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**Refining the chronostratigraphy of the Karoo Basin, South Africa:
magnetostratigraphic constraints support an Early Permian age for the Eccca Group**

Belica, Mercedes E.^{1}; Tohver, Eric¹; Poyatos-Moré, Miquel²; Flint, Stephen²; Parra-Avila, Luis A.^{3,4}; Lanci, Luca⁵; Denyszyn, Steven¹; Pisarevsky, Sergei A.^{1,6}*

¹ School of Earth Sciences, University of Western Australia, Perth WA 6009, Australia

² School of Earth and Environmental Sciences, University of Manchester, U.K.

³ Centre for Exploration Targeting, School of Earth Sciences, The University of Western Australia, Perth, WA 6009, Australia

⁴ Australian Research Council Centre of Excellence for Core to Crust Fluid Systems (CCFS)

⁵ Dept. of Pure and Applied Science, University of Urbino “Carlo Bò”, Urbino, Italy

⁶ Earth Dynamics Research Group, ARC Centre of Excellence for Core to Crust Fluid Systems (CCFS) and The Institute for Geoscience Research (TIGeR), Department of Applied Geology, Curtin University, GPO Box U1987, WA 6845, Australia

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“Magnetostratigraphy of the Eccca Group”

*Corresponding author: +61417175165; Mercedes.belica@research.uwa.edu.au

“Magnetostatigraphy of the Eccca Group”

Summary:

The Beaufort Group of the Karoo Basin, South Africa provides an important chrono- and biostratigraphic record of vertebrate turnovers that have been attributed to the End-Permian mass extinction events at ca. 252 Ma and ca. 260 Ma. However, an unresolved controversy exists over the age of the Beaufort Group due to a large dataset of published U-Pb SHRIMP zircon results that indicate a ca. 274-250 Ma age range for deposition of the underlying Eccca Group. This age range requires the application of a highly diachronous sedimentation model to the Karoo Basin stratigraphy and is not supported by published paleontologic and palynologic data. This study tested the strength of these U-Pb isotopic datasets using a magnetostatigraphic approach. Here we present a composite ~1500 m section through a large part of the Eccca Group from the Tanqua depocentre, located in the southwestern segment of the Karoo Basin. After the removal of two normal polarity overprints, a likely primary magnetic signal was isolated at temperatures above 450°C. This section is restricted to a reverse polarity, indicating that it formed during the Kiaman Reverse Superchron (ca. 318-265 Ma), a distinctive magnetostatigraphic marker for Early–Middle Permian rocks. The Eccca Group has a corresponding paleomagnetic pole at 40.8°S, 77.4°E ($A_{95} = 5.5^\circ$). U-Pb SHRIMP ages on zircons are presented here for comparison with prior isotopic studies of the Eccca Group. A weighted mean U-Pb age of 269.5 ± 1.2 Ma was determined from a volcanic ash bed located in the uppermost Tierberg Formation sampled from the OR1 research core. The age is interpreted here as a minimum constraint due to a proposed Pb-loss event that has likely influenced a number of published results. A comparison with the Geomagnetic Polarity Time Scale as well as published U-Pb TIMS ages from the overlying Beaufort Group supports a ca. 290-265 Ma age for deposition of the Eccca Group.

Keywords: palaeomagnetism; magnetostatigraphy; magnetic fabrics and anisotropy; reversals: process, time scale, magnetostatigraphy; sedimentary basin processes; tectonics and landscape evolution

1. Introduction

The Karoo Basin of South Africa (Fig. 1) represents an extensive depositional record of Pennsylvanian–early Jurassic fossiliferous sedimentary (Rubidge et al., 1999) rocks that have been critical for understanding the rate of vertebrate turnovers for continental extinction events during the End-Guadalupian and across the Permian-Triassic boundary (Botha and Smith, 2006; Day et al., 2015; Gastaldo et al., 2015; Rubidge et al., 2013; Smith and Botha, 2005; Smith and Botha-Brink, 2014; Smith and Ward, 2001; Ward et al., 2005). The excellent preservation of the associated vertebrate assemblages has led to a large library of published faunal information, complemented by palynological data, that has been extrapolated globally to understand the patterns of tetrapod extinctions and their timing relative to the end-Permian marine extinction at ca. 252 Ma (Burgess et al., 2014; Lucas, 2010; Mundil et al., 2004; Shen et al., 2011; Ward et al., 2005).

The Permian-Triassic boundary (PTB) is defined by marine conodont assemblages, and has a corresponding age of 251.9 ± 0.0 Ma determined from high resolution U-Pb TIMS (zircon) dating of ash beds from the Meishan section in south China (Burgess et al., 2014). The terrestrial PTB is considered to be broadly represented by the Last Appearance Datum of the late Permian tetrapods of the *Daptocephalus* (formerly *Dicynodon*; Viglietti et al., 2016) Assemblage Zone and the First Appearance Datum (FAD) of early Triassic taxa in the *Lystrosaurus* Assemblage Zone (Lucas, 2010; Smith and Botha-Brink, 2014), of which the Beaufort Group of the Karoo Basin has long been considered the prime example. However, in the past decade a number of U-Pb SHRIMP (zircon) results have been published from this region that support a ca. 274-250 Ma age range for deposition of the underlying Ecca Group, and therefore a Triassic age for the Beaufort Group (Fig. 2) (Fildani et al., 2007; Fildani et al., 2009; McKay et al., 2016; McKay et al., 2015).

This depositional age assignment requires that the PTB be placed within the upper Skoorsteenberg Formation of the Eccca Group, ~2000 m below the biostratigraphically constrained PTB in the upper Beaufort Group (Fig. 2). This assignment is in direct conflict with an extensive body of paleontologic data (Barbolini, 2014; Rubidge et al., 1999; Rubidge, 1990; Smith and Keyser, 1995), as well as a series of ca. 262–268 Ma U-Pb SHRIMP ages on zircon grains from the overlying Abrahamskraal Formation of the lower Beaufort Group (Lanci et al., 2013) and ca. 262–255 Ma ages from the Adelaide Subgroup of the Beaufort Group (Rubidge et al., 2013). The apparent age controversy was explored by McKay et al. (2015; 2016) who demonstrated that volcanic tuffs from the Eccca Group yield consistently younger ages (250 to 274 Ma) than tuffs from the conformably overlying Beaufort Group (257 to 452 Ma) along a 650 km transect across the southern margin of the Karoo Basin. This “inverted” basin stratigraphy was ascribed to an episode of zircon exhaustion and magmatic recycling (as opposed to a disturbed U-Pb system). The placement of the PTB within the upper Eccca Group was further reinforced by a presumed “tuff gap” in the Karoo Basin located above this horizon (Waterford Formation), similar to other Permian–Triassic depositional sites along the southern Panthalassan margin of Gondwana (McKay et al., 2016; McKay et al., 2015; Veevers, 2004).

In order to resolve this apparent discrepancy between the biostratigraphically-constrained and geochronologically-constrained PTB, we applied the technique of magnetostratigraphy, which has proven very useful in the correlation and timing of major geologic events (Glen et al., 2009; Horacek et al., 2010; Hounslow et al., 2016; Nawrocki, 2004; Ogg et al., 2016; Opdyke and Channell, 1996; Opdyke et al., 2000; Steiner et al., 1989; Steiner, 2006; Szurlies, 2013; Szurlies et al., 2012; Taylor et al., 2009; Ward et al., 2005). Magnetostratigraphy is a geophysical relative-dating method that relies on the global synchronicity of geomagnetic reversals (Opdyke and Channell, 1996). Sedimentary rocks

may record a Depositional Remanent Magnetization (DRM) that preserves the polarity and direction of Earth's magnetic field during the time of formation (Butler, 1992). The Geomagnetic Polarity Time Scale (GPTS) is the record of reversals tied to the biostratigraphically-constrained geologic stage boundaries (Fig. 3). The magnetic field reverses its polarity on average every 0.1-1 Ma, but reversal rates through time can be highly variable. Several periods of stable field polarity have been observed, the longest of which is known as the Kiaman Reverse Superchron (KRS; Fig. 3), which lasted more than 50 million years (Alva-Valdivia et al., 2002; Belica et al., 2017; Diehl and Shive, 1979; Diehl and Shive, 1981; DiVenere and Opdyke, 1990; DiVenere and Opdyke, 1991; Gialanella et al., 1997; Gose and Helsley, 1972; Irving and Parry, 1963; Khramov and Tarling, 1987; Lozovsky and Molostovsky, 1993; McFadden et al., 1988; Menning et al., 1988; Nawrocki, 1997; Opdyke and Channell, 1996; Opdyke et al., 2001; Opdyke et al., 2000; Steiner, 2006; Steiner et al., 1993; Szurlies et al., 2003).

