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# Thin-section detrital zircon geochronology mitigates bias in provenance investigations

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**Abstract:** Detrital zircon U–Pb geochronology has enabled advances in the understanding of sediment provenance, transportation pathways, and the depositional age of sedimentary packages. However, sample selection and processing can result in biasing of detrital zircon age spectra. This paper presents a novel approach using *in situ* detrital zircon U–Pb measurements on thin-sections to provide greater confidence in maximum depositional ages and provenance interpretations. New U–Pb age data of 310 detrital zircon grains from 16 thin-sections of the Triassic Mungaroo Formation from two wells in the Northern Carnarvon Basin, Australia, are presented. Whilst detrital zircon age modes are consistent with previous work, there are some differences in the relative proportions of age modes, which are partly attributed to a lack of small grains in handpicked grain mounts. The relative sample bias is quantified via grain size comparison of dated zircon (in thin-sections or handpicked mounts) relative to all zircons identified in bulk-mounts and thin-sections. The youngest age mode (*c*. 320–195 Ma) is consistent with an active margin to the north. The dated zircons reveal a single grain defined maximum depositional age of 197 Ma for the upper part of the Mungaroo Formation, suggesting deposition may have continued into the Early Jurassic.

Supplementary material: Additional figures, tables, datasets and programming scripts are available at https://doi.org/10.6084/m9.figshare.c.5628911

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Detrital zircon U-Pb geochronology has become ubiquitous in investigations of clastic sedimentary systems due, in part, to the robustness of zircon against physical and chemical modification, and its ability to function as an excellent geochronometer (e.g. Fedo et al. 2003; Gehrels 2012). Detrital zircon U-Pb geochronology is a widely implemented tool and has revolutionized interpretations of source-to-sink relationships (provenance), maximum depositional ages, stratigraphy, and palaeogeographic reconstructions (e.g. Cawood and Nemchin 2000; Nelson 2001; Dickinson and Gehrels 2009; Gehrels 2014). However, biasing of detrital zircon age populations may be caused by natural geological and anthropogenic laboratory factors (Fedo et al. 2003; Sláma and Košler 2012; Chew et al. 2020; Barham et al. 2021; Dröllner et al. 2021). While geological biases such as variable source rock fertility, erosivity, transportational sorting or sediment recycling may be intrinsic to detrital zircon investigations, these biases may be partly mitigated through sampling strategies and awareness of interpretive limitations (Spencer et al. 2018). Laboratory biases, including sampling and subsequent processing (e.g. disaggregation, mineral concentration, grain selection, picking and mounting) may simultaneously make a sample less representative of the primary source yet enhance its amenability to rapid imaging and geochronology (Sircombe and Stern 2002; Moecher and Samson 2006; Hietpas et al. 2011; Sláma and Košler 2012; Malusà et al. 2016; Dröllner et al. 2021). Collectively, sample processing can decrease the representativeness of detrital zircon populations, as well as the robustness and resolution of any provenance interpretation based on them. Therefore, conclusions based on the interpretation of relative age mode significances, which indicate the degree of a source's contribution to the detrital record, need to be considered with care.

Distortion of detrital zircon age spectra also impacts interpretations of maximum depositional ages (Barbeau et al. 2009; Dickinson and Gehrels 2009; Sharman and Malkowski 2020; Vermeesch 2020). Differences in the determination of maximum depositional age may reflect the strategy used in their age determination. Whilst the youngest individual grain age represents a maximum depositional age for a sediment, in that the sediment can be no older than its youngest detrital component, more conservative approaches have been advocated (Coutts et al. 2019; Vermeesch 2020), for example, taking the youngest age peak represented by more than one grain age, or taking the mean of the three youngest ages with analytical errors  $(1\sigma \text{ or } 2\sigma)$  within each other's uncertainty. The real depositional age of sedimentary rocks can be revealed using detrital zircon geochronology by dating zircon grains in tuff deposits, which provide an isochronous time marker (e.g. Page 1981; Bowring et al. 2006). It is especially challenging to confidently determine accurate maximum depositional ages in subsurface basin sequences due to sample recovery limitations from drill hole material, which may require composite sampling over hundreds of metres (e.g. MacDonald et al. 2013). Such sampling may cause stratigraphic smearing and loss of age resolution through the stratigraphy, and even contamination of age populations from drill chippings through collapse of material down the hole (e.g. Krueger and Vogel 1954; Kallmeyer et al. 2006; Hilbert-Wolf et al. 2017). Therefore, developing techniques to ensure recovery of representative zircon age populations is essential for scientifically robust determinations of sediment provenance and depositional age based on a sample's zircon cargo.

Australia's North West Shelf (Fig. 1) hosts world-class hydrocarbon resources (e.g. Purcell and Purcell 1988). The Mesozoic Mungaroo Formation is a remarkably prolific gas-

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**Fig. 1** Tectonic setting of Australia during the Late Triassic (**a**), and present location of the Northern Carnarvon Basin, NW of Australia, with sampled well locations (**b**). Grey line in (**a**) shows the outline of East Gondwana, with other map elements modified from Morón *et al.* (2019) and Haines *et al.* (2016), and tectonic plate reconstruction with GPlates after Matthews *et al.* (2016). Pie-charts in (**b**) illustrate zircon U–Pb age groups (1: 2000–1500 Ma, 2: 1300–800 Ma, 3: 650–490 Ma, 4: 350–200 Ma) for each well. Well names in bold (Geryon-2 and Yodel-1) represent the location of new data reported herein and compared to that of Lewis and Sircombe (2013). Abbreviations and numbers in (**a**): WAC: West Australian Craton, NAC: North Australian Craton, SAC: South Australian Craton; 1: Northern Carnarvon Basin, 2: Roebuck Basin, 3: Canning Basin, 4: Officer Basin, 5: Edmund Basin, 6: Perth Basin, 7: Kuunga Orogen, 8: Wilkes Land, 9: Albany Fraser Orogen, 10: Yilgarn Craton, 11: Pinjarra Orogen, 12: Gascoyne Province, 13: Pilbara Craton, 14: Capricorn Orogen, 15: Paterson Orogen, 16: King Leopold Orogen, 17: Halls Creek Orogen, 18: Tanami Region, 19: Arunta Orogen, 20: Musgrave Orogen.

condensate reservoir unit, but understanding its stratigraphy, depositional history and reservoir connectivity is hampered by poor biostratigraphic age control (e.g. Payenberg *et al.* 2013). Several wells penetrate the unit, and preliminary traditional detrital zircon data make the basin an ideal case study to address sources of bias in provenance investigations and evaluate their implications for geochronological interpretation through a novel contextualised *in situ* geochronology approach.

