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Precise and accurate measurement of U and Th isotopes via ICP-MS using a single solution

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U-series isotope measurements by ICP-MS commonly utilize separate runs for U and Th and standard-sample bracketing to determine correction factors for mass fractionation and ion counter yields. Here we present an approach where all information necessary to calculate an age (aside from background/baseline levels) is determined while analyzing a single solution containing both U and Th. This internally calibrated procedure should reduce any bias caused by distinct behavior of sample versus standard solutions during analysis and offers advantages including simplicity of operation, calculation of preliminary ages in real time, and simplified analysis of errors and their sources. Hellstrom (2003) developed a single-solution, internally-calibrated technique for an ICP-MS with multiple ion counters, but to our knowledge no such technique is available for an ICP-MS with a single ion counter.

We use a Thermo Neptune Plus multi-collector ICP-MS with eight movable Faraday cups and a fixed center cup/ion counter equipped with a high abundance-sensitivity filter (RPQ). We use Faraday cups to measure all masses except 230 and 234, which are measured on the ion counter with the RPQ detuned (i.e., Suppressor voltage = 9950 V). ²³⁸U is maintained in a cup throughout the analysis to avoid reflections and is used to normalize signal instabilities related to sample introduction. Each analysis has a three-part structure, i.e. 1) background/baseline levels, 2) sample composition, and 3) peak-tails are sequentially determined. In step 1, multiplier dark noise/Faraday baselines plus background intensities at each mass are determined while aspirating running solution. During sample measurement in step 2, ion counter yields for Th and U are determined using signals of 300-400 kcps for ²²⁹Th and ²³³U by measuring ²²⁹Th/²³⁸U and ²³³U/²³⁸U ratios first with the minor masses on the ion counter and then with both masses in cups. Mass bias can be determined using the ²³³U/²³⁶U ratio of the spike, allowing the sample's ²³⁸U/²³⁵U ratio to be measured. In step 3, we monitor peak-tails at half-mass positions (229.5, 231.5, 234.5) and on mass 237 while aspirating sample solution. Tail measurement requires a distinct cup configuration to maintain ²³⁸U in the cups; however, no sample is consumed during automated cup reconfiguration.

We monitor the accuracy of $^{234}\text{U}/^{238}\text{U}$ ratios using CRM 145, which gives a weighted mean atom ratio of $(5.2846 \pm 0.0029) \times 10^{-5}$ (all errors 2σ), consistent with published and reference values. The reproducibility of $^{230}\text{Th}/^{238}\text{U}$ ratios is monitored using the Schwartzwalder Mine secular-equilibrium standard (SM). We detect no bias in $^{230}\text{Th}/^{238}\text{U}$ or $^{234}\text{U}/^{238}\text{U}$ ratios measured for SM at beam intensities ranging over a factor of four, consistent with accurate correction for IC yields. Aladdin's cave coral (AC-1) was analyzed to check our ICP-MS method (and the preceding purification by ion exchange) on a carbonate and yields a mean age of 125.43 ± 0.38 ka, in agreement with published values.

We are currently applying the method to corals, speleothems, pedogenic coatings, and tufas.