The start of the KRS has been assigned an age of ca. 318 Ma from two U-Pb SHRIMP zircon ages of 317.8 ± 2.8 Ma (Eastons Arm Rhyolite) and 318.0 ± 3.4 Ma (Peri Rhyolite) in the northern Tamworth Belt of eastern Australia (Opdyke et al., 2000). The end of the KRS has recently been constrained by a ^{40}Ar - ^{39}Ar plateau (plagioclase) age of 265.1 ± 0.5 Ma from the Bumbo Latite near Kiama, Australia (Belica et al., 2017). Beginning in the Capitanian (ca. 265 Ma), the geomagnetic field transitioned back into a frequently reversing state, and this transition was thought to occur over a short (~10 Ma) interval (Hounslow, 2016). The Late Permian through Early Triassic, therefore, marks a period of increasing reversal frequency (~2-4/Ma; Hounslow, 2016), providing a strong chronostratigraphic marker (Fig. 3).

We present a composite, ~1500-m-thick magnetostratigraphic section through a large part of the Eccra Group from the Tanqua depocentre, located in the southwestern segment of

the Karoo Basin, and provide a test of the published U-Pb isotopic datasets through comparison with the GPTS. Since the geomagnetic field was restricted to a reverse polarity prior to 265 Ma, the presence of reversals would indicate that deposition of the ash beds occurred in the late Permian, whereas a uniform reverse polarity would support the existing biostratigraphic age data, as well as provide strong evidence for disturbance of the U-Pb system.

2. Geologic background

The Karoo Basin (~700,000 km²) has been classically interpreted to be a retro-arc foreland basin that developed behind an inferred magmatic arc and fold-thrust belt (Catuneanu et al., 1998; Johnson et al., 1996; Lindeque et al., 2011; Milani and De Wit, 2008). In this interpretation, the basin formed ~1000 km inland from the southern Panthalassan margin of Gondwana and to the north and east of the two branches of the Cape Fold Belt (Fig. 1). A more recent interpretation by Tankard et al. (2009; 2012) suggested that subsidence during the deposition of the Ecca Group and lower Beaufort Group was a function of mantle flow associated with dynamic topography of the subducting plate where foreland basin formation (linked to emergence of the Cape Fold Belt) did not take place until the Triassic. This model is consistent with provenance studies (Van Lente, 2004), gross thickness trends, and low rates of deposition in the lower Ecca Group (Flint et al., 2011). Tectonic activity related to the Cape Orogeny has been suggested to have occurred in two separate deformation events, the first at ca. 275-260 Ma, and the second at ca. 255-245 Ma (Hansma et al., 2016). The southern Cape Fold Belt is associated with low temperature metamorphism and deformation folds that decrease in amplitude northward toward the Karoo foreland basin (Paton et al., 2006). Deformation in the Cape Fold Belt has been linked to synchronous sedimentation in the foreland basin (Catuneanu et al., 1998), though the

provenance of the Eccca Group has also been interpreted to be the Patagonian batholith (Van Lente, 2004). The basin fill succession shows paleocurrents consistent with a long-term northeasterly progradation of the depositional systems (Flint et al., 2011; Johnson et al., 1996; Poyatos-Moré et al., 2016; Wild et al., 2009).

The base of the Karoo Supergroup is defined by the glaciogenic Dwyka Group that consists of diamictites formed during the Carboniferous–Permian glaciation of southern Gondwana (Fig. 3). Two U-Pb SHRIMP zircon ages of 288.0 ± 3.0 Ma and 289.6 ± 3.8 Ma reported from tuffs in the overlying Prince Albert Formation (Fig. 2) suggest a ca. 290 Ma minimum age for this unit (Bangert et al., 1999), and paleomagnetic data support deposition during the KRS (Opdyke et al., 2001). The Lower Eccca Group overlies the Dwyka Group (Fig. 2), and in the Tanqua depocentre (present study location; Fig. 1) is composed of shales to fine grained turbidites of the Prince Albert, Whitehill, and Collingham formations, which indicate the transition to prolonged postglacial sea-level rise and the initiation of deep-water deposition (Hodgson et al., 2006).

The middle-upper Eccca Group (Fig. 2) comprises ~1500 m of basin floor to deltaic strata; this includes basinal mudstones of the Tierberg Formation (Fig. 2), four basin floor turbidite fans (Fans 1-4) and a channelized lower slope complex (Unit 5; Fig. 2) of the Skoorsteenberg Formation, overlain conformably by submarine slope/shelf edge strata of the Kookfontein Formation, and deltaic deposits of the Waterford Formation (Gomis-Cartesio et al., 2016; Hodgson et al., 2006; Poyatos-Moré et al., 2016; Wickens, 1994; Wild et al., 2009; Wild, 2005). U-Pb ages published from the Eccca Group yield maximum depositional ages ranging from ca. 274–250 Ma (Fildani et al., 2007; Fildani et al., 2009; McKay et al., 2016; McKay et al., 2015). The younger ages in this range (ca. 260–250 Ma) are problematic in that they require the PTB to be placed within the middle Eccca Group, as opposed to the

biostratigraphically-supported placement in the overlying terrestrial fluvio-lacustrine strata of the Beaufort Group (Figs. 1-2).

Some authors have explained this apparent age inversion by applying a diachronous sedimentation model across the Karoo Basin, where the submarine fans of the Ecca Group in the southwest are considered correlative with the terrestrial Permian-Triassic sections located farther east (Fildani et al., 2007; Fildani et al., 2009). However, this interpretation is not supported by recent regional mapping that shows continuity of the marine Ecca Group for over 500 km across the basin, with a series of interfingering submarine fan deposits, all overlain by eastward prograding deltaic deposits (Flint et al., 2017). This proposed diachronous sedimentation model is also inconsistent with the biostratigraphic constraints on basin infill starting in the southwest during the deposition of the Beaufort Group (Rubidge, 2005).

3. Sampling and methodology:

3.1. Paleomagnetism

Paleomagnetic cores were collected from three partially overlapping sections comprising ~1500 m of the Tierberg, Skoorsteenberg, Kookfontein, and Waterford Formations (~600 samples) of the Ecca Group in the Tanqua depocentre (Figs. 1-2). The Ecca Group sedimentary rocks sampled here are almost flat-lying with regional dips of 1-3°, and lie outside the main region of Cape Fold Belt deformation (Figs. 1-2). Approximately 950 m of the Tierberg and Skoorsteenberg Formations were sampled from the OR1 core with a sampling interval of about 4 m (~200 samples). Magnetic susceptibility measurements were taken for the top 230 m of section at a resolution of 0.25 m. The overlying Kookfontein (Pienaarsfontein outcrop locality) and Waterford Formations (SL1 outcrop locality) were sampled from two field sections located ~20 km apart (Fig. 4). Lithologic correlation

between the two sections (Fig. 4) was made using the interpreted maximum regional flooding surfaces of Wild et al., (2009) and Poyatos-Moré et al. (2016). At Pienaarsfontein and SL1, the Kookfontein and Waterford Formations were sampled at a resolution of 2 m (~300 samples) and magnetic susceptibility measurements were taken with a SM-20 portable magnetic susceptibility meter to determine the variability of magnetic properties along the section at a resolution of 0.25 m.