This paper presents new U–Pb detrital zircon age data derived from thin-sections, which are compared with zircon age spectra derived from mounts of previous studies, and discusses differences and implications for provenance and maximum depositional age interpretations based on the implemented sampling strategy.

#### Geological background

The North West Shelf of Australia is a hydrocarbon-rich passive margin comprising a series of basins: Northern Carnarvon Basin, Roebuck Basin, Browse Basin and Bonaparte Basin (Fig. 1). The Northern Carnarvon Basin's evolution is related to the breakup of Gondwana from the late Permian to the Early Cretaceous during which it evolved from an intra-cratonic rift basin to a passive margin (Tao et al. 2013). During the Late Triassic, Australia was located on the eastern margin of Gondwana, forming a continent with Antarctica to the south and Greater India to the west (Fig. 1). The Lhasa Terrane was one of several terranes to the north, possibly associated with a southward-directed subduction zone. Substantial rifting during the Permian created a depocentre in the Northern Carnarvon Basin (Driscoll and Karner 1998; Bailey et al. 2006; Paumard et al. 2018) that was subsequently filled by a complete depositional cycle from marine to fluvio-deltaic and back to marine conditions during the Triassic through the Locker Shale, Mungaroo Formation and Brigadier Formation (Fig. 2). The Mungaroo Formation is a several kilometres thick fluvio-deltaic sequence comprising complex mud-rich, shaly and coarser sandstones with interbedded channel belts. These large-scale intracontinental channel belts are mainly east-west oriented (Martin et al. 2018),

suggesting sediment transport through the Canning Basin. The Mungaroo and Brigadier formations are overlain by marine sediments of Early and Middle Jurassic age, but are truncated by a mid-Jurassic unconformity at the crest of fault blocks and on the flanks of Mesozoic graben structures, as is the case with some of the wells sampled in this study (Fig. 2).

Based on U-Pb geochronology of detrital zircon grains from cutting samples, Lewis and Sircombe (2013) proposed the Capricorn Orogen, Arunta Orogen, Albany Fraser Orogen, Musgrave Province and Pinjarra Orogen (Fig. 1) as potential source regions. The data of Lewis and Sircombe (2013) also contain a large proportion of Neoproterozoic ages that they have suggested may have been derived from more distal sources in Antarctica and Greater India (Fig. 1). Known Proterozoic sources in Australia include several tectono-magmatic events within the broader Capricorn Orogen (Korhonen and Johnson 2015). The Arunta Province records several intrusions dated between 1690 and 1600 Ma and metamorphism at 1590-1560 Ma, potentially accounting for Paleoproterozoic-Mesoproterozoic zircon grains (Scrimgeour 2013a, b). The Albany Fraser Orogen's major tectonomagmatic events occurred at 1710-1650 Ma, 1345-1260 Ma and 1215-1140 Ma (Kirkland et al. 2011, 2015). The Musgrave Province contains 1600 and 1500 Ma crystalline rocks but dominantly comprises younger magmatic rocks at 1220-1150 Ma (Kirkland et al. 2013). Younger zircons may be related to magmatic/metamorphic events in the Pinjarra Orogen at 1140-1090 Ma (Fitzsimons 2003; Collins and Pisarevsky 2005). Neoproterozoic to Cambrian zircon growth is also recognized in parts of the Pinjarra Orogen, associated with the broader Gondwanan Kuunga Orogeny (Collins 2003). In particular, distinctive magmatic and metamorphic ages within the Leeuwin Complex (SW Western Australia) between 780 and 520 Ma correlates to the main proportion of detrital zircon ages from the Northern Carnarvon Basin in this time period. In addition to the conjugate equivalent of the Pinjarra Orogen, less significant contributions may have come from elsewhere in the Indian subcontinent (Hall 2012) and Australia (Lewis and Sircombe 2013). The Lhasa Terrane at



Fig. 2 Interpreted seismic data with drill core and sampling depths of Lady Nora-2, Yodel-1 and Goodwyn-6 located within the Rankin Platform of the Northern Carnarvon Basin.

Australia's northwestern margin drifted northward during Late Triassic rifting accompanied by active volcanism. Triassic age volcanism has also been recorded in the Roebuck Basin (MacNeill *et al.* 2018). Based on the possible source regions of the Mungaroo Formation, Lewis and Sircombe (2013) proposed several sediment transportation routes: (i) A south–north route transporting zircon grains from the Capricorn, Pinjarra and Albany Fraser orogens, (ii) an east–west route transporting zircons from the Arunta and Musgrave regions of central Australia, (iii) a possible north–south route transporting zircons from the Lhasa Terrane or the South West Borneo Block, and (iv) a west–east route transporting zircons from the Indian subcontinent.

