

Mid- to lower-crustal architecture of the northern Lachlan and southern Thomson orogens: evidence from O–Hf isotopes

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Supplementary papers

Appendix A. Sample selection and preparation, analytical methods and data procession.

Table A1. Lu–Hf session information (Excel file).

Table A2. Oxygen isotope session information (Excel file).

Table A3. Lu–Hf reference results (Excel file).

Table A4. Oxygen isotope reference results (Excel file).

Table A5. Lu–Hf and oxygen isotope sample results (Excel file).

Appendix A Supplementary Information

Sample collection and preparation

The locations of field sites are reported as decimal latitude and longitude and refer to the Geocentric Datum of Australia 1994 (GDA94). Site locations are labelled using relevant identifiers (GA SampleNo) in Geoscience Australia databases. Samples are located using hand-held GPS units, and are accurate to about 100 m. Sample preparation methods (sampling, crushing and zircon separation) were based on those documented by Chisholm, Sircombe and DiBugnara (2014), and mount preparation methods were based on DiBugnara (2016).

Isotopic data acquisition and processing

Isotopic data acquisition

Data acquisition and reduction procedures for SHRIMP U–Pb analyses are detailed in the publications from which those datasets were sourced (cited in Table 1 of the main manuscript). Following the selection of samples for O-isotope and Lu–Hf analyses, O-isotope analyses were conducted first, using either SHRIMP II or SHRIMP SI at the Research School of Earth Sciences (RSES) at the Australian National University (ANU). Once O-isotope analyses were completed on each mount, Lu–Hf analyses were undertaken via Multiple Collector Laser Ablation Inductively Coupled Plasma Mass Spectrometry (MC-LA-ICPMS), also at the RSES, ANU. For each sample, the O-isotope and Lu–Hf analyses targeted the same zircons (and growth domains) originally selected for U–Pb SHRIMP analyses, to maximise comparability of the three datasets. Directly comparable analyses utilised equivalent spot labels across the three techniques.

Summaries of relevant analytical session information for Lu–Hf and O-isotope analyses are presented in Tables A1 and A2, respectively. Full tables of the session-specific suites of reference zircon analyses are presented in Table A3 (Lu–Hf) and Table A4 (O-isotopes). Finally, the full set of Lu–Hf and O-isotopic analyses on the 25 samples analysed during this study is presented in Table A5.

Calculation of weighted means and their 95% confidence intervals

Our data reduction procedures make extensive use of weighted mean values derived from the pooling of multiple individual analyses, with each individual analysis weighted inversely proportional to its variance. Each weighted mean has an associated Mean Square of Weighted Deviates (MSWD) value, which is a measure of the degree of scatter of the constituent analyses relative to their assigned uncertainties (Ludwig, 2003), and a probability of equivalence (p) value, which is the probability that all of the constituent analyses are equivalent within their uncertainties. By convention, scatter beyond the assigned uncertainties is assumed to be present when the probability of equivalence is less than 0.05.

Uncertainties of weighted mean values are expressed as 95% confidence intervals, which are defined herein as the 1σ uncertainty of the mean multiplied by Student's *t* for *n*–1 degrees of freedom (where *n* is the number of constituent analyses), for weighted means with MSWD values less than or equal to 1. In cases where the MSWD value exceeds 1, the implied dispersion of the data-points beyond their analytical uncertainties is acknowledged by expanding the 95% confidence interval of the mean, via multiplication by the square root of the MSWD (Ludwig, 2003).

Lu–Hf data processing

During each of our eight Lu–Hf analytical sessions, we collected a suite of analyses from each of five reference zircons: the primary reference (FC1) and four secondary references (91500, Mud Tank, Plešovice, and QGNG). Woodhead and Hergt (2005) established reference present-day $^{176}\text{Hf}/^{177}\text{Hf}$ values for four of these, via solution MC-ICPMS analysis: FC1 = 0.282184, 91500 = 0.282306, Mud Tank = 0.282507, and QGNG = 0.281612. Sláma et al. (2008) established the reference value for Plešovice, via combined laser ablation and solution MC-ICPMS analysis = 0.282482. A total of 818 reference zircon analyses were collected across 40 datasets (one per reference zircon per analytical

session), and just four analyses were excluded from their constituent populations on the basis of poor quality (Table A1; Table A3).

