

Crustal and thermal structure of the Thomson Orogen: constraints from the geochemistry, zircon U–Pb age, and Hf and O isotopes of subsurface granitic rocks

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SUPPLEMENTARY PAPERS

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Supplementary papers

Appendix 1. Analytical methods; A. Sample preparation and analytical technique for whole-rock chemistry; B. Sample preparation and analytical techniques for zircon U–Pb dating by LA-ICPMS and SHRIMP, Hf and O isotopes in zircons.

Appendix 2. Results – zircon U–Pb dating LA-ICP-MS & SHRIMP. (Excel workbook)

Appendix 3. Results – Interpretation of zircon age distribution for the granitic rocks sampled from drill cuttings.

Appendix 4. Results – Hf isotope in zircon. (Excel workbook)

Appendix 5. Results – O isotope in zircon. (Excel workbook)

Appendix 1: Analytical methods

A. Sample preparation and analytical technique for whole-rock chemistry

Selected whole-rock chemistry and heat production values from Thomson metasediments were analysed at Geoscience Australia using XRF for major elements and ICP-MS for trace elements. Details on the analytical methods can be found in Appendix 1 of Champion & Bultitude (2013). The following describe the analytical methods for the granitic rocks.

Rock sample preparation

Core intervals of the intersected granitic rocks range from ~3 to 30 m long. Approximately 2–4 kg of the freshest rocks were sampled for whole-rock chemistry analyses. Veins, fractures, and enclaves were removed and the rest was crushed in a large and small steel jaw crusher until the sample reached a rice grain size. The crushed rock was then split several times in order to select a ~200 g representative portion. The latter portion was then refined in an agate mill until a powder of <10 μm was obtained. To avoid cross sample contamination, prior preparation of the following sample, all equipment was cleaned using a brush and air compressor and then wiped with ethanol. Once dry, a small quantity of rock sample was selected for removing contaminants in both the large and small jaw crusher and was then discarded. For the agate mill, decontamination was first undertaken using quartz sand, then using a small portion of the sample to be processed. Sample preparation was undertaken using facilities at the Sample Preparation Laboratory, School of Earth Sciences at the University of Queensland.

XRD analyses

XRD analyses were undertaken on both bulk-rock sample and clay mineral separates (<2 μm). Rock powders were processed in an agate mill. No additional sample preparation was required for bulk sample analyses. Clay mineral separates were obtained after disaggregation in distilled water using an ultrasonic bath. Different clay size fractions (2–1, <1, 2–0.5 and <0.5 μm) were obtained by centrifugation, and the decanted suspensions were placed on a glass slide.

XRD analyses were carried out at the University of Queensland, on an X-Ray diffractometer with Bragg-Brentano geometry and CuK α radiation, operated at 40 kV and 30 mA at a scanning rate of 1°2 θ /min. Each sample was scanned from 2 to 52°2 θ for bulk-rock samples and 2 to 32°2 θ for mineral clay separates, in the step-scanning mode with a step size of 0.05 degrees and a step time of 1.2s for bulk-rock sample and 230.4s for mineral clay separates.

The illite crystallinity (IC) or Kübler index is defined as the width of the first order illite basal reflection (10 Å peak) at half height and expressed in $\Delta 2\theta$ values (Kübler, 1964). The Kübler index decreases with increasing illite crystallinity, with temperature being the most important controlling factor (Frey, 1987). Illite crystallinity values are given in Table 3 and XRD profiles for bulk rock samples and mineral clay separates are available upon request.

Loss on ignition

Loss on ignition was undertaken by Australian Laboratory Services Pty. Ltd. (ALS), using a LECO Thermal Gravimetric Analysis (TGA) furnace. Ceramic crucibles containing the rock powders were placed at ambient temperature in the furnace, heated to 105°C and weight and heated to 1000°C and weight again. LAT-CS9 was used as a standard and Relative Standard Deviation (RSD) is 0.31.

Major elements

Major element were analysed by ALS with Wavelength Dispersive Spectrometry (WDS) using a PANanalytical MagiXfast XRF. Fused glasses were prepared with a Modutemp using 0.6 to 0.72 g of rock powder and 7.1 to 7.5 g of flux with a 12:22 lithium tetraborate and including lithium nitrate as an oxidising agent. Four standards (NCSDC73303, SARM-3, SARM-32 and SARM-35) were analysed for determining accuracy and reproducibility. Accuracy and reproducibility are better than 1% (except MnO).

