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Reston Stable Isotope Laboratory

Report of Stable Isotopic Composition

Reference Material CBS (Caribou Hoof Standard)

(Hydrogen and Oxygen Isotopes in Caribou Hoof keratin)

This reference material (RM) is intended for normalization of stable hydrogen (δ^2H) and oxygen (δ^{18}O) measurements of keratin in organic materials in wildlife and forensics research with a TC/EA (thermal conversion/elemental analyzer) and an isotope-ratio mass spectrometer when used with KHS (Kudu Horn Standard) [1]. This RM consists of 0.5 g of CBS (Caribou Hoof Standard) [1]. There is no limit on distribution. CBS was prepared by Dr. L. I. Wassenaar and Dr. K. Hobson [1] of Environment Canada, and this RM is distributed by the Reston Stable Isotope Laboratory (RSIL) of the U.S. Geological Survey, Reston, Virginia on their behalf.

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 and δ^{18}O values of SLAP (Standard Light Antarctic Precipitation) are –428 ‰ and –55.5 ‰, respectively [3,4]. The δ^2H value below was determined using pre-treatment with a preparation device designed to eliminate residual moisture and quantify exchangeable hydrogen [5]. The mass fraction of total hydrogen and oxygen, and non-exchangeable fraction of hydrogen in CBS are preliminary.

Stable hydrogen isotopic composition (non-exchangeable fraction):	$\delta^2H_{VSMOW\text{-}SLAP} = -157.0 \pm 0.9$ ‰ [5]
Nominal mass fraction of total hydrogen (ambient temperature techniques):	$w_H = 6.5$ %
Nominal mole fraction of exchangeable hydrogen:	$x_{H\text{-}ex} = 10.0 \pm 2.0$ % (ambient temperature techniques) $x_{H\text{-}ex} = 1.1 \pm 0.3$ % (under vacuum at a drying temperature of 105°C) [5]
Stable oxygen isotopic composition:	$\delta^{18}O_{VSMOW\text{-}SLAP} = +3.8 \pm 0.3$ ‰ [1]
Mass fraction of oxygen:	$w_O = 22.5$ %

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

Reston, Virginia 20192
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Distribution and stability: CBS is stable at normal room temperatures. To minimize the potential for contamination, it is recommended that this RM be stored in the container in which it is supplied. The recommended reference values given in this Report of Stable Isotopic Composition may change after further evaluation. Users should contact the RSIL for updates to this report.

Instructions for use: Unknown keratin samples and keratin reference materials analyzed for both δ^2H and δ^{18}O values typically need to be equilibrated with laboratory air at ambient temperature for at least 5 days. To minimize uncertainty caused by moisture that has not been removed, reference materials and unknowns need to be thoroughly dried prior to isotopic analysis based on experimental observations [6]. It is recommended that two keratin reference materials with different δ^2H and δ^{18}O values be used for normalizing isotopic measurement results to the VSMOW-SLAP scale. In addition to CBS, other RMs could be KHS, USGS42 Tibetan human hair, and USGS43 Indian human hair. It is suggested that the mass of CBS, other references (such as KHS), and unknowns should be identical to minimize or cancel biases. It is also recommended that one should make the unknown sample powder size as close as possible to the powder size of CBS, recognizing that in some studies the sample cannot be pulverized.

Experimental studies indicate that improved measurement results can be achieved by analyzing samples for δ^2H and δ^{18}O in separate runs because the operating conditions for δ^2H and δ^{18}O measurement with a TC/EA are different [6,7,8]. If unknown samples have the same chemical matrix as RMs, the δ^2H measurements can be carried out on either glassy carbon-filled reactor or chromium-filled reactor. Otherwise, users may want to use a chromium filled elemental analyzer as described by Gehre et al. [7] because the elemental chromium will prevent production of hydrogen cyanide and will promote quantitative conversion of hydrogen to molecular hydrogen (H_2).

Reporting of stable-isotope-delta values: The following recommendations are provided for reporting of stable hydrogen and oxygen isotope-delta values [2]. It is recommended that:

- The δ^2H values of all hydrogen-bearing substances be expressed relative to VSMOW-SLAP on a scale where $\delta^2H_{SLAP2} = -427.5$ ‰ or $\delta^2H_{SLAP} = -428$ ‰ exactly [3,4,9].
- 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 $\delta^{18}O_{SLAP2} = -55.5$ ‰ or $\delta^{18}O_{NBS19} = -2.2$ ‰, respectively.
- Authors should report δ 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. Example text is:

  > **"The δ^2H and δ^{18}O values of keratin samples are reported relative to the VSMOW-SLAP scales, and on these scales the δ^2H and δ^{18}O values of CBS and KHS are xxx, yyy, zzz, and uuu ‰, respectively [reference]."**

- Reporting of δ values relative to SMOW and PDB (Peedee belemnite) be discontinued [10].

References

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