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Early evolution of the Adelaide Superbasin

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Abstract

Continental rifts have a significant role in supercontinent breakup, and the development of sedimentary basins. The Australian Adelaide Superbasin is one of the largest and best-preserved rift systems that initiated during the breakup of Rodinia, yet substantial challenges still hinder our understanding of its early evolution and place within the Rodinian supercontinent. In the past decade, our understanding of rift and passive margin development, mantle plumes and their role in tectonics, geodynamics of supercontinent breakup, and sequence stratigraphy in tectonic settings has advanced significantly, however literature on the early evolution of the Adelaide Superbasin has not been updated to reflect these advancements. Using new detrital zircon age data for provenance, combined with existing literature, we examine the earliest tectonic evolution of the Adelaide Superbasin in the context of our modern understanding of rift system development. A new maximum depositional age of 893 ± 9 Ma from the lowermost stratigraphic unit provides a revised limit on the initiation of sedimentation and rifting within the basin. Our model suggests that the basin evolved through an initial pulse of extension exploiting pre-existing crustal weakness to form half-grabens. Tectonic quiescence and stable subsidence followed, with deposition of a sourceward-shifting facies tract. Emplacement and extrusion of the Willouran Large Igneous Province occurred at c. 830 Ma initiating a new phase of rifting. This rift renewal led to widespread extension and subsidence with deposition of the Curdimurka Subgroup, which constitutes the main cyclic rift sequence in the Adelaide Superbasin. Our model suggests that the Adelaide Superbasin formed through rift propagation an apparent triple junction, rather than apical extension outwards from this point. Additionally, we provide evidence suggesting a late Mesoproterozoic zircon source to the east of the basin, and show that the lowermost stratigraphy of the Centralian Superbasin, which is thought to be deposited coevally, had different primary detrital sources.

1 Introduction

The breakup of the supercontinent Rodinia, and subsequent formation of Gondwana, coincided with critical Earth system changes that led to the Phanerozoic world of extensive macroscopic mineralised life, significantly oxygenated atmosphere and hydrosphere, and a buffered climate devoid of whole-planet glaciations (Halverson et al. 2009; Shields et al. 2021; Tostevin & Mills 2020). Determining any interdependence between these phenomena (e.g., Alcott et al. 2020; Gernon et al. 2016; Halverson et al. 2009; Mills et al. 2019) requires constructing full-plate tectonic reconstructions of the globe (Merdith et al. 2021; Merdith et al. 2017b) that themselves need a fundamental

39 understanding of the temporal link between tectonically controlled geological features (such as rift
40 basins) and plate tectonic phenomena (such as continental plate sundering and ocean crust
41 formation; Collins et al. 2021; Merdith et al. 2017b).

42 The Adelaide Superbasin (Lloyd et al. 2020) is one of the largest and best preserved rift to passive-
43 margin successions to form during the Neoproterozoic breakup of Rodinia, which included large
44 continental rifts between the Australia, Amazonia, Baltica, Kalahari, Laurentia, and Siberia cratons
45 (Bogdanova et al. 2009; Cawood et al. 2016; Li et al. 2008). The Adelaide Superbasin is thought to
46 have formed the conjugate margin to western Laurentia in Rodinia (Brookfield 1993; Dalziel 1997;
47 Hoffman 1991; Karlstrom et al. 1999; Merdith et al. 2017b; Moores 1991; Wingate et al. 2002),
48 although other configurations for Rodinia have been suggested (e.g., Li et al. 2008; Li et al. 1995;
49 Wen et al. 2017; Wen et al. 2018). Poor chronological control, sparse and ambiguous palaeomagnetic
50 constraints, and a lack of young detrital zircon in the lower units of the Adelaide Superbasin have long
51 hindered the research and testing of these Rodinia reconstructions. Research on the tectonic
52 evolution of the Adelaide Superbasin has seen the geosyncline theory (Sprigg 1952), transition to
53 plate tectonics (Preiss 1987; 2000), and a few targeted (Counts 2016; Job 2011; Mackay 2011) or
54 more generalised (Keeman et al. 2020; Lloyd et al. 2020) studies since.

55 This research presents new detrital zircon U–Pb and trace element data for the lowermost units of
56 the Adelaide Rift Complex within the Adelaide Superbasin. We use these data, alongside existing
57 literature to provide a refined, early tectonic evolution of the rift system during the deposition of the
58 Callanna Group.

59 2 Geological Background

60 2.1 Adelaide Superbasin

61 The Adelaide Superbasin (Lloyd et al. 2020) is a large, Neoproterozoic to middle Cambrian
62 sedimentary system at the southeast margin of Proterozoic Australia, which formed as a result of the
63 breakup of the supercontinent Rodinia. The Adelaide Superbasin consists of several named basins
64 and sub-basins that span the Neoproterozoic to early Cambrian. The largest and oldest of these is the
65 Adelaide Rift Complex that is contiguous with the relatively undeformed rocks of the Torrens Hinge
66 Zone, Stuart Shelf (Sprigg 1952), and Coombarlarnie Platform (Callen 1990). Two Cambrian basins,
67 the Arrowie Basin, and the Stansbury Basin, are also considered as part of the Adelaide Superbasin
68 (Lloyd et al. 2020; Preiss et al. 2002) [Figure 1]. Whilst present day exposure of the sedimentary
69 basin is approximately 600 km north to south, the basin spans over 1,100 km from central Australia
70 through to Kangaroo Island. Deposition within the Adelaide Superbasin spans over 300 million years
71 of Earth's history and stretches from the northernmost regions of South Australia, narrowing in the
72 South Mount Lofty Ranges at the Fleurieu Peninsula and extending onto Kangaroo Island. Further
73 south, links with coeval sequences in Antarctica and eastern Tasmania are unclear, but possible
74 (Mulder et al. 2018). The basin began as an intracontinental rift system that successfully progressed
75 to a passive margin basin in its southeast region, yet remained a failed rift in the north. Deposition

76 within the basin ceased during the
77 Delamerian orogeny c. 514–490 Ma
78 (Drexel & Preiss 1995; Foden et al.
79 2006; Foden et al. 2020; Preiss
80 2000). The stratigraphy of the
81 Adelaide Superbasin is divided into
82 three supergroups (Preiss 2000), two
83 for the Neoproterozoic sequences
84 and the third for the Cambrian
85 sequences, with numerous group and
86 subgroup level divisions. In the
87 Neoproterozoic, the Warrina
88 Supergroup is comprised of the
89 Callanna, Burra, and Poolamacca
90 Groups, and the Heysen Supergroup
91 contains the Umberatana, Wilpena,
92 Torrowangee, and Farnell Groups.
93 Each of these groups are further
94 divided into numerous subgroups
95 (see Lloyd et al. 2020). Here we focus
96 on the Callanna Group, which is best
97 preserved in the failed arm of the rift
98 system. The reader is referred to
99 Preiss (1987), Preiss (2000), Counts
100 (2017), Lloyd et al. (2020), Cowley
101 (2020) and references therein for
102 further detail on the geological history
103 of the Adelaide Superbasin.

104 2.1.1 Callanna Group

105 The oldest stratigraphy of the Adelaide Superbasin is represented by the Callanna Group (Forbes et al. 1981), which is further subdivided into the Arkaroola Subgroup (Forbes et al. 1981; Mawson 1949; Thomson 1966), and the Curdimurka Subgroup (Forbes et al. 1981), that is inferred to be the younger of the two (Preiss 1993). For historical reference, the now outdated “Willouran Series” is equivalent to the Callanna Group, although this has not always been the case (Preiss 1987). In New South Wales, the Poolamacca Group (Cooper & Tuckwell 1971) is thought to be the equivalent of the Arkaroola Subgroup (Preiss 1987). The known depositional extent of the Callanna Group [Figure 2] is restricted to the eastern (NSW), central, and northern Adelaide Rift Complex (including the Davenport and Denison Ranges), Stuart Shelf, and possibly the eastern Officer Basin.

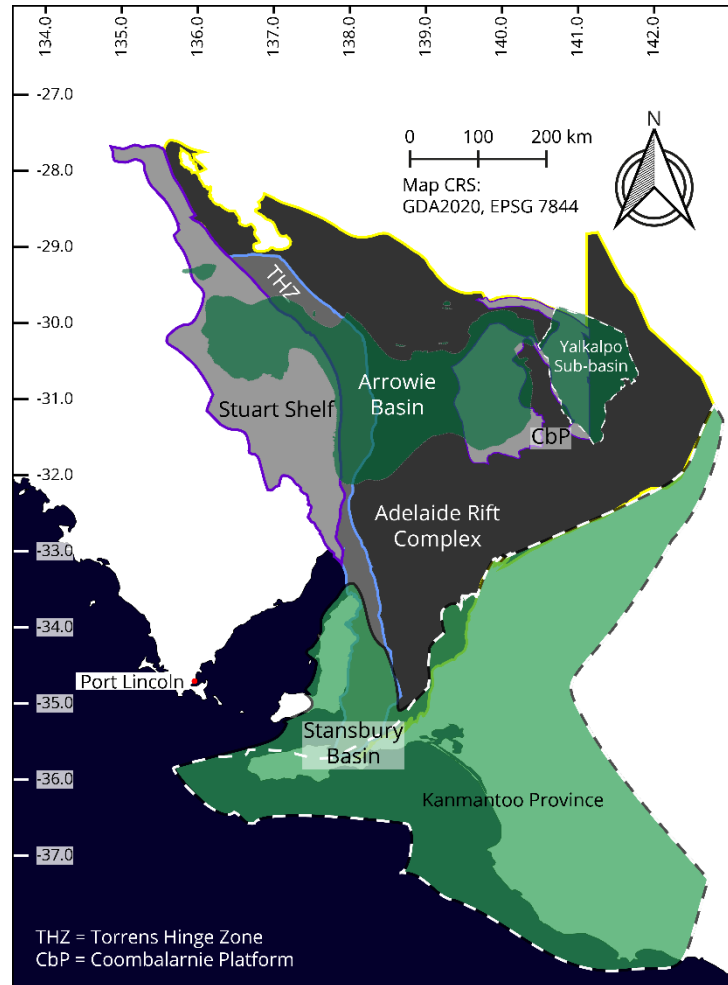


Figure 1 – Map of known Adelaide Superbasin extent and basin subdivisions (derived from Lloyd et al. 2020).