Trace elements

Trace and rare earth elements (REE) were analysed by Dr Alan Greig at the University of Melbourne using the following procedure. For each sample, 100 mg of sample was digested with HF–HNO₃ mixtures in high pressure bombs in an oven at 180°C for 60 hours. Solutions are then evaporated to dryness, then redissolved in HCl for 24 hours in the oven. Next, samples were dried down and refluxed twice with concentrated HNO₃, then dissolved in sealed vessels with 3N HNO₃ overnight. Solutions were transferred to transparent polycarbonate tubes, diluted with water and centrifuged, then inspected for undissolved fluorides. If present, the supernatant was transferred to new tubes for those samples containing fluorides, and the fluorides transferred to bombs and dissolved in HCl overnight in the oven. These solutions were then dried down, refluxed with nitric, then dissolved in 3N HNO₃. These solutions were recombined with their corresponding supernatant solutions and centrifuged to ensure no fluorides remained. An aliquot of the solution is further diluted with a 1.8% HNO₃ solution containing an internal standard mixture to give a total dilution factor of 14000 to 16000. Analytical and drift correction procedures are comprehensively described in Eggins et al. (1997). The method uses a natural rock standard for calibration, internal drift correction using multi-internal standards (Li⁶, Rh, Re and U²³⁵), external drift monitors and aggressive washout procedures. Differences from the Eggins et al. (1997) method are: 1) Tm, In and Bi were not used as internal standards as they are measured as analytes; (2) Two digestions of the USGS standard W-2 are used for instrument calibration. The preferred concentrations used for W-2 were mostly derived by analysing it against synthetic standards and a literature survey of isotope dilution analyses (Kamber et al., 2003, 2005). Because only a single calibration standard is used, data can be easily normalised to other sets of preferred values for standards. Samples were analysed at the School of Earth Sciences, University of Melbourne on an Agilent 7700x. The instrument was tuned to give Cerium oxide levels of <1%. Four replicates of 100 scans per replicate were measured for each isotope. Dwell times were 10 milliseconds, except for Be, Cd, In, Sb, Ta, W, Tl, Bi, which were 30 milliseconds. Long sample wash-out times of 6 minutes with solutions of 0.5% Triton X-100, 0.025% HF in 5% HNO₃ and 2% HNO₃ and long sample uptake times of 120 seconds were used. Digests of USGS standards BHVO-2 and GSP-2 and GSJ standard JA-2 were run as unknowns to check the long term accuracy and reproducibility. Absolute percent errors are generally less than 5%, with the exception of Cd, Sn and Sb for which percent errors are either <–5 or >5. Relative standard deviation (RSD) for standards analysed in 6 different analytical sessions are generally better than 5%. Elements with elevated RSD are Cu, As, Cd, In, Sn, Sb, Pb and Bi, but for each of those elements at least one standard have a RSD < 5.

B. Sample preparation and analytical techniques for zircon U–Pb dating by LA-ICPMS and SHRIMP, Hf and O isotopes in zircons

Rock sample preparation

Approximately 2 kg of rock sample were crushed in a large steel jaw crusher and further refined in a disc mill at the Sample Preparation Laboratory, School of Earth Sciences at the University of Queensland. The rock powder was sieved between 63 µm and 1 mm and processed by Apatite to Zircon Inc. (USA) for heavy mineral separation. The rock powder was first washed in plain water to remove silt and clay sized particles and then run through a lithium-metatungstate heavy liquid to allow separation of the light minerals (density less than 3 g/cc) from the heavier ones. The heavy minerals were run through a Frantz magnetic separator to remove magnetic minerals. Finally, the non-magnetic minerals were run through a Di-iodomethane (DIM) solution to separate the mineral apatite from the mineral zircon. Approximately 100 zircons from each rock samples were later subdivided into different groups based on morphological and colour variations. These zircons were mounted in epoxy resin blocks and the mounts were subsequently polished to expose the internal structures of the zircons.

LA-ICP-MS zircon dating and zircon chemistry

Two different batch of samples were analysed for LA-ICP-MS zircon dating. The first batch was completed at RSES, ANU in 2011 and include the following samples: BAL-01, ROS-01, WOL-01, BUD-01, STO-01 and TBC-01. The second batch was undertaken in 2017 at Central Analytical

Research Facility (CARF), Queensland University of Technology (QUT) and comprises VAL-01, TIC-01, LIS-01, CAI-01, ALB-01, LOL3-01, OMI-01, THO-01, NOC-01 and PIN-01.

