United States Geological Survey

Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Materials USGS54, USGS55, and USGS56

(Hydrogen, Oxygen, Carbon, and Nitrogen Isotopes in Wood)

These powdered wood reference materials (RMs) USGS54, USGS55 and USGS56 are mainly intended for isotope-delta normalization of stable hydrogen (δ^2H) and oxygen (δ^{18}O) isotopic measurements of unknown wood samples [1]. They also are suitable for measurements of the stable carbon (δ^{13}C) and nitrogen (δ^{15}N) isotopes [1]. A unit consists of 0.5 g powdered RM. There is no limit on distribution. These RMs were prepared by the Reston Stable Isotope Laboratory (RSIL) of the U.S. Geological Survey, Reston, Virginia [1].

Recommended values: Stable hydrogen and oxygen isotopic compositions are expressed herein as delta values [2] relative to VSMOW (Vienna Standard Mean Ocean Water) on a scale normalized such that the δ^2H value of SLAP (Standard Light Antarctic Precipitation) is –428 ‰ [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]. Stable nitrogen isotopic compositions are expressed relative to atmospheric nitrogen, which is isotopically homogenous [6]. On this scale, the $\delta^{15}N_{AIR\text{-}N_2}$ value of USGS32 KNO_3 is +180 ‰ exactly. The isotopic compositions, mass fractions of each element, and fractions of exchangeable hydrogen of USGS54, USGS55, and USGS56 woods RMs are [1]:

USGS54 (Canadian lodgepole pine)

$\delta^2H_{VSMOW\text{–}SLAP}$ = –150.4 ± 1.1 ‰ (n = 29), hydrogen mass fraction = 6.00 ± 0.04 % (n = 10)
Fraction of exchangeable hydrogen = 5.4 ± 0.6 % (n = 29)
$\delta^{18}O_{VSMOW\text{–}SLAP}$ = +17.79 ± 0.15 ‰ (n=18), oxygen mass fraction = 40.4 ± 0.2 % (n = 6)
$\delta^{13}C_{VPDB\text{-}LSVEC}$ = –24.43 ± 0.02 ‰ (n=18), carbon mass fraction = 48.3 ± 0.4 % (n = 12)
$\delta^{15}N_{air}$ = –2.42 ± 0.32 ‰ (n=17), nitrogen mass fraction = 0.05 % (n = 4)

USGS55 (Mexican ziricote)

$\delta^2H_{VSMOW\text{–}SLAP}$ = –28.2 ± 1.7 ‰ (n = 30), hydrogen mass fraction = 5.65 ± 0.06 % (n = 10)
Fraction of exchangeable hydrogen = 4.1 ± 0.5 % (n = 30)
$\delta^{18}O_{VSMOW\text{–}SLAP}$ = +19.12 ± 0.07 ‰ (n=18), oxygen mass fraction = 35.3 ± 0.2 % (n = 6)
$\delta^{13}C_{VPDB\text{-}LSVEC}$ = –27.13± 0.02 ‰ (n=18), carbon mass fraction = 53.3 ± 0.6 % (n = 12)
$\delta^{15}N_{air}$ = –0.3 ± 0.4 ‰ (n=16), nitrogen mass fraction = 0.25 % (n = 4)

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USGS56 (South African red ivorywood)

$\delta^{2}H_{VSMOW-SLAP}$ = –44.0 ± 1.8 ‰ (n = 30), hydrogen mass fraction = 5.65 ± 0.05 % (n = 10)
Fraction of exchangeable hydrogen = 6.6 ± 0.3 % (n = 30)
$\delta^{18}O_{VSMOW-SLAP}$ = +27.23 ± 0.03 ‰ (n=12), oxygen mass fraction = 41.1 ± 0.2 % (n = 6)
$\delta^{13}C_{VPDB-LSVEC}$ = –24.34± 0.01 ‰ (n=12), carbon mass fraction = 47.3 ± 0.2 % (n = 12)
$\delta^{15}N_{air}$ = +1.8 ± 0.4 ‰ (n=15), nitrogen mass fraction = 0.27 % (n = 4)

Technical coordination for this RM was provided by 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 USGS54, USGS55, and USGS56 are valid until December 31, 2036, 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 a RM is damaged, contaminated, or otherwise modified.

Source of the RMs: In the search for a wood relatively depleted in ^{2}H and ^{18}O, 30 lodgepole pine and spruce wood block samples were obtained (Sundre Forest Products, Sundre, Alberta, Canada). Eleven pieces of Canadian lodgepole pine having the lowest ^{2}H and ^{18}O abundances were combined as USGS54, having a total mass of 1.5 kg (Figure 1-a). For the wood relatively enriched in ^{2}H and ^{18}O, we purchased 46 exotic wood boards and blocks from a commercial vendor. The ziricote wood (about 1 kg) from Mexico was labelled USGS55 (Figure 1-b). The red ivorywood from South Africa having a mass of about 1.2 kg was labeled USGS56 (Figure 1-c).