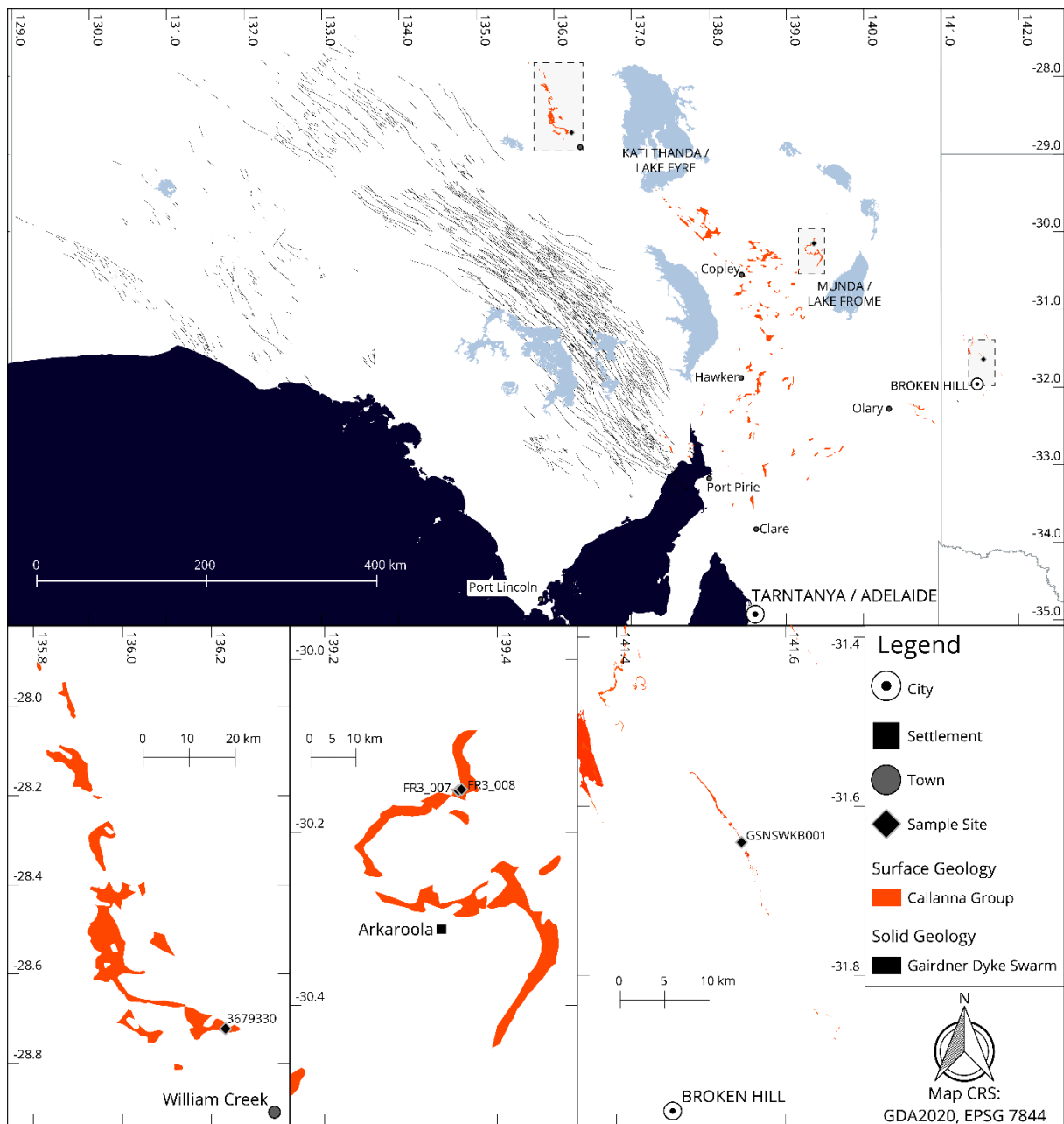


Figure 2 – Distribution of the exposed Callanna Group rocks (orange), the subsurface distribution of the Gairdner Dolerite (black lines), and insets showing sample locations (west to east).

114 The Callanna Group is characterised by initially siliciclastic sedimentation transitioning to carbonate
 115 and evaporite dominated deposition, with minor, interbedded, mafic to intermediate volcanic and
 116 volcanogenic sequences. The Arkaroola Subgroup [Figure 3, Supplementary Figure S1] comprises of
 117 basal siliciclastic units (e.g., Younghusband Conglomerate, Paralana Quartzite), overlain by a (meta-
 118 carbonate unit (e.g., Wywyana Formation) and finally capped by mafic (meta-)igneous rocks (e.g.,
 119 Wooltana Volcanics). The basal siliciclastic and middle carbonate sequences are thought to be
 120 deposited in sag basins from the gradual subsidence of a stable craton, prior to rifting (Preiss 2000),
 121 or alternately deposition as syn-rift sediments penecontemporaneous with faulting (Coats & Blissett
 122 1971). The igneous sequences at the top of the Arkaroola Subgroup are almost exclusively
 123 metabasaltic rocks with minor interbedded sediments (Hillyard 1990; Preiss 1987). These igneous
 124 sequences are inferred to have been extruded in subaerial settings (Hillyard 1990) as continental

125 tholeiitic (flood) basalts (Crawford & Hillyard 1990; Hillyard 1990; Powell 1998; Preiss 1987; 2000;
126 Wade, CE et al. 2014). The Wooltana Volcanics and its equivalent units of the uppermost Arkaroola
127 Subgroup are the most voluminous igneous rocks recognised in the Adelaide Superbasin and have
128 been termed the Willouran Large Igneous Province (LIP); alternately, Willouran Basic Province or
129 Gairdner LIP (Crawford & Hillyard 1990; Hillyard 1990; Huang et al. 2015; Wade, CE et al. 2014;
130 Wang, X-C et al. 2010; Werner et al. 2018; Wingate et al. 1998). Neoproterozoic mafic volcanics of
131 the Coompana Province, c. 860 Ma may also be part of the Willouran LIP (Travers 2015). The
132 Willouran LIP [Figure 2] is interpreted to represent the first major phase of rifting within the Adelaide
133 Superbasin, and thus initiation of Rodinia break-up at the eastern margin of Proterozoic Australia that
134 led to development of the palaeo-Pacific Ocean (Merdith et al. 2017b). At present day, the only
135 exposures of complete sections of the Arkaroola Subgroup [Figure 3] are located in the
136 Arkaroola/Mount Painter area, and the Davenport and Denison Ranges (Peake and Denison Inliers)
137 [Supplementary Figure S1]. Isolated blocks of Arkaroola Subgroup are recognised in carbonate
138 megabreccia (diapirs) throughout the Adelaide Superbasin, but particularly within the Willouran
139 Ranges. The equivalent Poolamacca Group crops out in the Barrier Ranges of New South Wales
140 [Figure 1].

141 The Curdimurka Subgroup is thought to
 142 overlie the Arkaroola Subgroup and locally
 143 exceeds 8 km stratigraphic thickness. As
 144 a result of tectonic, and salt tectonic
 145 dismemberment, no wholly intact section
 146 through the Curdimurka Subgroup has
 147 been identified (Ambrose et al. 1981;
 148 Forbes et al. 1981; Hearon IV et al. 2015;
 149 Mackay 2011; Preiss 1985; 1987; 2000).
 150 However, composite sections have been
 151 developed for the Willouran Ranges
 152 (Forbes et al. 1981) [Figure 3], the
 153 Davenport and Denison Ranges (Ambrose
 154 et al. 1981), the Worumba Anticline
 155 (Preiss 1985), and the Spalding Inlier
 156 (Preiss 1974). The most intact of these
 157 composite sections is within the Willouran
 158 Ranges [Supplementary Figure S1]. The
 159 Curdimurka Subgroup is comprised of a
 160 cyclical sequence of evaporitic mixed
 161 carbonate, and siliciclastic rocks, with
 162 minor intermediate to felsic igneous rocks
 163 (Ambrose et al. 1981; Fabris et al. 2005;
 164 Mackay 2011; Preiss 1987; 2000;
 165 Stüeken et al. 2019). The carbonate
 166 sequences comprise of stromatolitic
 167 limestones and dolostones, and cryptalgal
 168 dolostone with abundant evaporite
 169 mineral pseudomorphs and locally, tepee
 170 structures. The siliciclastic sequences
 171 include laminated, pyritic, and
 172 carbonaceous siltstone, and sandstones
 173 and siltstones with occasional graded
 174 bedding, halite casts and load casts.
 175 Additionally, feldspathic, and carbonate-
 176 cemented, cross-bedded sandstone, with
 177 occasional heavy mineral laminations and
 178 halite casts are present. The stratigraphic
 179 names of the Callanna Group, general
 180 geographic locations, and approximate
 181 relative stratigraphic positions are
 182 outlined in Supplementary Figure S1.