Transmitted light microscope images were acquired on the polished zircon grain mounts to reveal potential inclusions and fractures to be avoided. The mounts were later carbon-coated for cathodoluminescence (CL) imaging. CL images were obtained using a Robinson CL detector fitted JEOL JSM-6610A SEM at RSES, ANU and the Variable Pressure Secondary Electron (VPSE) detector on the ZEISS Sigma VP at CARF, QUT. All images were taken at 15 to 20 kV with a 60 μm spot size.

For the first batch, U–Th–Pb dating on zircons was undertaken at RSES, ANU, using a 193 nm ArF Lambda Physik Compex 110 excimer laser, connected to a Helix ablation cell, and coupled to an Agilent 7700 ICP-MS. For the second batch, the data were acquired using a ESI New Wave 193 nm ArF excimer laser with a Trueline cell, and coupled to an Agilent 8800 ICP-MS. Analytical conditions are described in Bryan et al. (2004), Allen and Campbell (2012) and references therein. Ablations were undertaken at 5 Hz using a 28 μm laser spot size. For each analysis, a ~ 25 s gas blank was acquired followed by a ~ 40 s analysis of the selected zircon domain. The following masses were measured simultaneously for the first batch: ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{232}Th , ^{238}U and ^{235}U , but ^{235}U was not used, for Th–U–Pb dating; and ^{29}Si , ^{31}P , ^{49}Ti , ^{89}Y , ^{91}Zr , ^{139}La , ^{140}Ce , ^{146}Nd , ^{153}Eu , ^{157}Gd , ^{163}Dy , ^{175}Lu and ^{177}Hf for chemistry. For the second batch, the following isotopes were measured: ^{29}Si , ^{31}P , ^{49}Ti , ^{93}Nb , ^{91}Zr , ^{139}La , ^{140}Ce , ^{146}Nd , ^{153}Eu , ^{163}Dy , ^{175}Lu , ^{177}Hf , ^{181}Ta , ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{232}Th , and ^{238}U . It is important to note here that the acquisition of 18/19 isotopes when only 5 are necessary for U–Pb dating result in a loss of precision. Temora 2 was used as the primary reference standard and R33 (Black et al., 2004) was used as an unknown standard to check on data quality. For the second batch Plešovice was also used as secondary standard (Sláma et al., 2008). NIST-610 glass was used as a standard for chemistry (Jochum et al., 2011). Overall, four analytical sessions were undertaken: two per batch. Each analytical session was initiated and finalised with two sequences of standards (NIST-610, R-33 and Temora 2), and bracketed by a sequence of standard every 10–15 unknowns.

The data reduction was carried out using the new freeware lolite v2.5 (Paton et al., 2010) under Igor Pro 6.22A for the first batch and lolite v3.63 under Igor Pro 7.06. After background subtraction, downhole fractionation was corrected for $^{206}\text{Pb}/^{238}\text{U}$, $^{207}\text{Pb}/^{235}\text{U}$ and $^{208}\text{Pb}/^{232}\text{Th}$ using Temora 2 as the primary standard and a smoothed cubic spline. The first and last portion of the signal was removed until the best Gaussian was obtained.

During ablation, distinct zones within the zircons may be analysed. For example, an analysis may begin by ablation of a rim and end by the ablation of the core. Alternatively, inclusions may be ablated. For this reason, each analysis was examined in lolite for any isotopic changes or anomalous P, La, Ce or Ti contents. Where necessary the automatic integration interval was refined to avoid inclusions or to separate rim vs core analyses. Comments on all analyses and integration intervals selected are available under request.

Once processed with lolite, the data were exported and treated further using the Excel add-on Isoplot 4.1. (Ludwig, 2008). Propagated uncertainties calculated in lolite were used for all diagrams and age determinations. Weighted mean ages obtained on Temora 2, and R33 are within uncertainties with the following accepted values: 416.78 ± 0.33 Ma for Temora, 418.26 ± 0.39 Ma for R33 (Black et al., 2004) and 337.13 ± 0.37 Ma for Plešovice (Sláma et al., 2008). Trace elements data for NIST610 are also in agreement with accepted values of Jochum et al. (2011).

Accuracy and external (session to session) reproducibility were determined using the secondary standard R33 for data acquired at ANU and Plešovice for data obtained at QUT. Accuracy for all analytical sessions range between 0.1 and 1.6% and the reproducibility of R33 and Plešovice is 1.2% and 3.2% respectively (2RSD). These reproducibilities have been propagated onto the uncertainties of the unknown.

