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U-Pb dating of calcite-aragonite layers in speleothems from hominin sites in South Africa by MC-ICP-MS

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Speleothems are found in association with early human (hominin) fossil-bearing cave deposits in South Africa and can be used to provide valuable chronological constraints. Such material is generally too old for U-Th dating and, although U-Pb geochronology presents a suitable alternative, bulk U concentrations are typically too low (< 0.05 μg/g) to provide useful ages. Very small amounts of radiogenic Pb accumulated in young samples, especially those with low U contents and a dominance of common Pb, even in Pb poor material, further complicates dating. U-Pb isochron age estimates can, however, be obtained by taking a number of sub-samples very close to each other (cm-scale) from growth layers. The close spacing makes it more likely that common Pb in the samples is homogeneous and minimizing scatter in isochron diagrams. Multi-collector ICP-MS is highly suitable for measuring isotope ratios of small (∼ng) quantities of Pb from such samples. Strong heterogeneity in U concentrations is frequent, rendering layers with sufficient U concentrations ($\geq$ 1 μg/g) rare and difficult to locate. For this reason, a pre-screening step is necessary: we used simple non-invasive beta-scanner imaging to identify U-rich ($\geq$ 1 μg/g) domains that could be analyzed with MC-ICP-MS techniques to provide U-Pb ages. Laser ablation scans for U and other trace elements is an alternative. A set of flowstone samples from the hominin bearing cave sediments at Sterkfontein Cave exhibit infrequent < 1 cm-thick layers with U concentrations $\geq$ 1 μg/g. These layers were thoroughly investigated using thin section petrography, scanning electron microscopy (SEM) and laser ablation ICP-MS. Relict aragonite needles are found exclusively in these U-rich layers. Apart from U, they are also enriched in Sr and Ba, due to the compatibility of large ions in the aragonite structure. They are also lower in common Pb than calcite layers above and below them. The layers occur in all cases in the lowermost few cm of flowstone profiles, and aragonite probably crystallized as a result of calcite growth inhibition due to high Mg content. Cave drip waters resulting from first rains leaching soil after very prolonged dry periods would be relatively enriched in elements such as U and Mg and may account for the positioning of the U-rich layer in the first few cms of flowstone above the underlying cave sediments. We recognize that open system behaviour during the partial transformation of aragonite to calcite is a potential problem and argue, on the basis of geochemistry and age consistencies, that recrystallization took place rapidly after speleothem formation and did not significantly affect the U-Pb ages. We also urge the use of thin sections in investigating speleothem fabrics prior to dating.