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In Situ Laser-Laser carbon and oxygen isotopes measurements in carbonates: A step forward field isotopic characterization

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Stable isotope ratios (d^{13}C and d^{18}O) of carbonates archived in the rock record are routinely used to reconstruct paleo-temperatures and the secular evolution of the biogeochemical carbon cycle through Earth History. The state-of-the-art technique, employed since the mid 20th century, to measure these isotopic ratios includes: micro-drilling and/or sawing and crushing, CO_2 release by wet acid digestion, gas equilibration, purification, and transfer, before gas phase IRMS measurements. While these steps are time- and resource-consuming, they provide accurate measurements from rock samples.

This study presents a new protocol involving a laser calcination system that decreases drastically the analysis time by reducing the number of preparation steps while offering the possibility of performing punctual in-situ analyses at the mm scale. This original method is based on the use of a fiber laser diode device emitting in near infrared at 880nm and inducing the decomposition of calcium carbonate into lime and carbon dioxide.

We analyzed 9 different types of carbonates encompassing a range of isotopic compositions, between -18.2 and+3.3 and between -1.7 and -14.6‰ for d^{13}C$_{carb}$ and d^{18}O$_{carb}$ VPDB, respectively. A comparison of isotopic results was performed between the classic (micro-drilling and acid digestion) vs. laser calcination methods and using both a magnetic sector IRMS and laser-based Isotope Ratio Infrared Spectrometer. The resulting isotopic cross-calibration shows a direct positive co-variation between both methods with a correlation coefficient of 0.99 and a regression slope of 1 within uncertainties for d^{13}C$_{carb}$ values. The d^{18}O$_{carb}$ values also compared well, with a correlation coefficient of 0.96, suggesting a constant gas-solid phase isotopic equilibrium. The reproducibility of our laser calcination method shows a 1sstandard deviation of 0.31 and 0.77 for d^{13}C$_{carb}$ and d^{18}O$_{carb}$, respectively.

We demonstrate that (i) laser calcination gives accurate and reproducible C and O isotope characterizations of carbonates, (ii) the physical effect during calcination does not introduce sensible isotopic fractionation for C and is accompanied by a constant isotopic equilibrium offset for O. These findings pave the way for a new range of possibilities for carbonate isotopic measurements using rapid, punctual and easy to manipulate laser preparation devices.