Due to isobaric interferences between ^{204}Pb and ^{204}Hg , common Pb was not directly measured by LA-

ICP-MS. Common Pb correction was performed using a modified version of the “ ^{208}Pb method” (Compston et al., 1984; Hinthorne et al., 1979; Wingate et al., 1998) with the Holmes-Houtermans model to calculate common Pb composition (Holmes, 1946; Houtermans, 1946), and primeval lead isotopic ratios of Pb troilite from the iron meteorite Canyon Diablo (Tatsumoto et al., 1973). However, this correction did not improve the degree of concordance for all analyses. Consequently, we used the common-Pb corrected age if the absolute difference between the corrected $^{206}\text{Pb}/^{238}\text{U}$ age and corrected $^{207}\text{Pb}/^{235}\text{U}$ age is less than the non-corrected one, otherwise the non-corrected ages were kept. These are reported in Appendix 2.

SHRIMP U–Pb dating of zircons

Sample preparation for U–Pb SHRIMP dating was completed by David DiBugnara (Mineral Separation Laboratory, GA) using the procedures described by Chisholm et al. (2014). Zircon separates used for LA-ICP-MS dating were further refined to obtain a high quality zircon separate. A zircon mount was prepared and zircons were imaged using transmitted and reflected light microscopy and cathodoluminescence technique. Zircons were analysed in one analytical session in a round-robin fashion, bracketed every 4 analyses by Temora 2 and every 8 unknown analyses by OG1. Temora 2 is used to calibrate the $^{206}\text{Pb}/^{238}\text{U}$ ratios and OG1 to monitor the $^{207}\text{Pb}/^{206}\text{Pb}$ ratios. For this session, the $^{206}\text{Pb}/^{238}\text{U}$ mean age measured for Temora and the $^{207}\text{Pb}/^{206}\text{Pb}$ mean age measured for OG1 are within error with the accepted values.

Data reduction was undertaken using the SQUID excel macro of Ludwig (2009) and the ISOPLOT excel macro of Ludwig (2008). Common Pb correction was applied using the model of Stacey & Kramers (1975). Common Pb corrected ratios and their propagated uncertainties were used for age determinations. The 1σ spot to spot error (0.91%) was propagated onto the $^{206}\text{Pb}/^{238}\text{U}$ uncertainty values of unknowns, and the 1σ session to session error (e.g. external error; 0.19% here) was propagated onto the weighted mean age uncertainties of the unknown. Further information on analytical conditions and data reduction are given in Bodorkos et al. (2013).

Lu–Hf isotopes in zircons

Lu–Hf isotopes on zircons were measured at the Research School of Earth Sciences (RSES) at Australian National University (ANU), using a laser ablation multicollector inductively coupled plasma mass spectroscopy (LA-MC-ICP-MS), with a Helex 193 nm ArF excimer laser ablation system coupled to a ThermoFinnigan Neptune MC-ICP-MS (Eggins et al., 2005). Ablations were performed at 5–8 Hz and with a 50 μm laser spot size. Each analysis was acquired for 80 to 90 s, but in cases for a shorter amount of time due to the small size of some zircons. Ten unknown analyses were bracketed with the acquisition of a gas blank, and the following standards: Mud tank, 91500, Temora 2 and R33. Masses ^{171}Yb , ^{173}Yb , ^{174}Hf , ^{175}Lu , ^{176}Hf , ^{177}Hf , ^{178}Hf , ^{179}Hf and ^{181}Ta were acquired simultaneously in a static collection mode. Isobaric interferences were corrected following the approach detailed in the supplementary material of Hollis et al. (2014a). Data were reduced offline using the software Lolite Package (Paton et al., 2010). 91500 was used as a primary standard and Mud tank, FC-1, QGNG and Plešovice were used as a check on data quality. Hf isotopes on reference materials are within uncertainty and agree very well with accepted values listed in Woodhead and Hergt (2005). Consequently, no correction factor was applied to the data. Reproducibility calculated using initial ϵ_{Hf} of 91500 is 6% (1RSD). $^{176}\text{Hf}/^{177}\text{Hf}$ is calculated using a decay constant $\lambda^{176}\text{Lu} = 1.867 \cdot 10^{-11} \text{ y}^{-1}$ (Söderlund et al., 2004), ϵ_{Hf} is calculated using chondritic values from Bouvier et al. (2008). Two-stage model ages are calculated using depleted mantle values from Griffin et al. (2000) and a Lu/Hf equal to 0.0125 after Chauvel et al. (2014).