#### Samples and methods

In total, 16 polished thin-sections (Table S1) were made of a range of facies from fine- to coarse-grained sandstone with shale partings from two separate cores penetrating the Triassic Mungaroo Formation in the Northern Carnarvon Basin. Thin-section blocks were cut from complete drill cores from Geryon-2 (Exmouth Plateau) and Yodel-1 (Rankin Platform, Fig. 1). Rock billets, remaining from thin-section preparation, were used to create bulk grain mounts. All thin-section billets (accounting for a volume of  $112 \times 10^3$  mm<sup>3</sup> in comparison to 326 mm<sup>3</sup> for the thin-sections, Table S2) had their constituent grains liberated via selfrag, with heavy minerals concentrated by heavy liquids (sodium polytung-state, 2.8 g cm<sup>-3</sup>) and magnetic separation (current less than 1.0 A). Resulting heavy mineral fractions were bulk mounted into epoxy resin without picking, and polished to half-grain thickness (of the smallest zircon grains).

#### Sample characterization

# *Light microscopy, backscattered electron and cathodoluminescence imaging*

An automated Zeiss AXIO Imager M2m microscope system was used to produce overview images of the thin-sections and bulk mounts in transmitted and polarized light. Backscattered electron (BSE) and cathodoluminescence (CL) images of representative zircon crystals in carbon-coated thin-sections were produced using a Tescan Mira3 field emission scanning electron microscope at the John de Laeter Centre (JdLC) at Curtin University (Perth, Australia) with HV of 12 kV and a working distance of *c*. 17 mm. CL images provide an overview of the internal textures in the zircons.

# TESCAN integrated mineral analyser phase mapping

Mineral maps of carbon-coated thin-sections and bulk mounts were produced using the TESCAN Integrated Mineral Analyser (TIMA) at the JdLC at Curtin University. The TIMA combines BSE images with energy-dispersive X-ray spectroscopy (EDS) to create mineral phase maps (Hrstka et al. 2018). Thin-sections and bulk mounts were analysed in 'dot-mapping' mode with a rectangular mesh at a step size of 3 and 1 µm respectively for BSE imaging. One thousand EDS counts were collected every ninth step (i.e. 27 µm for thinsection,  $9\,\mu m$  for bulk mount analysis) or when the BSE contrast changed (i.e. a change in mineral phase). For a given mineral grain, EDS counts were integrated across the entire grain. High-resolution settings (1 µm per pixel for both BSE and EDS) account for relatively long scanning times (several hours) for each sample. However, sufficient detail of the dominant mineral compositions and zircon targeting can be resolved for geochronological analyses from faster c. 30 min scans by lowering the sampling rate of EDS. TIMA analyses used an accelerating voltage of 25 kV, a beam intensity of 19, a probe current of 5.70-6.36 nA, a spot size of 41-52 nm and a nominal working distance of 15 mm. The EDS spectra were standardized using a measurement of a pure Mn standard. After imaging and EDS collection, BSE signals and EDS peaks were referenced to a mineral library for automatic mineral classification.

# U-Pb geochronology

U–Pb isotope ratios were measured using Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) at Curtin University's JdLC with 310 zircon grains analysed over the course of three analytical sessions. An excimer laser (resolution LR 193 nm ArF with a Lauren Technic S155 cell) with a spot diameter of 24 µm was used to sputter zircon targets. The system used onsample laser energy of 2.51 J cm<sup>-2</sup> with a repetition rate of 5 Hz for 30 s of total analysis time and *c*. 20 s of background capture. All analyses were preceded by two cleaning pulses. The sample cell was flushed by ultrahigh purity He (0.68 l min<sup>-1</sup>) and N<sub>2</sub> (2.8 ml min<sup>-1</sup>). An Agilent 8900 triple quadrupole detector was used to measure isotopic masses. The quadrupole used high-purity Ar as the carrier gas (flow rate = 0.98 l min<sup>-1</sup>) and measured the following mass stations: <sup>206</sup>Pb, <sup>207</sup>Pb, <sup>232</sup>Th and <sup>238</sup>U. All grains sufficiently large to host a 24 µm spot were chosen for measurement ('blind-dating' of Garzanti *et al.* 2018). Reference materials (GJ-1, OG-1 and Plešovice) were analysed after every 18 unknowns. The primary reference material was the natural zircon GJ-1 (608.5 ± 1.5 Ma; Jackson *et al.* 2004) and secondary reference materials were natural zircons OG-1 (3465.4 ± 0.6 Ma; Stern *et al.* 2009) and Plešovice (337.13 ± 0.37 Ma; Sláma *et al.* 2008), which were treated as unknowns. In the three analytical sessions, secondary reference materials yielded weighted mean ages of  $344 \pm 1$ ,  $340 \pm 1$  and  $339 \pm 1$  Ma ( $^{206}$ Pb/ $^{238}$ U ages  $\pm 2\sigma$ ) for Plešovice and  $3475 \pm 10$ ,  $3468 \pm 5$  and  $3471 \pm 7$  Ma ( $^{206}$ Pb/ $^{207}$ Pb ages  $\pm 2\sigma$ ) for OG-1 (Tables S3 and S4). Time-resolved mass spectra were reduced using the U–Pb geochronology data reduction scheme in iolite v.4.3.0 (Paton *et al.* 2011).

Uncertainties on the secondary reference materials isotope ratios (<sup>238</sup>U/<sup>206</sup>Pb of Plešovice and <sup>207</sup>Pb/<sup>206</sup>Pb of OG-1) were propagated into the measurements (Spencer et al. 2016). Grain ages are calculated with Isoplot R (Vermeesch 2018), guoted with a two sigma  $(2\sigma)$  absolute uncertainty, and considered concordant if the error ellipse intersected the concordia curve within the  $2\sigma$ uncertainty limit (literature data were reprocessed in the same way). Additionally, for our data we provide ages filtered using a 10% discordance limit, as a comparison to the  $2\sigma$  error ellipse filter (cf. Vermeesch 2021). Concordia ages are used in all further calculations, in the statistics for the U-Pb ages (Dzstats; Saylor and Sundell 2016), calculations for kernel density estimations (Isoplot R; Vermeesch 2018), cumulative probabilities (Isoplot R; Vermeesch 2018) and multidimensional scaling (Saylor et al. 2018). Results for different samples from the same well have been combined because of the similar composition of the thin-sections and the apparent continuity of the age spectra of the Mungaroo Formation samples within individual wells.