In order to assess the potential for session-specific instrumental mass bias in the measured $^{176}\text{Hf}/^{177}\text{Hf}$ values, we calculated an inverse-variance weighted mean $^{176}\text{Hf}/^{177}\text{Hf}$ for each population of reference zircon (RZ) analyses in each session (Table A1). For each population, the mass bias is expressed as a deviation from the solution MC-ICPMS value:

$$[\text{Mass bias factor}]_{\text{RZ}} = [\text{Weighted mean } ^{176}\text{Hf}/^{177}\text{Hf}]_{\text{RZ}}^{\text{session}} - [\text{Reference } ^{176}\text{Hf}/^{177}\text{Hf}]_{\text{RZ}}^{\text{solution}}$$

The mass bias factor for each session (Table A1) was calculated as a ‘grand’ weighted mean of the five RZ-specific mass bias factors for that session, with the 95% confidence interval of each RZ-specific mass bias factor being the same as the 95% confidence interval of the underlying weighted mean $^{176}\text{Hf}/^{177}\text{Hf}$ population.

If the 95% confidence interval of the ‘grand’ weighted mean mass bias factor for the session encompassed zero, no mass bias correction was applied to analyses of sample zircons collected during that analytical session (i.e. the individual $^{176}\text{Hf}/^{177}\text{Hf}$ and $^{176}\text{Hf}/^{177}\text{Hf}$ (corr.) values are the same in such sessions; Table A5). For sessions where the ‘grand’ weighted mean mass bias factor was distinct from zero at the 95% confidence level, its value was subtracted from the measured $^{176}\text{Hf}/^{177}\text{Hf}$ value of each individual analyses of unknown zircons collected during that analytical session:

$$[^{176}\text{Hf}/^{177}\text{Hf} \text{ (corr.)}]_{\text{spot}}^{\text{session}} = [^{176}\text{Hf}/^{177}\text{Hf}]_{\text{spot}}^{\text{session}} - [\text{Grand mean mass bias factor}]^{\text{session}}$$

The $^{176}\text{Hf}/^{177}\text{Hf}$ (corr.) values are given in Table A5. Because the mass bias correction is applied on a session-at-once basis, the $\pm 2\text{SE}$ values associated with $^{176}\text{Hf}/^{177}\text{Hf}$ (corr.) are unaffected.

The potential for excess scatter in the measured $^{176}\text{Hf}/^{177}\text{Hf}$ was assessed separately in each session, by reference to the population of analyses made on the well-characterised primary reference zircon FC1 (as it should be homogeneous in $^{176}\text{Hf}/^{177}\text{Hf}$ at a range of scales, which implies that measured populations should have MSWDs near unity). For FC1 populations with $p \leq 0.05$, the measured $\pm 2\text{SE}$ values are too small to account for the observed degree of scatter i.e. the analytical uncertainties are underestimated in going from spot to spot.

Once excess scatter has been demonstrated in the FC1 population, we calculate (using Isoplot 3.76; Ludwig, 2003) the constant *additional* uncertainty per spot that must be added in quadrature to each measured $\pm 2\text{SE}$ value, in order to produce $\text{MSWD} \sim 1$ for the session-specific population of $^{176}\text{Hf}/^{177}\text{Hf}$ values. This constant additional uncertainty is termed the ‘spot-to-spot error’ (or the external error, or ‘repeatability’), and where applicable, its session-specific 2SE values are presented in Table A1 and A2. The spot-to-spot error is added in quadrature to the other sources of error (principally related to counting statistics) for each value in the unknowns, and thus is incorporated in the uncertainties for all individual $^{176}\text{Hf}/^{177}\text{Hf}$ values presented in the Lu–Hf data tables (Table A5).