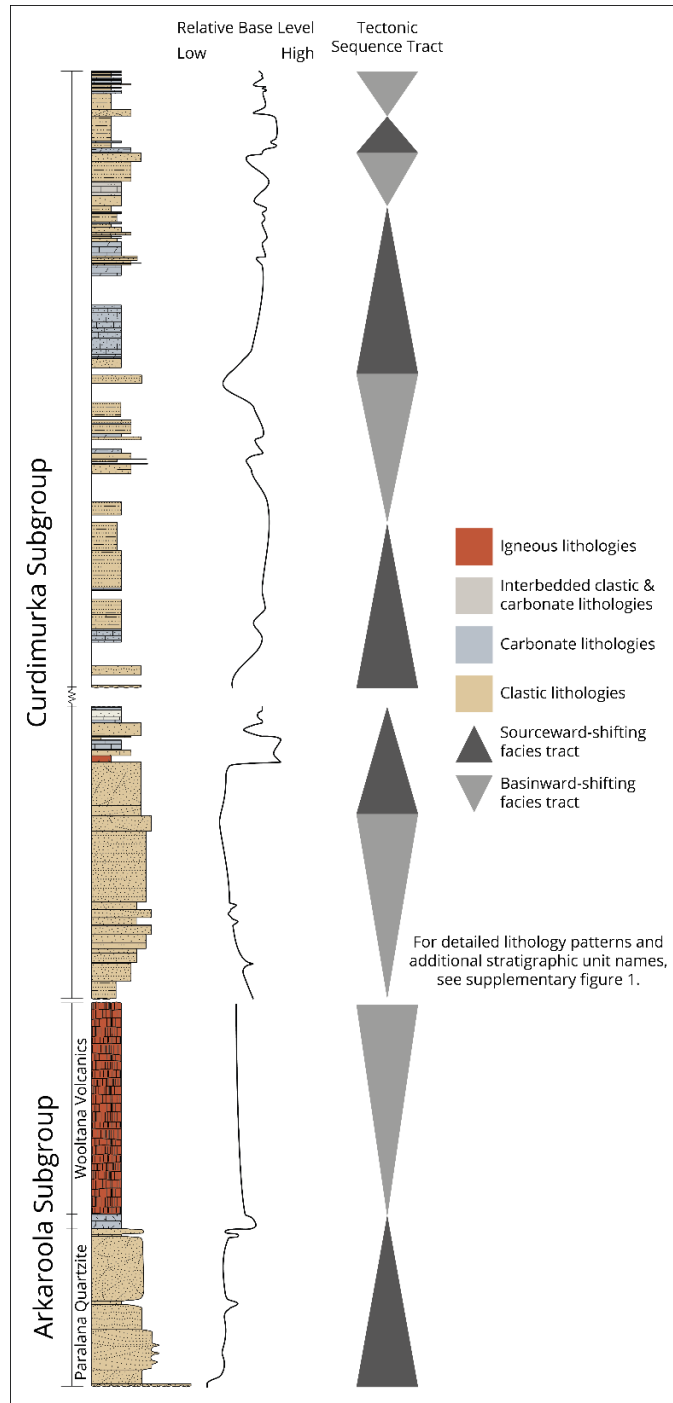


Figure 3 – Simplified (composite) stratigraphic log of the Callanna Group based on the type sections from Arkaroola and the Willouran Ranges. Relative base level utilises further information from Mackay (2011) and Preiss (1987; 2000). Tectonic successions follow terminology of Matenco and Haq (2020) sourceward-shifting facies tracts are where accommodation space is created faster than the rate of sediment supply ($\delta AS/SS = >1$) and basinward-shifting facies tracts are where the rate of sediment supply outdoes the creation of accommodation space ($\delta AS/SS = <1$).

183 3 Methods

184 Three samples were analysed for detrital zircon geochronology, two from the Paralana Quartzite
185 (FR3_007, FR3_008), and one from the Lady Don Quartzite in the eastern part of the superbasin
186 (GSNSWKB001; see Figure 2). A fourth volcano-sedimentary sample, 3679330, was also analysed in
187 the hope of obtaining an indication of the crystallisation age of the Cadlareena Volcanics—a
188 presumed equivalent of the Wooltana Volcanics (Ambrose et al. 1981; Figure 3). These samples were
189 selected to investigate the provenance earliest sedimentary rocks of the Adelaide Superbasin, and
190 any spatially related variations in coeval sequences.

191 Rock samples were first prepared for detrital zircon analysis by crushing the rock samples using a jaw
192 crusher, disk mill, then sieved using nylon mesh of 79 μm and 400 μm . All equipment was thoroughly
193 cleaned by vacuuming, ethanol, and compressed air between each sample. New sieve mesh was
194 used for each sample. Mineral separation was completed by water panning of the 79–400 μm
195 fraction and use of LST heavy liquid set to a density of $2.85 \pm 0.02 \text{ gcm}^{-3}$. Zircon was then handpicked
196 and mounted in an epoxy resin. Any grain that remotely resembled a zircon was picked to minimise
197 human bias, an issue highlighted by Slama and Košler (2012) and Dröllner et al. (2021). Where
198 permitted by zircon yields, at least 300 zircons were picked per sample, otherwise all zircons in the
199 sample were picked. The mounts were then imaged via cathodoluminescence on either a FEI Quanta
200 600 scanning electron microscope (for zircon analysed in 2020) or a Cameca SXFive Electron
201 Microprobe (for zircon analysed in 2021). The zircons were then analysed using Laser Ablation
202 Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) to obtain a suite of elemental data for
203 U–Pb geochronology and rare earth element (REE) analysis. All zircons were analysed using a
204 Resonetics M-50 (193 nm ArF excimer) laser ablation system coupled with an Agilent 7900x
205 inductively coupled plasma mass spectrometer. All analytical instruments used are housed at
206 Adelaide Microscopy, University of Adelaide, Australia.

207 Four standards were used during analysis, GEMOC GJ-1 (Horstwood et al. 2016; Jackson et al.
208 2004), Plešovice (Horstwood et al. 2016; Sláma et al. 2008), 91500 (Horstwood et al. 2016;
209 Wiedenbeck, M. et al. 1995; Wiedenbeck, Michael et al. 2004), and the NIST610 glass (Jochum et al.
210 2011). Unknowns were bracketed by two analyses of GJ-1, followed by a combined two to three
211 analyses of Plešovice and 91500, and two analyses of NIST610 every 20–30 unknowns. GJ-1 was
212 used as the primary calibration standard for U–Pb ratios and NIST610 was used as the primary
213 calibration standard for Pb isotope ratios and trace element data. ^{91}Zr was used as the internal
214 standard for trace element data with a value of 431,400 ppm (43.14 wt%) ^{91}Zr assigned to
215 unknowns. Plešovice and 91500 were used as validation standards. A 30 second gas blank followed
216 by either a 40 second or 30 second ablation (session on 2021-03-30) time was used with a laser
217 repetition rate of 5 Hz. A spot size of 29 μm and a nominal fluence of 2 Jcm^{-2} was used for zircon, and
218 a spot size of 43 μm using a nominal fluence of 3.5 Jcm^{-2} was used for NIST610. Data were
219 processed using LADR (Norris & Danyushevsky 2018), version 1.1.06 and output as “Full Analytical
220 Uncertainty”. No common Pb corrections were applied to the data. Reference material ratios for GJ-
221 1, Plešovice, and 91500 were set to the Chemical Abrasion Isotope Dilution Thermal Ionisation Mass
222 Spectrometry (CA-ID-TIMS) values (uncorrected for thorium disequilibria and common-Pb) of
223 Horstwood et al. (2016). Weighted averages and dispersion statistics for all standards are available
224 from the link in data availability.

225 Statistical analysis of the zircon U–Pb data follows the method of Lloyd et al. (2020). Data are
 226 considered concordant if within $\pm 10\%$, and a “meaningful” age if the 2σ uncertainty is $\leq 10\%$ —if a
 227 datum satisfies both parameters it is termed a *Filtered Age*. Maximum depositional ages are
 228 determined from a stricter 2% concordance filter and use the older age of the three isotope ratios
 229 ($^{207}\text{Pb}/^{235}\text{U}$, $^{206}\text{Pb}/^{238}\text{U}$, $^{207}\text{Pb}/^{206}\text{Pb}$) for a conservative estimate of the youngest single concordant
 230 grain. All ages are quoted with 2σ uncertainty. Kernel density estimates (KDEs), and multidimensional
 231 scaling plots (MDS) were generated using IsoplotR (Vermeesch 2018). Key zircon trace element data
 232 are presented graphically using methods following Verdel et al. (2021) and additionally lanthanoid
 233 data are represented using violin plots and lambda representation (Anenburg 2020; O’Neill 2016).

234 Metadata for the LA-ICP-MS sessions,
 235 data for all analyses,
 236 cathodoluminescence images, and R
 237 code used to generate plots are
 238 available from the links in data and code
 239 availability.

240 4 Results

241 A total of 161 analyses were conducted
 242 for sample FR3_008. Of these, 141
 243 analyses passed filtering parameters,
 244 with ages ranging from 2914 ± 46 Ma to
 245 892 ± 13 Ma [Figure 4]. The primary
 246 population peak of this sample is c.
 247 1550 Ma, with a secondary peak at c.
 248 1750 Ma, and tertiary peaks c. 1180 Ma
 249 and 935 Ma. Four analyses lie outside
 250 these populations, ranging from $2914 \pm$
 251 46 Ma to 2237 ± 57 Ma.

252 A total of 125 analyses were conducted
 253 for sample FR3_007. Of these, 99
 254 analyses passed filtering parameters,
 255 with ages ranging from 3090 ± 31 Ma to
 256 1305 ± 17 Ma [Figure 4]. The primary
 257 population peak of this sample is c.
 258 1680 Ma, with secondary population
 259 peaks c. 2480 Ma, 2000 Ma, and 1480
 260 Ma. Three analyses lie outside these
 261 populations, ranging from 3097 ± 27 Ma to 2819 ± 60 Ma.

262 A total of 114 analyses were conducted for sample GSNSWKB001. Of these, 85 analyses passed
 263 filtering parameters, with ages ranging between 3090 ± 31 Ma to 1302 ± 23 Ma [Figure 4]. The
 264 primary population peak of this sample is c. 1620 Ma, with a secondary peak c. 1840 Ma. These two
 265 peaks form a bimodal population ranging from 1999 ± 32 Ma to 1302 ± 23 Ma.