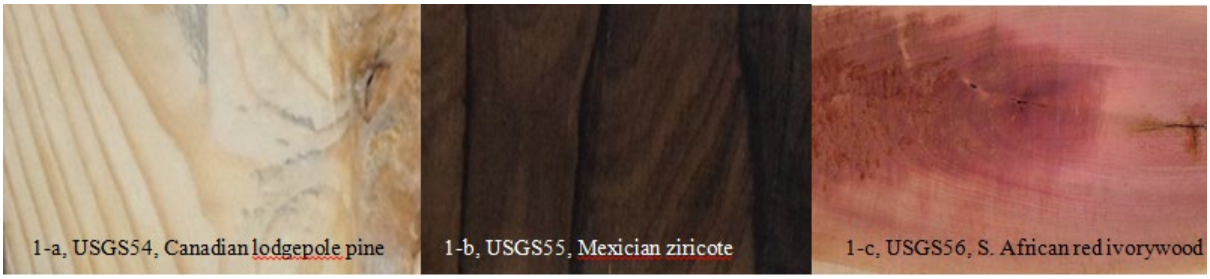


Figure 1. Raw wood materials used for preparation of three isotopic reference materials.



Figure 2. Powdered wood isotopic reference materials in vials

Preparation of the RMs: Each material was chopped into pieces smaller than 1 cm and dried in a vacuum oven at 40 °C overnight. The dried wood pieces were pulverized with a Retsch model MM301 ball mill. Once the wood was pulverized, it was homogenized using a 50-mesh (0.25 mm) or 70-mesh (0.21 mm) sieve shaker (Retsch model AS200). Any material for USGS54 not passing through the 50-mesh sieve and material for USGS55 and USGS56 not passing through the 70-mesh sieve was pulverized again. To ensure isotopic homogeneity of each RM, the following steps were carried out. First, the sieved material was divided into two fractions and collected in two 2-L glass containers. Second, about 50 g of material was taken from each container and combined on a 60-mesh or 70-mesh sieve, mixed, and sieved. The same steps were repeated until all material in the two original containers was thoroughly mixed and sieved. The second procedure was repeated three times to thoroughly homogenize the material. The homogenized batch of material is stored in several 2-L glass bottles in a dark, cool, and dry environment.

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 0.5 g in a glass vial (Figure 2). USGS54, USGS55, and USGS56 are stable at normal room temperatures when stored under dry conditions. To minimize the potential for contamination, it is recommended that each RM be stored in the container in which it was supplied. Storing in a dark, cool, and dry place is preferred.

Instructions for Use: USGS54, USGS55, and USGS56 wood isotopic reference materials are intended for δ^2H and δ^{18}O normalization of unknown wood samples. It is not necessary to use all three RMs in one analytical sequence. A pair of RMs (such as USGS54 and USGS55 for δ^2H, and USGS54 and USGS56 for δ^{18}O) can be selected to normalize unknown wood samples. It is suggested that masses of RMs and unknowns be identical to minimize biases. The typical sample sizes used for δ^2H, δ^{18}O, δ^{13}C, and δ^{15}N measurements are 0.46 mg, 0.5 mg, 0.35 mg, and 7 to 21 mg, respectively. It is recommended that unknown wood samples and wood RMs analyzed for both δ^2H and δ^{18}O values be equilibrated with laboratory air at ambient temperature for at least 5 days, then dried thoroughly prior to δ^2H and δ^{18}O measurements. Due to the present of nitrogen in wood, the chromium packed reactor on the TC/EA is required for δ^2H analysis [7, 8]. The original glassy carbon TC/EA should be used for δ^{18}O measurements. Because wood contains very small amounts of nitrogen, the N_2 interference with the CO background is insignificant. Thus, the use of an N_2-diverting valve was unnecessary.

Using wood isotopic reference materials, USGS54, USGS55, and USGS56, should improve the comparability of isotopic results of whole wood samples among laboratories. However, the uncertainty of isotopic results of wood samples may not be as small as one would hope for because of the variable fraction of exchangeable hydrogen [$x(H)_{ex}$ varied from 0.041 of USGS55 to 0.066 of USGS56] originating from variable concentrations of wood compounds in different tree species [1].

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

- The δ^2H and δ^{18}O values of all wood samples be expressed relative to VSMOW-SLAP on a scale where the δ^2H of SLAP = –428 ‰ exactly [4].
- 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 [4,5], even though LSVEC is no longer recommended as a RM for $\delta^{13}C$ measurement [9].

- The $\delta^{15}N$ values of all nitrogen-bearing substances be expressed relative to atmospheric nitrogen [6].
- When reporting results, authors should always use text in their reports such as:

  "The $\delta^{2}H$ and $\delta^{18}O$ values of wood samples are reported relative to the VSMOW-SLAP scale, and on this scale the $\delta^{2}H$ values of USGS54 and USGS55 are –150.4 ‰ and –28.2 ‰, respectively, and $\delta^{18}O$ values of USGS54 and USGS56 are +17.79 ‰, +27.23 ‰, respectively"

  In this manner, readers can re-normalize measurement results to current values of USGS54, USGS55, and USGS56, as they are improved with new techniques.
- Reporting of delta values relative to SMOW and PDB (Peedee belemnite be discontinued [10].

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