Timing and style of high-temperature metamorphism across the western Gawler Craton during the Paleo- to Mesoproterozoic

A. J. Reid^{a,b*}, J. A. Halpin^c, and R. A. Dutch^{a,b}

^a Geological Survey of South Australia, Department for Energy and Mining, Adelaide, SA 5001, Australia; ^b Department of Earth Sciences, The University of Adelaide, Adelaide, SA 5005, Australia; ^c ARC Centre of Excellence in Ore Deposits (CODES), School of Natural Sciences, University of Tasmania, Private Bag 79, Hobart, TAS 7001, Australia

*Corresponding author: anthony.reid@sa.gov.au

[^]Current address: Institute for Marine and Antarctic Studies, University of Tasmania, Private Bag 129, Hobart, TAS 7001, Australia

- A. J. Reid <u>https://orcid.org/0000-0002-9435-9342</u>
- J. A. Halpin https://orcid.org/0000-0002-4992-8681
- R. A. Dutch https://orcid.org/0000-0003-4247-7483

SUPPLEMENTARY PAPERS

Australian Journal of Earth Sciences (2019) 66, https://doi.org/10.1080/08120099.2019.1602565

Copies of Supplementary Papers may be obtained from the Geological Society of Australia's website (<u>www.gsa.org.au</u>), the Australian Journal of Earth Sciences website (www.ajes.com.au) or from the National Library of Australia's Pandora archive (<u>https://pandora.nla.gov.au/tep/150555</u>).

Supplementary paper

Appendix 1. Extended analytical methods.

- Appendix 2. Details of reference standards analysed during zircon and monazite geochronology (Excel spreadsheet).
- Appendix 3. Results of zircon and monazite geochronology (Excel spreadsheet).
- Appendix 4. Whole rock XRF results.
- Appendix 5. Representative mineral chemical analyses (Excel spreadsheet).
- Appendix 6. Details of mineral mode and compositional isopleths calculated using the program TCInvestigator v1.0 (Pearce, White, & Gazley, 2015).

Appendix 1. Extended analytical methods

Geochronology

Monazite geochronology was undertaken at the University of Tasmania utilizing in situ using polished rock chips mounted in epoxy resin within a 2.5 cm diameter mould, polished and washed in distilled water in an ultrasonic bath. A carbon coat was applied to the resin mounts prior to analysis on the SEM. Monazite was identified using a FEI Quanta 600 SEM controlled by an automated software package (Mineral Liberation Analyser or MLA) at the Central Science Laboratory, University of Tasmania. MLA software independently controls the instrument for an automated quantitative mineralogical identification and analysis. For each sample, a high-resolution back scattered electron (BSE) image was meshed with an image overlay containing the identified monazite grains to provide a base map that could then be registered using the LA-ICPMS (Geostar) software for geochronological analysis. High resolution, high contrast BSE images were obtained for individual monazite grains using a Hitachi SU-70 field emission (FE)-SEM at the Central Science Laboratory, University of Tasmania.

U-Pb monazite analyses were performed on an Agilent 7500cs quadrupole ICPMS with a 193 nm Coherent Ar-F gas laser and the Resonetics S155 ablation cell at the University of Tasmania. Each analysis was pre-ablated with 5 laser pulses to remove the surface contamination then the blank gas was analysed for 30 s followed by 30 s of monazite ablation at 5 Hz and \sim 2 J/cm² using a spot size of 13 µm; keeping U and Th in the pulse counting mode of detection on the electron multiplier. Elements measured included ²⁹Si, ³¹P, ⁴³Ca, ⁸⁹Y, ¹⁴⁰Ce, ¹⁷²Yb, ²⁰²Hg, ²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb, ²³²Th and ²³⁸U with each element being measured sequentially every 0.16 s with longer counting time on the Pb isotopes compared to the other elements. The down hole fractionation, instrument drift and mass bias correction factors for Pb/U and Pb/Th ratios on monazites were calculated using two analyses on the primary standard (RGL4B of Rubatto, Williams, & Buick, 2001) analysed at the beginning of the session and every 15-20 unknowns, using the same spot size and conditions as used on the samples to provide an independent control to assess accuracy and precision. The correction factor for the ²⁰⁷Pb/²⁰⁶Pb ratio was calculated using 13 analyses of the international glass standard NIST610 analysed throughout analytical session and corrected using the values recommended by Baker et al. (2004). LA-ICPMS setup and conditions, and monazite data reduction and reproducibility, are described in detail in Halpin et al. (2014).