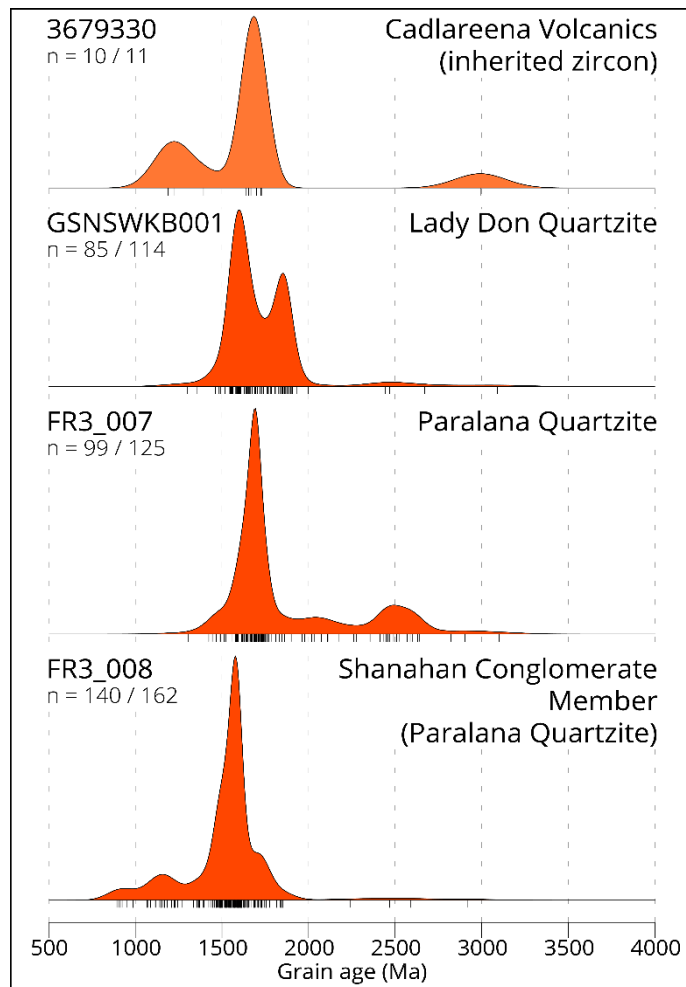


Figure 4 – Kernel density estimate plots of the four samples analysed in this study. These are in ascending stratigraphic order. Tick marks below each plot represent an analysis. n = filtered analyses / total analyses. Created using IsoplotR (Vermeesch 2018)

266 From the small quantity of sample that was crushed for sample 3679330, 11 zircons were obtained
267 and analysed, with ten of these within
268 filtering parameters. The oldest grain
269 yielded an age of 2992 ± 27 Ma, the
270 youngest grain was 1189 ± 18 Ma, and
271 the remainder range between 1222 ± 22
272 Ma and 1725 ± 24 Ma with a cluster of
273 four grains c. 1680 Ma [Figure 4].

274 Lanthanoid concentrations are typical for
275 zircons, with several orders-of-magnitude
276 increase in concentration from light to
277 heavy elements, a slight negative
278 deviation in europium (Eu), and a positive
279 deviation in cerium (Ce) [Figure 5].

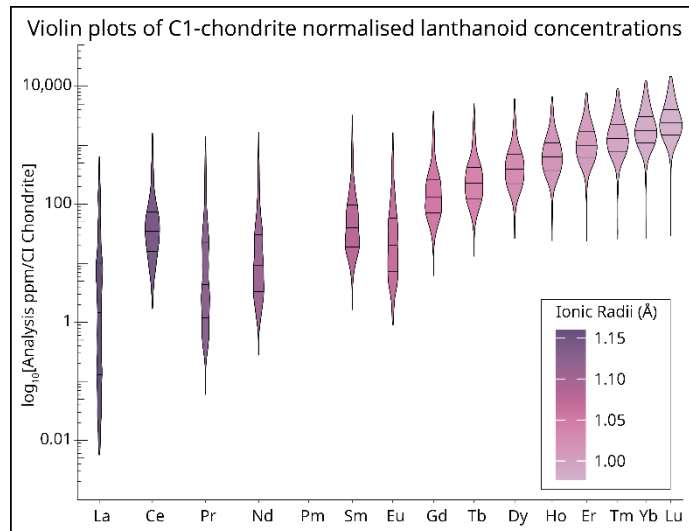


Figure 5 – Violin plots of CI chondrite (O'Neill 2016) normalised lanthanoids for all filtered zircon analysed in this study. X-axis is spaced by ionic radii (Shannon 1976) and ordered by atomic number. Black lines across the fill of each plot represent the 0.25, 0.5, and 0.75 quantiles. Bandwidth of the density estimates is calculated using the Botev algorithm from the Provenance package (Vermeesch et al. 2016).

280 5 Discussion

281 5.1 Provenance and 282 maximum depositional 283 ages

284 5.1.1 Paralana Quartzite, incl. Shanahan Conglomerate Member

285 Samples FR3_007 and FR3_008 were both sampled from the Paralana Quartzite; however, FR3_008
286 was sampled from a stratigraphically lower position, mapped as the Shanahan Conglomerate
287 Member. The MDA of the Paralana Quartzite will combine the results of both samples.

288 The youngest zircon in FR3_008 (analysis FR3_008 - 090, Figure 6) originally yielded $^{207}\text{Pb}/^{235}\text{U}$,
289 $^{206}\text{Pb}/^{238}\text{U}$, and $^{207}\text{Pb}/^{206}\text{Pb}$ ages of 897 ± 46 Ma, 896 ± 18 Ma, and 889 ± 39 Ma, respectively. To
290 verify the age obtained, this zircon was reanalysed on a subsequent analytical session with two
291 additional analyses. The second analysis (FR3_008_run2 - 003, Figure 6) yielded $^{207}\text{Pb}/^{235}\text{U}$,
292 $^{206}\text{Pb}/^{238}\text{U}$, and $^{207}\text{Pb}/^{206}\text{Pb}$ ages of 893 ± 39 Ma, 892 ± 13 Ma, 886 ± 30 Ma, respectively. The third
293 analysis (FR3_008_run2 - 004, Figure 6) yielded a younger discordant age likely due to a small

294 inclusion that can be seen in Figure 6. The
 295 two concordant signals have Th/U ratios
 296 of ~0.55, and the discordant analysis has
 297 a Th/U ratio of ~1.3. A concordia age of
 298 893 ± 9 Ma, MSWD 0.067, $p(\chi^2)$ 0.98 is
 299 calculated from the two concordant
 300 analyses and a traditional uncertainty
 301 weighted mean yields a $^{206}\text{Pb}/^{238}\text{U}$ age of
 302 893 ± 10 Ma, MSWD 0.14 Ma, $p(\chi^2)$ 0.71—
 303 both age determinations propagate
 304 external uncertainties. The zircon is
 305 euhedral with simple regular growth
 306 zoning with a {101} form (Corfu et al.
 307 2003; Pupin 1980). Although one end of
 308 the zircon appears to have broken off, the
 309 aspect ratio is at least 3.3:1. As the
 310 concordia age is the statistically “most
 311 likely” age, (Ludwig 1998; Vermeesch
 312 2021), uses the most amount of available
 313 analytical data from the multiple analyses
 314 of the single grain, and is in good
 315 agreement with individual calculated
 316 decay ages and the $^{206}\text{Pb}/^{238}\text{U}$ weighted
 317 mean, this is used as the age of
 318 crystallisation, and subsequently the
 319 maximum depositional age for the
 320 Paralana Quartzite. This revises the
 321 maximum depositional age of the
 322 Paralana Quartzite down from 1177 ± 28
 323 Ma (Lloyd et al. 2020) to 893 ± 9 Ma.

324 Both samples, FR3_008 (Shanahan
 325 Conglomerate Member) and FR3_007
 326 (Paralana Quartzite), have an overlapping
 327 population of zircons c. 1800–1300 Ma,
 328 with their primary population peaks
 329 centred c. 1580 Ma and c. 1690 Ma
 330 respectively [Figure 4]. These primary
 331 zircon populations are likely to be derived
 332 locally from the Ninnerie Supersuite and/or Radium Creek Group (Armit et al. 2014; Kromkhun et al. 2013; Wade, CE 2011; Wade, CE et al. 2012). The two sample populations differ significantly with the direction of the population tails. Sample FR3_008 tails toward younger ages with an additional minor population peak c. 1150 Ma and small cluster of grains c. 900 Ma [Figure 4]. There are only four zircons older than c. 1850 Ma present in sample FR3_008. In contrast, sample FR3_007 tails toward older ages, with an additional minor population peak c. 2500 Ma [Figure 4] and no zircon younger

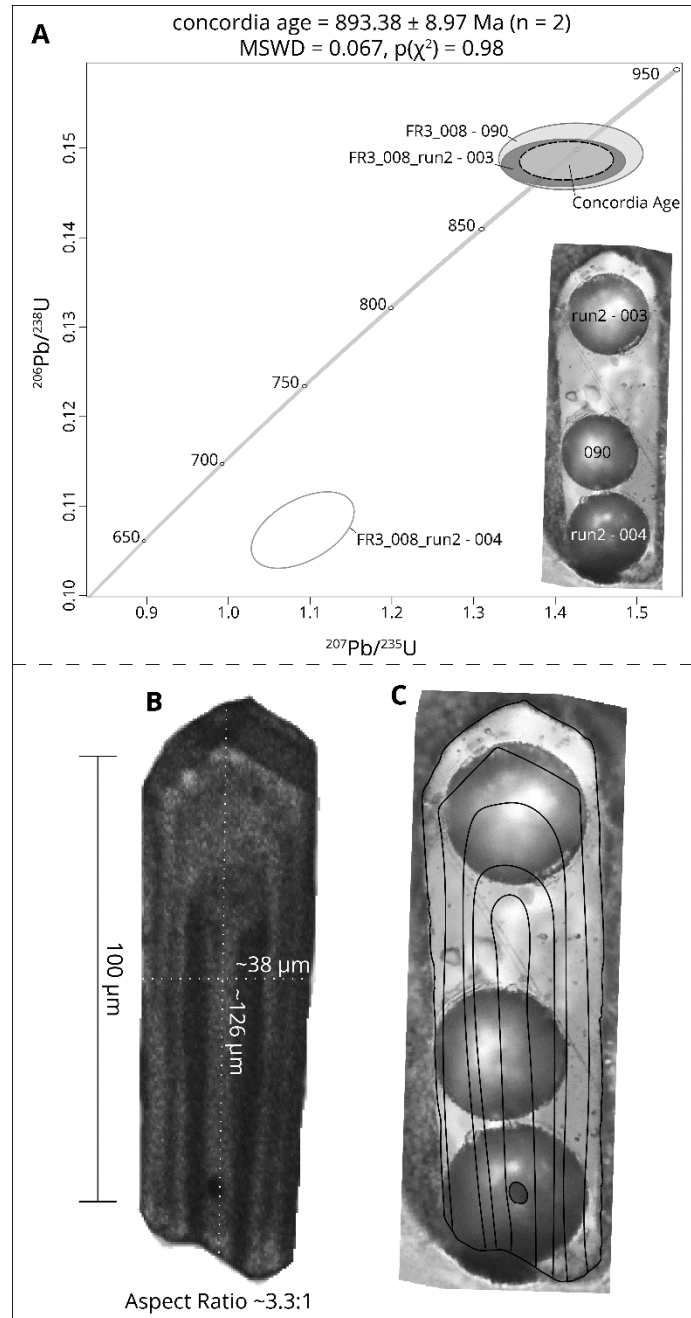


Figure 6 – A) Concordia plot and age of the three spots analysed on the youngest single zircon for FR3_008 (Shanahan Conglomerate Member). Spots are labelled on the reflected light image. B) Cathodoluminescence image overlain with measurements for aspect ratio. C) Outlines of growth zones and inclusion overlain on reflected light image. Concordia plot generated using IsoplotR (Vermeesch 2018).

