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Proof of concept: External calibration ICP-MS analysis in apatite (U–Th)/He thermochronometry

BJARNE FRIEDRICHS, FLORA BOEKHOUT, NICOLAS VILLAMIZAR ESCALANTE, BIANCA HEBERER, JOHANN GENSER AND CHRISTOPH VON HAGKE

University of Salzburg

The (U–Th)/He system is a popular low-temperature thermochronometer, and apatite is the mineral dated most using this decay system. With a typical closure temperature around 70°C corresponding to a depth of approximately 2–3 km for common continental geothermal gradients, apatite (U–Th)/He thermochronometry is a powerful tool to date shallow crustal cooling. Apatite (U–Th)/He data are therefore used to reconstruct the thermal history of sedimentary basins, the exhumation of cratonic plateaus, or the spatial and temporal thermal evolution of mountain chains. It can also be employed to constrain the timing of past meteorite impacts or volcanic activity.

The analytical workflow in (U–Th)/He thermochronometry consists of dimension measurements (to correct dates for ^{4}He lost out of the dated crystals upon alpha decays) and two subsequent mass spectrometric analyses. Daughter nuclides (^{4}He) are released from single crystals packed into Pt or Nb microtubes by infrared laser heating and quantified with a gas mass spectrometer. Parent nuclides (particularly ^{238}U and ^{232}Th), on the other hand, are quantified, after dissolving these single crystals, by inductively coupled plasma mass spectrometry (ICP-MS). Both these analyses are typically conducted by isotope dilution techniques, adding spike isotopes such as ^{3}He, ^{233}U or ^{235}U, and ^{229}Th or ^{230}Th, respectively.

Here, we present the wet chemical routines developed at the University of Salzburg for external calibration instead of isotope dilution ICP-MS analysis. Intensity variations of commonly 10% and sometimes up to 30% for repeated analyses of calibration solutions throughout individual, one-day analytical sessions are overcome by on-line addition of Cr and Bi internal standards. Normalizing analytes' intensities to internal standards' count rates smoothes sensitivity fluctuations and reduces these variations by about an order of magnitude, permitting an ICP-MS precision of <1% (1σ SD), similar to that obtained by established isotope dilution techniques. Accuracy of this procedure is documented by Durango and MK-1 reference apatite (U–Th)/He dates. Whereas sample preparation for external calibration analysis is slightly more delicate relative to isotope dilution techniques due to the need for full-quantitative liquid handling, our routines are easier to establish and, most importantly, independent of increasingly critical radionuclide spike supplies.