Samples were drilled in the field using a portable, petrol-powered hand drill and oriented using a Brunton magnetic compass and a solar compass to correct for local magnetic declination. Thickness of the stratigraphic succession was measured using a Jacob's staff perpendicular to bedding to account for tectonic dip, and a detailed stratigraphic log was completed at a 1 m resolution with GPS points tagged every 10 m for correlation with the logged section of Wild (2005). The section was photographed and structural attitudes were measured in order to correct for any post-magnetization tilting. Samples were then cut into 10 cm³ cylindrical cores, and measured at the Alpine Laboratory of Paleomagnetism in Peveragno, Italy, where a pilot study was conducted to determine the best treatment for isolation of the individual magnetic components. To avoid the acquisition of a viscous remanence over 400°C, the samples were demagnetized in a field free space and measured directly after cooling.

Stepwise thermal demagnetization was conducted using an ASC TD48 thermal demagnetizer to temperatures up to 670°C. Samples were measured on a 2G DC-SQUID magnetometer, and paleomagnetic directions were isolated via principal component analysis (Kirschvink, 1980) using PaleoMag 3.1 (Jones 2006). Individual components were calculated from three or more points, and directions were restricted to Mean Angular Deviation (MAD) values $\leq 10^\circ$ for lines and $\leq 15^\circ$ for planes in calculation of the mean paleomagnetic pole. A minimum of three points (4 for planes) defined a stable endpoint magnetization, and anchored

fits were avoided due to the anomalously low uncertainty estimations (Heslop and Roberts, 2016). Where MAD values were greater than 15° , or where samples did not reach stable end-points, the arc method of McFadden and McElhinny (1988) was used to isolate the primary component of magnetization. Final iterative directions along the arc constraints were combined with lines for the stratigraphic polarity interpretation.

The reversal stratigraphy (declination, inclination, and VGP Latitude) was then plotted against sampling level and a composite magnetostratigraphic barcode was completed and compared with the GPTS (Hounslow and Balabanov, 2016; Ogg et al., 2016). Magnetic reversals were interpreted from remanence directions that crossed the magnetic equator (defined as 90° along a great circle from the mean direction) where three or more consecutive samples define a magnetozone. Single-sample polarity reversals (marked by a half-bar to represent uncertainty) indicate either 1) a geomagnetic subchron; 2) a magnetic reversal with insufficient adjacent sampling, or 3) possible remagnetization of the individual sample. Paleomagnetic directional data are provided in Table 1.

3.2 Magnetic Fabric

Eighty-seven samples were processed from the Eccla Group (Pienaarsfontein and SL1) to determine the anisotropy of magnetic susceptibility (AMS). Principal axes of AMS were measured on a MFK1-FA Kappabridge using the 3-axis spinning protocol at the University of Western Australia. Results were plotted using the Anisoft 42® software (Chadima and Jelínek 2009), and analysed using Jelínek statistics (Jelínek, 1981; Jelínek and Kropáček, 1978). Results are provided in Table 2.

3.3 SHRIMP U-Pb geochronology

Ten ash beds from the Tierberg and Skoorsteenberg Formations of the Ecca Group were sampled and processed for geochronological work. Samples were collected from the continuous 800-m-thick core of the research borehole OR1 (Fig. 1). Ash layers are typically 2-6 cm thick, and consist of fine grained siliciclastic and well-consolidated clay-rich volcaniclastic material interbedded within siltstones. Zircon grains were isolated from each ash layer using conventional mineral separation techniques at the University of Western Australia. Each lithified ash sample was pulverized with a rock hammer, processed through a disc mill, washed and placed in a sonic bath for 30 minutes and dried. The crushed material was separated using 40 and 150 μm sieves and a bar magnet to remove ferromagnetic components before processing through a Frantz Isodynamic Separator. The non-magnetic portion was then put through a heavy liquid density separation with multiple agitation periods using LST (Lithium heteropolytungstates) heavy liquid ($\rho = 2.85 \text{ g/cm}^3$ at 25°C) for four hours to isolate grains in the higher density fractions. The heavy fraction was washed and handpicked under a binocular microscope.

Six of the ten ash beds yielded zircon grains considered suitable for analysis. Within the six samples, three main populations of zircon morphology were identified, including a population of idiomorphic and elongate grains with oscillatory cathodoluminescent zoning and an aspect ratio of 4:1 (Fig. 5a). Two other populations of euhedral zircons with oscillatory zoning were also sampled with aspect ratios of 2-3:1 (Fig. 5ab). Around 50 randomly picked zircon grains from each sample were mounted alongside the zircon standards M257 (561.3 Ma; 840 ppm U; Nasdala et al., 2008) and Temora 2 (416.8 Ma; Black et al., 2004) in 2.5 cm diameter epoxy discs (3–4 samples per disc) as external standards for U/Pb ratios and U concentrations. Discs were then polished and gold coated (20 μm) for imaging with the back scatter electron (BSE) and cathodoluminescence (CL) detectors of a Tescan Vega 3 analytical SEM system at the Centre for Microscopy,

Characterization and Analysis (CMCA) at the University of Western Australia.

Microphotographs were used to identify magmatic features such as oscillatory zoning and euhedral/elongate form (Fig. 5). Apparent inherited cores were identified from CL images as well as fractures and damaged grains (Fig. 5). Highly fractured and metamict grains can produce unreliable U-Pb age data, and were avoided in the subsequent SHRIMP analysis.

The discs were then cleaned using ethanol, petroleum spirits, and a soap solution, and rinsed with deionized water. Discs were dried in an oven (1 h at 60°C) and the surface was gold coated at 40 µm and processed on the SHRIMP II A (Sensitive High Resolution Ion Microprobe) at Curtin University, Western Australia, following the procedure described by Wingate and Kirkland (2017). Analytical conditions and operational procedures have previously been described (Claoué-Long et al., 1995; Compston et al., 1984; De Laeter and Kennedy, 1998; Kennedy and De Laeter, 1994; Wingate and Kirkland, 2017). Rastering of the O²⁻ beam was conducted for 120-150 s to reduce common Pb contamination before each analysis, and each spot was focused using a primary ion beam of 1.5-1.9 nA with a diameter of ~25 µm. SHRIMP spots were made on both inherited cores and rims to determine the level and types of inheritance, and a common Pb correction was applied using the measured amount of ²⁰⁴Pb. Data reduction and correction were conducted as described by Wingate and Kirkland (2017) using the SQUID2 and Isoplot3 software (Ludwig, 2008; Ludwig, 2009). All calculated ages are reported at 95% confidence. To reduce the problems caused by zircon grains with potentially disturbed U-Pb systems, analyses that comprised more than 1.5% of non-radiogenic ²⁰⁶Pb or U concentrations over 1000 ppm were rejected. Analytical data are presented in Table 3, and the complete isotopic dataset is available in the supplementary material.

4. Results and interpretation

4.1 Paleomagnetism

4.1.1. *Ecca Group, Pienaarsfontein*

The outcrop section spanning the upper Skoorsteenberg and Kookfontein formations (Fig. 4) displayed the least complicated thermal demagnetization spectra of all the studied sections (Figs. 6-8abd). Mean paleomagnetic directions are reported in Table 1. The Natural Remanent Magnetization (NRM; magnetization before demagnetization) intensities of the sedimentary rocks range from 0.3 to 113 mA/m, with an average value of 4.0 mA/m. NRM intensities were highest for the top 50 m of section (~1430 mA/m), and randomly oriented and shallow directional data suggest that this segment has been completely remagnetized by lightning. These uppermost samples were discarded from further consideration. Magnetic susceptibility data revealed a mainly ~300 m rhythmic susceptibility profile with an average value of 0.15 ($\times 10^{-3}$ SI units; Fig. 9).

