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Continuous-flow fluid inclusion oxygen and hydrogen isotope analysis: a progress report

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We present a new design continuous-flow preparation device for on-line δ^2 H and δ^{18} O analyses of fluid inclusions in speleothem carbonate. The design is based on a relatively low-cost adaptation to a Finnigan TC-EA pyrolysis furnace. Standard specifications of the Finnigan TC-EA allow for the simultaneous δ^2 H and δ^{18} O analysis of ~0.2 microliter of water, reproducible within 2%, for δ^2 H and 0.5%, for δ^{18} O (1SD).

The fluid inclusion extraction line that we have connected to the inlet of the TC-EA contains an adapted vacuum valve that serves as a hand-operated crusher, and a cold trap operating at liquid nitrogen temperature. In contrast with most existing devices, crushing and cryogenic capture of fluid inclusion water take place under a helium atmosphere, rather than in vacuum. Subsequent flash heating of the cold trap releases the frozen water sample into a helium flow, which carries the sample to the TC-EA.

First experiments with this system focused on δ^2 H only, and involved direct injection of sub-microliter amounts of standard waters via a septum-port attached to the crusher. Repeated analyses resulted in accurate standard water isotope values, reproducible within the standard specifications of the TC-EA. This may be interpreted to confirm the proper functioning of the cryogenic trap and sample transfer to the TC-EA. A second set of experiments then focused on the analyses of speleothem calcite from a cave with known drip water composition. Crushing and analysing a set of samples cut from a single sub-recent stalagmite growth interval gave δ^2 H values that compare remarkably well to the drip waters from that cave, at a reproducibility well within the TC-EA specs.

Although these first results look promising, a small number of technical problems still need attention; particularly a solution for the memory effects known to occur for TC-EA analysis may still improve this technique considerably. Pre-conditioning the TC-EA with standard water of an isotope value as close as possible to the fluid inclusions to be analysed currently minimizes memory effects.

In summary, these first results suggest that this system will potentially be able to produce rapid and accurate fluid inclusion $\delta^2 H$ and $\delta^{18} O$ data of speleothems or other geological materials, which would be an important step forward in our attempts to reconstruct paleotemperatures from speleothem records.