338 than c. 1300 Ma. Zircons from the older tail of sample FR3_008, particularly the c. 2500 Ma,
 339 population are most likely derived from the Gawler Craton [Figure 7], namely the Mulgathing Complex
 340 and Sleaford Complex (Reid et al. 2014; Reid & Payne 2017; Williams, MA & Reid 2021), as has been
 341 previously suggested (Lloyd et al. 2020). The younger c. 1300–1050 Ma zircon population in sample
 342 FR3_008 is most likely derived from the Musgrave Province [Figure 7] (Howard et al. 2015; Smithies
 343 et al. 2008; Smithies et al. 2011; Smits et al. 2014; Wade, BP et al. 2008); however, they could
 344 alternately be sourced from a yet undiscovered but inferred Musgrave like, late Mesoproterozoic (c.

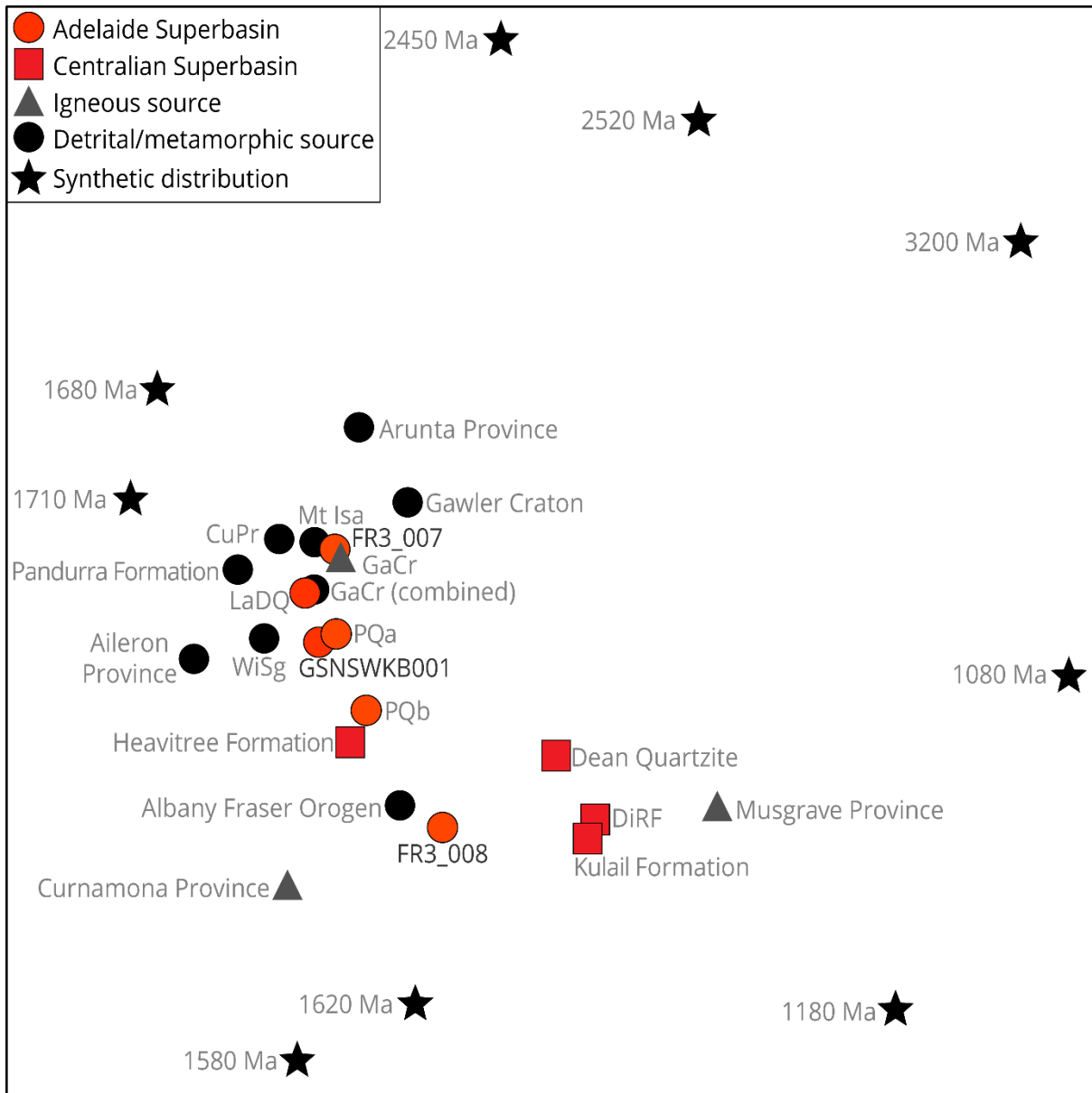


Figure 7 – Non-metric multidimensional scaling plot of samples analysed (n > 40) in this study (orange circles) with data from potential correlative formations of the Centralian Superbasin (red squares), potential source regions (black and grey circles and triangles), and synthetic distributions of main population peaks and key zircon growth events in the region. This plot shows relative similarity of all data to each other and are intended as a visual guide. Points that plot closer together suggest greater similarity. Axes are omitted as the algorithm used produces normalised values with no physical meaning and can be safely removed. Produced using IsoplotR (Vermeesch 2018). Abbreviations: CuPr = Curnamona Province; GaCr = Gawler Craton (combined signifies detrital, metamorphic, and igneous data); WiSg = Willyama Supergroup, DiRF = Dixon Range Formation, PQ = Parana Quartzite data from this study and existing literature (a = does not include Shanahan Conglomerate Member data, b = includes Shanahan Conglomerate Member data).

345 1300–1000 Ma) source to the east (Fergusson et al. 2007; Korsch et al. 2012; Mackay 2011;
346 Wysoczanski & Allibone 2004). The five youngest zircons present in sample FR3_008, younger than
347 1000 Ma, are enigmatic. They have no known local source terrane and given the euhedral to
348 subhedral nature of these grains, and the breccia-conglomerate nature of the rock, it is unlikely they
349 have been transported a great distance. It is possible these zircons are derived from an, as yet,
350 undiscovered or previously destroyed minor magmatic sequence that would mark initial volcanism of
351 the Adelaide Superbasin that precedes flood basalt emplacement. The zircon populations and
352 lithological differences between the two samples, which were sampled approximately 350 m from
353 each other, suggest a change in sediment source up stratigraphy to include a greater percentage of
354 more distal source areas, and a loss of the younger source material.

355 5.1.2 Lady Don Quartzite

356 Sample GSNSWKB001 was sampled from the Lady Don Quartzite in New South Wales. Based on
357 lithology and stratigraphic position it is believed that this formation and the Christine Judith
358 Conglomerate are equivalents to the basal Callanna Group. The maximum depositional age obtained
359 for sample GSNSWKB001 is 1497 ± 52 Ma. There are a few zircons younger than this in the sample
360 with the youngest being 1302 ± 23 Ma; however, all these younger zircons are slightly discordant
361 ($>2\%$, $<10\%$). The sample's zircon age population is similar to that of the Paralana Quartzite [Figure
362 4, Figure 7] samples, with a primary population peak c. 1580 Ma but includes an additional
363 prominent population peak c. 1850 Ma. There are a few zircons with ages older than 2000 Ma, with
364 one c. 3090 Ma, one c. 2670 Ma, and two c. 2450 Ma. The primary zircon population c. 1580 Ma is
365 likely to be derived locally from the Ninnerie Supersuite and Radium Creek Group (Armit et al. 2014;
366 Kromkhun et al. 2013; Wade, CE 2011; Wade, CE et al. 2012), lending support to stratigraphic
367 correlation of the basal Adelaide Superbasin sequences [Figure 7]. The additional population c. 1850
368 Ma is potentially derived from the underlying Willyama Supergroup (Conor & Preiss 2008; Page et al.
369 2005) that has been suggested to ultimately be derived from the Arunta Province (Barovich & Hand
370 2008; Payne et al. 2006). The few zircon grains older than 2000 Ma are also potentially derived from
371 recycling of the underlying Willyama Supergroup. The rarity of these >2000 Ma zircons suggests
372 direct transport from the Gawler Craton where these ages are found, namely the Mulgathing
373 Complex, Sleaford Complex, and Cooyerdoo Granite (McAvaney 2012; Reid et al. 2014; Reid & Payne
374 2017; Williams, MA & Reid 2021), is unlikely.

375 5.1.3 Cadlareena Volcanics

376 The small Cadlareena Volcanics sample, 3679330, only yielded 10 zircons that are all interpreted to
377 be inherited/detrital as there is significant spread with no apparent clustering in the individual ages
378 [Figure 4], and most of the zircon are subhedral and fragmented. The sample's physical appearance
379 suggests that the rock is a silicified, intermediate volcano-sedimentary rock, so this result is
380 unsurprising. From this we interpret a maximum depositional age of 1189 ± 18 Ma. The ages of the
381 zircon align with those found in the broader region, namely that of the Pitjantjatjara Supersuite of the
382 Musgrave Province (Close 2013; Smithies et al. 2011) and the Tunkillia Suite of the Gawler Craton
383 (Hand et al. 2007; Morrissey et al. 2019; Payne et al. 2010).