Progressive thermal demagnetization of the samples revealed up to three discernible paleomagnetic components, with all samples becoming unstable at $T > 580^{\circ}\text{C}$ (Figs. 6-8). The first, referred to as Component A, is present in about 20% of the samples, and is stable from about 110-250°C. The mean direction is broadly consistent with a present field overprint, although we note that the small number of isolated directions may not be representative of the true mean (Fig. 10a). In some cases, this component is absent or is preceded by a randomly-oriented low temperature direction stable below 150°C that may be representative of goethite or a Viscous Remanent Magnetization (VRM). The second component, referred to as Component B, is present in nearly every sample, and is stable from about 300-450°C (Figs. 7-9). Directional data (Declination = 341.0° ; Inclination = -60.8° ; $\alpha_{95} = 2.5$) are similar to both reported Jurassic overprint directions and a modern field component (De Kock and Kirschvink, 2004b; Lanci et al., 2013; Moulin et al., 2017). When both components A and B are recorded by the same sample, Component B always shows higher

unblocking temperatures, and is the last direction isolated before the unblocking of the Characteristic Remanent Magnetization (ChRM) direction (Component C), which is interpreted to be primary (Fig. 10b). Component C ($D = 116.0^\circ$; $I = 62.1^\circ$; $\alpha_{95} = 4.2^\circ$) is stable from about 475-580°C, and is restricted to reverse polarity, with the exception of a few samples throughout the section which display normal polarity. Some of these normal polarity samples showed stable end-point magnetizations isolated from 565-580°C after the removal of both normal Components A and B (Fig. 7d), and may be correlative with previously documented ca. 275 Ma normal subchrons within the KRS (Hounslow and Balabanov, 2016).

Intensity decay diagrams show demagnetization curves consistent with magnetite as the main magnetic phase (Fig. 8). Isolation of this ChRM component within the unblocking temperatures of magnetite excludes secondary hematite as a possible source of magnetization (Butler, 1992). Components B and C are statistically distinct and fail a common distribution test at the 95% confidence level ($\lambda_o = 21.1^\circ$; $\lambda_c = 7.0^\circ$; McFadden and McElhinny 1990). Previous paleomagnetic work in the Tanqua depocentre revealed up to three paleomagnetic components, including a low-temperature ($\leq 175^\circ\text{C}$) recently-acquired VRM and a higher temperature ($\leq 475^\circ\text{C}$) remagnetization that may be related to Jurassic Large Igneous Province (LIP) emplacement (Lanci et al., 2013; Tohver et al., 2015). Reverse polarity ChRM data reported from the overlying Abrahamskraal Formation in the Beaufort Group (Fig. 2) were similarly isolated at temperatures of 450-580°C (Lanci et al., 2013).

The mean equal area projection of the ChRM directions (Fig. 10b) shows a somewhat N-S elongation. No pattern in stratigraphic level was observed with respect to this elongation, or any difference in directional data when Bingham statistics were applied. Typically inclination shallowing expected from sedimentary rocks which have undergone compaction is recognized by an E-W directed elongation of the paleomagnetic dataset (Tauxe and Kent, 2004). This section lies within a geomagnetic superchron, and the exact behaviour of the

Earth's magnetic field during these types of events is still poorly understood (Granot et al., 2012; Hounslow, 2016; Lhuillier et al., 2016). Contributions from a non-zero non-GAD (Geocentric Axial Dipole) field can change the distribution of directions, where a non-zero axial octupole of the same sign as the axial dipole will enhance the N-S elongation of the observed directional data resulting in a shallow polarity bias (Tauxe and Kent, 2004). The superchron recorded here was of reverse polarity, and it has been documented that at least for the past 5 million years, the reverse polarity field has displayed a larger non-dipole component than the normal field (Johnson et al., 2008; Merrill and McElhinny, 1977; Opdyke and Henry, 1969; Schneider and Kent, 1988), which may be attributable to a persistent axial octupole contribution (Parés and Van der Voo, 2012), though we note this observation is speculative.

4.1.2. *Ecce Group, SL1*

The outcrop section spanning the upper Waterford Formation at the SL1 locality reveals more complicated demagnetization spectra (Figs. 6-8c). The average NRM intensity of this section is slightly higher than that recorded from the Pienaarsfontein section, with an average value of 40 mA/m. The magnetostratigraphic profile was completed through the stratigraphic succession with some offsets due to lack of exposure along the ridgeline leading up to the SL1 research well (Fig. 4). The third segment of this sampling transect (425-475 m) seems to have been mostly remagnetized by lightning, with elevated NRM intensities and randomly oriented and shallow directional data. Magnetic susceptibility data collected toward the base of the section shows a less regular pattern than that of the Kookfontein Formation, and ranges from 0.03 to a peak of 0.35 ($\times 10^{-3}$ SI units) at the 375 m mark (Fig. 9).

Progressive thermal demagnetization of the samples revealed two distinct paleomagnetic components identical to Components B and C from the Kookfontein

Formation (Pienaarsfontein locality). A few samples exhibit a random low-temperature direction up to 150°C, however, this overprint is harder to detect here due to the generally less coherent demagnetization data. Component B is present at temperatures from 150-350°C, and is removed above 400°C (Figs. 6-8c). Component C is stable from 475°C to 575°C, and is fully demagnetized above 580°C (Figs. 6-8c). The demagnetization curves from this section indicate some combination of magnetite and maghemite as the main magnetic phases (Fig. 8c). An equal area projection of the mean ChRM data shows a Fisherian distribution, with the reverse polarity sites (positive inclination) displaying a higher scatter in direction due to the overlap in unblocking spectra with the normal polarity overprint (Fig. 11ab). This section is also restricted to reverse polarity, with two potential normal subchrons from two single samples (Fig. 9).

4.1.3 Eccu Group, OR1 Core

The samples collected from the long-core of the OR1 research well (Fig. 1) show similar demagnetization behaviour to Pienaarsfontein and SL1, with the exception that these samples generally became unstable at temperatures greater than 500°C. This is attributed to partially fractured samples that were destroyed during heating above 500°C (before they were completely demagnetized). However, the majority of samples had recoverable remanences and showed a clear removal of a normal polarity overprint direction (Component B) below 475°C, with isolation of a reverse polarity ChRM direction (Component C) at temperatures ranging from 500-580 °C (Fig. 12). The drill core is vertically oriented, so only inclination data are available from this section. Negative inclinations represent normal polarity and positive inclinations represent reverse polarity. This section is restricted to reverse polarity, with one normal polarity remanence (stable at high temperatures) at 200 m depth (Fig. 13). The top of the OR1 core contains the upper sandstones of Fan 4 as well as Unit 5 from the

Skoorsteenberg Formation which overlaps with the first 115 m of section at Pienaarsfontein (Poyatos-Moré et al., 2016; Wild et al., 2009). Both overlapping sections show good agreement with respect to the variation in inclination values, with the steepest inclinations observed near the top of Unit 5, a steady and shallower average inclination of $\sim 50^\circ$ for the base, and an average inclination value of $\sim 60^\circ$ for the uppermost part of Fan 4 (Figs. 9 and 13). Magnetic susceptibility measurements range from -0.2 to $0.4 (\times 10^{-3}$ SI units), with the largest recorded values correlative with mudstone intervals against a rhythmic background susceptibility profile centred at $0.15 (\times 10^{-3}$ SI units) similar to the pattern observed at Pienaarsfontein (Fig. 9). Overlapping segments of magnetic susceptibility measurements are in strong agreement for Unit 5 (Figs. 9 and 13). NRM intensity of the drill-core ranges from 0.25 to 240 mA/m, with an average value of 3.5 mA/m.

