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Isotopic measurement of speleothem inclusion water in nano-liter quantities: application to stalagmites from Okinawa, Japan

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Speleothem inclusion water isotopic compositions are promising new climatic proxies. The applicability, however, is limited by low water content and challenging analytical difficulties. We have developed a precise and accurate isotopic technique based on cavity ring-down spectroscopy with a low sample-amount requirement of 20-260 nL of inclusion water from only 77-286 mg of stalagmite deposits in Gyokusen Cave, Okinawa Island, Japan. The 1σ reproducibility is $\pm$0.24permil for δ18O and $\pm$1.8permil for δD. The small sample size requirement demonstrates that our analytical technique can offer high-resolution inclusion water-based paleoclimate reconstructions. The δ18O and δD values of inclusion water samples from the two most recently layers are within the expected range of isotopic monitoring data for drip water and rainwater at the island. Data inferred from coupled stalagmite δ18O records of coeval inclusion water and carbonate indicate that a cave temperature over the past few decades agrees with instrumental observations. Inferred temperatures at 9-10 thousand years ago (ka) and 26 ka are consistent with previous marine sediment records. Our results support the application fidelity of the proposed technique for speleothem formation deposited under oxygen isotopic equilibrium conditions. However, we observed two biased temperature at the last glacial period: an enrichment of 0.5-1permil in δ13C and δ18O on the fringe carbonate deposition can bias the derived temperature by up to -4 °C. This kinetic fractionation should be carefully evaluated to avoid misleading interpretations.