384 5.1.4 Comparison to basal Central Superbasin sequences

385 The Centralian Superbasin developed as an intracontinental basin coeval with the Adelaide
386 Superbasin (Munson et al. 2013; Walter et al. 1995), which developed relatively independently from
387 each other (Preiss 2000). Geochronologic control, and thus correlation, of several stratigraphic units
388 across the lower Adelaide Superbasin and Centralian Superbasin remain poor (Lloyd et al. 2020;
389 Normington & Donnellan 2020). However, the lowermost units are commonly correlated based on
390 stratigraphic similarity and position (Drexel et al. 1993; Normington & Donnellan 2020; Preiss 1987;
391 Walter et al. 1995). In the Centralian Superbasin these formations are the Heavitree Formation, Dean
392 Quartzite, Vaughn Springs Quartzite, Amesbury Quartzite, Munyu Sandstone, and Kulail Sandstone
393 (Haines & Allen 2017; Normington & Donnellan 2020; Normington & Edgoose 2018). These are
394 thought to be equivalents to the Adelaide Superbasin formations from which samples analysed in this
395 study were obtained. When the detrital zircon age populations are compared, two main groupings
396 appear, the units of the Centralian Superbasin form one group separate from those of the Adelaide
397 Superbasin [Figure 7]. This suggests that the two basins received detritus from differing sources.
398 However, two exceptions occur, the Heavitree Formation and the Shanahan Conglomerate Member.
399 These two units both plot [Figure 7] as an intermediary to the more obvious groupings of the
400 Centralian Superbasin and Adelaide Superbasin sequences, suggesting a shared or similar primary
401 detritus source. This is more easily explained for the Heavitree Formation, a relatively mature sandy
402 unit, as the Arunta region, which has somewhat similar zircon age populations as the Gawler Craton,
403 is inferred to be a major source of detritus for the Heavitree Formation (Al-Kiyumi 2018; Maidment et
404 al. 2007). This intermediary position on [Figure 7] is much harder to reconcile for the Shanahan
405 Conglomerate Member, as this unit is an immature, breccia–conglomerate, and is unlikely to have
406 received detritus from distal sources, and as stated earlier, no local source of young zircon is known.
407 The lends some support to the notion of a potential (Fergusson et al. 2007; Korsch et al. 2012;
408 Mackay 2011; Wysoczanski & Allibone 2004) Stenian–Tonian source to the east.

409 5.2 Zircon trace element geochemistry

410 Zircon trace element chemistry, particularly of the lanthanoids, uranium (U), thorium (Th), yttrium (Y),
411 oxygen (O), and hafnium (Hf), can be useful in understanding their petrogenesis and provenance, and
412 for crustal evolution (Campbell et al. 2020; Fernandes et al. 2021; Grimes, Craig B. et al. 2009;
413 Grimes, C. B. et al. 2007; Grimes, C. B. et al. 2015; Hawkesworth & Kemp 2006; Verdel et al. 2021).
414 While lanthanoid geochemistry is not thought to be particularly useful in assisting with provenance
415 determinations (Hoskin & Ireland 2000), it is useful at a broader scale for understanding the
416 continental history of a region. Here we make general observations about the trace element
417 geochemistry of detrital zircon from the lowermost Adelaide Superbasin analysed in this study.

418 First, as a straightforward measure of continental or oceanic affinity for zircon generation, one can
419 use U/Yb plotted against Y (Grimes, C. B. et al. 2007; Grimes, C. B. et al. 2015). All zircons analysed

420 in this study are inferred to have been
 421 generated in continental crust as shown by
 422 [Figure 8]. C1 chondrite normalised (O'Neill
 423 2016) concentrations of lanthanoids are
 424 typical of zircon [Figure 5] with a positive
 425 pattern slope (decreasing λ_1 values) from
 426 light to heavy lanthanoids, a positive cerium
 427 anomaly, and negative europium anomaly
 428 (Hoskin & Ireland 2000; Hoskin &
 429 Schaltegger 2003). Nearly all zircons have a
 430 Th/U >0.07 and are inferred to be originally
 431 generated as magmatic rather than
 432 metamorphic zircon (Collins et al. 2004;
 433 Rubatto 2002). There is no apparent trend
 434 in lanthanoid pattern slope or curvature
 435 [Figure 9], denoted as λ_1 (linear slope), λ_2
 436 (quadratic slope), and λ_3 (cubic slope)
 437 (Anenburg 2020), with time or sample. Both
 438 Eu and Ce anomalies (denoted by Eu* and
 439 Ce*) show a significant spread through time.
 440 However, while statistical confidence is
 441 limited due to the low number of samples <1000 Ma, it is observed that these generally have low Eu*
 442 and Ce* values ("low" is used as in Verdel et al. 2021, i.e. "strongly negative"). The positive
 443 correlation of low Eu* and Ce* values may suggest crystallisation in reduced conditions, thick crust,
 444 sediment incorporation, deep mantle plume, effects of fractional crystallisation, and/or competition
 445 with plagioclase and/or monazite (Verdel et al. 2021). The slight increase in Yb/U [Figure 9] in these
 446 younger zircons suggests the addition of MORB like, or juvenile mantle-derived magmatism, which is
 447 consistent with this type of magmatism accompanying Rodinia rifting.

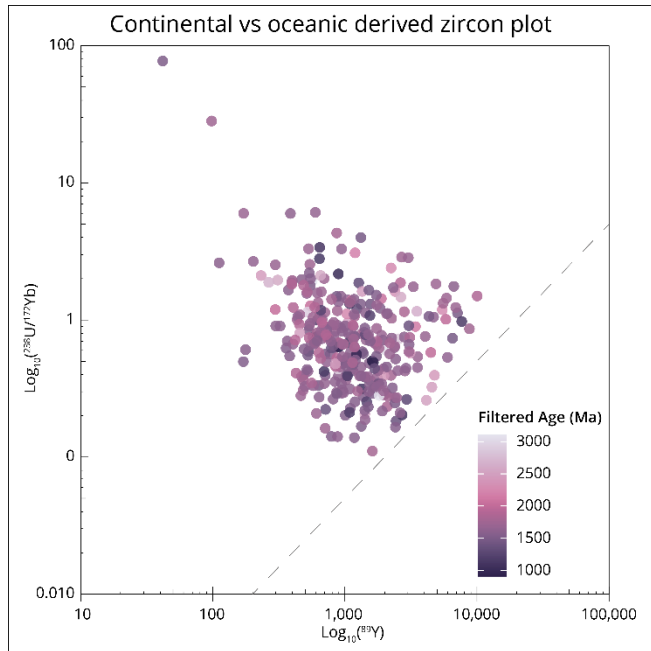


Figure 8 – Plot based on (Grimes, C. B. et al. 2007) used as an indicator of zircon crustal origin. This plots Y against U/Yb, with the dashed reference line dividing the “oceanic” (below line) and “continental” (above line) fields. All data plot above the reference line, suggesting zircon formation in crust of continental affinity. Coloured by filtered age where light is older and darker is younger.

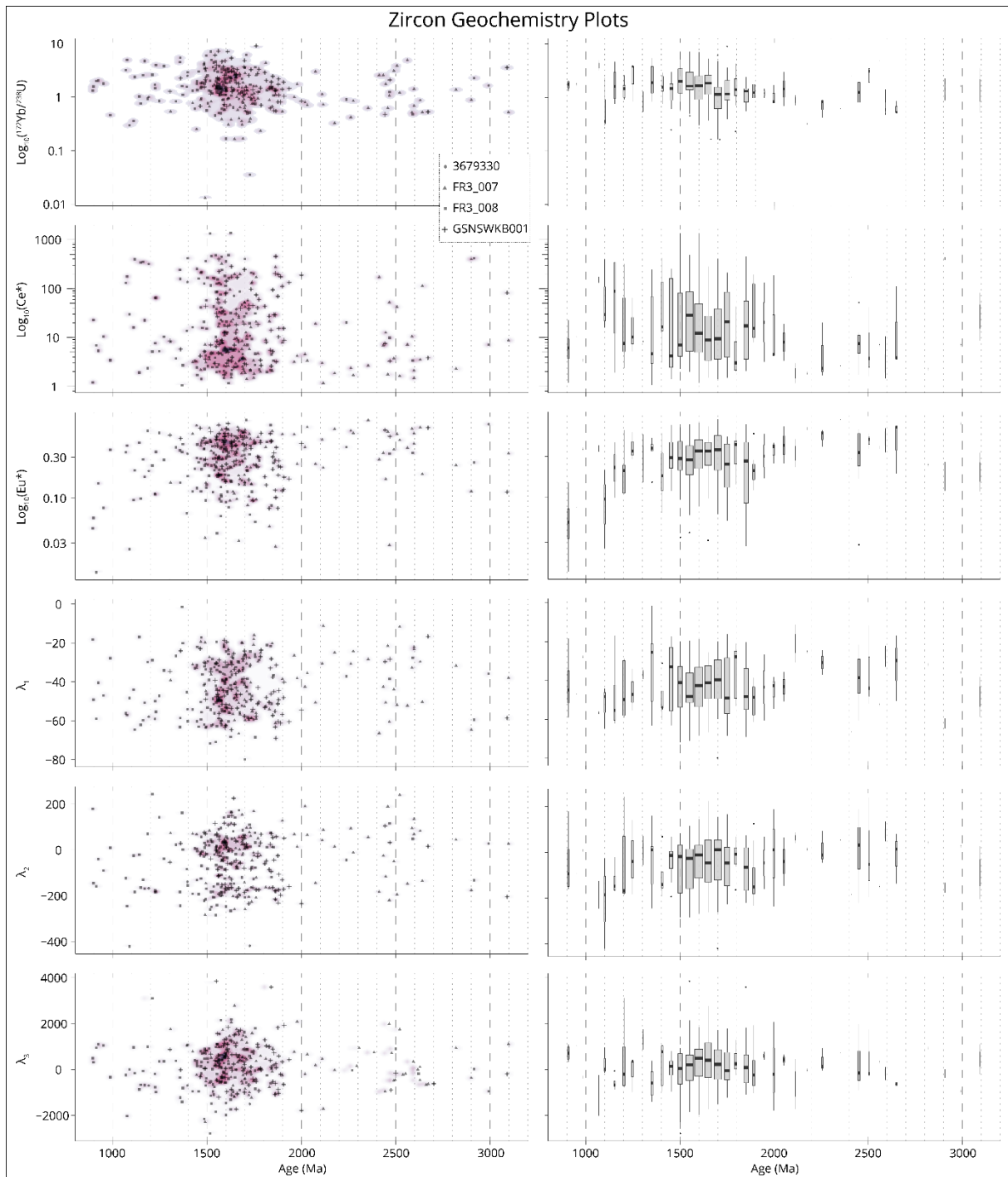


Figure 9 – Key zircon geochemistry plots for zircon analysed in this study. Left: Scatter plots underlain with 2D density estimation. Right: 50 million year binned boxplots with width scaled by the count of values in the bin. Top to bottom: Yb/U, Ce*, Eu* and λ_1 –3. λ_1 –3 are measures of lanthanoid pattern shapes, with λ_1 –3 representing the linear slope, quadratic slope, and cubic slope, respectively. Ce*, Eu* and λ_1 –3 are calculated using BLambdaR (Anenburg & Williams 2021).

