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Speleothem fluid inclusion isotope analysis: improving its accuracy

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Fluid inclusion isotope analysis of speleothem $CaCO_3$ gives the parent water $d^{18}O$ and d^2H. Kinetic isotope effects do not influence parent water $d^{18}O$ or d^2H as opposed to $CaCO_3$ isotopes. During fluid inclusion isotope analysis speleothem chips are heated and subsequently crushed to release the water as vapor. However, pre-crushing evaporation and/or post-crushing absorption can affect the isotope values and bias temperatures based on the calculated isotope fractionation between $CaCO_3$ and fluid inclusion $d^{18}O$. The fractionation associated with analytical (pre-crushing) evaporation can induce trendlines with shallow slopes (<1.4) in $d^{18}O$ - d^2H crossplots. Natural evaporation in the soil or cave typically results in steeper trendlines.

In this study, we quantify the water loss related to analytical evaporation and apply 4 different crushing temperatures (90, 100, 110, 125°C) to replicates of flowstone and stalagmite layers to test whether analytical evaporation can be prevented. Our results indicate that high yield samples (>1-2 µl/g) are generally more reliable compared to low yield samples (<0.5 µl/g). However, samples with high water content can still be altered by in crusher evaporation. For our samples, results indicate that crushing at 90°C prevents most analytical evaporation and increases the sample yield by up to 30%. Our low yield samples crushed at temperatures of 110°C and 125°C result in different trendlines. At 110°C, the trendline can even be parallel to the global meteoric water line. These different slopes may be explained by various amounts of absorption of water to freshly crushed calcite powder.

Generally, data that plot in the vicinity of the global meteoric water line are regarded as trustworthy. However, we show that this is not always a suitable quality criterion for fluid inclusion isotope analysis. High yield samples crushed at 90°C produce accurate $d^{18}O$, d^2H, and d-excess values. Furthermore, by reducing and quantifying analytical evaporation we can resolve in-cave evaporation in speleothem $CaCO_3$. Cave air temperatures, calculated from our flowstone $CaCO_3$ - drip water oxygen isotope fractionation are reliable when crushed at 90°C, supported by TEX_{86} temperatures and modern drip water isotope compositions.