Analytical Methods, Version 1, Little Devil's Postpile Project

Origins of Data

This document clarifies where portions of the dataset were collected, with what methods, and provides contact information for those seeking additional information about analysis procedures.

***XRF geochemical analyses of LDP basalt**:* Washington State University, via Peter Zeitler (Lehigh University). Methods reference**:** https://environment.wsu.edu/facilities/geoanalytical-lab/technical-notes/xrf-method/.

***U-Pb zircon ages for Cathedral Peak Granodiorite**:* LA/!CPMS analyses at Laserchron facility, University, via Peter Reiners (University of Arizona). Methods reference**:** https://sites.google.com/a/laserchron.org/laserchron/home.

***LDP10 U-Th/He apatite, zircon, and titanite analyses, and LDP12 U-Th/He apatite, and zircon analyses**.* Peter Reiners (University of Arizona). Methods reference: Reiners, P.W., Carlson, R.W., Renne, P.R., Cooper, K.M., Granger, D.E., McLean, N.M., and Schoene, B., 2018. Geochronology and Thermochronology, Wiley, 480 pp., ISBN: 978-1-118-45585-2

***LDP12 U-Th/He and $^{4}He/^{3}He$ apatite analyses**.* David Shuster (University of California – Berkeley). Methods reference: Tremblay, M.M., Fox, M., Schmidt, J.L., Tripathy-Lang, A., Wielicki, M.M., Harrison, T.M., Zeitler, P.K. and Shuster, D.L., 2015. Erosion in southern Tibet shut down at ~10 Ma due to enhanced rock uplift within the Himalaya. Proceedings of the National Academy of Sciences 112, 12030-12035, doi: 10.1073/pnas.1515652112.

***LDP southern contact reset U-Th/He apatite and zircon analyses**.* Daniel Stockli (University of Texas at Austin). Methods references, apatite: Lee, J.L., Stockli, D.F., Owen, L.A., Finkel, R.C., and Kislitsyn, R., 2009. Exhumation of the Inyo Mountains, California: implications for the timing of extension along the western boundary of the Basin and Range Province and distribution of dextral fault slip rates across the Eastern California shear zone. Tectonics, 28(1), TC1001, doi:10.1029/2008TC002295. Methods references, zircon: Wolfe, M.R., and Stockli, D.F., 2010. Zircon (U-Th)/He thermochronometry in the KTB drill hole, Germany, and its implications for bulk He diffusion kinetics in zircon. Earth and Planetary Science Letters, 295, 69-82, doi: 10.1016/j.epsl.2010.03.025.

***LDP $^{40}Ar/^{39}Ar$ analyses**.* Peter Zeitler (Lehigh University). Methods reference, biotite and K-feldspar: Schmidt, J.L., 2018. Revealing a Cenozoic history of landscape change and differential unroofing in the southeastern Lhasa block: Applications of thermochronometry along the Tibetan Plateau margin. Unpublished Ph.D. dissertation, Lehigh University, Bethlehem, PA, USA, 297 pp. Methods reference, whole-rock basalt: Ancuta, L.D., Zeitler, P.K., Idleman, B.D., and Jordan, B.T., 2018. Whole-rock $^{40}Ar/^{39}Ar$ geochronology, geochemistry, and stratigraphy of intraplate Cenozoic volcanic rocks, Central Mongolia. Geological Society of America Bulletin, 130 (7-8), 1397-1408, 10.1130/B31788.1.

LDP apatite fission-track analyses. Richard Ketcham (University of Texas at Austin). Methods described in this document:

Fission-track Methods

Data acquisition

The sample preparations, experiments and measurements were carried out at the Fission Track and UTChron Laboratories in the Department of Geological Sciences at the University of Texas at Austin.