All data reduction calculations and error propagations were done within Microsoft Excel® via macros designed at the University of Tasmania and summarized in Halpin *et al.* (2014). Secondary monazite standard N3364-1.5 m, equivalent to the Broken Hill Hores Gneiss, yields a ²⁰⁷Pb/²⁰⁶Pb upper intercept age of 1601 ± 6 Ma (n = 9, MSWD = 1.2) within error of the reference age of 1599 ± 9 Ma (Page, Stevens & Gibson, 2005). The Jefferson County secondary standard yields a ²⁰⁷Pb corrected ²⁰⁶Pb/²³⁸U weighted mean age of 363 ± 11 Ma (n = 4, MSWD = 2.0), within error of the reference age of 362 ± 4 Ma (Alagna, Petrelli, Perugini, & Poli, 2008). In-house standard 14971 yields a ²⁰⁷Pb/²⁰⁶Pb weighted mean age of 908 ± 6 Ma (n=15, MSWD = 0.92), within error of the recommended age of 912 ± 3 Ma.

Zircon geochronology was undertaken at the Adelaide Microscopy, University of Adelaide. Zircons were extracted from sample 2159307 from drill hole Lake Maurice East 1, at a commercial mineral

separation facility, Geotrack International (<u>http://www.geotrack.com.au/</u>), Melbourne, Australia. Samples were processed using standard crushing, magnetic and density separation techniques. The zircon concentrates were mounted into epoxy resin discs and polished so as to expose the grains approximately in half. Zircons were imaged using transmitted and reflected plain light and their internal structure determined by cathodoluminescense (CL) imaging at Adelaide Microscopy, the University of Adelaide.

U–Pb isotopes were analysed at Adelaide Microscopy, Adelaide University, with an ASI RESOlution ArF excimer laser ablation system with a S150 large format sample chamber. The laser was coupled to an Agilent 7900x ICPMS. The spot size utilised was 30 micron with a fluence at the sample of ~2 J/cm² and a repetition rate of 5 Hz. Samples were ablated in a He atmosphere (flow rate 0.35 L/min) and the aerosol mixed with Ar carrier gas (flow rate 1.01 L/min) for transport to the ICPMS. An analysis consisted on 30 seconds of gas background collected while the laser was not firing followed by 30 seconds of ablated signal. The GJ zircon (Jackson, Pearson, Griffin, & Belousova, 2004) was used as the primary standard to correct for down hole fractionation and instrument drift and mass bias. Four analyses of GJ-1 were analysed every 10 unknown zircons. The following isotopes were measured with counting times in brackets: ⁹⁰Zr (5 ms), ²⁰²Hg (20 ms), ²⁰⁴Pb (20 ms), ²⁰⁶Pb (30 ms), ²⁰⁷Pb (30 ms), ²⁰⁸Pb (10 ms), ²³²Th (10 ms), and ²³⁸U (20 ms), giving a total sweep time of ~0.16 seconds.

Results of analysis of reference zircons 91500 (1065.4 \pm 0.3 Ma; Wiedenbeck *et al.*, 1995), Plešovice (337.13 \pm 0.37 Ma; Sláma *et al.*, 2007) and QGNG (1851.6 \pm 0.6 Ma; Black *et al.*, 2003) are detailed in Appendix 2; the majority of analyses for the reference zircons were within analytical uncertainty of the reference values. Data from the ICPMS were processed using the Iolite v2.5 software using the VisualAge DRS package (Paton, Hellstrom, Paul, Woodhead, & Hergt, 2011; Paton *et al.*, 2010). U and Th decay constants of Jaffey, Flynn, Glendenin, Bentley and Essling (1971), as recommended by Steiger and Jäger (1977) have been utilised. All time-resolved isotope analyses were reviewed to check the integrity and consistency of the down-hole analytical trace. Where zones of compositional change and or increased ²⁰⁴Pb were encountered during the analysis, portions of the 30 second analysis were selected to integrate for age information for the individual zircon that excluded such compromised domains. Although ²⁰⁴Pb was monitored during the analysis, isobaric interference by ²⁰⁴Hg present in the Ar–He carrier gas and the low overall ²⁰⁴Pb counts, mean that no correction for common (non-radiogenic) Pb has been made for these data.