4.2 Magnetic Fabric

AMS results for the Eccra Group are displayed in Figure 14 along with the corresponding Jelínek statistics and mean directions in Table 2. At Pienaarsfontein (Kookfontein Fm.), the measured anisotropy factors do not show any significant variation along section or for any particular lithology. The shape of the AMS ellipsoid is oblate, with a shape factor (T) = 0.669 (Fig. 14a). The fabric shows K_3 values clustered around the vertical axis consistent with horizontally-layered sediments unaffected by ductile deformation (Fig. 14a). The similarity between the shape factors (T) and difference shape factors (U) indicates a very low degree of anisotropy for the sedimentary rocks (Jelínek, 1981). At SL1 (Waterford Fm.), the AMS ellipsoid shows a mostly oblate shape (Fig. 14b); however, the Jelínek diagrams indicate a more complex geometry than expected from typical sedimentary burial and compaction ($T = 0.310$). The K_3 values here are much shallower ($< 35^\circ$), and are

clustered in a N-S direction (Fig. 14b). This fabric may be a result of an intersection lineation between sub-horizontal bedding and a vertical foliation (Parés et al., 1999).

4.3. SHRIMP U-Pb geochronology

Of the four samples analysed by SHRIMP (Table 3), three samples yielded at least 10 zircon grains with ages < 300 Ma. Two of these samples yielded internally consistent mean weighted $^{206}\text{Pb}/^{238}\text{U}$ ages suitable for interpretation and are discussed below.

For sample OR72 (331.1 m), 14 standard spots (2 rejected from initial instrument calibration) were taken on M257 (MSWD = 3.19), with an external spot-to-spot error of 0.95% (1σ) and a $^{238}\text{U}/^{206}\text{Pb}$ calibration uncertainty of 0.5178% (1σ). Of 18 analyses from zircon grains, two spots of ca. 360 and 770 Ma were interpreted as inherited grains (Table 3). $^{232}\text{Th}/^{238}\text{U}$ ratios did not indicate any metamorphic resetting, with a minimum observed value of 0.42. One grain was rejected due to a high concentration of U (1070 ppm; Table 3). No grains were rejected based on the proportion of ^{206}Pb ; however, one grain (ca. 248 Ma) was rejected based on presumed Pb-loss, likely resulting from a nearby fracture in the grain. The remaining 14 spots gave a weighted-mean $^{206}\text{Pb}/^{238}\text{U}$ age of 264.6 ± 2.9 Ma with a MSWD of 2.25. This age is interpreted here as a minimum constraint due to suspected Pb-loss. A linear trend from the oldest to the youngest grains is observed sub-parallel to Concordia, and the range in zircon ages prevents the identification of a single and discrete age population (Fig. 13).

For sample OR98 (452.8 m), 9 standard spots (2 rejected from initial instrument calibration) were taken (MSWD = 1.10), with an external spot-to-spot error of 0.50% (1σ) and $^{238}\text{U}/^{206}\text{Pb}$ calibration uncertainty of 0.4973% (1σ). $^{232}\text{Th}/^{238}\text{U}$ ratios did not indicate any metamorphic resetting, with a minimum observed value of 0.55. Of 18 unknown zircon analyses, two were rejected based on CL images that showed that the beam diameter

overlapped with fractures. Two analyses were rejected based on ^{206}Pb values ($>1.5\%$; Table 3). No analyses were rejected based on U concentration or inheritance. The remaining 14 spots revealed a distinct age population at ca. 271 Ma, with a sub-population at ca. 266 Ma that is likely a result of Pb-loss (Fig. 13). The 14 spots yielded a weighted-mean $^{206}\text{Pb}/^{238}\text{U}$ age of 269.5 ± 1.2 Ma with a MSWD of 1.02. This calculated age is in good agreement with both the reported magnetostratigraphy (Figs. 9 and 13) and the GPTS (Fig. 3), and so provides a good age estimate for the base of the Skoorsteenberg Formation in the Tanqua depocentre.

5. Discussion

5.1 Magnetostratigraphy

Individual magnetostratigraphic columns for each section are plotted in Figures 9 and 13, with a composite of the Eccra Group and overlying Abrahamskraal Formation (Beaufort Group) presented in Figure 15. The magnetostratigraphy shows an extended reverse polarity interval for over 1500 m of section that is consistent with formation during the Pennsylvanian–Middle Permian KRS (Hounslow and Balabanov, 2016; Irving and Parry, 1963; Menning et al., 1988; Opdyke et al., 2000; Steiner, 2006). This interval of constant magnetic polarity was followed by a return to a frequently reversing field at ca. 265 Ma (Belica et al., 2017), thus providing a minimum age constraint for the Eccra Group (Fig. 15).

To provide the best age estimate for deposition of the Eccra Group in the Tanqua depocentre (20°E), we compare this section with the GPTS, as well as the published isotopic ages that do not conflict with the resolved magnetostratigraphy (i.e. > 265 Ma; Fig. 15). The normal polarities reported here cannot be confirmed as primary as they are only represented by one consecutive sample; however, they are broadly consistent with the occurrence of normal polarity subchrons within the KRS (Fig. 15).

Our composite 1500 m section from the Eccca Group terminates within the upper Waterford Formation, which underlies a previously published section at Ouberg Pass (upper Waterford-lower Abrahamskraal Formation; Fig. 15) that shows mostly stable reverse polarity end-points (80%) in the magnetostratigraphy similar to Pienaarsfontein (Lanci et al., 2013). Normal polarity magnetozone were also reported (2-3 consecutive samples) for this section, and passed a reversal test. U-Pb SHRIMP (zircon) ages range from ca. 262-268 Ma, and the section was interpreted to span ~7 Ma (Lanci et al., 2013). Hounslow and Balabanov (2016) proposed two magnetostratigraphic options for this section. The first is that the reported normal polarities from Ouberg Pass are correlative with a late Wordian magnetostratigraphy, with a documented normal subchron at ca. 269 Ma. The second is that the normal magnetozone are correlative with post-Kiaman reversals at ca. 265 Ma.

U-Pb SHRIMP (zircon) ages of ca. 290 Ma are reported from the Prince Albert Formation (lowermost Eccca Group; Fig. 15), while ages of ca. 262-268 Ma are reported from the lower Beaufort Group (above the Eccca Group; Fig. 15). A U-Pb SHRIMP (zircon) age of 270.4 ± 2.7 Ma has been reported (Fig. 15) from the middle Tierberg Formation (McKay et al., 2016), as well as an age of 274.8 ± 1.5 Ma from the Collingham Formation of the Laingsburg depocenter, 50 km to the southeast (Fildani et al., 2007; 20.75°E). The magnetostratigraphy and corresponding ages, therefore, support an age range of ca. 290-265 Ma for the deposition of the Eccca Group of the southern Karoo Basin. This age range is consistent with the accepted depositional history for the Karoo Basin, as well as the large body of published biostratigraphic data, and re-establishes the location of the geochronologically-constrained PTB of the Tanqua depocentre to within the overlying terrestrial sediments of the Beaufort Group (Fig. 15).

5.2 SHRIMP U-Pb geochronology

Samples OR72 and OR98 reported here from the Eccca Group are plotted against the corresponding sampling level for comparison with the magnetostratigraphy (Fig. 13). The U-Pb results from OR72 indicate that the system has likely been exposed to some level of Pb-loss (Fig 13). Similar to other published isotopic analyses from the Eccca Group, we isolated several late Permian-aged grains from each sample (Table 3). These grains formed a distinct sub-population in OR98, but were not excluded from the final age calculation in order to provide a minimum age constraint (Fig 13). The age reported here is ca. 10 Ma older than those previously reported from the Eccca Group (Fig. 2), and we interpret this as mainly resulting from the contrasting interpretations of complicated U-Pb datasets. (Fildani et al., 2007; Fildani et al., 2009; McKay et al., 2016; McKay et al., 2015). The former age calculations were based on the single youngest grain (Fildani et al., 2007; Fildani et al., 2009) or sub-population of youngest grains (McKay et al., 2016; McKay et al., 2015), whereas our analysis was based on the weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age of all isolated Permian grains (< 280 Ma) that met the specified quality criteria (section 3.3). We identified several inherited grains with ages of 1130 Ma, 880 Ma, 590 Ma, 450 Ma, and 380 Ma (Table 3). The grains selected for our age calculation showed no obvious signs of inheritance, and SHRIMP spots were taken on rims to avoid contamination from potential inherited cores (Fig. 5b).