# Lithologically contextualizing measured grains; a computational methodology

Mineral specific RGB values of TIMA mineral phase panorama images were extracted pixelwise by a Python script (https://github. com/Isabel-Zu/approach\_TIMA-ArcGIS-Python and supplementary material A), which resulted in a point grid of mineral phases from the thin-section mineral phase image (Fig. 3). Specific grain-level information (such as U-Pb age data) was joined with the position of the measured zircons using ArcGIS software (geographic information system). An ArcGIS model was generated, which aggregated zircon points from the point grid. The model was optimized for aggregation of a minimum of three points, based on the comparison between the model and the images. The 'buffer analysis' tool in ArcGIS was then used to quantify the proximal mineral phase assemblage within the specified proximity of the measured zircon. The buffer tool creates a larger polygon around the zircon polygon at a specified distance. To ensure a representative selection of grid points that adequately captures the mineralogical variation in the buffer, c. 500 grid points were considered, which equates to a buffer distance of c. 66 µm. Thus, the buffer polygons capture the mineral content proximal to each dated zircon.

### Grain size analysis

Grain size analysis was conducted using three different methods depending on the source of the data. Grain area values of all zircons within a thin-section or bulk mount were exported from TIMA, while the area of the U–Pb dated zircons was determined from the polygons generated by the ArcGIS model (see above). In order to compare the data of this study with published detrital zircon geochronology of the Mungaroo Formation from well cuttings



Fig. 3 Process of sample characterization automated mineralogy (see https://github.com/Isabel-Zu/approach\_TIMA-ArcGIS-Python and supplementary material A for Python script and ArcGIS model). (1) Overview image of the thin-section. A Python script creates a point grid (3) from the TIMA phase image (2), which is aggregated to phase grain-polygons (4) based on the ArcGIS model. At this stage measured data can be georeferenced to the mineral polygons and their measured grain sizes compared to the overall grain sizes within the thin-section, or further buffer analyses can define the lithology around dated zircons.

(Lewis and Sircombe 2013), grain areas of zircon grains in mount images were determined using a MATLAB script (https://github. com/Isabel-Zu/approach\_TIMA-ArcGIS-Python and supplementary material A), which first identifies the contours of the grains and then determines their area by converting the pixel count to square micrometers using the pixel count of a known 100 µm<sup>2</sup> area.

# Results

### Sample description and composition

Thin-sections from all samples, on average, range from medium silt (>25  $\mu$ m) to granule-bearing very coarse sand (<2000  $\mu$ m). The majority of samples show well to moderately sorted sub- to well-rounded quartz grains defining individual laminations within a finer matrix. Coarser feldspar grains are mainly altered showing kaolinization textures, whereas smaller feldspar grains are mainly unaltered. Thin-sections from some samples are poorly sorted and contain shell fragments as well as minor seams of organic matter. Each sample's compositional variation was quantitatively analysed via TIMA. Six main minerals, which in total make up more than 50% of the total mineral content are listed in Table S5, except sample TR-61 in which minerals could not be reliably classified due to the very fine grain size. Overall, quartz (<88.01%), feldspar (<34.80%) and kaolinite (<14.34%) are the three dominant constituent minerals.

#### Zircon characterization

Zircon grains are angular to sub-rounded, but most grains are subhedral. CL images reveal various internal textures within single zircon crystals, including primary oscillatory, distinct sector and patchy zonation (Fig. 4). The majority of grains display primary magmatic zonation patterns, and some show relict cores and overgrowths. Rarely are the internal textural domains less than the LA-ICP-MS ablation spot size. Zircon fragments and discrete crystals with homogeneous textures, which may reflect metamorphic growth, are a minor component of the population.

#### Zircon grain size analysis

Based on the new approach presented in this work and considering all 16 thin-sections, 6151 zircon grains were detected. The majority, 98.4% of the total zircon grains, have a grain area below 3000  $\mu$ m<sup>2</sup> (Fig. 5), with 87.5% of the grains having an area below 1000  $\mu$ m<sup>2</sup>. U–Pb measured zircon grains with concordant U–Pb systematics reveal a similar grain size distribution. 83.7% of concordant grains have an area below 1000  $\mu$ m<sup>2</sup>; in contrast, only 12.5% have an area between 1000 and 2000  $\mu$ m<sup>2</sup>. The number of concordant grains >4000  $\mu$ m<sup>2</sup> (1.9%) is over two times the proportion of grains of this size in the overall zircon population (0.8%), consistent with smaller grains being more discordant. In order to address a potential grain size bias in the measured thin-sections caused by random grain



Fig. 4 Cathodoluminescence images from dated zircons showing primary magmatic zonation patterns. A laser spot with Concordia age is indicated.



Fig. 5 Histograms of grain size distribution. (a) Analysed zircons on handpicked mounts from Lewis and Sircombe (2013), (b) all zircons within bulk mounts, (c) all zircons within thin-sections, (d) analysed zircons of the thin-sections in this work. All grain sizes are determined in this study (see https://github.com/Isabel-Zu/approach\_TIMA-ArcGIS-Python and supplementary material A).

orientation and incomplete grain sectioning, the zircon size distribution within the remaining thin-section billets was also investigated. The zircon grain size population for the billets is highly comparable to that in the thin-sections. Of the total 42 209 detected zircon grains within the 16 bulk mounts, 98.8% have an area below 3000  $\mu$ m<sup>2</sup>, with 84.5% having an area below 1000  $\mu$ m<sup>2</sup> (Fig. 5).

# Mineralogical content within zircon grain buffers

The buffered areas around the analysed zircon grains in the thinsections contain a mineral assemblage that matches the composition of the entire thin-section and is relatively consistent across the different zircon age groups (Fig. 6). Furthermore, a general observation is that zircon grains are in proximity to other zircon grains, especially in sample TR-23 (Fig. 6), in which the grains are concentrated at a boundary between a coarser quartz-rich and a fine feldspar-rich layer.

