

United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials USGS32, USGS34, and USGS35

(Nitrogen and Oxygen Isotopes in Nitrate)

These reference materials (RMs) are intended for normalizing stable nitrogen ($\delta^{15}\text{N}$) and oxygen ($\delta^{18}\text{O}$) relative isotope-ratio measurements of unknown nitrate samples. A unit of USGS32, USGS34, and USGS35 consists of approximately 0.3 g. USGS32 KNO_3 was prepared by Böhlke et al. [1]. USGS34 KNO_3 and USGS35 NaNO_3 were prepared by Böhlke et al. [2]. USGS32, USGS34, and USGS35 also may be available from the International Atomic Energy Agency (IAEA) [3,4,5] or from the U.S. National Institute of Standards and Technology (NIST) [6], respectively, as RM 85658, RM 8568, and RM 8569.

Recommended values: Stable nitrogen isotopic compositions are expressed herein as delta values [7] relative to atmospheric nitrogen, which is isotopically homogenous [8]. On this scale, the $\delta^{15}\text{N}_{\text{AIR}}$ value of USGS32 KNO_3 has a consensus value of +180 ‰ exactly, and that of IAEA-NO-3 is +4.7 ‰ [9,10]. Stable oxygen isotopic compositions are expressed herein as delta values relative to VSMOW (Vienna Standard Mean Ocean Water) on a $\delta^{18}\text{O}$ scale normalized such that the $\delta^{18}\text{O}$ value of SLAP (Standard Light Antarctic Precipitation) is –55.5 ‰ [11]. Two $\delta^{18}\text{O}_{\text{VSMOW-SLAP}}$ values for each material are listed [2,12]. The uncertainties of Böhlke et al. [2] are the estimated one-sigma reproducibilities. The uncertainties of $\delta^{18}\text{O}_{\text{VSMOW-SLAP}}$ values of Brand et al. [12] are estimated combined uncertainties that have about a 68 % probability of encompassing the mean. $\Delta^{17}\text{O}_{\text{VSMOW}}$ values listed below are defined by $\Delta^{17}\text{O} = \delta^{17}\text{O} - 0.52 \times \delta^{18}\text{O}$ [13]; other definitions may yield slightly different values [2].

Description	$\delta^{15}\text{N}_{\text{AIR}}$	$\delta^{18}\text{O}_{\text{VSMOW-SLAP}}$	$\Delta^{17}\text{O}_{\text{VSMOW}}$	Data source
USGS32	+180 ‰ exactly	+25.7 ± 0.2 ‰	no data	[2,9]
		+25.4 ± 0.2 ‰		[12]
USGS34	–1.8 ± 0.1 ‰	–27.9 ± 0.3 ‰	–0.3 ± 0.2 ‰	[2]
		–27.78 ± 0.37 ‰		[12]
USGS35	+2.7 ± 0.1 ‰	+57.5 ± 0.3 ‰	+21.6 ± 0.2 ‰	[2,13]
		+56.81 ± 0.31 ‰		[12]

Technical coordination for this RM was provided by J.K. Böhlke, S. Mroczkowski, and H. Qi of the RSIL.

Reston, Virginia 20192
August 17, 2026

Tyler B. Coplen, Director
Reston Stable Isotope Laboratory

Maintenance of RM certification: The Reston Stable Isotope Laboratory (RSIL) will monitor these RMs. The RSIL will notify the purchaser if substantive technical changes are observed that affect the stable nitrogen or oxygen isotopic compositions over time.

Distribution and stability: Units of USGS32, USGS34, and USGS35 are supplied in glass vials that are vacuum sealed in plastic pouches. Nitrate salts should be kept in a dry environment, as they may attract water when exposed to air. There is no exchange of oxygen in nitrate with oxygen in water except in strong acid conditions, so wetting and drying these RMs should not affect their isotopic compositions unless other contaminants are present.

Instructions for use: These RMs can be dried in a low-temperature oven or under vacuum before being weighed for analysis. They can be used at the beginning, the middle, and the end of the analysis sequence to enable satisfactory correction of drift with time [14]. Commonly, two or three of the nitrate isotopic reference materials are used together in an analytical batch, depending on the ranges of $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ values in the batch [2,14]. **NOTE:** Because USGS35 has a large positive $\Delta^{17}\text{O}$ value, it can yield anomalous apparent $\delta^{15}\text{N}$ values when analyzed as N_2O if data are not adjusted appropriately [2,15]. In such cases, USGS34 and USGS32 can be used for $\delta^{15}\text{N}$ calibration of samples with normal mass-dependent oxygen-isotope variations.