Mineral chemistry and metamorphic phase equilibria modelling

Petrographic studies were carried out on a transmitted light microscope and was supplemented by quantitative chemical analyses were performed on a Cameca SX100 equipped with 5 tunable wavelength dispersive spectrometers at the Central Science Laboratory, University of Tasmania, operating with a 40° takeoff angle, beam energy of 15 keV, beam current of 20 nA and beam diameter of 2 microns. Fe³⁺ was estimated on the basis of ideal stoichiometric constraints (after Droop, 1987) for garnet, sillimanite, magnetite and ilmenite.

The *P*–*T* evolution of samples were investigated through a forward modelling approach. Pressure– temperature (*P*–*T*) pseudosections were calculated in the model chemical system MnO–Na₂O–CaO– K_2O –FeO–MgO–Al₂O₃–SiO₂–H₂O–TiO₂–Fe₂O₃ (MnNCKFMASHTO) using THERMOCALC 3.40i

(Powell & Holland, 1988) with the recently updated internally consistent dataset of Holland and Powell (Holland & Powell, 2011; ds62, created 6/2/2012), All activity-composition models for phases considered in the modelling (g, garnet; cd, cordierite; opx, orthopyroxene; bi, biotite; mu, muscovite; pl, plagioclase: ksp. K-feldspar: ilm-hem, ilmenite-hematite: mt. magnetite: sp. spinel: lig. silicate melt) are from White et al. (2014a, 2014b). The aluminosilicates (sill/ky/and), guartz (g), rutile (ru) and aqueous fluid (H₂O) are pure end-member phases. Whole rock geochemical data was obtained from samples crushed with a hydraulic crusher and tungsten-carbide ring-mill, then prepared as pressed-powder discs and analysed for major elements using PANalytical Phillips X-ray fluorescence (XRF) spectrometer at the University of Tasmania. The XRF-derived bulk composition was converted to molar oxide percent and normalised to 100% in the MnNCKFMASHTO chemical system. The Fe₂O₃ content (recast as "O" in the chemical system) was estimated independently for each sample, based on the opaque minerals observed in thin section, and a sensitivity analysis to the chosen XFe3+ value was undertaken to investigate the effect on the boundaries of key equilibria (i.e. those bounding the inferred peak assemblage field). The Loss On Ignition (LOI) value from XRF analysis was taken as an upper limit for H₂O content for the samples; the actual amount of H₂O used was estimated on the basis of T-M(H₂O) pseudosections - the value chosen allows the inferred peak assemblage to be stable above the solidus. The exception to this is sample 2115322 (COL 20D) where subsolidus and suprasolidus conditions were investigated. In this case the H2O-content for suprasolidus conditions was based on the minimum value required in order to calculate the solidus to P = 10 kbar. Mineral mode and compositional isopleths were calculated using the program TCInvestigator v1.0 (Pearce et al., 2015). Some samples contain contours that fluctuate within a given field, likely due to problems with extrapolating the initial starting guesses that TCInvestigator uses to calculate contours, however, a broad appreciation of the trends (in composition or mode) are still apparent.