Interestingly, the work by McKay et al. (2015; 2016) did not identify any ages older than ca. 280 Ma in the Eccca Group, and so early Permian-aged zircons were excluded from their age calculations by reasoning of assumed inheritance. This assumption was based on the hypothesis that the zircons from the overlying Beaufort Group (ca. 262-268 Ma) were sourced from the same volcanic center as the Choiyoi Group of South America, that they proposed to have experienced a period of Triassic zircon exhaustion and recycling of older inherited grains (McKay et al., 2015). However, recent Rare Earth Element (REE) data indicate that the Karoo Basin ash beds were more likely sourced from Antarctica, and do not

share a common magmatic history with the South American ash beds (McKay et al., 2016). Without clear documentation of the location of the magma chamber from which the ashes were generated, applying a hypothetical inheritance cut-off to the total analyses would skew the calculation towards a younger age.

Here we provide a more straightforward explanation for this apparent age inversion: that the zircon grains from ash beds in the middle Eccra Group have been affected by Pb-loss, the degree of which is difficult to assess at the resolution of the SHRIMP method for samples <500 Ma (Tohver et al., 2015). For example, for Phanerozoic samples, subsequent loss of radiogenic Pb will cause analyses to plot in an array between the magmatic age and the age of disturbance along a line subparallel to the Concordia curve, and with uncertainty ellipses still overlapping with Concordia, so the degree of Pb-loss is potentially impossible to resolve (Tohver et al., 2015). The reproducibility of consistently younger ages from the underlying Eccra Group and older ages from the overlying Beaufort Group, along the southern margin of the Karoo Basin, can be explained by an episode of Pb-loss. Our paleomagnetic analysis from the Tierberg and Skoorsteenberg Formations (OR1 core) shows a much larger degree of remagnetization with generally more unstable demagnetization behaviour than in the overlying Kookfontein and Waterford Formations, suggesting that the lower to middle Eccra Group was more heavily affected by this Pb-loss event than the upper Eccra Group, though this may also be a result of the drill string.

Some published illite crystallinity data from the middle Eccra Group support low-grade metamorphic temperatures of 170-200°C (Hälbich et al., 1983), which is within range of when fluid-mediated lattice damage initiates in zircon (Geisler et al., 2002; Geisler et al., 2007). However, we did not find any clear evidence for reabsorption or alteration of the oscillatory zoning in the zircons (Fig. 5ab), and the measured $^{232}\text{Th}/^{238}\text{U}$ ratios are not consistent with a lattice disturbed by metamorphism (Table 3). The CL and BSE images of

the analysed zircon grains (Fig. 5) do not show extensive U damage, metamorphism or any other feature that indicates zircon damage, so Pb-loss is a reasonable explanation to the spread of ages observed in the data collected. The Pb-loss hypothesis stems from the observation that the previously published SHRIMP ages from the Eccra Group are consistently too young when compared to the overlying Beaufort Group (Fildani et al., 2007; Fildani et al., 2009; McKay et al., 2016; McKay et al., 2015), and if their inaccuracy came from heterogeneous standards or other analytical errors, the age difference would be non-systematic.

As this Pb-loss event has only been observed so far in the Eccra Group, we conclude that this episode was most likely an isolated incident. However, another possibility is that this Pb-loss event was more extensive, and the preponderance of younger grains isolated from the Eccra Group could be explained by a lower proportion of co-eruptive zircon (McKay et al., 2015) than in the overlying Beaufort Group. Zircon has been shown to be present in variable concentrations in different tectonostratigraphic terranes (Moecher and Samson, 2006). This variability also has the potential to create an age bias in the study of magmatic systems (McKay et al., 2015). For example, the Beaufort Group may have been sourced by zircon-rich volcanic eruptions, while the Eccra Group was sourced by zircon-poor eruptions. In this scenario, the Beaufort Group would have been exposed to the same Pb-loss event; however, the abundance of available zircon grains would dilute these disturbed analyses in the final age calculation, since they would not fit within the main population (or sub-population), but represent outliers. Interestingly, Pb-loss was similarly detected in the Transantarctic Mountains of Antarctica, where a large number of Triassic grains were present in the interpreted Permian-aged beds (Elliot et al., 2017). As REE data seem to support a common magmatic origin for the Karoo Basin and Antarctic ash beds (McKay et al., 2016), further research in these areas is required. Regardless of a direct demonstration or mechanism for the

proposed Pb-loss, the magnetostratigraphy presented here provides an independent test against potentially disturbed U-Pb isotopic data, and confirms the Early Permian age for the Eccca Group.

5.3 *The Gondwana APWP*

In order to further constrain the relative timing of the interpreted primary and secondary magnetizations, the mean poles reported from Component B (overprint) and Component C (ChRM) are plotted alongside the recommended APWP of Torsvik et al. (2012) for reference (Fig. 16). The reported ChRM direction is consistent with the 270 Ma segment of the Gondwana APWP, and forms a critical angle (λ_c) of 13.6° with the reported ca. 270 Ma RM (Running Mean) Gondwana pole (no applied corrections; Torsvik et al., 2012). Component B is consistent with the 190 Ma segment ($\lambda_c = 8.3^\circ$) of the Gondwana APWP, as well as recent results reported from the Karoo LIP (Moulin et al., 2017). This suggests that this component represents a partial (low-temperature) Jurassic remagnetization of the Eccca Group during the initial stages of LIP emplacement. The eruption of the Karoo LIP likely resulted in the mobilization of heated fluids which could leach Pb, and recent paleothermal studies of the basin have indicated regional elevation of paleotemperatures of the sedimentary rocks of the Eccca Group to 200°C (Maré et al., 2016). This temperature interval is consistent with potential Pb-loss, which should not occur in zircons except at low temperatures (Schoene, 2014), such as during low temperature hydrothermal dissolution and reprecipitation (Geisler et al., 2002; Geisler et al., 2003).

Though Karoo Basin paleomagnetic directions were originally considered to be representative of a complete remagnetization (demagnetization to 350°C) due to a negative fold test in the Cape Fold Belt (Ballard et al., 1986), a number of paleomagnetic studies have since been published which resolved primary magnetization components suitable for

comparison with the GPTS (De Kock and Kirschvink, 2004a; Gastaldo et al., 2015; Lanci et al., 2013; Tohver et al., 2015; Ward et al., 2005). The Cape Orogeny (ca. 275-265 Ma), though significant in deformation in some areas, does not seem to have significantly remagnetized the Eccca Group strata, which in the present study location (Tanqua depocentre; Fig. 1) are almost flat-lying and unaltered. Paleomagnetic results from a stratigraphically overlying section at Ouberg Pass (Lanci et al., 2013) revealed magnetizations with several antipodal reversed and normal polarities that would be impossible to obtain if the remanence represented a blanket remagnetization acquired during KRS-aged Cape Fold Belt activity. However, if the magnetizations were reset during the KRS, then this would imply an even older age (>270 Ma) for the Eccca Group, consistent with the proposed hypothesis of Pb-loss and the main conclusions of this work. If the Cape Orogeny had caused the Eccca Group Pb-loss, then it had to have occurred directly after the deposition of the Eccca Group and before the deposition of the Beaufort Group. Hence it would be more or less coeval with sediment deposition. As the Eccca Group is outside the main area of Cape Fold Belt deformation (Fig. 1), we attribute the thermal overprint marker to the eruption of the Karoo LIP, which due to the vicinity of numerous dykes and sills in the field area, had the potential to make a more significant impact on resetting the magnetizations than distal Cape Fold Belt activity.

