

Data repository Item

U-Pb isotope, Lu-Hf isotope and trace element data for zircons of the Narraburra Complex, southeastern Australia

1. U-Pb isotope SIMS analysis of zircons

The U-Pb data was obtained with a Cameca ims1270 ion microprobe housed at the Swedish Museum of Natural History, Stockholm. Operating conditions and analytical protocols are essentially the same as described by Whitehouse et al. (1999). The Pb/U calibration was performed relative to the Geostandards zircon 91500, which was analysed repeatedly throughout each session. Analyses of the Temora 2 standard were also interspersed throughout the session to monitor data quality, and 16 analyses yielded a concordia age of 418.0 ± 3.0 Ma (2σ), indistinguishable to that quoted by Black *et al.* (2003). Isotope ratios in Table 1 are not corrected for common Pb, and apparent magmatic ages include uncertainty in the Pb/U calibration. ' ^{207}Pb corr' represents the $^{206}\text{Pb}/^{238}\text{U}$ age corrected for common lead contamination by the ^{207}Pb method, whereby each analysis is extrapolated to concordia along a mixing line projected from the assumed modern terrestrial lead composition of Stacey and Kramers (1975) ($^{207}\text{Pb}/^{206}\text{Pb} = 0.83$, with an arbitrary error of ± 0.1). Intercept and concordia ages were computed by Isoplot (Ludwig, 2001). Errors are quoted at 1σ precision.

2. Lu-Hf isotope analysis of zircons by laser ablation

All Hf isotope analyses were conducted in the Department of Earth Sciences, University of Bristol, using a Finnigan Neptune multi-collector ICP-MS and 193 nm ArF laser operating at a 4 Hz laser pulse repetition rate. The power density at the sample was around 6-7 mJ/cm², which translated into an estimated drilling rate of between 0.5-1 $\mu\text{m/sec}$. The Faraday cup configuration is the same as used by Woodhead et al. (2004). Ablation was conducted in He, this being combined with argon in a small glass mixing chamber prior to transport into the ICP. A small N₂ flow was introduced into the Ar carrier gas upstream of the mixing chamber, which suppressed oxide formation in the plasma and enhanced sensitivity by a factor of two. Subsequent tests on pure REE solutions have established that formation of nitride molecules is negligible. Final quoted $^{176}\text{Hf}/^{177}\text{Hf}$ ratios were derived by integration over a 60 second ablation period. In cases of isotopic zoning (as in the Barmedman granite zircons) and with intersecting cracks/inclusions, only the flattest, most stable portions of the time-resolved signal were selected for integration.

The single most critical factor in obtaining accurate $^{176}\text{Hf}/^{177}\text{Hf}$ ratios by laser ablation concerns the ability to correct for the isobaric interference of the rare earth elements Lu and

Yb on ^{176}Hf . The latter may exceed 300 000 ppm on mass 176, depending on the REE content of the analysed zircon. This is particularly critical for zircons of the Barmedman Granite, which have elevated Yb/Hf ratios (Table DR2). To interpret Hf isotope data with confidence it is therefore imperative to establish the veracity of the interference correction over the range of $^{176}\text{Yb}/^{177}\text{Hf}$ ratios encountered in these zircons. The overlap correction is performed by in a similar fashion to that advocated by Woodhead *et al.* (2004), by measuring the interference-free ^{171}Yb during the analysis and then calculating the magnitude of the ^{176}Yb interference using the 'known' $^{176}\text{Yb}/^{171}\text{Yb}$ ratios, in this case that independently determined by Segal *et al.* (2003) ($^{176}\text{Yb}/^{171}\text{Yb} = 0.897145$). The Lu correction is performed in the same fashion by monitoring ^{175}Lu , and using $^{176}\text{Lu}/^{175}\text{Lu} = 0.02669$ (De Bievre and Taylor, 1993). To correct for instrumental mass fractionation, Yb isotope ratios were normalised to $^{173}\text{Yb}/^{171}\text{Yb} = 1.130172$ (Segal *et al.* 2003) and Hf isotope ratios to $^{179}\text{Hf}/^{177}\text{Hf} = 0.7325$. The mass bias behaviour of Lu was assumed to follow that of Yb. This protocol was tested by replicate analysis of a solution of known Hf isotope composition (JMC 475, $^{176}\text{Hf}/^{177}\text{Hf} = 0.282152 \pm 3$, $n=16$, 1σ) that was spiked with variable amounts of pure Yb. Figure A1 shows that the interference correction using the Yb isotope compositions of Segal *et al.* (2003) yields accurate Hf isotope compositions over a large range of Yb/Hf ratios that exceeds that of the Narraburra complex zircons, though precision degrades somewhat as the size of the correction increases. This effect is responsible for the greater uncertainty of zircon analyses from the Barmedman granite. Any correlation between $^{176}\text{Hf}/^{177}\text{Hf}$ and Yb/Hf (or Lu/Hf) is ascribed a geological origin.