#### U–Pb geochronology

Given the insignificant variation of age spectra within wells, concordant U–Pb ages from individual samples (Tables S6 and S7) are combined with others from the same well to generate composite datasets for each well for ease of discussion. Combined U–Pb data sets are used to generate composite age populations for each well (Figs 1 and 7, S1–S3).

Seventy-two grains from Geryon-2 and 32 grains from Yodel-1 are concordant. Whilst many recent detrital zircon studies utilize larger numbers of grains in order to capture all sources, we aim to depict only the statistically significant fractions that can be



Fig. 6 Boxplots showing the amount of zircon grains in percent within  $66 \ \mu m$  of measured zircons of different ages in thinsections (**a**), amount of zircons in the whole thin-section (**b**), and zircons within the buffer area of other zircons across the whole thin-section (**c**). The ends of the boxes are the interquartile range from the 25th percentile to the 75th percentile, the median is marked by the red line, the whiskers are the minimum and maximum values and the outliers are symbolized by open circles.

recognized with fewer grains analysed (Vermeesch 2004). Statistically, the population of 32 grains dated from Yodel-1 will have captured all age component fractions  $\geq 10\%$  with 70% certainty (Vermeesch 2004). Assuming random sampling, more prevalent components that are source region significant will still be captured at lower analysis numbers. Hence, even lower-*n* data sets can be geologically informative, especially when no grains can be interpreted as processing contaminants as they are analysed *in situ* in thin-sections.

The 72 concordant detrital zircon ages acquired from Geryon-2 and Yodel-1 range from Paleoarchean to Early Jurassic in age. The oldest concordant ages for each well are ~1546 ± 14 Ma (Yodel-1 TR61–23) and ~2981 ± 27 Ma (Geryon-2 TR12–30), and the youngest ages are ~206 ± 3 Ma (Yodel-1 TR60a–32) and ~197 ± 2 Ma (Geryon-2 TR14–19). Consequently, the age of ~197 ± 2 Ma constrains the maximum depositional age of the sampled part of the Mungaroo Formation. A more conservative estimate of the maximum depositional age is provided by calculating the weighted mean of the youngest age group,  $209 \pm 2$  Ma (MSWD = 0.99, n = 6, Geryon-2) and  $213 \pm 2$  Ma (MSWD = 2.1, n = 3, Yodel-1).

Detrital zircon Th–U ratios, which have been considered by some as indicative of growth environment (e.g. magmatic if >0.5 or metamorphic if <0.1; Hoskin and Black 2000; Rubatto 2002; Hoskin and Schaltegger 2003), scatter around one and show a slight trend towards lower values with increasing U content (Fig. S5).

Detrital zircon age populations are polymodal with four principal age modes of variable intensities across the kernel density estimates (Fig. 7, \*-panels, Yodel-1 and Geryon-2). Group 1 comprises grains within 2000–1500 Ma (group 1) defining a minor unimodal or bimodal broad peak, group 2 is represented by grains within 1300–800 Ma, group 3 is defined by 650–490 Ma grains and 350–200 Ma grains are assigned to group 4.

Peak age intensities in groups 4 and 3 are of similar significance to each other in both samples; however, these peaks are the dominant age modes in Yodel-1 but subordinate in Geryon-2 (Fig. 7). Furthermore, the Yodel-1 age spectra contain a smaller subordinate age peak at ~900 Ma that is absent in the Geryon-2 spectra.

A Kolmogorov–Smirnov test statistic yields a D-value of 0.410 for Yodel-1 and Geryon-2 zircon ages and a *P*-value of 0.0006,

which is lower than 0.05 and thus the null hypothesis is rejected that both samples are from the same distribution (Table S8). The biggest influence on this similarity relationship is age mode 3, which comprises 22% and 17% of the age spectra in Yodel-1 and Geryon-2, respectively (cf. pie charts in Fig. 1 and cumulative probability curves in Fig. S2).

# Discussion

Detrital zircon age modes provide useful information on provenance through correlation to crystallization ages in crustal provinces and comparison with detrital zircon age spectra in other sedimentary basins. Relative detrital zircon age mode significances have been widely used to interpret dominant source regions (e.g. Fedo et al. 2003; Voice et al. 2015). However, such interpretations may lead to spurious conclusions if the dated zircon grain populations are not representative of the true prominence of age-distinctive source regions supplying detritus. The relative height of age peaks (significance of age modes) may retain less geological information than the presence of an age alone, given the potential for geological or laboratory distortion of relative age proportions (Vermeesch 2004; Reiners et al. 2017). Many previous studies have linked the degree of statistical robustness of age components to the number of dated zircon grains (e.g. Pullen et al. 2014; Nie et al. 2018; Chew et al. 2020). In this work, we introduce a different approach to evaluate how representative the dated zircon component is to the total zircon cargo. We achieve this by comparing grain sizes between the dated zircons in thin-sections and all detected zircons within thin-sections (Fig. 5). In order to determine the effect of sectioning on grain-size bias in thin-sections, we compare grain sizes in thin-sections with grain sizes in bulk mounts from rock billets of the same sample material. The resulting grain size distributions are almost identical and support an interpretation in which section orientation does not meaningfully bias the grain size population (Fig. 5). Another interesting aspect of this finding is that a three orders of magnitude increase in the volume of sampled material has only a minor effect on the measured grain size population. Furthermore, zircon grain sizes from thin-sections are compared to those in the conventionally prepared hand-picked zircon mounts of Lewis and Sircombe (2013). In addition, the age



**Fig. 7** Kernel density plots of concordant zircon U–Pb ages for each well (Lewis and Sircombe 2013; \*new data); new data also shown with a 10% discordance filter (dashed lines with corresponding labels in italic). For well locations see Figure 1. S = number of samples, n = number of concordant grains/total number of grains. Note: Figure 7 and Fig. S1–S3 are colour coded by wells.