6. Conclusions

In an attempt to better resolve the Permian chronostratigraphy of the Karoo Basin, we have presented magnetostratigraphic and geochronologic data from a large part of the Eccca Group in the Tanqua depocentre. A uniformly-reverse polarity magnetostratigraphic profile of a composite ~1500 m section confirms that deposition occurred during the Kiaman Reverse Superchron (ca. 318 to 265 Ma), providing a minimum depositional age constraint of ca. 265 Ma for the upper Eccca Group. This age is not compatible with the

geochronologically-indicated PTB in the upper Skoorsteenberg Formation, but supports the existing biostratigraphic data in the overlying Beaufort Group. A U-Pb age of 269.5 ± 1.2 Ma is reported here from the uppermost Tierberg Formation, and is interpreted as a minimum age constraint due to a proposed Pb-loss event in the Eccra Group of the southern Karoo Basin.

A combination of published U-Pb ages from the overlying Abrahamskraal Formation (ca. 262 to 268 Ma), the Prince Albert Formation (ca. 290 Ma) and the Collingham Formation (ca. 275 Ma), compared with our magnetostratigraphic composite of the Tanqua depocentre, support an age range of ca. 290-265 Ma for the ~1.5 km thick Skoorsteenberg/Kookfontein/Waterford Formation section of the Eccra Group.

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Table 1. Paleomagnetic Results

Section	Component	S _{lat} (°S)	S _{long} (°E)	N(n)	P	D (°)	I (°)	α_{95}	k	P _{lat} (°N)	P _{long} (°E)	d _p /d _m
<i>SL1</i> (Waterford Fm.)	B	32.631	20.145	48	N	319.5	-64.5	7.7	8.08	-56.3	74.1	9.9/12.3
	C	32.631	20.145	63 (50)	R	122.9	65.5	10.0	5.05	-45.0	73.2	13.2/16.2
	**C	32.631	20.145	37	R	124.7	65.3	12.9	4.18	-46.2	73.6	16.9/20.9
<i>Pienaarsfontein</i> (Kookfontein Fm.)	A	32.780	19.993	34	N	3.1	-69.0	6.47	15.44	-70.2	14.4	9.3/11
	B	32.780	19.993	158	N	341.0	-60.8	2.50	21.16	-72.4	73.6	2.9/3.8
	C	32.780	19.993	163 (149.5)	R	116.0	62.1	4.22	8.47	-39.8	78.2	5.1/6.6
	**C	32.780	19.993	136	R	114.4	61.8	4.54	8.08	-38.6	78.4	5.4/7
Ecca Group (SL1 +Pienaarsfontein)	C	32.706	20.069	226 (199.5)	R	117.3	62.8	3.98	7.27	-40.8	77.4	4.9/6.2
	**C	32.706	20.069	173	R	116.2	62.5	4.46	6.76	-40.0	77.7	5.4/7.0

Notes: S_{lat} = site latitude, S_{lon} = site longitude, N = number of samples used for calculation of the mean direction, n = L + ½ p (L = lines; p = planes; McFadden and McElhinny, 1988), P = polarity, D = declination, I = inclination, α_{95} = radius of the cone of 95% confidence about the mean direction, k = precision parameter (Fisher, 1953), P_{lat} = pole latitude, P_{long} = pole longitude, d_p/d_m = semi-minor and semi-major axes of oval of 95 per cent confidence, ** = mean excluding arcs, bold = recommended paleomagnetic mean.

Table 2. AMS Results

Site	N	K_m	L	F	P	T	U	$K_{max}D$	$K_{max}I$	$K_{int}D$	$K_{int}I$	$K_{min}D$	$K_{min}I$
Pienaarsfontein	60	1.97e-4	1.005	1.027	1.033	0.669	0.665	126.1	19.0	216.8	2.0	312.6	70.9
SL1	27	1.50e-4	1.008	1.015	1.023	0.310	0.306	93.0	7.8	259.0	82.0	2.7	1.9
Ecca Mean*	87	1.83e-4	1.006	1.023	1.030	0.558	0.553	107.8	17.3	202.2	13.8	328.8	67.6

Notes: N = number of samples; K_m = average bulk volume susceptibility in SI units; L = Magnetic lineation: K_{max}/K_{int} ; F = foliation: K_{int}/K_{min} ; P = anisotropy degree: K_{max}/K_{int} ; T = Shape factor: $T=2\ln(K_{int}/K_{min})/\ln(K_{max}/K_{min})-1$; U = Difference shape factor: $(2K_{int}-K_{max}-K_{int})/(K_{max}-K_{int})$; K_{max} (K_{min}); Dec/Inc = Declination/Inclination. *Mean tensor directions were calculated from combined sample-level data. (Jelínek, 1981; Jelínek and Kropáček, 1978).

Table 3. U-Pb SHRIMP Results

Spot	% $^{206}\text{Pb}_c$	ppm U	ppm Th	^{232}Th / ^{238}U	$\pm\%$	^{207}Pb / ^{206}Pb	$\pm\%$	^{206}Pb / ^{238}U	$\pm\%$	(1) $^{206}\text{Pb}/^{238}\text{U}$ Age	
<i>Sample OR 98</i>											
D-8	0.32	613	787	1.33	0.16	0.0546	1.3	0.0590	3.6	265	± 2
D-7	0.31	223	118	0.55	0.31	0.0540	2.1	0.0652	1.3	266	± 2
D-3	0.07	867	675	0.80	0.62	0.0533	1.0	0.0775	2.9	266	± 2
D-10	0.27	497	278	0.58	0.49	0.0585	1.2	0.0681	2.9	268	± 3
D-4	0.03	814	972	1.23	0.28	0.0534	1.0	0.0806	2.0	268	± 2
D-5	0.55	425	312	0.76	0.30	0.0581	1.4	0.0738	2.5	268	± 3
D-13	1.21	636	696	1.13	0.45	0.0654	1.1	0.0676	5.0	269	± 3
D-6	0.93	377	330	0.90	0.74	0.0623	1.6	0.0558	6.4	270	± 2
D-14	0.07	401	254	0.65	0.22	0.0539	2.4	0.0770	3.4	270	± 2
D-11	0.74	574	579	1.04	0.16	0.0530	1.3	0.0727	3.0	271	± 4
D-15	0.28	892	667	0.77	0.27	0.0565	1.0	0.0554	4.2	271	± 2
D-1	0.29	518	383	0.76	0.81	0.0550	1.3	0.0717	3.2	271	± 2
D-12	0.45	668	802	1.24	0.43	0.0534	1.2	0.0706	1.7	272	± 2
D-9	0.60	449	510	1.17	1.12	0.0537	2.3	0.0739	2.8	272	± 3
<i>Rejected analyses</i>											
D-16	0.25	909	928	1.05	0.36	0.0543	1.0	0.0657	6.2	260	± 4
D-2	0.40	381	292	0.79	0.64	0.0560	1.5	0.0704	3.3	261	± 2
D-17	1.84	370	418	1.17	1.20	0.0730	1.3	0.0754	2.7	264	± 2
D-1	1.98	194	111	0.59	0.65	0.0678	3.8	0.0713	1.2	267	± 3
<i>Sample OR 72</i>											
OR72-18	0.55	359	176	0.51	0.26	0.0592	2.0	0.0636	1.3	258	± 3
OR72-9	0.00	451	248	0.57	0.67	0.0527	1.9	0.0635	0.8	259	± 3
OR72-13	0.07	500	271	0.56	0.22	0.0532	1.8	0.0713	1.9	260	± 5
OR72-7	0.08	430	211	0.51	0.23	0.0536	1.8	0.0700	1.8	262	± 3
OR72-17	0.27	438	177	0.42	0.25	0.0531	1.9	0.0629	2.0	263	± 3
OR72-2	0.09	403	229	0.59	0.70	0.0543	1.9	0.0667	1.7	263	± 4
OR72-5	0.31	612	408	0.69	1.18	0.0519	1.5	0.0721	1.9	263	± 4
OR72-6	0.06	627	407	0.67	0.28	0.0504	2.7	0.0671	1.1	263	± 3
OR72-1	0.14	695	530	0.79	0.16	0.0510	1.5	0.0709	0.9	265	± 4
OR72-3	0.28	384	529	1.42	0.92	0.0520	1.7	0.0796	0.7	265	± 4
OR72-15	0.21	484	253	0.54	0.62	0.0513	1.7	0.0723	1.2	266	± 4
OR72-4	0.00	414	265	0.66	0.53	0.0538	1.7	0.0768	1.5	269	± 3
OR72-14	0.23	458	188	0.42	0.24	0.0514	1.8	0.0723	2.0	270	± 4
OR72-11	0.19	380	183	0.50	0.68	0.0531	2.0	0.0678	0.8	276	± 3
<i>Inherited population</i>											
OR72-8	0.08	1394	361	0.27	0.65	0.0635	0.8	0.0858	3.0	359	± 4
OR72-10	0.03	576	272	0.49	0.19	0.0709	0.8	0.2330	1.6	768	± 9
<i>Rejected analyses</i>											
OR72-12	0.23	322	149	0.48	0.26	0.0530	2.2	0.0587	0.9	248	± 3
OR72-16	0.00	1070	756	0.73	0.24	0.0532	2.3	0.0714	2.0	264	± 3
<i>Sample OR 105</i>											
OR-15	0.91	134	102	0.79	0.81	0.0566	3.4	0.0632	1.4	251	± 4
OR-7	1.36	139	145	1.08	0.38	0.0667	3.0	0.0617	1.3	252	± 4
OR-12	0.31	362	471	1.34	1.14	0.0561	2.0	0.0679	2.2	253	± 3
OR-10	0.96	266	217	0.85	0.26	0.0595	2.3	0.0626	3.1	257	± 3

