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Characterization of fluid inclusions in speleothems: heating experiments and isotopic hydrogen composition of the inclusion water.

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The study gives a better understanding of the release of water by speleothem calcite during recovery of fluid inclusions. We followed the water released by uncrushed and crushed speleothem calcite with a quadripole mass spectrometer and measured the δD of the inclusion water, recovered with the decrepitation as well as with the crushing and heating technique.

During heating of an uncrushed piece of calcite, water is released between 25 and 550˚C and between 550 and 900˚C. We relate the first water peak to fluid inclusion water and the second peak to structurally bound water, released during decrepitation (decomposition) of the calcite to lime (CaO). The δD of recovered water is close to the cave seepage water and, in contrast to previous authors, we observed no important negative shift in δD for the water recovered with the decrepitation method. However, in the Han-sur-Lesse calcite, the "decrepitation" water (550-900˚C) has a δD, which is on the average 7 permil lighter than the fluid inclusion water, released between 25 and 550˚C, which is close to the cave seepage water. Our study suggests that the recovery of water from uncrushed calcite between 25 and 550˚C would be a better representa-

tive of the inclusion water. The study suggests that the 20 to 30 permil offset observed by previous authors is a methodological artefact due to contamination with isotopically light atmospheric moisture adsorbed on lime powder present in the extraction line, which is released when temperature in the extraction cell attains 400˚C.

The water release curve of crushed calcite is much smoother than for uncrushed calcite and water is released at higher temperatures indicating a “buffering” effect of the powder. With the crushing technique, water is released up to 360˚C, a second water release, probably due to the opening of inclusions smaller than the grain size is not well individualised with the decrepitation peak. To recover all the water released by crushing, the powder should be heated up to 360˚C.

The study suggests that a simple heating technique could recover water more representative of the seepage water than the now used decrepitation method and with a double water yield than the used crushing and heating method.