Dumoulin under the direction of Prof. H. A. J. Meijer (Centrum voor IsotopenOnderzoek (CIO), University of Groningen, The Netherlands) [7]. The mixing process was conducted along the lines described in Faghihi et al. [7] in which the same batch of natural water was used. Their uncertainty is inherited from the uncertainty in the natural water, with only negligible influence by the uncertainty in the water enriched in ^{2}H. Each reference water was homogenized at the RSIL, loaded into 5-mL glass ampoules, and sealed with a torch. Each ampoule was inverted and autoclaved at about 120 °C for a minimum of 20 minutes to terminate biological activity. Each ampoule was weighed to identify and eliminate any leaking ampoules.

Stability: GFLES-1, GFLES-2, GFLES-3, and GFLES-4 are stable at normal room temperatures. The reference values in this Report of Investigation apply only to freshly opened ampoules.

Instructions for Use: This RM is intended for calibration of instrumentation and for calibrating unknown waters by interspersing aliquots of the RM among water sample unknowns. The unused fraction of this RM should be discarded after opening an ampoule due to the strong possibility of evaporative losses causing significant isotopic fractionation.

Reporting of Stable-isotope-delta Values: The following recommendations are provided for reporting stable hydrogen and oxygen isotope-delta values [4]. It is recommended that:

- The δ^2H values of all hydrogen-bearing substances be expressed relative to VSMOW-SLAP on a scale where δ^2H_{SLAP} = –428 ‰ exactly or δ^2H_{SLAP2} = –427.5 ‰ [8].
- The δ^{18}O values of all oxygen-bearing substances be expressed relative to VSMOW-SLAP or relative to Vienna Peedee belemnite (VPDB; for carbonates) on a scale such that the δ^{18}O of SLAP = –55.5 ‰ relative to VSMOW, and for carbonates, such that δ^{18}O of NBS 19 calcium carbonate = –2.2 ‰.
- Authors report δ 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 δ values relative to SMOW and PDB (Peedee belemnite) be discontinued [9].

REFERENCES

[1] Coplen, T. B., 1988, Normalization of oxygen and hydrogen isotope data: Chemical Geology (Isotope Geosciences Section), v. 72, p. 293–297, last accessed May 8, 2020 at https://doi.org/10.1016/0168-9622(88)90042-5).

[2] 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, 2538–2560, (last accessed May 8, 2020 at http://onlinelibrary.wiley.com/doi/10.1002/rcm.5129/abstract).

[3] Gonfiantini, R., 1978, Standards for stable isotope measurements in natural compounds: Nature, v. 271, p. 534–536, last accessed May 8, 2020 at https://doi.org/10.1038/271534a0).

[4] Coplen, T. B., 1994, Reporting of stable hydrogen, carbon, and oxygen isotopic abundances: Pure and Applied Chemistry, v. 66, p. 273–276, last accessed May 8, 2020 at https://doi.org/10.1016/0375-6505(95)00024-0).

[5] Meijer, H. A. J., and Li, W. J., 1998, The use of electrolysis for accurate $\delta^{17}O$ and $\delta^{18}O$ isotope measurements in water: Isotopes in Environmental and Health Studies, v. 34, 349–369, last accessed May 8, 2020 at https://doi.org/10.1080/10256019808234072).

[6] Schoenemann, S. W., Schauer, A. J., and Steig, E. J., 2013, Measurement of SLAP2 and GISP ^{17}O and proposed VSMOW-SLAP normalization for ^{17}O and $^{17}O_{excess}$: Rapid Communications in Mass Spectrometry, v. 27, 582–590, last accessed May 8, 2020 at https://doi.org/10.1002/rcm.6486).

[7] Faghihi, V., Verstappen-Dumoulin, B.M.A.A., Jansen, H.G., van Dijk, G., Aerts-Bijma, A.T., Kerstel, E.R.T., Groening, M., and Meijer, H.A.J., 2015, A new high-quality set of singly (^{2}H) and doubly (^{2}H and ^{18}O) stable isotope labeled reference waters for biomedical and other isotope-labeled research, Rapid Communications in Mass Spectrometry, v. 29, 311–321, last accessed May 8, 2020 at https://doi.org/10.1002/rcm.7108).

[8] International Atomic Energy Agency (IAEA), Reference Sheet for International Measurement Standards, last accessed May 8, 2020 at http://nucleus.iaea.org/rpst/Documents/VSMOW2_SLAP2.pdf).

[9] Coplen, T. B., 1995, Discontinuance of SMOW and PDB: Nature, v. 375, 285, last accessed May 8, 2020 at https://doi.org/10.1038/375285a0).