

DEVELOPMENT AND CHARACTERIZATION OF AN IN-HOUSE STANDARD FOR STABLE CARBON AND OXYGEN ISOTOPES ANALYSES

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ABSTRACT: The actual precision and accuracy of an isotope ratio mass spectrometry (IRMS) laboratory and its scientific production heavily depends on the fabrication and maintenance of their in-house calibration material. However, the specification and characterization of the in-house standard and analytical details of the laboratorial routine are rarely published or accompanied in the scientific studies that rely on IRMS measurements. Here we describe the development of the in-house laboratory standard (SHP2L) to be used for calibrating measurements of carbon and oxygen stable isotopes (i.e. $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) on carbonate material at the newly formed Paleoceanography and Paleoclimatology Laboratory (P2L) of the School of Arts, Sciences and Humanities, University of São Paulo, Brazil. The P2L laboratory is equipped with a Thermo™ MAT253 isotope ratio mass spectrometer coupled to an automated carbonate preparation device Thermo™ Kiel IV. First, we illustrate a detailed overview of the analytical setup and performance at P2L. The equipment is stable in the long term and ensures analytical precision better than 0.07 ‰ up to masses of 10 mg. Second, we describe the production and characterization of our in-house laboratory standard. The material chosen for the production of the SHP2L is a calcium carbonate plate from the German Jurassic Solnhofen Formation. After removing the weathered layer, an array of 25 micro-samples was obtained from the plate surface in order to assess the spatial variability of its isotopic composition. Overall, the $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ 1 σ spatial variability is better than 0.11 ‰. The plate was then fragmented, grounded and sieved to isolate the 63-125 μm grain-size fraction. The chemical and mineralogical purity of the SHP2L in-house standard was established by x-ray fluorescence and diffraction. Batches of ca. 50 samples from the 63-125 μm fraction were analyzed for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ in three laboratories. Established SHP2L isotopic composition is $-0,77 \pm 0,043$ ‰ for $\delta^{13}\text{C}$ e $-5.74 \pm 0,067$ ‰ for $\delta^{18}\text{O}$. The SHP2L in-house standard proves to be a homogeneous and reliable material to be used as in-house standard for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ stable isotopes analyses.

Keywords: CARBON AND OXYGEN STABLE ISOTOPES, CARBONATE, WORKING STANDARD, ISOTOPE RATIO MASS SPECTROMETRY