Fission Track Analysis

The samples were mounted in araldite epoxy resin, the internal surfaces of the mounts were polished with Buehler TexMet and MasterTex polishing papers., using 0.3 and 0.05 µm aluminum oxide polishing solutions on Buehler 10965-00 polisher. Etching procedures were carried out by using the standard etch protocol by Carlson et al. 1999, 5.5 M HNO_3 at 21.0±0.1 °C for 20 s. The etchant temperature was measured using an MN Measurement Instruments K-type thermocouple thermometer, model DM6801A+. The samples were agitated during etching and submerged in water to stop the procedure. To remove any residual liquid within the tracks, the samples were left to dry at ~50 °C for at least an hour in a Baxter DX-41 drying oven. Grain images and coordinates were captured at 1000x magnification on a Zeiss M2m Axio-Imager microscope using TrackWorks 2.2.0 software. The fission track analysis was executed on the captured images using FastTracks v 2.2.0 software. To provide more confined fission track lengths, after image capture, track counting and laser ablation, the samples were repolished to remove the etched surface and irradiated by ^{252}Cf with a Cf track density of ~ 10^7 tracks/cm^2. Confined tracks were etched, captured and measured in the same way described above.

LA-ICP-MS Analysis

The recorded grain coordinates were transferred to the laser ablation facilities, using an in-house developed coordinate transformation spreadsheet. Ablation spots were manually verified by checking the captured images. The spots were ablated at Excimer laser ablation system with a Element2 HR-ICP-MS along with NIST612 standard for ^{44}Ca internal calibration. Finally, the data were reduced by Iolite v2.5. **Table 1** shows the details of the laser ablation process.

Table 1. Details of the laser ablation process

Laser	
Instrument	Photon Machines Analyte.G2, 193 nm wavelength
Software	Chromium2 V2013.5.8
Laser carrier gas	1.127 L/min
Washout and background	1 sec / 10000 cps for ^{43}Ca
Energy density	~2.0 J/cm^2
Laser energy	4 mJ
Spot size	20 µm
Repetition rate	10 Hz
Inductively coupled plasma mass spectrometer	

Instrument	Thermo Element2 single detector HR-ICP-MS
Software	Thermo Element v3.1.7.278
Plasma ratio frequency power	1353 W
Sample gas flow	1.127 L/min
Operating mode	Low Resolution, Mass Accuracy mode
Effective mass sweep time	1.1 sec
Total dwell time	0.67 sec
Tuning conditions	U/Th <1.2
Data reduction	
Software	Iolite 2.5
Primary standard	NIST 612
Data Reduction Scheme	Trace Elements IS
Internal Standard	^{43}Ca

Laser ablation data analysis

The laser ablation data are reduced in Iolite v2.5. The washout intervals of the final portions of the laser signals are used as baseline integration to reduce the laser signals. A standard area of interest is applied for a certain time interval for all the ablation data. NIST 612 silicate glass was used as the primary reference material. U concentrations are finally normalized to ^{43}Ca via the data reduction scheme to correct variations in ablation yield.

Zeta calibration

We used a zeta-type calibration for age determination, based on Hasebe et al. (2004) and Donelick et al. (2005) but adapted to work with data in ppm U rather than U/Ca ratios. To calculate a zeta factor we used equation (1), conducting the necessary measurements on Durango (31.4 Ma) and Fish Canyon Tuff (~27.9 Ma) apatites.

$$\zeta = \frac{(e^{t_s \lambda_D} - 1) \sum \Omega U}{\lambda_D \sum N_S}. \quad (1)$$

Symbol definitions are given in **Table 2**. Measured track densities and the U concentrations of known age standards are used to calculate the zeta factor. The zeta factor based on Durango data was used to calculate the fission track age of Fish Canyon Tuff apatites as an internal check. For this study, the measured zeta value was 1695.6 ± 48.7, based on measuring 1295 tracks.

Age equation

Single-grain and pooled ages were calculated with equation (2):

$$t = \frac{1}{\lambda_D} \ln\left(\frac{P_s \lambda_D \zeta}{U} + 1\right) \quad (2)$$

Table 2. Description of symbols used in equations 1 and 2.

Symbol	Description	Unit
t	Age	Ma
$\mathrm{P_s}$	Spontaneous track density	number/cm^2
λ_D	Total decay constant of U	Ma^{-1}
ζ	Zeta factor	-
U	^{238}U concentration measured by LA-ICP-MS	ppm, or µg/g
t_s	Age of the standard	Ma
Ω	Area counted	cm^2
N_s	Number of spontaneous tracks	-