References

- Alagna, K. E., Petrelli, M., Perugini, D., & Poli, G. (2008). Micro-analytical zircon and monazite U-Pb isotope dating by laser ablation-inductively coupled plasma-quadrupole mass spectrometry. *Geostandards and Geoanalytical Research*, 32(1), 103–120. doi:10.1111/j.1751-908X.2008.00866.x
- Baker, J., Peate, D., Waight, T., & Meyzen, C. (2004). Pb isotopic analysis of standards and samples using a Pb-207–Pb-204 double spike and thallium to correct for mass bias with a double-focusing MC-ICP-MS. *Chemical Geology*, *211*(3–4), 275–303. doi:10.1016/j.chemgeo.2004.06.030
- Black, L. P., Kamo, S. L., Williams, I. S., Mundil, R., Davis, D. W., Korsch, R. J., & Foudoulis, C. (2003). The application of SHRIMP to Phanerozoic geochronology; a critical appraisal of four zircon standards. *Chemical Geology*, 200(1–2), 171–188. doi:10.1016/S0009-2541(03)00166-9
- Droop, G. T. R. (1987). A general equation for estimating Fe3+ concentrations in ferromagnesian silicates and oxides from microprobe analyses, using stoichiometric criteria. *Mineralogical Magazine*, *51*, 431–435. doi:10.1180/minmag.1987.051.361.10
- Halpin, J. A., Jensen, T., McGoldrick, P., Meffre, S., Berry, R. F., Everard, J. L., . . . Whittaker, J. M. (2014). Authigenic monazite and detrital zircon dating from the Proterozoic Rocky Cape Group, Tasmania: Links to the Belt-Purcell Supergroup, North America. *Precambrian Research*, 250(0), 50–67. doi:10.1016/j.precamres.2014.05.025
- Holland, T. J. B., & Powell, R. (2011). An improved and extended internally consistent thermodynamic dataset for phases of petrological interest, involving a new equation of state for solids. *Journal of Metamorphic Geology, 29*(3), 333–383. doi:10.1111/j.1525-1314.2010.00923.x
- Jackson, S. E., Pearson, N. J., Griffin, W. L., & Belousova, E. A. (2004). The application of laser ablation-inductively coupled plasma-mass spectrometry to in situ U–Pb zircon geochronology. *Chemical Geology*, 211(1–2), 47–69. doi:10.1016/j.chemgeo.2004.06.017
- Jaffey, A. H., Flynn, K. F., Glendenin, L. E., Bentley, W. C., & Essling, A. M. (1971). Precision Measurement of Half-Lives and Specific Activities of 235U and 238U. Physical Reviews, C4, 1889–1906. doi:10.1103/PhysRevC.4.1889

- Page, R. W., Stevens, B. P. J., & Gibson, G. M. (2005). Geochronology of the sequence hosting the Broken Hill Pb–Zn–Ag orebody, Australia. *Economic Geology, 100*(4), 633–661.
- Paton, C., Hellstrom, J., Paul, B., Woodhead, J., & Hergt, J. (2011). lolite: Freeware for the visualisation and processing of mass spectrometric data. *Journal of Analytical Atomic Spectrometry*, 26(12), 2508–2518. doi:10.1039/c1ja10172b
- 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*(3), n/a–n/a. doi:10.1029/2009GC002618
- Pearce, M. A., White, A. J. R., & Gazley, M. F. (2015). TCInvestigator: Automated calculation of mineral mode and composition contours for thermocalc pseudosections. *Journal of Metamorphic Geology*, 33(4), 413–425. doi:10.1111/jmg.12126
- Powell, R., & Holland, T. J. B. (1988). An internally consistent dataset with uncertainties and correlations: 3. Applications to geobarometry, worked examples and a computer program. *Journal* of Metamorphic Geology, 6, 173–204. doi:10.1111/j.1525-1314.1988.tb00415.x
- Rubatto, D., Williams, I. S., & Buick, I. S. (2001). Zircon and monazite response to prograde metamorphism in the Reynolds Range, central Australia. *Contributions to Mineralogy and Petrology*, 140(4), 458–468. doi:10.1007/PL00007673
- Sláma, J., Košler, J., Condon, D. J., Crowley, J. L., Axel, G., Hanchar, J. M., . . . Whitehouse, M. J. (2007). Plešovice zircon – A new natural reference material for U–Pb and Hf isotopic microanalysis. *Chemical Geology*, 249, 1–35. doi:10.1016/j.chemgeo.2007.11.005
- Steiger, R. H., & Jäger, E. (1977). Subcommission of Geochronology: Convention on the Use of Decay Constants in Geo- and Cosmochronology. *Earth and Planetary Science Letters*, 36, 359– 362. doi:<u>10.1016/0012-821X(77)90060-7</u>
- White, R. W., Powell, R., Holland, T. J. B., Johnson, T. E., & Green, E. C. R. (2014a). New mineral activity-composition relations for thermodynamic calculations in metapelitic systems. *Journal of Metamorphic Geology*, *3*2(3), 261–286. doi:10.1111/jmg.12071
- White, R. W., Powell, R., & Johnson, T. E. (2014b). The effect of Mn on mineral stability in metapelites revisited: new a–x relations for manganese-bearing minerals. *Journal of Metamorphic Geology*, 32(8), 809–828. doi:10.1111/jmg.12095
- Wiedenbeck, M., Alle, P., Corfu, F., Griffin, W. L., Meier, M., Oberli, F., . . . Speigel, W. (1995). Three natural zircon standards for U–Th–Pb, Lu–Th trace element and REE analyses. *Geostandards Newsletter, 19*, 1–23.