spectra of the thin-section samples are compared to those in the hand-picked grain mounts, with the aim of better understanding grain size biases on age populations. Zircon grain sizes vary throughout the thin-sections and range from 170  $\mu$ m (23 400  $\mu$ m<sup>2</sup>) to 28  $\mu$ m (625  $\mu$ m<sup>2</sup>) for the long-axis of a grain-fitted ellipse. The thin-sections contain 87.5% of small grains (<1000  $\mu$ m<sup>2</sup>), which contrasts with only ~3.0%, of small grains in the hand-picked zircon

mounts of Lewis and Sircombe (2013) (Fig. 5). Whilst large grains  $(>2000 \,\mu\text{m}^2)$  in the thin-sections account for only a minor proportion of the total exposed zircon population (<5%), the majority of grains on the hand-picked mounts are large (3000- $5000 \,\mu\text{m}^2$ ). Given the representative grain size/lithologies sampled in this work, these results reveal a clear bias in the hand-picked sample mounts towards larger zircon grains (rather than simply initial sampling of coarser facies). This implies that small zircon grains are less likely to be retained through conventional rock sample selection, mineral-separation (e.g. jaw crushing, heavy mineral separation, hand-picking) and grain-mounting. Hence, we contend that zircon spectra produced from grains dated in thinsection, if of sufficient number, can be more representative than those dated on picked grain separate mounts. Whilst it may be assumed that the missing smaller grain size component in the conventional mounts accounts for a specific age group, we do not see any clear, simplistic link between grain size and age (cf. Lawrence et al. 2011). Such a finding is consistent with Leary et al. (2020), who observed that detrital age spectra in sandstone and mudstone were independent of the grain sizes. Analytical limitations will always place a limit on the minimum size of grain accessible to geochronology; however, by analysing material in thin-section and having knowledge of the entire grain size population, at least the magnitude of this bias can be evaluated and its effect minimized.

# Provenance

The following provenance interpretation encompasses U–Pb data derived from hand-picked zircon mounts of Lewis and Sircombe (2013) and new U–Pb data from thin-sections (Fig. 7, S1–S3, Tables S6 and S7). All age spectra show age modes within specific ranges 2000–1500 Ma (group 1), 1300–800 Ma (group 2), 650–490 Ma (group 3) and 350–200 Ma (group 4).

# Archean

Concordant analyses within the thin-sections show minor agedispersed grains >2.5 Ga with oldest grain ages of c. 3.28 Ga (Fig. 7). These ages are consistent with the oldest concordant ages in Lewis and Sircombe (2013), which range from 3.4 Ga (North Rankin-5) to 2.72 Ga (Hijinx-1). Similar ages are recognized in the crystalline basement of the Yilgarn Craton and the Pilbara Craton. The Yilgarn Craton comprises several assembled terranes with ages mainly between 3.2 and 2.8 Ga and some younger terranes, notable in the Eastern Goldfields Superterrane (c. 2.75–2.65 Ga; Kinny et al. 1988, 1990; Griffin et al. 2004; Mole et al. 2019; Wyman 2019). The Pilbara Craton consists of a series of granite, greenstone terranes with granite magmatic crystallization ages of 3.72 to 2.85 Ga, 3.27 to 2.92 Ga, and <3.29 Ga (Williams and Collins 1990: Blake and Barley 1992: Thorpe et al. 1992). Based on the U-Pb ages, it is difficult to definitively distinguish between Yilgarn Craton and Pilbara Craton sources, but a proximal Pilbara Craton source most easily accounts for the measured Archean grains.

#### Paleo- and Mesoproterozoic, group 1 (2000–1500 Ma)

Between 1825 and 1765 Ma, the Western Australian Craton collided with the North Australian Craton generating several intrusive magmatic and metamorphic events (Smith 2001; Cawood and Korsch 2008). Zircon grains of this age could be sourced from the Gascoyne Province, as well as from the Arunta–Tanami–Tennant region (Black 1984; Young *et al.* 1995; Cross and Crispe 2007), and further north the 1855–1820 Ma Halls Creek and King Leopold Orogens. In the northern and central part of the Arunta Orogen (Fig. 1), major tectonism occurred between 1790 and 1730 Ma. In proximity to the Arunta Orogen, the Musgrave

Province was formed at the suture zone of the North, West and South Australian cratons in central Australia (Fig. 1). One of the oldest basement rocks of the Musgrave Province, the 1600–1550 Ma Warlawurru Supersuite orthogneiss, has a comparable age to age group 1.

#### Meso- and Neoproterozoic, group 2 (1300-800 Ma)

Mesoproterozoic age group 2 is defined by variable magnitudes of unimodal dominant to bimodal broad subordinate age peaks within the different wells (Fig. 7). Zircon grains within age group 2, which represent the more dominant peak at ~1190 Ma, are possibly sourced from the Albany Fraser Orogen (Stage II 1215-1140 Ma; Kirkland et al. 2011, 2015) and the Musgrave Orogen (1220-1150 Ma; Haines et al. 2016). Equivalent rocks to those of the Albany Fraser Orogen are identified in Wilkes Land of East Antarctica, defining a long-lived, highly extended margin (Morrissey et al. 2017). Zircon ages of ~1300-1160 Ma from magmatic rocks from Wilkes Land extend the possible provenance of detrital components of the Mungaroo Formation to more distal parts of the formerly contiguous landmass. The zircon grains of the younger peak, ~1050 Ma in age group 2, which cannot be observed in all kernel populations, are possibly sourced from the Pinjarra Orogen (1095-990 Ma, Collins 2003; Fitzsimons 2003; Markwitz et al. 2017a, Fig. 1).