O isotopes in zircons

O isotopes were analysed by Dr Richard Armstrong using the SHRIMP II at RSES, ANU, following similar analytical conditions to those described in Ickert et al. (2008). O isotope analyses were undertaken on the exact same location as U–Pb dating SHRIMP pits. $^{18}\text{O}/^{16}\text{O}$ measurements are reported in the $\delta^{18}\text{O}$ notation, in standard per mil (‰), relative to Vienna-Standard Mean Ocean Water

(VSMOW). Standards FC1, Temora 2 and OG1 were analysed in a round robin fashion. Uncertainties from the standard Temora 2 were propagated onto the uncertainties of the unknowns. Reproducibility is 3.2% using $\delta^{18}\text{O}$ of Temora 2. $\delta^{18}\text{O}$ values on reference materials agree well with the accepted values.

References

- Allen, C. M., & Campbell, I. H. (2012). Identification and elimination of a matrix-induced systematic error in LA-ICP-MS $^{206}\text{Pb}/^{238}\text{U}$ dating of zircon. *Chemical Geology*, 332–333, 157–165.
- Black, L. P., Kamo, S. L., Allen, C. M., Davis, D. W., Aleinikoff, J. N., Valley, J. W., Mundil, R., Campbell, I. H., Korsch, R. J., Williams, I. S., & Foudoulis, C. (2004). Improved $^{206}\text{Pb}/^{238}\text{U}$ microprobe geochronology by the monitoring of a trace-element-related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards. *Chemical Geology*, 205 (1–2), 115–140.
- Bodorkos, S., Blevin, P. L., Simpson, C. J., Gilmore, P. J., Glen, R. A., Greenfield, J. E., Hegarty, R., & Quinn, C. D. (2013). *New SHRIMP U–Pb zircon ages from the Lachlan, Thomson and Delamarian orogens, New South Wales: July 2009–June 2010*. Canberra ACT: Geoscience Australia Record 2013/29, 124 p.
- Bouvier, A., Vervoort, J. D., & Patchett, P. J. (2008). The Lu–Hf and Sm–Nd isotopic composition of CHUR: Constraints from unequilibrated chondrites and implications for the bulk composition of terrestrial planets. *Earth and Planetary Science Letters*, 273 (1–2), 48–57.
- Bryan, S. E., Allen, C. M., Holcombe, R. J., & Fielding, C. R. (2004). U–Pb zircon geochronology of Late Devonian to Early Carboniferous extension-related silicic volcanism in the northern New England Fold Belt. *Australian Journal of Earth Sciences*, 51 (5), 645–664.
- Champion, D. C., & Bultitude, R. J. (2013). The geochemical and Sr–Nd isotopic characteristics of Paleozoic fractionated S-types granites of north Queensland: Implications for S-type granite petrogenesis. *Lithos*, 162–163 (0), 37–56.
- Chauvel, C., Garçon, M., Bureau, S., Besnault, A., Jahn, B. M., & Ding, Z. (2014). Constraints from loess on the Hf–Nd isotopic composition of the upper continental crust. *Earth and Planetary Science Letters*, 388, 48–58.
- Chisholm, E. I., Sircombe, K. N., & DiBugnara, D. L. (2014). *Handbook of Geochronology Mineral Separation Laboratory Techniques*. Canberra ACT: Geoscience Australia, Record 2014/046, 45 p.
- Compston, W., Williams, I., & Meyer, C. (1984). U–Pb geochronology of zircons from lunar breccia 73217 using a sensitive high mass-resolution ion microprobe. *Journal of Geophysical Research: Solid Earth (1978–2012)*, 89 (S02), B525–B534.
- Eggins, S. M., Woodhead, J. D., Kinsley, L. P. J., Mortimer, G. E., Sylvester, P., McCulloch, M. T., Hergt, J. M., & Handler, M. R. (1997). A simple method for the precise determination of ≥ 40 trace elements in geological samples by ICPMS using enriched isotope internal standardisation. *Chemical Geology*, 134 (4), 311–326.
- Eggins, S. M., Grün, R., McCulloch, M. T., Pike, A. W., Chappell, J., Kinsley, L., Mortimer, G., Shelley, M., Murray-Wallace, C. V., & Spötl, C. (2005). In situ U-series dating by laser-ablation multi-collector ICPMS: new prospects for Quaternary geochronology. *Quaternary Science Reviews*, 24 (23), 2523–2538.
- Frey, M. (1987). Very low-grade metamorphism of clastic sedimentary rocks. In M. Frey (Ed.) *Low temperature metamorphism* (pp. 9–58). Glasgow: Blackie & Sons.
- Griffin, W. L., Pearson, N. J., Belousova, E., Jackson, S. E., van Achterbergh, E., O'Reilly, S. Y., & Shee, S. R. (2000). The Hf isotope composition of cratonic mantle: LAM-MC-ICPMS analysis of zircon megacrysts in kimberlites. *Geochimica et Cosmochimica Acta*, 64 (1), 133–147.
- Hinthorne, J. R., Andersen, C. A., Conrad, R. L., & Lovering, J. F. (1979). Single-grain $^{207}\text{Pb}/^{206}\text{Pb}$ and U/Pb age determinations with a 10- μm spatial resolution using the ion microprobe mass analyzer (IMMA). *Chemical Geology*, 25 (4), 271–303.
- Hollis, J. A., Carson, C. J., Glass, L. M., Kositcin, N., Scherstén, A., Worden, K. E., Armstrong, R. A.,