448

449 5.3 Willouran Large Igneous Province and Palaeogeography

450 Previous authors (Li & Powell 2001; Li et al. 1995; Mackay 2011; Wang, X-C et al. 2010) have
 451 advocated for a spatial link of the Willouran Large Igneous Province (LIP) and the Guibei LIP primarily
 452 based on igneous geochemistry, palaeomagnetic poles, and geochronology. These authors advocated

453 for a link between southeast Proterozoic Australia and South China within Rodinia, the “missing link”
454 model (Li et al. 1995). Wen and co-authors (Wen et al. 2017; Wen et al. 2018) developed an
455 alternative missing link model placing Tarim between Australia and Laurentia instead of South China.
456 However, an increasing number of studies examining detrital zircon (e.g., Cawood et al. 2020;
457 Cawood et al. 2018; Hui et al. 2021; Wang, P et al. 2020), geodynamic and kinematic studies (e.g.,
458 Merdith et al. 2017a; Merdith et al. 2017b; Wu et al. 2021), and a recent comprehensive review and
459 update to palaeomagnetic poles (Park et al. 2021) suggest that this position of South China (or Tarim)
460 within the centre of Rodinia is unlikely. Further, this infers that the Willouran LIP and Guibei LIP are
461 not spatially linked as has been previously suggested (Li & Powell 2001; Li et al. 1995; Mackay 2011;
462 Wang, X-C et al. 2010; Werner et al. 2018). The new detrital zircon data in this study further supports
463 that the missing link model with Tarim or South China for Rodinia is unlikely. Our detrital zircon data
464 [Figure 4] lack the prominent c. 800 Ma population that is present in samples from Tarim (Wang, P et
465 al. 2020) and South China (Cawood et al. 2013; Cawood et al. 2018). Our data also preserve
466 prominent populations at c. 1580 Ma and c. 1840 Ma that are not prominent within samples from
467 either Tarim or South China.

468 5.4 Early evolution of the Adelaide Superbasin

469 Much of the Callanna Group has been either eroded, tectonically dismembered or disrupted by
470 diapirs, and geochronologic controls on deposition remain poor. There are also limited seismic
471 surveys that cross the Adelaide Superbasin (Korsch & Kositsin 2010), and none over key areas where
472 good stratigraphic control on the Callanna Group is possible (e.g., Willouran Ranges). This makes
473 reconstructing the earliest sequences of the Adelaide Superbasin and its evolution particularly
474 difficult. Here, using existing research on the basin, drawing on literature concerning modern (e.g.,
475 East African Rift: Boone et al. 2021; Corti 2009; Keranen & Klempner 2008; Purcell 2018; Varet
476 2018; Williams, FM 2016; Zwaan et al. 2020) and ancient (e.g., Midcontinent Rift: Allen et al. 2006;
477 Stein et al. 2018) rift systems, and new detrital zircon data presented in this paper, we present an
478 updated model for the early evolution of the Adelaide Superbasin.

479 Initiation of deposition within the Adelaide Superbasin began sometime between 893 ± 9 Ma and c.
480 830 Ma. The initial, thin, and geographically restricted, mostly brecciated/conglomeratic clastic
481 sediments (e.g., Shanahan Conglomerate Member) were likely deposited in a series of small,
482 somewhat asymmetric half grabens with a local detrital source that contain enigmatic young (<1000
483 Ma) zircons. The half grabens are thought to have developed by lithospheric thinning under an initial
484 pulse of minor extension focussed along pre-existing crustal weaknesses (e.g., Norwest Fault, Isan-
485 Olarian orogen: Conor & Preiss 2008; Mackay 2011; Meaney 2017; Morrissey et al. 2013; Nordsvan
486 et al. 2018; Rutherford et al. 2007; Tiddy & Giles 2020; Volante et al. 2020). This initial extension
487 was most probably a result of far-field forces (Cawood et al. 2016), although a mantle plume may
488 have played some role through thermal doming (Hill et al. 1992) or lithospheric weakening. Tectonic
489 quiescence followed, with stable subsidence in the newly created rift, culminating with deposition of
490 alluvial to fluvial sands and shallow water, sometimes stromatolitic, carbonates (e.g., Paralana
491 Quartzite, Wywyana Formation). This is initially reflected in the change in zircon spectra of the
492 Paralana Quartzite [Figure 4] to include a greater diversity of detrital sources before shallower water
493 sediments were laid down. It is likely there was transtensional (Armit et al. 2012; Job 2011; Paul et
494 al. 1999) movement along the Paralana Fault (and its splays) at this time, accounting for the

495 significant thickness variation (~700 m) of the Paralana Quartzite across the fault plane in the
496 Arkaroola area (Preiss 1987). This interpretation differs from that of Preiss (2000), but agrees with
497 Mackay (2011) and Job (2011), in that we consider the Arkaroola Subgroup to be early syn-rift, rather
498 than pre-rift, deposition, but note that the amount of extension was minor. The Arkaroola Subgroup is
499 here considered to reflect a sourceward-shifting facies tract (SFT) (Matenco & Haq 2020), which
500 fines upward after the initial phase of rift basin development [Figure 3]. Dyke emplacement (Gairdner
501 Dolerite, Amata Dolerite) and extrusion of flood basalts (e.g., Wooltana Volcanics, Beda Basalt)
502 occurs at the top of this first SFT and represents the first major phase of extension in the basin. The
503 flood basalts were extruded in subaerial environments and may have originally formed a continuous
504 sheet (Preiss 1987; 2000). After extrusion of the Willouran LIP, rift development continued, at an
505 accelerated rate within well-developed grabens, with the deposition of cyclic clastic-carbonate-
506 evaporative sequences of the Curdimurka Subgroup [Figure 3]. This is consistent with detrital zircon
507 and Nd provenance suggesting a gradual transition from evolved to juvenile, and broad to restricted
508 detrital sources (Barovich & Foden, 2000; Lloyd et al., 2020) over the long-term evolution of the
509 basin. Evacuation of the magma chambers is thought to be partially responsible for major graben
510 subsidence (Hillyard 1990). The Curdimurka Subgroup is at least 8 km thick, much greater in
511 thickness than the Arkaroola Subgroup [Supplementary Figure S1], with significant variations across
512 the basin. Magmatism is known to have continued during deposition of the Curdimurka Subgroup
513 with bimodal volcanics known from the Willouran Ranges (Rook Tuff), a thin basalt flow in the
514 Spalding Inlier, and xenoclasts of dolerite (thought to belong to the Curdimurka Subgroup) in
515 diapirs/carbonate megabreccia zones (Preiss 1987). Constraints on the end of Curdimurka Subgroup
516 deposition, and thus Callanna Group, remain poor. While an exact stratigraphic position has not been
517 determined due to a lack of intact contact relationships (Fabris et al. 2005), the Oodla Wirra
518 Volcanics provide the best determination of a maximum age for the final deposition of the
519 Curdimurka Subgroup where two independent samples yielded ages of 798 ± 5 Ma and 799 ± 4 Ma
520 (Fabris et al. 2005). This is within uncertainty of the 802 ± 10 Ma age of the Rook Tuff (Fanning et al.
521 1986) of the lower to mid Curdimurka Subgroup. However, the age determination from the Rook Tuff
522 needs revising as is not reproducible due to the unavailability of the isotopic data from the original
523 analyses, and increased precision and accuracy can be obtained on modern analytical equipment.
524 The minimum age estimate for deposition of the Curdimurka Subgroup is constrained by the Boucaut
525 Volcanics (Armistead et al. 2020) and a porphyry in a basal member of the Skillogalee Dolomite
526 (Preiss et al. 2009) to c. 790 Ma. The exact stratigraphic position of the Boucaut Volcanics remains to
527 be resolved (Lloyd et al. 2020); however, the position of the Skillogalee Dolomite is well constrained.
528 As such, the Callanna Group–Burra Group transition must occur between c. 800 Ma and c. 790 Ma
529 and allow for deposition of the upper Curdimurka Subgroup and entire Emeroo Subgroup. Deposition
530 of the Emeroo Subgroup marks a southward propagation of the Adelaide Rift Complex during upper
531 Curdimurka Subgroup times, with the most southerly deposition occurred near Spalding (Preiss
532 1987; 2000). A renewed pulse of magmatism (e.g., Boucaut Volcanics, Jarrold Basalt Member,
533 Koorunga Member) occurs at c. 790 Ma (Armistead et al. 2020; Preiss et al. 2009) in the southern and
534 eastern areas of the basin and likely marks a southern shift in tectonic activity and a period of
535 tectonic quiescence of c. 70–80 million years in the northern Adelaide Superbasin.

536 In this model the rift system did not develop as a classic triple junction system through apical
537 extension as was suggested by von der Borch (1980) and Zhao et al. (1994). Instead, the northern
538 and central areas of the Adelaide Rift Complex initiated as an intra-continental rift that formed along

539 pre-existing crustal weakness and failed to progress to continental breakup, resulting in the present
540 day aulacogen. Later development of the Adelaide Rift Complex expanded the extent of the rift
541 system to the south with wider deposition of the Burra Group. This southern region is suggested to
542 represent the successful rift axis of the Adelaide Superbasin where the proto-Pacific later formed
543 (consistent with kinematic constraints suggested by Merdith et al. 2017b). In this model, the triple
544 junctions suggested by von der Borch (1980) are a result of the intersection of propagating rifts to
545 form a geometric triple junction.