#### Neoproterozoic and Cambrian, group 3 (650-490 Ma)

Age group 3 comprises zircons of early Neoproterozoic to late Cambrian age; it is identified across all samples and represents the dominant age mode in six of the 10 well age distributions (Fig. 7). Yodel-1, Geryon-2, Noblige-1 and Dalia South-1 contain more minor late Neoproterozoic age components. Zircon grains clustering around 600 Ma are dominant in the Mungaroo Formation and may correlate with zircon growth in the 600-550 Ma Paterson Orogeny (Fig. 1). Magmatism at 550 Ma is prolific in the NW of the Paterson Orogen, proximal to the Northern Carnarvon Basin. Additionally, the ~750-520 Ma Kuunga Orogeny (Fig. 1) took place when India-Africa collided with Australia-Antarctica (Collins 2003; Markwitz et al. 2017c). Thus, a more distal source to the south in East Antarctica is feasible, where crystalline rocks of this age exist (Boger et al. 2002; Liu et al. 2006). Morón et al. (2019) proposed a long-lived sediment transportation pathway from Antarctica to the Northern Carnarvon Basin through the Canning and/or Perth Basin, whereas Kirkland et al. (2020) argued for a more dissected transport system originating from Antarctica, with numerous recycling stages across a Yilgarn Craton veneer.

# Post-Cambrian, group 4 (350-200 Ma)

Age group 4 comprises Late Triassic to Early Jurassic aged zircon grains and generally represents a subordinate age peak across the wells, except in Yodel-1 and Geryon-2 where it is similar in intensity to the age group 3 peak (Fig. 7). In Noblige-1 age group 4 is non-existent. Age group 4 could be sourced from the Lhasa Terrane (Pullen *et al.* 2011; Zhu *et al.* 2011), which drifted northwards due to rifting processes in the Late Triassic, or from the South West Borneo Block, which was still assembled with Gondwana (Fig. 1). Alternatively, age group 4 may reflect derivation from Triassic igneous rocks encountered in the Anhalt-1 and Hannover South-1 wells in the Roebuck Basin (Curtis *et al.* 2019). The Triassic detrital age peak appears temporally close to the depositional age, ~200 Ma, of these sedimentary rocks.

### Recycled sediment

Zircons of all age groups may also be transported into the Northern Carnarvon Basin via recycling from intermediate sedimentary storage, i.e. basins. Basins in the Capricorn Orogen such as the Edmund Basin contain zircon grains sourced from the Gascoyne Province (1820–1770 Ma; Johnson 2013), which would correlate with age group 1 zircons. Another possible source of recycled material is the Perth Basin, which comprises zircon grains from the Yilgarn Craton, Albany Fraser Orogen, Pinjarra Orogen and the East Antarctica Craton (Cawood *et al.* 2012; Olierook *et al.* 2019). Basins in central and NW Australia (e.g. Officer and Canning basins) also contain distinctive zircon age signatures associated with the Musgrave Province and more distal Neoproterozoic to earliest Paleozoic Antarctican sources (Fig. 1; group 3; Haines *et al.* 2013; Morón *et al.* 2019).

#### Age distribution synthesis

No specific systematic geographical pattern exists in the age spectra of the samples collected from different wells, suggesting fundamentally similar sedimentary sources across the Mungaroo Delta during the interval sampled (Fig. 1). Additionally, no specific changes within age spectra were observed throughout the sampled stratigraphy within individual wells (50 m Yodel-1 and 240 m Geryon-2), suggesting temporal stability of source material supply to the delta. Although the four age groups are consistent in terms of their occurrence in the samples across all wells, the age peak intensities vary (Fig. 7, Tables S6 and S7).

Our new detrital zircon age data emphasize the significance of a Triassic source, which is reflected by age group 4. This supports the possibility of a north-south sediment transportation pathway from the Lhasa Terrane or the South West Borneo Block or the Roebuck Basin to the Northern Carnarvon Basin (Lewis and Sircombe 2013). The contribution to the Mungaroo Formation of age group 4 zircons in Yodel-1 and Geryon-2 is equivalent to that of age group 3 zircons, which would suggest a less dominant Neoproterozoic-Cambrian/ Ordovician component than reported in the age populations of Noblige-1, Dalia South-1, Hijinx-1, Guardia-1, Alaric-1 and Goodwyn-6 (Fig. 1 and 7). Furthermore, the similar magnitudes of the two youngest age groups hint at similar source dominance and therefore at provenance drainages with comparable sediment flux. In contrast, the Mesoproterozoic age group, c. 1.3 Ga, is clearly dominant in Geryon-2 (similar in North Rankin-5 and Lady Nora-2), which implies significant sediment transportation ultimately from rocks affected by Stage II of the Albany Fraser Orogen, the Musgrave Orogeny or comparable magmatic systems in East Antarctica (or reworked intermediate basins).

# Maximum depositional age

The youngest zircon age within a sediment is very important as it constrains the maximum depositional age of the bed in which it occurs (e.g. Bingen et al. 2001; Williams 2001; Fedo et al. 2003) or, in some circumstances, may even reflect a first-cycle volcanic component, which would then directly date the deposition. The Mungaroo Formation could potentially contain tuff deposits due to its proximal location to a subduction zone to its north associated with arc-volcanism during the Late Triassic (e.g. Veevers 2006; Zeng et al. 2019). Whilst some zircon grains do congregate in layers (e.g. TR-8; Fig. 6), the minor rounding on these grains and the fact that the local compositional environment around such grains is no different to the entire thin-section (Fig. 6) strongly imply they are not primary airfall crystals and are likely hydrodynamically concentrated. As these samples lack directly emplaced volcanic zircon crystals, we suggest that the measured youngest zircon grain provides only a maximum depositional age constraint for the Mungaroo Formation. There are various means of calculating maximum depositional age; however, in this work, we argue that in situ measured zircons are more representative and immune from



Fig. 8 Schematic diagram illustrating how in situ zircon U–Pb measurements in thinsections are more representative of the detrital sink than measurements on processed hand-picked mounts.

sample contamination issues (mainly related to drilling mud and laboratory processes like crushing). The youngest single grain determined in this work yields a concordia age of  $197 \pm 2$  Ma (Sinemurian, Early Jurassic, Geryon-2), which is younger than the Norian age (Late Triassic) traditionally assigned to the upper part of the Mungaroo Formation (e.g. Iasky et al. 2002). This grain preserves primary oscillatory zonation (Fig. 4), has a moderate to high Th/U ratio (2.72), and is interpreted to provide a magmatic crystallization age. The youngest concordant zircon grain in Yodel-1 is  $206 \pm 3$  Ma (Rhaetian, Upper Triassic) and older than in Geryon-2, but the sample itself does not represent the very top of the formation due to an unconformity above this sample location in Yodel-1 (Fig. 2). Except for Noblige-1, which has the youngest age peak of ~600 Ma, all other wells are consistent with a ~200 Ma maximum depositional age. Thus, the maximum depositional age of the base of the overlying Brigadier Formation may not be Rhaetian (Backhouse et al. 2002) as previously believed.

