Geophysical Research Abstracts, Vol. 9, 05702, 2007
SRef-ID: 1607-7962/gra/EGU2007-A-05702

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 δ^2H and δ^{18}O 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 δ^2H and δ^{18}O analysis of 0.2 microliter of water, reproducible within 2 permil for δ^2H and 0.5 permil for δ^{18}O (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 δ^2H analyses, but not enough experiments were done at the time to evaluate the applicability of the device for δ^{18}O analyses of the same samples.

Over the last year we have run extensive experiments with the so-called jump routine, which allows combined δ^2H and δ^{18}O 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 H_2 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 $\sim$10 ml/min flow entering at the botttom connector of the TCEA reactor, and B) a $\sim$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 $\sim$ -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:

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