(U-Th)/He analytical procedure at Dalhousie University

Apatites for (U-Th)/He analyses were manually selected under high-magnification stereoscopic microscope. Preference was given to the euhedral, transparent, inclusion- and cracks-free grains with a smallest dimension being not less than 70 □m. All selected grains were packed in Pt tubes. Before packing grains were measured and photographed. These measurements were used to calculate an a-ejection correction (Farley et al., 1996). He measurements were completed on a home-built He extraction line equipped with 45W diode laser and Pfeiffer Vacuum Prisma quadrupole mass-spectrometer. Each Pt packet was heated using laser to release He. Apatites were heated to 1050° C for 5 minutes. After He extraction a precisely measured aliquot of 3He was added to the sample and 3He/4He ratio was measured using quadrupole mass-spectrometer. This procedure was repeated once for apatites to make sure no He is left in the grain. Typical errors are in range of 1.5-3% (1σ). Samples were analyzed in groups of 36, each group included 2 Durango apatite standards. These standards went through same analytical procedures as unknown samples to ensure accuracy, reproducibility, and reliability of the data.
After He extraction apatites were dissolved in 7N HNO3 at 80° C for 1.5 hour. Prior dissolution all samples were spiked with mixed 235U, 230Th, and 149Sm spike. Isotopic ratios were measured using iCAP Q ICP-MS.
All the raw data were reduced using Helios software package developed by R. Kislitsyn and D. Stockli specifically for (U-Th)/He data reduction. α-ejection correction was calculated based on surface to volume ratio (Farley at al., 1996).

(U-Th)/He analytical procedure at Göttingen University

Intact, euhedral, inclusion- and fissure-free apatite crystals were hand-picked and inspected under 250x magnification and cross-polarized light. To calculate the alpha ejection correction factor (Farley et al. 1996) microphotographs were taken for determining shape parameters (width, total length, and length of prismatic section). After proper documentation, each crystal was loaded in ca. 1x1 mm platinum capsules and degassed in high vacuum by heating with an infrared laser for 2 min in high vacuum. The extracted gas was purified using a SAES Ti–Zr getter kept at 450 °C. The 4He content of the purified gas extracted from the crystals was measured on a Hiden triple-filter quadrupole mass

spectrometer. He blanks were estimated using the same procedure on empty Pt tubes, and the crystals were checked for degassing of He by sequential reheating and He measurement. For the detection of the alpha-emitting elements (U, Th and Sm) the degassed crystals were spiked with calibrated ^{230}Th and ^{233}U solutions. After dissolution in a 4 % HNO_3+ 0.05 % HF acid mixture in Savillex Teflon vials, spiked solutions were analysed by a Thermo iCAP-Q ICP-MS. The ejection correction factors (Ft) were determined for the single crystals by a modified algorithm of Farley et al. (1996) using an in-house spreadsheet.

Farley, K.A.,Wolf, R.A., and Silver, L.T., 1996, The effects of long alpha-stopping distances on (U-Th)/He ages: Geochimica et Cosmochimica Acta, v. 60, p. 4223–4229.