

Data Repository Item 1: Analytical Methods used in U-Pb Geochronology

Zircon was separated from bulk rock samples by standard crushing, heavy liquid, and magnetic separation techniques, and was handpicked using a binocular microscope with selection based on clarity and crystal morphology. To minimize the effects of Pb-loss in zircon, grains were pre-treated using thermal annealing and chemical leaching (chemical abrasion or CA-TIMS technique: Mattinson, 2005) designed to preferentially remove the high-U parts of the zircon crystal that are most susceptible to Pb-loss. Annealing took place inside a furnace at 900°C for 60 hours. The annealed grains were loaded into 200µl FEP Teflon® microcapsules and leached in 29M HF at 180°C in high-pressure Parr® vessels for 12 hours. The partially dissolved sample was then transferred into 3ml Savillex® FEP beakers, fluxed with 4N HNO₃ on a hot plate, placed in an ultrasonic bath, and thoroughly rinsed with several millilitres of ultra-pure water. The grains were then loaded back into their microcapsules, spiked with a mixed ²⁰⁵Pb-²³³U-²³⁵U tracer solution, and dissolved completely in 29M HF at 210°C for 48 hours.

Dissolved Pb and U were chemically separated using a miniaturized HCl-based ion-exchange chromatography procedure modified after Krogh (1973), using 50 ml columns of AG1x8 anion-exchange resin. The FEP dissolution vessels were cleaned in between analyses in four consecutive steps using concentrated HF and 6N HCl solutions at dissolution temperatures over a period of four days.

Both Pb and U were loaded with a silica gel - H₃PO₄ emitter solution (Gerstenberger and Haase, 1997) on single degassed Re filaments and their isotopic compositions were measured on the VG Sector 54 multi-collector thermal ionization mass spectrometer at MIT. Lead isotopic measurements were made in a peak-switching mode by ion counting using a Daly photomultiplier detector with a ²⁰⁶Pb ion beam intensity of 0.5 to 2.0 x 10⁻¹³ amps usually maintained during data acquisition. Uranium isotopes were measured as oxide ions on three Faraday detectors in a static mode with an average ²³⁵U¹⁶O₂⁺ ion-beam intensity of 1.0 x 10⁻¹² Amps.

Measured isotopic ratios were corrected for mass-dependant isotope fractionation in the mass spectrometer, as well as for U and Pb contributions from the spike and laboratory procedural blanks.

References

- Gerstenberger, H. and Haase, G., 1997, A highly effective emitter substance for mass spectrometric Pb isotope ratio determinations: *Chemical Geology*: v.136, n. 3-4, p. 309-312.
- Jaffey, A.H., Flynn, K.F., Glendenin, L.E., Bentley, W.C. and Essling, A.M., 1971, Precision measurements of half-lives and specific activities of ²³⁵U and ²³⁸U. *Physical Reviews C*, v. 4, p. 1889-1906.
- Krogh, T. E., 1973, A low contamination method for hydrothermal decomposition of zircon and extraction of U and Pb for isotopic age determination: *Geochimica et Cosmochimica Acta*, v. 37, p. 485-494.
- Ludwig, K.R., 1980, Calculation of uncertainties of U-Pb isotopic age data: *Earth and Planetary Science Letters*, v. 46, p. 212-220.
- Mattinson, J.M., 2005, Zircon U-Pb chemical abrasion (CA-TIMS) method: Combined annealing and multi-step partial dissolution analysis for improved precision and accuracy of zircon ages: *Chemical Geology*, v. 220 p. 47-66.

Table DR1. U-Pb isotopic data.

Sample	$\frac{\text{Pb}^*}{\text{Pb}_c}$	Pb_c (pg)	$\frac{\text{Th}}{\text{U}}$	Isotopic ratios									Dates (Ma)						
				$\frac{^{206}\text{Pb}}{^{204}\text{Pb}}$	$\frac{^{208}\text{Pb}}{^{206}\text{Pb}}$	$\frac{^{206}\text{Pb}}{^{238}\text{U}}$	% err	$\frac{^{207}\text{Pb}}{^{235}\text{U}}$	% err	$\frac{^{207}\text{Pb}}{^{206}\text{Pb}}$	% err	corr. coef.	$\frac{^{206}\text{Pb}}{^{238}\text{U}}$	±	$\frac{^{207}\text{Pb}}{^{235}\text{U}}$	±	$\frac{^{207}\text{Pb}}{^{206}\text{Pb}}$	±	% disc.
(a)	(b)	(c)	(d)	(e)	(f)	(f)	(g)	(f)	(g)	(f)	(g)		(h)	(i)	(h)	(i)	(h)	(i)	(j)
z1	58	0.91	0.57	3485	0.183	0.036486	0.08	0.255849	0.14	0.050857	0.11	0.60	231.02	0.18	231.32	0.29	234.40	2.58	1.44
z3	108	0.44	0.59	6396	0.188	0.036479	0.06	0.255540	0.12	0.050806	0.10	0.50	230.97	0.13	231.07	0.25	232.00	2.37	0.44
z4	75	0.86	0.61	4441	0.196	0.036467	0.06	0.255689	0.12	0.050852	0.09	0.58	230.90	0.15	231.19	0.24	234.16	2.19	1.40
z5	27	0.42	0.40	1689	0.130	0.036480	0.13	0.255985	0.29	0.050893	0.24	0.53	230.98	0.30	231.43	0.59	236.00	5.62	2.13
z6	38	0.56	0.58	2258	0.189	0.036466	0.09	0.255986	0.23	0.050913	0.20	0.48	230.89	0.21	231.43	0.47	236.88	4.65	2.53
z10	135	0.41	0.62	7928	0.198	0.036466	0.05	0.255561	0.08	0.050828	0.07	0.63	230.89	0.12	231.08	0.17	233.04	1.51	0.92
z11	49	0.33	0.47	3032	0.152	0.036461	0.08	0.255774	0.16	0.050878	0.14	0.51	230.86	0.17	231.26	0.34	235.28	3.22	1.88
z12	57	0.88	0.64	3377	0.203	0.036453	0.07	0.255332	0.10	0.050800	0.07	0.70	230.81	0.16	230.90	0.21	231.76	1.70	0.41

(a) z1, z2 etc. are single grains of zircon; (b) Ratio of radiogenic Pb to common Pb; (c) Total weight of common Pb; (d) Model Th/U ratio calculated from radiogenic $^{208}\text{Pb}/^{206}\text{Pb}$ ratio and $^{207}\text{Pb}/^{206}\text{Pb}$ age; (e) Measured ratio corrected for spike and fractionation only. Mass fractionation corrections were based on analysis of NBS-981 and NBS-983. Corrections of $0.25 \pm 0.04\%$ /amu (atomic mass unit) were applied to single-collector Pb Daly analyses; (f) Corrected for fractionation, spike, blank, and initial common Pb. All common Pb was assumed to be procedural blank; (g) Errors are 2σ , propagated using the algorithms of Ludwig (1980); (h) Calculations are based on the decay constants of Jaffey et al. (1971); (i) Errors are 2σ ; (j) % discordance = $100 - (100 \times ^{206}\text{Pb}/^{238}\text{U} \text{ date} / ^{207}\text{Pb}/^{206}\text{Pb} \text{ date})$.