

METHODOLOGY C, N, S, H

For $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ determinations:

Subsamples of powdered materials were weighed to the nearest μg and placed into tin capsules for $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$ determinations. Isotopic analyses were carried out at the Laboratorio de Isótopos Estables of the Estación Biológica de Doñana (LIE-EBD, Spain; www.ebd.csic.es/lie/index.html). All samples were combusted at 1020 °C using a continuous flow isotope-ratio mass spectrometry system by means of Flash HT Plus elemental analyzer coupled to a Delta-V Advantage isotope ratio mass spectrometer via a CONFLO IV interface (Thermo Fisher Scientific, Bremen, Germany). The isotopic composition are reported in the conventional delta (δ) per mil notation (‰), relative to Vienna Pee Dee Belemnite ($\delta^{13}\text{C}$) and atmospheric N_2 ($\delta^{15}\text{N}$). Replicate assays of standards routinely inserted within the sampling sequence indicated analytical measurement errors of $\pm 0.1\text{‰}$ and $\pm 0.2\text{‰}$ for $\delta^{13}\text{C}$ and $\delta^{15}\text{N}$, respectively. The standards used were: EBD-23 (cow horn, internal standard), LIE-BB (whale baleen, internal standard) and LIE-PA (feathers of Razorbill, internal standard). These laboratory standards were previously calibrated with international standards supplied by the International Atomic Energy Agency (IAEA, Vienna).

For $\delta^{34}\text{S}$:

Subsamples of powdered materials were weighed to the nearest μg and placed into tin capsules for $\delta^{34}\text{S}$ determinations. Isotopic analyses were carried out at the Laboratorio de Isótopos Estables of the Estación Biológica de Doñana (LIE-EBD, Spain; www.ebd.csic.es/lie/Home.html). All samples were combusted at 1020 °C using a

continuous flow isotope-ratio mass spectrometry system by means of Flash HT Plus elemental analyzer coupled to a Delta-V Advantage isotope ratio mass spectrometer via a CONFLO IV interface (Thermo Fisher Scientific, Bremen, Germany). Stable isotope ratios are expressed in the standard δ -notation (‰) relative to Vienna Canyon Diablo Troilite (VCDT). Replicate assays of laboratory standards routinely inserted within the sampling sequence, and previously calibrated with international standards, indicated analytical measurement errors of $\pm 0.3\text{‰}$.

For $\delta^2\text{H}$ in Keratin materials:

Subsamples of powdered materials were weighed to the nearest μg and placed into silver capsules for $\delta^2\text{H}$ determinations. Isotopic analyses were performed at the Laboratorio de Isótopos Estables of the Estación Biológica de Doñana (LIE-EBD, Spain; www.ebd.csic.es/lie/index.html). Isotope measurements were performed on H_2 derived from high-temperature flash pyrolysis at 1450°C by means of Flash HT Plus elemental analyzer coupled to a Delta-V Advantage isotope ratio mass spectrometer via a CONFLO IV interface (Thermo Fisher Scientific, Bremen, Germany). The isotopic composition is reported in the conventional delta (δ) per mil notation (‰), relative to Vienna Standard Mean Ocean Water (VSMOW). Replicate assays of laboratory standards routinely inserted within the sampling sequence indicated analytical measurement errors of $\pm 3\text{‰}$. The standards used were: CBS, KHS (keratin standards supplied by Environment Canada) and LIE-PA2 (feathers of Razorbill, internal standard). The $\delta^2\text{H}$ analyses were carried out using the comparative equilibration approach described in Wassenaar & Hobson (2003)*, and by using calibrated keratin isotope reference materials in order to avoid effects of H exchange with ambient water vapor.

* Wassenaar, L. I., & Hobson, K. A. (2003). Comparative equilibration and online technique for determination of non-exchangeable hydrogen of keratins for use in animal migration studies. *Isotopes in Environmental and Health Studies*, 39(3), 211-217.