Goldschmidt 2023 Abstract
https://doi.org/10.7185/gold2023.14625

Long-term performance of the Break Seal method for measuring Carbonate clumped isotopes

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During the last 19 years, Carbonate clumped isotope thermometry (Δ_{47}) has grown in popularity popular due to temperature dependence of isotopic ordering of ^{13}C-^{18}O within carbonate lattice rather than its parental fluid composition [1], along with the parallel adaptation of different carbonate-acid reaction techniques and mass spectrometer analytical methods[2]. Here, we present the long-term precision of three carbonate reference materials tested alongside inter-laboratory ETH carbonates and Heated Gas (HG). Our routine analytical protocol involves zero enrichment using Working Gas (WG) (Linde CO_2), analysis of one carbonate reference material along with samples, and HG prepared from refreezing of CO_2 post analysis. Occasionally, we tuned our protocol by analyzing water-equilibrated CO_2 at different temperatures. In this experiment, we used three different carbonate references; MARJ1 refers to Carrara marble powder (<250 mesh), OMC (<100 mesh) refers to cap carbonate from Otavi Meieberg, and OASIS Calcite from Sittampudi complex, India. Our analytical protocol is a Break Seal method where Carbonate is allowed to completely react with ∼105% orthophosphoric acid in an enclosed chamber at 25±0.1°C for a minimum of 12 hours [3]. The long-term mean value of Δ_{47} (CDES) and precision of analysis (mean ± 1SE; Number of analysis) are as follows MARJ1 (0.402±0.002; n= 88), OMC (0.580±0.004, n=33), and OASIS Calcite (0.505±0.004, n=18). Similarly, the long term mean value and reproducibility (1SE) for $\delta^{13}C$ (‰VPDB) and $\delta^{18}O$ (‰VPDB) are as follow: a) MARJ1 (2.06±0.01, n=88 and -2.02±0.03, n=88), b) OMC (-4.22±0.01, n=33 and -8.34±0.05, n=33), and c) OASIS Calcite (1.34±0.01, n=18 and -10.87±0.02, n=18). During our analysis, we monitored Δ_{49}/Δ_{44} ratio and accepted only those where it is less than 0.1 to define the level of contamination during sample preparation following the protocol of Gas Chromatography cleaning. Our analytical procedure is reproducible and robust, providing the best method for high-precision analysis of clumped isotopes in carbonate samples.

References:

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