Appendix 4. Whole-rock XRF results

Jacqueline Halpin	CODES/SES	26/05/2015	Analyst: Jay Thompson
XRF ANALYSES			XRF sample preparation: Katie McGoldrick
10 samples			
Axios Advanced XRF	spectrometer with 4kW	/ Rh tube	

Sample	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na₂O	K ₂ O	P ₂ O ₅	BaO	Loss	Total	S%
2115503	55.7	1.00	16.6	9.31	0.17	4.24	7.91	3.78	0.40	0.17	<0.01	0.980	100.32	0.08
2115505	47.5	1.35	14.1	16.4	0.26	6.86	11.1	2.60	0.35	0.11	<0.01	-0.238	100.40	0.17
2115510	51.6	1.05	16.4	11.5	0.23	4.65	10.9	3.80	0.28	0.12	<0.01	-0.136	100.38	<0.01
2115512	63.9	1.04	15.3	7.98	0.44	3.01	2.67	2.79	2.09	0.03	0.09	0.509	99.82	0.04
2115516	64.2	0.95	14.1	10.3	1.04	2.91	1.35	1.44	2.68	0.08	0.13	0.553	99.76	<0.01
2115313	56.2	1.10	17.6	8.55	0.07	3.47	1.47	1.60	8.04	0.09	0.20	1.359	99.67	0.13
2115319	65.1	0.72	14.6	7.08	0.09	2.85	1.29	2.27	4.32	0.06	0.08	1.924	100.40	0.02
2115318	56.4	0.67	18.2	12.0	0.26	4.09	0.31	0.47	5.45	0.07	0.11	0.861	98.90	<0.01
2115322	69.2	0.62	13.1	7.73	0.25	2.14	1.63	1.42	2.41	0.16	0.06	0.940	99.68	<0.01
2115492	60.9	0.65	15.1	12.1	0.74	3.31	1.55	1.58	3.49	0.08	0.09	0.570	100.13	0.48
Detection Limits	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01			0.01

Appendix 6. Details of mineral mode and compositional isopleths calculated using the program TCInvestigator v1.0



Figure A8-1. Calculated pseudosections (left) and isopleths (right) in the chemical system MnNCKFMASHTO. The interpreted peak metamorphic assemblage field(s) is outlined in bold red. The arrow shows an approximated post-peak path. The bulk composition shown is in molar oxide %. Measured garnet compositional parameters (bottom) are outlined in grey fill and in black outline on legend.



Figure A8-2. Calculated pseudosections (left) and isopleths (right) in the chemical system MnNCKFMASHTO. The interpreted peak metamorphic assemblage field(s) is outlined in bold red. The arrow shows an approximated post-peak path. The bulk composition shown is in molar oxide %. Measured garnet compositional parameters (bottom) are outlined in grey fill and in black outline on legend.



Figure A8-3. Calculated pseudosections (left) and isopleths (right) in the chemical system MnNCKFMASHTO. The interpreted peak metamorphic assemblage field(s) is outlined in bold red. The arrow shows an approximated post-peak path. The bulk composition shown is in molar oxide %. Measured garnet compositional parameters (bottom) are outlined in grey fill and in black outline on legend.



Figure A8-4. Calculated pseudosections (left) and isopleths (right) in the chemical system MnNCKFMASHTO. The interpreted peak metamorphic assemblage field(s) is outlined in bold red. The arrow shows an approximated post-peak path. The bulk composition shown is in molar oxide %. Measured garnet compositional parameters (bottom) are outlined in grey fill and in black outline on legend.