Fluid inclusion hydrogen and oxygen isotope analyses using the “Amsterdam Device”: a progress report

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The Amsterdam Device is a continuous-flow preparation device for on-line δ²H and δ¹⁸O analysis of fluid inclusions in speleothem carbonate. The design is based on a relatively low-cost adaptation to a Thermo Finnigan TC-EA pyrolysis furnace. Standard specifications of the Thermo Finnigan TC-EA allow for simultaneous δ²H and δ¹⁸O analysis of 0.2 microliter of water, reproducible within 2 permil for δ²H and 0.5 permil for δ¹⁸O (1SD). The technical description of the Amsterdam device was published in 2006, together with the results of some first crushing experiments. These experiments showed that the technique works well for δ²H analyses, but not enough experiments were done at the time to evaluate the applicability of the device for δ¹⁸O analyses of the same samples.

Over the last year we have run extensive experiments with the so-called jump routine, which allows combined δ²H and δ¹⁸O analysis of the water released from a single crush. The jump routine is a standard feature in the software for our ThermoFinnigan TC-EA and Delta XP, and involves a calibrated rapid magnet jump of the mass spectrometer in the time between the entry of the GC-separated H₂ and CO peaks.

In these recent experiments we have made two main changes compared to the Vonhof et al (2006) protocol:

1: We have installed a reverse-flow carrier gas setup, which has decreased the memory effect on TC-EA analyses (Gehre et al., 2004). To accommodate that, we split the incoming carrier gas in two flows: A) a ~10 ml/min flow entering at the bottom connector of the TCEA reactor, and B) a ~100 ml/min carrier flow that leads through the crusher and cold trap to enter in the top of the glassy carbon reactor.
2: For the freezing protocol we apply an ethanol slush at ~ -90 degrees Celsius, instead of liquid nitrogen applied in the older experiments. This is essential because isotopically distinct CO$_2$ released from the crushed CaCO$_3$ sample is trapped along with the inclusion water under liquid nitrogen temperature and contributes to the $\delta^{18}$O value analysed with the pyrolysis method. At ethanol slush temperatures, water will be trapped quantitatively and CO$_2$ will not, leading to a inclusion water $\delta^{18}$O analysis unaffected by CO$_2$ derived oxygen.

With this new protocol we have analysed speleothem material from different settings and a selection of results is presented. Often, $\delta^2$H and $\delta^{18}$O data plot near the Global Meteoric Water Line (GMWL), which lends support to the accuracy of the analyses. If data plot away from the GMWL it is usually the $\delta^{18}$O data that are off-set. These $\delta^{18}$O off-sets are believed to be analytical artefacts. Our experiments did not provide evidence for oxygen exchange between fluid-inclusion water and surrounding carbonate.

References:
