

Evolution of the Lewisian Complex: Geochronological Data

Analytical Methods

Monazite ICP-MS

Around 200 µg of crack- and inclusion-free air-abraded monazite crystals were hand picked and divided into five roughly equal fractions (the individual fractions were not weighed) which were repeatedly cleaned in an ultrasonic bath in dilute HNO₃, then ultrapure H₂O. These were then transferred into clean 2 ml Savillex beakers to which a few drops of ultrapure H₂SO₄ was added, and then heated for several hours at ~220°C until dissolved. Dissolution was confirmed by examining the solutions under a microscope. Each fraction was then split into two roughly equal aliquots to one of which a ²⁰⁴Pb-²³⁶U tracer prepared at the University of Oxford was added and allowed to equilibrate. Both aliquots were dried down and redissolved in 2% HNO₃ for analysis. Because of the comparatively high U and radiogenic Pb content expected, preconcentration was not carried out, and the dissolved monazite was diluted to ~20 µg monazite/ml solution and measured directly on a Nu Plasma ICP-MS at the University of Oxford.

For the spiked aliquots, Pb and U were measured in the same analysis in alternate static steps with all beams measured on Faraday collectors. ²⁰²Hg and ²⁰⁰Hg were also measured to allow for online ²⁰⁴Hg correction. Analyses comprised 15 cycles of alternating 10s integrations measured on U and Pb steps. Instrumental mass fractionation was estimated using bracketing U (CRM145) and Pb (SRM983) standards analysed twice between each sample. Pb in the unspiked aliquots was measured in a similar way but using ion counters for ²⁰⁴Pb, ²⁰²Hg, and ²⁰⁰Hg. Both mass fractionation and ion counter gain on the ²⁰⁴Pb were corrected by standard bracketing. The procedural blank was ~0.5 pg for Pb and ~0.06 pg for U.

Natural ²⁰⁴Pb in the spiked aliquots was corrected for using the ²⁰⁶Pb/²⁰⁴Pb measured in the unspiked cuts. Likewise the total non-radiogenic Pb was corrected for using the unspiked ²⁰⁶Pb/²⁰⁴Pb ratio and an assumed modern day Stacey & Kramers (1975) composition. The analyses were all radiogenic, and insensitive to the choice of ²⁰⁶Pb/²⁰⁴Pb and ²⁰⁷Pb/²⁰⁴Pb ratios for the non-radiogenic component, hence no separate blank correction has been made.

The uncertainty in the ²⁰⁷Pb/²⁰⁶Pb has been estimated from the reproducibility of the unspiked versus the spiked (after correction for minor isotopes in the spike) measurements (the reproducibility of the bracketing SRM983 standards gave an unrealistically small uncertainty of ~180 ppm), plus a 50% uncertainty on the non-radiogenic Pb correction. Uncertainties on the ²⁰⁶Pb/²³⁸U are estimated as 50% of the total correction (i.e. mass fractionation correction plus non-radiogenic Pb correction). The uncertainty associated with the spike calibration is not propagated and is 1.1 permil (95%) on the critical ²³⁶U/²⁰⁴Pb ratio.

The data were treated using Isoplot (Ludwig 2003) and the decay constants of Steiger & Jaeger (1977).

Zircon TIMS

Sample preparation, analysis and data reduction follows well documented procedures (Parrish 1987; Parrish *et al.* 1987; Noble *et al.* 1993) and has not been notably modified here. Measurements were carried out on a VG354 multicollector TIMS instrument at the NERC Isotope Geology Laboratory (NIGL), Keyworth, UK. Pb and U were measured on the same filament, mostly in mixed Daly – Faraday measurements. Daly gain was determined internally by peak jumping a beam that could be measured both on the Daly and Faraday collector. Mass fractionation was corrected using typical fractionation values determined by measuring standards. U-Pb ratios were calculated with reference to a $^{235}\text{U}/^{205}\text{Pb}$ tracer calibrated to ~1 permil uncertainty.

Blanks range from 2.1 to 7.1 pg for Pb, and 0.1 to 0.8 pg for U and have been corrected for. Common Pb has been corrected using the Stacey & Kramers (1975) model, but the analyses contain >99% radiogenic ^{206}Pb so this correction is small. The decay constants of Steiger & Jaeger (1977) were used.

Isotopic data

Isotopic data are summarised in Table 1. Details of the analysed samples are given in the main text.

References

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Table 1. U-Pb data

Fraction No. ^a	Fraction weight (mg)	U conc. (ppm)	Pb conc. (ppm)	$^{206}\text{Pb}/^{204}\text{Pb}$	% non-rad. $^{206}\text{Pb}^c$	Th/ U^d	$^{206}\text{Pb}/^{238}\text{U}^e$	$^{207}\text{Pb}/^{235}\text{U}^e$	$^{207}\text{Pb}/^{206}\text{Pb}^e$	$^{206}\text{Pb}/^{238}\text{U}$ age (Ma) ^f	$^{207}\text{Pb}/^{235}\text{U}$ age (Ma) ^f	$^{207}\text{Pb}/^{206}\text{Pb}$ age (Ma) ^f	Correlation coefficient	% discordance ^g
OH04-3 Ness pelite [NB 5585 6108]^h - monazite														
27-1(M,A,I)	-	-	-	59382	0.031	-	0.3293 ± 0.09	5.169 ± 0.3	0.1138 ± 0.09	1835 ± 3	1848 ± 6	1861 ± 3	0.90	1.6
27-3(M,A,I)	-	-	-	207000	0.009	-	0.3426 ± 0.08	5.506 ± 0.3	0.1166 ± 0.05	1899 ± 3	1901 ± 5	1904 ± 2	0.90	0.3
27-4(M,A,I)	-	-	-	29053	0.064	-	0.3156 ± 0.09	4.852 ± 0.4	0.1115 ± 0.14	1768 ± 3	1794 ± 6	1824 ± 5	0.90	3.5
27-5(M,A,I)	-	-	-	59453	0.031	-	0.3394 ± 0.09	5.418 ± 0.3	0.1158 ± 0.08	1884 ± 3	1888 ± 6	1892 ± 3	0.90	0.5
SH01-64 - Langavat Pegmatite [NG 0792 8639]^h - zircon														
Z26 (M,U,P)	0.0123	500.6	165.8	13950	0.13	0.36	0.3163 ± 0.15	4.906 ± 0.1	0.1125 ± 0.11	1772 ± 5	1803 ± 2	1840 ± 4	0.70	4.2
Z27 (M,U,P)	0.0144	1271.0	411.8	33590	0.055	0.36	0.3088 ± 0.12	4.85 ± 0.1	0.1139 ± 0.03	1735 ± 4	1794 ± 2	1862 ± 1	0.97	7.8
Z110 (U,P)	-	-	-	2556	0.73	0.18	0.3104 ± 0.15	4.572 ± 0.2	0.1068 ± 0.15	1743 ± 5	1744 ± 3	1746 ± 6	0.65	0.2
Z111 (U,P)	-	-	-	6319	0.3	0.52	0.335 ± 0.12	5.311 ± 0.1	0.115 ± 0.07	1862 ± 4	1871 ± 2	1880 ± 3	0.83	1.1
Z112 (A,I)	0.0108	116.4	41.2	9027	0.21	0.52	0.3258 ± 0.11	5.017 ± 0.1	0.1117 ± 0.05	1818 ± 4	1822 ± 2	1827 ± 2	0.92	0.6
Z113 (A,I)	0.0055	112.9	43.3	3469	0.54	0.72	0.3368 ± 0.23	5.321 ± 0.2	0.1146 ± 0.11	1871 ± 7	1872 ± 4	1873 ± 4	0.89	0.1

^aM=multigrain analysis, U=euhedral, A=anhedral, P=prismatic, I=irregular

^bCorrected for spike contribution and instrumental bias

^cEstimated percentage of non-radiogenic lead in analysis, including blank.

^dDetermined based on measured $^{208}\text{Pb}/^{206}\text{Pb}$

^eDetermined ratios corrected for instrumental biases, spike, blank, and common lead. Errors are 1 sigma in percent.

^fErrors are 2 sigma in Ma, decay constants used are those recommended by Steiger and Jaeger (1977)

^gDiscordance calculated with respect to the origin

^hLocations given with respect to the U.K. Ordnance Survey grid system.