

Detrital zircon age and provenance constraints on late Paleozoic ice-sheet growth and dynamics in western and central Australia

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SUPPLEMENTARY PAPERS

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Supplementary papers

Appendix 1. Zircon dating methodology.

Table S1. Heavy mineral count for detrital zircon samples with cored petroleum well/sample name (DRO: Drosera-1, CAP: Capparis-1, CYC: Cycas-1), depth of sample (m), number, N, of grains counted, and heavy mineral percentage of total. An, anatase; Ap, apatite; Bi, biotite; Cd, chloritoid; Ct, chlorite; Ep, epidote; Ga, garnet; Mo, monazite; Ru, rutile; Sp, sphene; To, tourmaline; Zr, zircon. (excel file)

Table S2. All Grant Group detrital zircon U–Pb laser ablation MC-ICP-MS analyses. (excel file)

Table S3. U–Pb laser ablation MC-ICP-MS analyses of two youngest detrital zircons from sample CAP. (excel file)

Table S4. Hf-isotope laser ablation MC-ICP-MS analyses of two youngest detrital zircons from sample CAP. (excel file)

Table S5. All Grant Group detrital zircon Hf-isotope laser ablation MC-ICP-MS analyses. (excel file)

Appendix 1. Zircon dating methodology

Samples were crushed, sieved, and separated using gravitational and centrifugal heavy liquid techniques. Zircons were further separated from the heavy mineral fraction using Frantz magnetic techniques, hand picked randomly under a binocular microscope, mounted on an epoxy block and polished to expose an equatorial section. Every grain was imaged using cathodoluminescence (CL) to identify compositional zonation and the ablation target area. U–Pb ages were determined using an Elemental Axiom double focussing MC-ICPMS coupled to a New Wave Research Microprobe UP193SS nm, Nd:YAG laser ablation system, at the Natural Environment Research Council Isotope Geosciences Laboratory (NIGL), UK. Analytical procedure for U–Pb dating using the Axiom is fully outlined in Horstwood *et al.* (2003). A minimum of 60 U–Pb ages was determined for each sample (Table S2); analysis of 60 grains provides a 95% probability of finding a population comprising 5% of the total (Dodson *et al.*, 1988). Spot size and laser repetition rate were maintained at 50 μm and 5 Hz generating $\sim 2.6\text{--}3.4\text{ J.cm}^{-2}$ per pulse, with ablated material transferred from the sample cell to the Axiom plasma source by argon. Elemental fractionation was minimised by adopting standard protocol such as ablating pits with a low aspect ratio and calculating Pb/U ratios with reference to standard zircon 91500, which was analysed after every fifth detrital zircon analysis. Ages were calculated using the Isoplot add-in (Ludwig, 2003) for Microsoft Excel using the decay constants recommended by Steiger and Jäger (1977). Ages quoted in this paper are $^{206}\text{Pb}^*/^{238}\text{U}$ for $< 1000\text{ Ma}$ and $^{207}\text{Pb}^*/^{206}\text{Pb}^*$ for $> 1000\text{ Ma}$, with 2σ error (Figures 8 and 9). The ^{204}Pb signal for each analysis was used to assess the amount of common Pb (Horstwood *et al.*, 2003), with correction applied as necessary.

Hf isotope analyses were performed (Table S5) after U–Pb geochronology using a Nu Plasma HR (Nu Instruments) MC-ICP-MS coupled to a UP193SS (New Wave Research) laser ablation system. Ablations were targeted adjacent to the U–Pb ablation spots where possible using CL images. Similar zones within the same grains were targeted for Hf analyses placement immediately adjacent to the pre-existing U–Pb ablation pit was not possible (e.g. Figure 5). The analytical methodology was modified after Woodhead *et al.* (2004) to allow for the use of the U–Pb collector block on the Nu Plasma HR at NIGL. This collector block is limited to 7 central Faraday detectors for use with isotope systems other than U–Pb, necessitating the sacrifice of the ^{180}Hf and ^{172}Yb peaks. Only the $^{178}\text{Hf}/^{177}\text{Hf}$ stable isotope ratio is therefore used to monitor the accuracy of the Hf mass bias correction, whilst a modified $^{176}\text{Yb}/^{173}\text{Yb}$ ratio is determined prior to analysis through Yb-doping of the JMC475 Hf isotope reference material. This characterises the difference between Hf and Yb instrumental mass bias and allows correction of the ^{176}Yb isobaric interference through measurement of the ^{173}Yb peak, without the need to measure both the ^{172}Yb and ^{173}Yb peaks simultaneously. Peak jumping experiments to allow simultaneous Yb mass bias correction of the Yb isobaric interference correction show no difference within uncertainty to those data determined using the doping approach. Monitoring of the $^{180}\text{Hf}/^{177}\text{Hf}$ ratio through a dynamic acquisition also demonstrates accurate stable isotope values.

Mass spectrometer performance was checked each session by running reference material JMC475 with and without Yb doping. Session averages for corrected $^{176}\text{Hf}/^{177}\text{Hf}$ and $^{178}\text{Hf}/^{177}\text{Hf}$ were $0.282148\text{--}63 \pm 40\text{--}110\text{ ppm}$ and $1.467210\text{--}38 \pm 10\text{--}32\text{ ppm}$ 2SD, respectively. Laser spot size and repetition rate were maintained at 50 μm and 5 Hz, respectively, using a fluence of $6\text{--}8\text{ J.cm}^{-2}$ per pulse. Sample data were normalised relative to reference material 91500 assuming $^{176}\text{Hf}/^{177}\text{Hf} = 0.282306$ (Woodhead *et al.*, 2004). $^{178}\text{Hf}/^{177}\text{Hf}$ for 91500 gave session averages of $1.46724\text{--}27$ with average reproducibility of $\sim 35\text{ ppm}$ 2SD.

Interferences on ^{176}Hf were corrected by measuring ^{173}Yb and ^{175}Lu and using $^{176}\text{Yb}/^{173}\text{Yb} = 0.79462$ and $^{176}\text{Lu}/^{175}\text{Lu} = 0.02653$. Lu–Hf sample data were normalised using reference material 91500 relative to an expected $^{176}\text{Lu}/^{177}\text{Hf}$ of 0.000311 (Woodhead *et al.*, 2004).

All uncertainties were propagated using quadratic addition to reflect the reproducibility of replicate measurements of the Mudtank reference material with the εHf and T_{DM} uncertainties incorporating components to reflect the uncertainty on the determined $^{176}\text{Hf}/^{177}\text{Hf}$, $^{176}\text{Yb}/^{177}\text{Hf}$ and $^{176}\text{Lu}/^{177}\text{Hf}$ ratios

as well as the uncertainty on the U–Pb age. The ^{176}Lu decay constant and chondrite and depleted mantle values of Blitchert-Toft and Albarede (1997) were used to calculate ε_{Hf} and T_{DM} model ages.

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