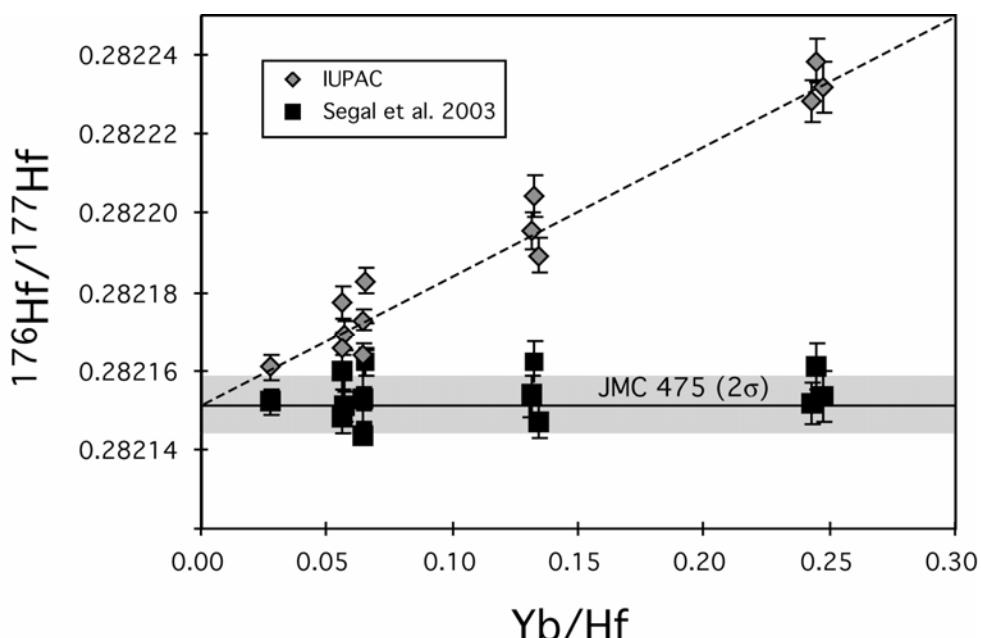


Figure DR1. Hf isotope data for solutions of JMC475 doped with Yb.

Table DR3 summarises the mean interference-corrected $^{176}\text{Hf}/^{177}\text{Hf}$ ratios of the zircon standards utilised at Bristol, measured over a nine-month period, and during the course of this study. These values are indistinguishable to those obtained by solution analysis by Woodhead et al. (in press), and as quoted by Ping et al. (2004) using the same instrumentation.

Standard	$^{176}\text{Hf}/^{177}\text{Hf}$ meas (laser)	$^{176}\text{Hf}/^{177}\text{Hf}$ meas (solution)
91500	$0.282299 \pm 13^*$ (n = 71)	0.282306 ± 4
Temora 2	0.282680 ± 15 (n = 110)	0.282686 ± 4
Mud Tank	0.282487 ± 9 (n = 34)	0.282507 ± 3
CZ3	0.281697 ± 6 (n=5)	0.281703 ± 12 (n=16) [#]

* all data are quoted at 1 standard deviation

[#] laser ablation data from Ping et al. (2004)

3. Trace element analysis of zircons

Trace element analyses were performed by a 266nm solid-state laser and PlasmaQuad II ICP-MS at the University of Bristol using a 25 μm beam size and 5 Hz repetition rate. Data collection involved a 60 second acquisition time followed by 2.5 minute wash-out, to ensure that count-rates dropped to background levels before commencing the next analysis. Isotopes of Ba, Ca and P were monitored to screen for the effects of inclusions and contamination by secondary material in cracks, and those analyses showing unacceptably high count rates of these were either truncated to omit the contaminated portion of the signal or rejected. Apatite inclusions posed the most problem for zircons of the Barmedman Granite. Data were calibrated according to the measured count-rates for each element yielded by NIST610 under the same analytical conditions (bracketing 10-12 unknown analyses) and using electron microprobe data for Hf as the internal standard in the unknowns. Analyses of NIST612 and standard zircon 91500 were interspersed as a secondary check on data quality.

