United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Material USGS44

(Carbon Isotopes in Calcium Carbonate)

This high purity, powdered, calcium carbonate reference material (RM) is intended for normalization of stable carbon ($\delta^{13}C$) measurements of unknown calcium carbonate and similarly behaving carbon-bearing substances [1]. A unit consists of 0.5 g in a glass vial. There is no limit on distribution. This RM was prepared by the Reston Stable Isotope Laboratory (RSIL) of the U.S. Geological Survey, Reston, Virginia [1].

Recommended value: Stable carbon isotopic compositions are expressed herein as delta values [2] relative to VPDB (Vienna Peedee belemnite) on a scale normalized such that the $\delta^{13}C$ values of NBS 19 calcium carbonate and LSVEC lithium carbonate are +1.95 ‰ and –46.6 ‰, respectively [3, 4]. The stable carbon isotope delta value of USGS44 calcium carbonate with combined standard uncertainty is:

Reference	$\delta^{13}C_{VPDB\text{-}LSVEC}$	Data source
USGS44	–42.21 ± 0.05 ‰	[1]

Nominal value: Due to its fine grain size (< 63 μm), it is not suitable as a $\delta^{18}O$ RM because its oxygen can exchange with atmospheric water, changing its $\delta^{18}O$ value. Nevertheless, some users may be interested in a nominal $\delta^{18}O$ value. The nominal $\delta^{18}O_{VPDB}$ value of USGS44 is –15.7 ‰. On this scale, the $\delta^{18}O_{VPDB}$ value of NBS 19 calcium carbonate is –2.2 ‰ exactly [5].

Technical coordination for this RM was provided by Haiping Qi of the RSIL.

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August 21, 2026

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Expiration of Reference Value: The reference value for the isotopic composition of USGS44 is valid until December 31, 2036, provided the RM is handled in accordance with the instructions given in this Report of Stable Isotopic Composition (see "Instructions for Use"). The reference value is nullified if the RM is damaged, contaminated, or otherwise modified.

Source of the RM: Sixteen bottles of high purity calcium carbonate powder with a total mass of 8 kg were purchased from Merck. To ensure isotopic homogeneity of the RM, the following steps were carried out. First, approximately 20 g of material was removed from each of these sixteen 500-g bottles, combined, and passed through a 170-mesh (88 μm) stainless steel sieve with a sieve shaker (Retsch model AS200, 11 Penns Trail, Suite 300, Newtown, PA 18940, USA) to homogenize the material. The very small amount of material larger than 88 μm was discarded. The sieved material was divided and collected in four 4-L glass containers. The same steps were repeated until all materials from the original 16 bottles was combined and either passed through the 88-μm sieve or was discarded after not passing through the sieve. Second, approximately 50 g of material was removed from each of the four 4-L containers, combined, and passed through a 170-mesh sieve, mixed, sieved again and distributed evenly in four 4-L new glass containers. Third, about 50 g of material was taken from each of these four containers, combined, passed through a 230-mesh (63 μm) stainless steel sieve, distributed among nine new 2-L glass containers, and repeated until all material passed through the sieve. The third step was repeated three times to thoroughly homogenize the material. Then, samples were taken from the top, middle, and bottom of each of these jars for use in homogeneity testing. The large batch of material is stored in several 1-L vacuum-sealed glass flasks. From these flasks, individual aliquots of 0.5–0.6 g each were distributed into 4-mL glass vials, with Polyseal caps, and vacuum sealed in plastic pouches. All RMs are stored in a cool, dry, dark environment.

Maintenance of RM Report of Stable Isotopic Composition: The U.S. Geological Survey RSIL will monitor this RM 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 0.5 g in a glass vial that is vacuum sealed in a plastic pouch. USGS44 is stable at normal room temperatures when stored under dry conditions. To minimize the potential for contamination, it is recommended that USGS44 be stored in the container in which it was supplied.

Instructions for Use: USGS44 can be interspersed among every 10–15 unknowns. It can be used with other carbon isotopic RMs. To prevent USGS44 from degrading over time, after it is opened, it is recommended that users always close the cap tightly after usage and store in a dry desiccator.

Reporting of stable-isotope-delta values: The following recommendations are provided for reporting stable carbon isotope-delta values. It is recommended that:

- 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 [3,4], even though LSVEC is no longer recommended as a RM for $\delta^{13}C$ measurement [6].
- 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 [7].

REFERENCES

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