For each sample, a weighted mean $^{176}\text{Hf}/^{177}\text{Hf}$ value is calculated from zircon domains interpreted (based on their U–Pb SHRIMP analyses) to have formed during magmatic crystallisation. In each case, the 95% confidence interval of the weighted mean $^{176}\text{Hf}/^{177}\text{Hf}$ has been augmented (via quadratic addition) by the uncertainty associated with the session-specific mass bias factor (although its effect is very minor at six decimal places).

Uncertainties associated with the values of (1) the measured $^{176}\text{Lu}/^{177}\text{Hf}$, (2) the magmatic crystallisation age of the sample, and (3) the ^{176}Lu decay constant (Scherer, Münker, & Mezger, 2001), all relate solely to the very minor proportion of ^{176}Hf that has accumulated since crystallisation (maximum 0.012%, median 0.003% across the entire population of individual ‘magmatic’ analyses presented herein). These three systematic uncertainties are each of the order of 1% of this tiny fraction, which means that they will not affect $^{176}\text{Hf}/^{177}\text{Hf}$ values at the six decimal places; these

sources of uncertainty are therefore neglected in the calculation of the sample-specific mean uncertainties.

O-isotope data processing

During each of our 10 mount-specific O-isotope analytical sessions, we collected a suite of analyses from our comounted primary reference zircon TEMORA2 ($\delta^{18}\text{O} = 8.20\text{‰}$, Black et al., 2004), relative to the Vienna Standard Mean Ocean Water (VSMOW) baseline (Baertschi, 1976); and occasional datasets from secondary reference zircons (FC1 and OG1). A total of 347 reference zircon analyses were collected across 14 datasets (one per reference zircon per analytical session); overall, 23 analyses were excluded from their constituent populations on the basis of poor analytical quality (Tables A2 and A4).

Significant excess scatter is nearly always present in the measured $^{18}\text{O}/^{16}\text{O}$ in any given analytical session, even within populations of analyses of well-characterised primary reference zircons such as TEMORA2, which should be homogeneous in $^{18}\text{O}/^{16}\text{O}$ at a range of scales. Such homogeneity would imply that measured populations should have MSWDs near unity when weighted mean values are calculated using the measured uncertainties; this is very rarely observed. TEMORA2 populations routinely have $p \leq 0.05$, which means the measured $\pm 2\text{SE}$ values are too small to account for the observed degree of scatter i.e. the analytical uncertainties are significantly underestimated in going from spot to spot.

Once excess scatter has been demonstrated in the TEMORA2 population, we calculate (using Isoplot 3.76; Ludwig, 2003) the constant *additional* uncertainty per spot that must be added in quadrature to each measured $\pm 2\text{SE}$ value, in order to produce MSWD ~ 1 for the session-specific population of $\delta^{18}\text{O}$ values. This constant additional uncertainty is termed the ‘spot-to-spot error’ (or the external error, or ‘repeatability’), and its session-specific 2SE values are presented in Table A2. The spot-to-spot error is added in quadrature to the other sources of error (which are generally much smaller, and principally related to counting statistics) for each value in the unknowns, and thus is incorporated in the uncertainties for all individual $\delta^{18}\text{O}$ values presented in the O-isotope data-tables (Table A5).

For each sample, a weighted mean $\delta^{18}\text{O}$ value is calculated from zircon domains interpreted (based on their U–Pb SHRIMP analyses) to have formed during magmatic crystallisation. In order to maximise intersession comparability of the sample mean results, the 95% confidence interval of the weighted mean $\delta^{18}\text{O}$ has been augmented (via quadratic addition) by the uncertainty associated with each of the session-specific means of the TEMORA2 measurements (i.e. the internal error, or ‘session-to-session error’; Table A2) applicable to that sample, even though the contribution of this systematic uncertainty is minor in $\delta^{18}\text{O}$ values specified at two decimal places.

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