#### Composition

The samples of the Mungaroo Formation are fine-grained alternating with coarser-grained sandstones. Similar compositions across different well depths (Tables S1 and S5) suggest the sediment supply of the Mungaroo Formation is well-mixed. However, there are differences between the wells in terms of feldspar content (14% Geryon-2, 7% Yodel-1). The occurrence of feldspar implies that some of the detritus is likely first cycle and could point to a more proximal source region for framework minerals (e.g. Tyrrell et al. 2009). In contrast, highly altered feldspar, mineral fragments, and clay minerals are interpreted as products of longer transportation paths, greater chemical weathering and/or energetic transportation (e.g. Attal and Lavé 2009). Because the thin-sections contain angular as well as sub-rounded zircon grains, primary and recycled zircon sources are implicated (Markwitz et al. 2017b). Grain size variation between laminations could indicate subtle changes in depositional conditions, e.g. changes in fluvial current velocities.

Whereas *in situ* thin-section by automated phase mapping has already been described for crystalline rocks (e.g. Vermeesch *et al.* 2017), the analytical approach applied herein quantifies the proximal detrital mineral assemblage around the dated zircon grains. The comparison between the *c*. 66  $\mu$ m buffer around the dated grains and the broader thin-section composition reveals a

similar mineral assemblage, implying that the dated zircon grains are not embedded in specific lithological fragments, nor concentrated in tuff bands (Fig. 6).

### Implications

Interpretation of U–Pb age data of detrital zircon grains may be limited by a number of factors including mineral separation, analytical spot size, statistical robustness (amount of dated grains), and a wide array of other geological and laboratory biases. Nonetheless, in this study we provide a methodology that can evaluate whether or not dated zircon grain sizes are representative of the overall grain sizes preserved within a sample (Fig. 8). Additionally, quantitative mineral assemblage assessment within specific thin-section areas yields further lithological context for zircon provenance interpretations.

The application of this new approach might not be suitable in large basin-scale provenance reconstruction projects, nor challenging low zircon concentration sedimentary rocks due to cost- and time-efficiency factors. However, the approach shows potential in detecting and/or quantifying the mineral composition of zirconbearing rock fragments within a sedimentary rock, which in turn could be compared to possible source region rock compositions. Furthermore, relatively thin tuff layers, which are important in determining absolute depositional ages, can be more easily detected. Likewise, the determination of maximum depositional ages of in situ measured grains are more robust compared to grain ages measured on conventional mounts. Thus, the most appropriate sampling strategy depends on the ultimate geological question addressed and the nature of the geological material. In general, while stratigraphic fingerprinting would appear best addressed via dating on bulk mounts, maximum depositional ages are most accurately defined via dating in thin-section.

### Conclusions

This study identifies grain size biasing of detrital zircon populations introduced by the choice of sample preparation, e.g. thin-section vs. hand-picked-/bulk mounts. The comparison of grain size distributions from different sample preparation approaches reveals the effect of grain size bias on detrital zircon age signature. Here we compare dated zircon grain sizes in thin-sections to disaggregated sample zircon mounts and also to hand-picked zircon mounts. Furthermore, the local lithological context of detrital zircons can be assessed by the mineralogical mapping of the grains' proximal environment. This approach enables greater confidence in identifying volcanic contributions or grains as clastic inclusions. Furthermore, analysis in thin-section circumvents some issues associated with maximum depositional ages as determined conservatively via a young group, giving greater confidence in the youngest individual analysis.

Possible provenances of the Mungaroo Formation are most likely the Albany Fraser, Arunta, Pinjarra and Musgrave orogens, indicating major sediment transportation from the south or east to the Northern Carnarvon Basin. More distally, grains may have been supplied from East Antarctica (Wilkes Land, Kuunga Orogen). Besides first-cycle direct transportation from magmatic sources, multi-cycle sediment transportation through reworking of intermediate sediment storages, e.g. the Edmund Basin, the Perth Basin and/or the Canning Basin, is also feasible. The Lhasa and South West Borneo terranes are potential sources of Triassic zircon reflecting a north-south transportation pathway. Our findings support a greater contribution from an active margin to the north than previously assumed. Generally, sediment transportation to the Northern Carnarvon Basin was via a stable deltaic river system with a steady sediment supply. The maximum depositional age of the upper part of the sampled Mungaroo Formation may be younger than the previously suggested Norian age based on a single grain age of  $197 \pm 2$  Ma.

The new methodology demonstrates *in situ* U–Pb geochronology of detrital zircons measurements in thin-sections is more robust against sample processing bias than hand-picked grain mounts, which appear biased towards larger grain sizes. An incorporated buffer analysis applied on dated zircon grains provides quantitative mineral assemblage information in specific thin-section areas, thus extracting greater lithological information. Whilst undoubtedly more time consuming, thin-section analysis provides greater confidence in geochronological interpretation of the relative detrital zircon peak heights.

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**Data availability** All data generated or analysed during this study are included in this published article (and its supplementary information files). Computer code developed in this work is hosted in GitHub and linked in the text and supplementary materials.

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