- Yaxley, G. M., & Kemp, A. I. S. (2014). Detrital zircon U–Pb–Hf and O isotope character of the Cahill Formation and Nourlangie Schist, Pine Creek Orogen: Implications for the tectonic correlation and evolution of the North Australian Craton. *Precambrian Research*, 246, 35–53.
- Holmes, A. (1946). An estimate of the age of the earth. *Nature*, 157, 680.
- Houtermans, F. G. (1946). Die Isotopenhäufigkeiten im natürlichen Blei und das Alter des Urans. *Naturwissenschaften*, 33 (6), 185–186.
- Ickert, R. B., Hiess, J., Williams, I. S., Holden, P., Ireland, T. R., Lanc, P., Schram, N., Foster, J. J., & Clement, S. W. (2008). Determining high precision, in situ, oxygen isotope ratios with a SHRIMP II: Analyses of MPI-DING silicate-glass reference materials and zircon from contrasting granites. *Chemical Geology*, 257 (1–2), 114–128.
- Jochum, K. P., Weis, U., Stoll, B., Kuzmin, D., Yang, Q., Raczek, I., Jacob, D. E., Stracke, A., Birbaum, K., Frick, D. A., Günther, D., & Enzweiler, J. (2011). Determination of Reference Values for NIST SRM 610–617 Glasses Following ISO Guidelines. *Geostandards and Geoanalytical Research*, 35 (4), 397–429.
- Kamber, B. S., Greig, A., Schoenberg, R., & Collerson, K. D. (2003). A refined solution to Earth's hidden niobium: implications for evolution of continental crust and mode of core formation. *Precambrian Research*, 126 (3), 289–308.
- Kamber, B. S., Greig, A., & Collerson, K. D. (2005). A new estimate for the composition of weathered young upper continental crust from alluvial sediments, Queensland, Australia. *Geochimica et Cosmochimica Acta*, 69 (4), 1041–1058.
- Ludwig, K. R. (2008). *Manual for Isoplot 3.7*. Berkeley Geochronology Center Special Publication. 4, 77 p.
- Paton, C., Woodhead, J. D., Hellstrom, J. C., Hergt, J. M., Greig, A., & Maas, R. (2010). Improved laser ablation U–Pb zircon geochronology through robust downhole fractionation correction. *Geochemistry Geophysics Geosystems*, 11, Q0AA06.
- Sláma, J., Košler, J., Condon, D. J., Crowley, J. L., Gerdes, A., Hanchar, J. M., Horstwood, M. S. A., Morris, G. A., Nasdala, L., Norberg, N., Schaltegger, U., Schoene, B., Tubrett, M. N., & Whitehouse, M. J. (2008). Plešovice zircon — A new natural reference material for U–Pb and Hf isotopic microanalysis. *Chemical Geology*, 249 (1), 1–35.
- Söderlund, U., Patchett, P. J., Vervoort, J. D., & Isachsen, C. E. (2004). The ¹⁷⁶Lu decay constant determined by Lu–Hf and U–Pb isotope systematics of Precambrian mafic intrusions. *Earth and Planetary Science Letters*, 219 (3), 311–324.
- Stacey, J. S., & Kramers, J. D. (1975). Approximation of terrestrial lead isotope evolution by a two-stage model. *Earth and Planetary Science Letters*, 26 (2), 207–221.
- Tatsumoto, M., Knight, R. J., & Allegre, C. J. (1973). Time differences in the formation of meteorites as determined from the ratio of lead-207 to lead-206. *Science*, 180 (4092), 1279–1283.
- Wingate, M. T. D., Campbell, I. H., Compston, W., & Gibson, G. M. (1998). Ion microprobe U–Pb ages for Neoproterozoic basaltic magmatism in south-central Australia and implications for the breakup of Rodinia. *Precambrian Research*, 87 (3–4), 135–159.
- Woodhead, J. D., & Hergt, J. M. (2005). A preliminary appraisal of seven natural zircon reference materials for in situ Hf isotope determination. *Geostandards and Geoanalytical Research*, 29 (2), 183–195.