Additional References:-

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- Segal I, Halicz L and Platzner IT 2003. Accurate isotope ratio measurements of ytterbium by multi-collector inductively coupled plasma mass spectrometry applying erbium and hafnium in an improved double external normalisation procedure. J Anal At Spectrom 18: 1217-1223.
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- Woodhead JD, Hergt J M, Shelley M, Eggins S, & Kemp R 2004. Zircon Hf-isotope analysis with an excimer laser, depth profiling, ablation of complex geometries, and concomitant age estimation. Chemical Geology 209: 121-135

Table DR1. U-Pb isotope data of zircons from the Narraburra Granite.

Analysis	Apparent Ages (Ma)														
	[U] ppm	[Th] ppm	[Pb] ppm	Th/U	f ₂₀₆ % [†]	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb/ ²⁰⁶ Pb	±	²⁰⁷ Pb/ ²⁰⁶ Pb	±	²⁰⁶ Pb/ ²³⁸ U	±	²⁰⁷ Pb corr	±
1a	419	96	28	0.229	0.03	0.05343	0.0008	0.05900	0.0008	347.1	20.1	369.5	4.7	369.8	9.4
1b	489	74	32	0.151	0.13	0.05449	0.0008	0.05830	0.0008	391.2	16.7	365.3	4.6	365.0	9.3
2	714	185	52	0.260	0.04	0.05346	0.0008	0.06350	0.0008	348.4	19.7	396.9	5.0	395.6	10.1
3	548	216	40	0.395	0.04	0.05369	0.0008	0.06102	0.0008	358.0	17.9	381.8	4.8	382.1	9.7
4	968	477	66	0.493	15.75	0.05287	0.0008	0.05837	0.0008	323.1	165.3	365.7	4.7	309.0	10.3
5	383	69	26	0.181	0.03	0.05388	0.0008	0.06002	0.0008	366.2	24.7	375.8	4.7	375.9	9.5
6	1021	230	65	0.225	0.03	0.05438	0.0009	0.05683	0.0009	387.0	16.3	356.3	5.3	356.0	10.6
7	1789	883	138	0.493	0.06	0.05372	0.0009	0.06025	0.0009	359.3	14.2	400.3	5.9	400.6	12.0
8	1381	209	91	0.151	0.12	0.05333	0.0009	0.06006	0.0009	342.9	16.1	376.0	5.6	375.9	11.3
9	1977	486	132	0.246	0.02	0.05417	0.0009	0.05907	0.0009	377.9	11.7	369.9	5.4	369.8	11.0
10	753	221	49	0.293	0.06	0.05414	0.0009	0.05636	0.0009	376.8	20.1	353.5	5.2	353.2	10.6
11	1449	455	106	0.314	0.37	0.05695	0.0010	0.06353	0.0010	373.1	20.9	397.0	5.8	395.9	11.8
12	156	17	10	0.108	0.39	0.05515	0.0010	0.05857	0.0010	418.2	47.4	366.9	6.0	366.4	12.2
13	586	96	38	0.164	0.09	0.05408	0.0009	0.05930	0.0009	374.5	23.7	371.3	5.5	371.3	11.1
15	560	135	38	0.242	0.09	0.05445	0.0008	0.06086	0.0008	389.7	23.1	380.9	4.8	380.7	9.7
16	3006	662	210	0.220	0.03	0.05422	0.0008	0.06215	0.0008	380.2	10.3	388.7	4.9	388.7	9.9
17	641	122	43	0.190	0.04	0.05423	0.0008	0.05988	0.0008	380.4	21.9	374.9	4.7	374.8	9.6
18	580	307	40	0.530	0.09	0.05407	0.0007	0.05587	0.0007	374.1	23.3	350.5	4.4	350.2	9.0
19	425	93	28	0.219	0.03	0.05460	0.0008	0.05956	0.0008	395.7	27.2	373.0	4.8	372.7	9.7
20	1140	245	77	0.215	0.03	0.05368	0.0008	0.06036	0.0008	357.8	16.1	377.8	4.8	378.1	9.6

T178-8	0.282848	0.000009	0.002875	0.000037	0.282827	10.49	0.181	21.5
T178-9a#	0.282848	0.000009	0.000815	0.000006	0.282842	11.01	0.049	13.2
T178-9b	0.282864	0.000007	0.001156	0.000015	0.282856	11.49	0.067	23.0
							5001	823.6
							103.2	8567
							513.2	754.8
							0.680	0.584

Barmedman Granite (170)

Table DR2. Lu-Hf isotope and selected trace element data from zircons of the Narraburra Granite and Barmedman Granite. All Hf isotope analysis were conducted with a 50 μm beam size except for those marked with an * (65 μm) or # (40 μm). ΣHf values were calculated at 375 Ma, using the chondritic values of Blichert-Toft and Albarede (1997). Analytical uncertainties are at 1 s.e.