546 This model is similar to recent ideas about the development of the Afar triple junction, where the Red
547 Sea meets the Gulf of Aden and the East African Rift system. Traditionally, this area has been viewed
548 as the classic triple junction rift-rift-rift system formed by apical extension away from the triple
549 junction centre (McKenzie et al. 1970; Tesfaye et al. 2003). However, the geological evidence
550 suggests that at least two of the three arms (Aden Rift, Ethiopian Rift) propagated inwards towards
551 the now seen geometric triple junction, and the chronology of the rift systems does not fit with plume
552 driven apical extension from a central point (e.g., Barberi et al. 1972; Corti 2009; Varet 2018; Zwaan
553 et al. 2020). It appears that the modern Afar triple junction is a geometric place where three rifts,
554 with their predetermined geometries, happened to cross rather than being the point of initiation (e.g.,
555 Barberi et al. 1972; Corti 2009; Varet 2018; Zwaan et al. 2020), similar to our model for the Adelaide
556 Rift Complex.

557 6 Conclusions

558 The development of the Adelaide Superbasin initiated between c. 890–830 Ma with the deposition of
559 the Arkaroola Subgroup in a series of structurally controlled half-grabens in what now constitutes the
560 Adelaide Rift Complex. These structures are likely a manifestation of northeast-southwest (present
561 day) orientated extensional strain from far-field forces, and potentially also stress from a mantle
562 plume. This phase of extension was limited, and tectonic quiescence followed until extrusion of the
563 Willouran Large Igneous Province (LIP). The Willouran LIP may have been the result of a mantle
564 plume, and its emplacement led to extensive rifting and subsequent deposition of the Curdimurka
565 Subgroup.

566 Key findings of this research are:

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- Revised constraints on timing of initial deposition within the Adelaide Superbasin, between $\geq 893 \pm 9$ Ma and c. 830 Ma.
 - Identification of an enigmatic source of young (<1000 Ma) zircon, in the basal stratigraphic unit
 - The Arkaroola Subgroup represents early, syn-rift, deposition within half-grabens, developed in an initial pulse of extension that likely exploited pre-existing crustal weakness.
 - The central and northern Flinders Ranges formed the initial arm of the rift system, but failed to progress to continental breakup.
 - Basal Centralian Superbasin and Adelaide Superbasin stratigraphic units had different primary detrital sources.
 - Support for a potential late Mesoproterozoic source region to the east of the basin.

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582 Supplementary Material

583 Supplementary Figure S1 is available as both an EPS file and a PNG file hosted on Figshare:
584 <https://doi.org/10.6084/m9.figshare.19153274>

585 Data Availability

586 Complete data for this publication are freely available for download from Figshare at the following
587 links. These datasets contain all the U–Pb geochronology data, trace element data, and basic sample
588 metadata.

589 Zircon and NIST standards data for all analytical sessions:
590 <https://doi.org/10.6084/m9.figshare.18131432>

591 Callanna Group (this study only) detrital zircon data: <https://doi.org/10.6084/m9.figshare.18131420>

592 Zircon CL images: <https://doi.org/10.6084/m9.figshare.19181024>

593 Code Availability

594 R code used to generate the zircon geochemistry plots is available on GitHub at
595 <https://github.com/jarredclloyd/zircon-trace-element-plots>

596 CRediT author statement

597 **Jarred C. Lloyd:** Conceptualisation, investigation, writing - original draft, writing - review & editing,
598 methodology, formal analysis, data curation, visualisation. **Morgan L. Blades:** Investigation, writing -
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600 writing - review & editing. **Sarah E. Gilbert:** Formal analysis, methodology, investigation, writing -

601 review & editing. **Kathryn J. Amos:** Conceptualisation, funding acquisition, supervision, writing -
602 review & editing.

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617 This work is conducted with the appropriate permissions and scientific permits from the relevant
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1134

1135 Appendix - Use of NIST610 as primary $^{207}\text{Pb}/^{206}\text{Pb}$ standard

1136 Matrix matched reference materials are essential for the accurate determination of U/Pb ratios, and
1137 thus calculated ages, of accessory minerals such as zircon via laser ablation mass spectrometry
1138 (Allen & Campbell 2012; Kuhn et al. 2010; Miliszkiewicz et al. 2015; Thompson et al. 2018). This is
1139 due to the offset in ratio and subsequently age determinations caused by “matrix effects” (Kořler et
1140 al. 2005; Marillo-Sialer et al. 2014; Schaltegger et al. 2015). Primarily, this is a result of downhole
1141 fractionation (Paton et al. 2010; Ver Hoeve et al. 2018) with one of the major causes being laser
1142 induced elemental fractionation (LIEF) of U from Pb in the crystal lattice (Kořler et al. 2005; Marillo-
1143 Sialer et al. 2014). However, it has been determined that there is negligible to no fractionation of Pb
1144 isotopes during laser ablation of various accessory minerals and silicate glasses (Allen & Campbell
1145 2012; Guillong et al. 2020; Miliszkiewicz et al. 2015; Souders & Sylvester 2010), thus allowing the
1146 use of non-matrix matched silicate glasses (e.g., NIST610) as external reference materials for the
1147 determination of accurate Pb isotope ratios. Methodology using NIST610, or other silicate glasses, as
1148 the $^{207}\text{Pb}/^{206}\text{Pb}$ primary reference material has been successfully used in past (Guillong et al.
1149 2020; Halpin et al. 2014; Large et al. 2013; Standish et al. 2013). We further validate this as the
1150 NIST610 corrected $^{207}\text{Pb}/^{206}\text{Pb}$ ratio and calculated age for every natural zircon reference material
1151 analysed is within uncertainty at high accuracy [Figure A1] of their CA-ID-TIMS determined values
1152 (Horstwood et al. 2016). The use of NIST610 allows for more precise determination of Pb isotope
1153 ratios due to the better homogeneity and characterisation of the reference material (Jochum et al.
1154 2011) while retaining accuracy. This is useful in overcoming the higher degrees of uncertainty
1155 associated with natural reference materials that are measurably heterogenous (Horstwood et al.
1156 2016; Schaltegger et al. 2021), which is likely the result of radiation damage induced lead loss,
1157 zonation in zircon crystallinity, or protracted growth.

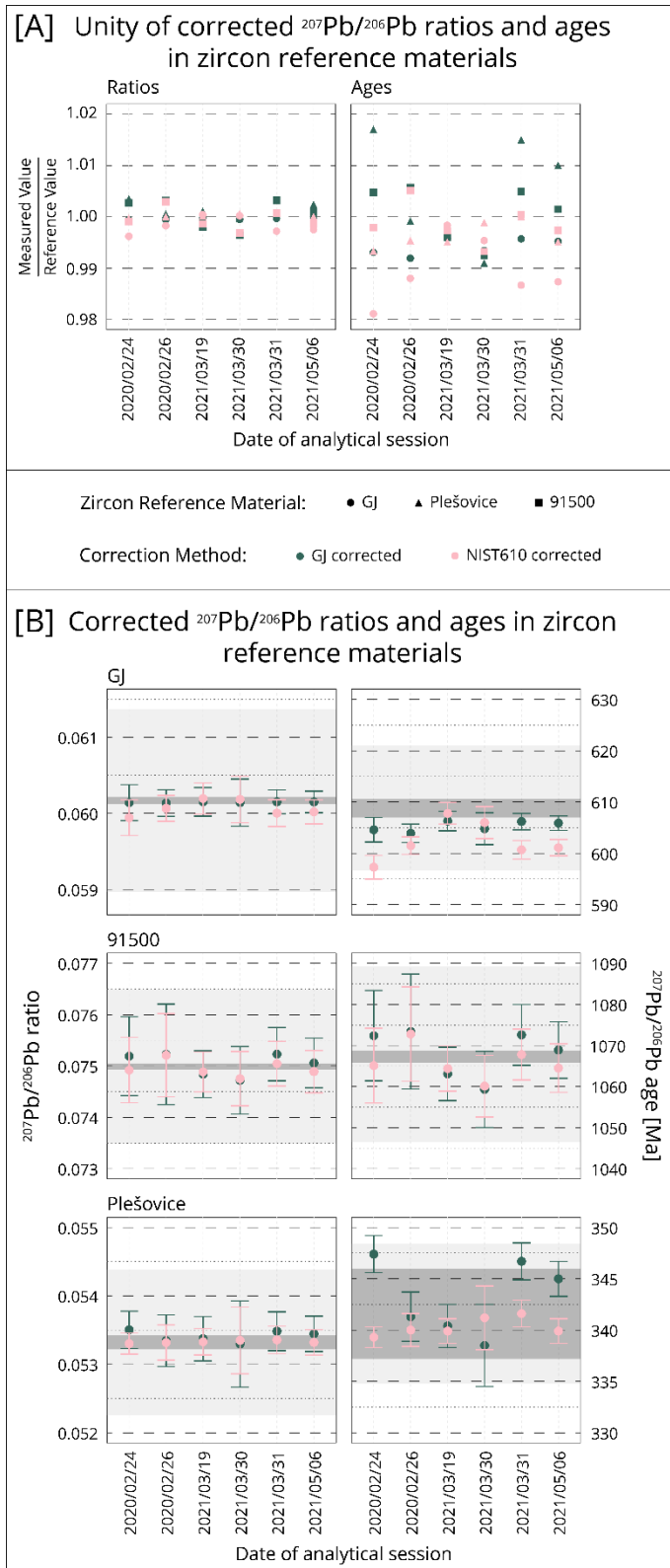


Figure A1—A: Unity measure (observed/reference value) of corrected Pb isotope ratios and ages. **B:** individual observed corrected ratios and ages for each natural zircon reference material used. Dark grey shading is the reference value material range (2x standard error), light grey shading is the 2% unity range (reference value \pm 2%). Uncertainties on observations are 2 standard error. Reference values are from (Horstwood et al. 2016)