Appendix 3. Interpretation of zircon age distribution for the granitic rocks sampled from ditch cuttings

In this appendix we provide a description of the U–Pb geochronology of zircons obtained from granites sampled as drill cuttings. While the drill cuttings were sampled as deep as possible to avoid any cross-contamination with the above sedimentary formations, we identified a potential contamination with the naked-eye in some samples. This was removed as best as possible, however it is highly likely that some degree of contamination remains in the samples studied here. Consequently, to properly interpret the ages obtained here, we first examine the depositional age of the sedimentary formation sitting above the granite (Table 3.1). Most granites are unconformably overlain by the sedimentary formations, and must therefore be older than their respective depositional ages. The youngest zircon age populations represented by at least three zircon grains and older than the depositional age are tentatively interpreted as the emplacement age. This may, however not be true if those grains derive from the above sedimentary formation. Consequently, the ages presented here must be used with a high degree of caution and can be later refined by providing zircon detrital age spectra from the sedimentary formations overlying those granites. Table 3.1 indicate the interpreted emplacement age, the inherited zircon age populations are summarised in Table 4 (main text).

Table 3.1. Summary of zircon geochronological data for the granites sampled from drill cuttings

Sample name	Well name	Sedimentary formation above granite	Age sedimentary formation	Relationship to granite	Interpreted emplacement age (Ma)
CAI-01	BRP Cairnhope 1	Colinlea sandstone	Roadian to Wordian (272.95 to 265.1)	Overlies unconformably	369 ± 49
LIS-01	PPC Lissoy 1	Log Creek Formation	Givetian to Frasnian (393.3 to 382.7 Ma)	Overlies unconformably	426 ± 18
ALB-01	LEA Albilbah 1	Joe Joe Group	Early Permian	Overlies unconformably	453.8 ± 4.4
NOC-01	PPL Noccundra 1	Undifferentiated	Permian	Overlies unconformably	373.3 ± 7.2
OMI-01	PPL Omicron 1	Hutton sandstone	166 to 178 Ma	Overlies unconformably	369.1 ± 5.3
PIN-01	PPL Pinkilla 1	Hutton sandstone	166 to 178 Ma	Overlies unconformably	560 ± 92
THO-01	SSL Thoar 3	Toolachee Formation	Wordian to Changhsingian (268.8 to 251.9)	Overlies unconformably	419 ± 52
LOL3-01	LOL (Longreach) 3	Sequence of shales and sandstones	Jurassic	Overlies unconformably	422.6 ± 8.0

CAI-01

Fifty-four analyses on CAI-01 zircons yielded 33 concordant analyses with ages ranging from 284 to 1834 Ma. Twelve analyses correspond either to inherited cores or detrital grains derived from sedimentary formations above the granites and provide Precambrian (n = 10) and Ordovician to Cambrian (n = 2) ages. One younger Early Permian outlier is also rejected and could result either from Pb loss or cross-contamination of the above sedimentary layers. The remaining 20 analyses yield a wide range of ages from 317 to 407 Ma with a weighted average age of 363 ± 11 (MSWD = 11.5; prob = 0.000). The high MSWD (11) suggest several age populations, however, they form a normal distribution (slope of 1.043 ± 0.073 on a linearised probability plot) providing an age of 369 ± 49 Ma. This is considered as the emplacement age for CAI-01.