Reporting of stable-isotope-delta values: The following recommendations are provided for reporting stable oxygen and nitrogen isotope-delta values:

- The $\delta^{18}\text{O}$ values of all oxygen-bearing substances should be expressed relative to VSMOW-SLAP on a scale where the $\delta^{18}\text{O}$ of SLAP = -55.5 ‰ exactly [11].
- The $\delta^{15}\text{N}$ values of all nitrogen-bearing substances should be expressed relative to atmospheric nitrogen gas (N_2) [8].
- Authors should report delta values of internationally distributed (secondary) isotopic reference materials that were assumed for normalization of data for samples of similar chemical composition, 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.

REFERENCES

- [1] Böhlke, J.K., Gwinn, C.J., Coplen, T. B., 1993, New reference materials for nitrogen-isotope-ratio measurements: Geostandards Newsletter, v. 17(1), p. 159–164. <https://doi.org/10.1111/j.1751-908X.1993.tb00131.x>
- [2] Böhlke, J.K., Mroczkowski, S. J., Coplen, T. B., 2003, Oxygen isotopes in nitrate: New reference materials for $18\text{O}:17\text{O}:16\text{O}$ measurements and observations on nitrate-water equilibration: Rapid Communications in Mass Spectrometry, v. 17(16), p. 1835–1846. <https://doi.org/10.1002/rcm.1123>
- [3] IAEA, Reference Products for Environment and Trade, GISP. <https://analytical-reference-materials.iaea.org/usgs32/>
- [4] IAEA, Reference Products for Environment and Trade, GISP. <https://analytical-reference-materials.iaea.org/usgs34/>

- [5] IAEA, Reference Products for Environment and Trade, GISP. <https://analytical-reference-materials.iaea.org/usgs35/>
- [6] National Institute of Standards and Technology (NIST), SRM. <https://www.nist.gov/srm>
- [7] 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>
- [8] Mariotti, A., 1983, Atmospheric nitrogen is a reliable standard for natural ^{15}N abundance measurements: Nature, v. 303, p. 685–687. <https://doi.org/10.1038/303685a0>
- [9] Böhlke, J.K., and Coplen, T.B., 1995, Interlaboratory comparison of reference materials for nitrogen-isotope-ratio measurements, Reference and intercomparison materials for stable isotopes of light elements, IAEA TECDOC 825: International Atomic Energy Agency, Vienna, p. 51-66. <https://inis.iaea.org/records/9rbw1-xwr71>
- [10] Brand, W. A., Coplen, T. B., Vogl, J., Rosner, M., and Prohaska, T., 2014, Assessment of international reference materials for stable isotope ratio analysis 2013: Pure and Applied Chemistry, v. 86(3), p. 425-467. <https://doi.org/10.1515/pac-2013-1023>
- [11] 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>
- [12] Brand, W. A., Coplen, T. B., Aerts-Bijma, A. T., Böhlke, J.K., Gehre, M., Geilmann, H., Gröning, M., Jansen, H. G., Meijer, H. A. J., Meijer, Mroczkowski, S. J., Qi, H., Soergel, K., Stuart-Williams, H., Weise, S. M., Werner, R. A., 2009, Comprehensive inter-laboratory calibration of reference materials for $\delta^{18}\text{O}$ versus VSMOW using various on-line high-temperature conversion techniques: Rapid Communications in Mass Spectrometry, v. 23(7), p. 999–1019. <https://doi.org/10.1002/rcm.3958>
- [13] Michalski, G., Savarino, J., Böhlke, J.K., and Thiemens, M.H., 2002, Determination of the total oxygen isotopic composition of nitrate and the calibration of a $\Delta^{17}\text{O}$ nitrate reference material: Analytical Chemistry, v. 74(19), p. 4989-4993. <https://doi.org/10.1021/ac0256282>
- [14] Coplen, T.B., Qi, Haiping, Révész, Kinga, Casciotti, Karen, and Hannon, J.E., 2012, Determination of the $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ of nitrate in water; RSIL lab code 2900, chap. 17 of Stable isotope-ratio methods, sec. C of Révész, Kinga, and Coplen, T.B. eds., Methods of the Reston Stable Isotope Laboratory (slightly revised from version 1.0 released in 2007): U.S. Geological Survey Techniques and Methods, book 10, 35 p. <https://pubs.usgs.gov/publication/tm10C17>
- [15] Coplen, T.B., Böhlke, J.K., and Casciotti, K.L., 2004, Using dual bacterial denitrification to improve $\delta^{15}\text{N}$ determinations of nitrates containing mass independent ^{17}O : Rapid Communications in Mass Spectrometry, v. 18(3), p. 245–250. <https://doi.org/10.1002/rcm.1318>