LIS-01

Twenty-seven of 49 analyses are concordant yielding ages from 381 to 2031 Ma. Seven older outliers ranging from 541 to 2031 Ma are rejected, the remaining analyses indicate three zircon age populations: (1) 390.7 ± 5.1 (MSWD = 5.1; prob = 0.86; n = 10), (2) 426 ± 18 (MSWD = 2.8; prob = 0.026; n = 5), and (3) 469.4 ± 8.2 (MSWD = 0.51; prob = 0.73; n = 5). The former is similar in age to

the age of the overlying sedimentary formation and as such is considered to derive from that formation by cross-contamination. The Silurian population is considered to represent the emplacement age of LIS-01, whereas the Ordovician population is either inherited or derived from the overlying sedimentary formation. Additional constraints is needed to better interpret the emplacement age for LIS-01 and may be obtained by performing U–Pb detrital zircon dating of the Log Creek Formation.

ALB-01

Fifty-four analyses were undertaken on ALB-01 zircons with 39 giving concordant ages. Seven older outliers (492 to 1048 Ma) and three younger outliers (297 to 385 Ma) were excluded from further consideration. The remaining 29 analyses provide a weighted average age of 453.8 ± 4.4 Ma (MSWD = 1.9; prob = 0.002) considered as the emplacement age of ALB-01.

NOC-01

Only 17 analyses are concordant from the 55 analyses completed on NOC-01 zircons. These zircons yield a complex geochronological signatures with numerous age populations. After excluding one younger outlier (195 Ma), and four Precambrian outliers (570 to 2198 Ma), three main zircon age populations can be identified at 373.0 ± 7.2 Ma (MSWD = 0.47; prob = 0.63; n = 3), 414.5 ± 6.4 (MSWD = 0.58; prob = 0.68; n = 5) and 480 ± 49 (MSWD = 7.3; prob = 0.000; n = 4). The latter two zircon age populations are represented essentially by resorbed zircon cores and are thus considered inherited. Additional constraints could be provided by dating the zircons from the overlying sedimentary formation. The emplacement age of NOC-01 is interpreted to be 373.0 ± 7.2 Ma.

OMI-01

Fourty-eight analyses are concordant amongst the sixty undertaken yielding 206/238 ages from 255 to 445 Ma. Three younger outliers (255 to 320 Ma) are rejected and the remaining 46 analyses can be subdivided into three age populations as suggested in a linearised probability plot: (1) 369.1 ± 5.3 Ma (MSWD = 0.53; prob = 0.79; n = 7), (2) 395.1 ± 5.1 (MSWD = 1.3; prob = 0.2; n = 14, and (3) 429.1 ± 3.6 Ma (MSWD = 1.17; prob = 0.26; n = 24). This is consistent with the unmix age function in Iolite: (1) 371.9 ± 6.3 , (2) 398.1 ± 5.7 and (3) 429.8 ± 3.5 Ma (relative misfit: 0.544).

PIN-01

Sixty analyses were performed on PIN-01 zircons yielding 47 concordant ages ranging from 242 to 3439 Ma. Twenty-eight analyses are clearly older and the other 19 analyses yield a weighted average age of 557 ± 21 Ma (MSWD = 17). The MSWD is high and suggests several age populations, however they depict a normal age distribution with a slope of 1.048 ± 0.065 on a linearised probability plot giving an age of 560 ± 92 Ma. The latter is considered as the emplacement age for PIN-01.

THO-01

Fourty analyses are concordant from the fifty undertaken on THO-01 zircons yielding ages from 299 to 2664 Ma. Three young outliers are rejected and are either explained by Pb loss or cross-contamination from above sedimentary formations. Another 26 analyses are older than the main age population yielding two key age population at 506 ± 9.4 Ma and 578 ± 15 Ma and a myriad of ages from 703 to 2664 Ma. The remaining 11 analyses yield a weighted average age of 415 ± 13 Ma, but the MSWD is high (6.2). However, those analyses form a normal distribution with a slope of 1.07 ± 0.15 providing an age of 419 ± 52 Ma, considered as the emplacement age of THO-01.

LOL3-01

Fifty-eight analyses were undertaken on LOL3-01 zircons resulting in 50 concordant analyses ranging from 103 to 1931 Ma. The youngest zircon population represented by four zircon grain analyses yield an age of 422.6 ± 8.0 (MSWD = 0.23; prob = 0.88), tentatively interpreted as the emplacement age. Most analyses provide an older age of 467.2 ± 8.4 Ma (MSWD = 4.8; prob = 0.000), considered as inherited. Another four grains represent an older zircon age population at 541 ± 25 Ma (MSWD = 2.4; prob = 0.067), and the remaining 11 analyses scatter from 794 to 1931 Ma.