Table 3. U-Pb SHRIMP Results

Spot	% ²⁰⁶ Pb _c	ppm U	ppm Th	²³² Th / ²³⁸ U	±%	²⁰⁷ Pb / ²⁰⁶ Pb	±%	²⁰⁶ Pb / ²³⁸ U	±%	(1) ²⁰⁶ Pb/ ²³⁸ U Age	
OR-8	1.26	276	273	1.02	0.42	0.0582	2.3	0.0654	1.0	258	±4
OR-1	0.52	198	204	1.06	0.65	0.0571	2.6	0.0775	1.1	259	±6
OR-3	0.63	392	450	1.19	0.21	0.0549	2.1	0.0621	0.8	260	±4
OR-2	0.58	294	244	0.86	0.24	0.0543	2.2	0.0697	2.1	261	±4
OR-4	0.68	220	393	1.84	0.56	0.0588	3.8	0.0729	1.0	272	±5
OR-11	3.14	322	311	1.00	1.12	0.0762	4.4	0.0413	4.4	296	±5
OR-9	0.18	1399	365	0.27	1.11	0.0603	0.7	0.1271	1.1	425	±6
OR-13	0.08	501	548	1.13	0.35	0.0571	1.3	0.1254	1.2	445	±5
OR-14	0.16	680	107	0.16	0.48	0.0577	1.1	0.1175	2.6	450	±6
OR-6	0.64	290	324	1.16	0.38	0.0545	2.3	0.0726	2.2	259	±3
OR-5	0.53	420	309	0.76	0.23	0.0617	1.5	0.1006	0.7	444	±5
<i>Sample OR 107</i>											
C-7	0.60	1764	614	0.36	0.96	0.0595	0.7	0.0596	3.9	224	±2
C-9	0.19	1793	819	0.47	0.11	0.0556	0.7	0.0689	3.3	251	±2
C-4	0.08	365	64	0.18	0.33	0.0514	1.6	0.0762	1.7	271	±2
C-3	0.42	1393	78	0.06	0.93	0.0638	0.6	0.1139	6.2	380	±2
C-11	0.00	1059	558	0.54	0.35	0.0548	0.7	0.1209	1.8	389	±2
C-2	0.15	109	45	0.42	0.45	0.0579	2.0	0.1301	3.2	468	±5
C-6	0.37	298	83	0.29	0.31	0.0566	2.2	0.1403	2.6	470	±4
C-5	0.04	1012	141	0.14	1.16	0.0631	0.7	0.1104	4.3	478	±4
C-1	0.14	459	156	0.35	11	0.0617	0.8	0.1920	1.4	589	±11
C-8	0.11	527	206	0.40	0.21	0.0758	0.6	0.2570	2.9	884	±6
C-10	0.23	104	132	1.31	0.56	0.0802	1.1	0.3349	3.1	1136	±13

Notes: Errors are 1-sigma; Pb_c indicates the common Pb portions. Error in Standard calibration was 0.40% (not included in above errors but required when comparing data from different mounts) for OR72 and OR105 and 0.22% for OR98 and OR107. (1) Common Pb corrected using measured ²⁰⁴Pb. Bold = Samples with calculated ages (Fig. 13).

FIGURE CAPTIONS

Fig 1. a) Geological map of the Karoo Basin after Johnson et al. (1996). b) Regional inset of the sampling area located in the Tanqua depocentre. Sample sections from the present study are shown as red stars. For a general lithostratigraphic summary see Figure 2.

Fig. 2. Lithostratigraphy and published U-Pb ages for the Permian Eccca Group of the Tanqua depocentre (20°E) and the Permian-Triassic Beaufort Group vertebrate type section (~26°E). Biostratigraphy after Smith and Botha-Brink (2014) and Gastaldo et al. (2017). Note the large stratigraphic inconsistency between the reported biostratigraphy and U-Pb SHRIMP ages.

Fig. 3. The Geomagnetic Polarity Time Scale (GPTS) modified from Gradstein (2012) showing the extent of the Kiaman Reverse Superchron (KRS).

Fig. 4. Stratigraphic correlation between the two field sections SL1 and Pienaarsfontein spanning the Kookfontein and Waterford Formation using measured sections from Wild (2005). The top of Unit 5 is represented by a regionally extensive mudstone. Section location and sampling sites (GPS-tagged) shown by yellow lines on Google Earth® images. Sections are located approximately 20 km apart.

Fig. 5. Backscattered Electron (BSE) and Cathodoluminescence (CL) images of zircon grains from the lower Eccca Group (OR1 core). SHRIMP spots are shown by the black ovals. a) OR 72 (331.1 m); b) OR 98 (452.8 m).

Fig. 6. Representative Zijderveld demagnetization diagrams for the Waterford Formation. a-b) Reverse polarity samples from the Kookfontein Fm. (Lower Waterford) at Pienaarsfontein, with removal of the Jurassic overprint direction at 475°C; c) Reverse polarity sample from the Upper Waterford at SL1 showing the removal of two normal polarity overprint directions before isolation of the ChRM from 525-540°C; d) Normal polarity sample from the upper Kookfontein Fm. at Pienaarsfontein showing the removal of two overprint components before isolation of the ChRM at 580°C. Solid (open) squares represent projections on the horizontal (vertical) plane. For the accompanying sample equal area projections and intensity decay diagrams see Figs. 7 and 8.

Fig. 7. Representative equal area projections for the Waterford Formation. Up (down) pointing paleomagnetic directions are indicated by open (closed) squares. Numbers represent the thermal demagnetization steps in °C.

Fig. 8. Representative intensity decay diagrams for the Waterford Formation. Demagnetization curves are consistent with magnetite as the main magnetic phase.

Fig. 9. Magnetostratigraphy of the upper Eccca Group (Kookfontein and Waterford Formations) from the field sections SL1 and Pienaarsfontein. ChRM declination and inclination (polarity) data are plotted against sampling level, VGP latitude, lithology, and magnetic susceptibility measurements (SI units). The section is restricted to reverse polarity (white), with three potential normal subchrons (black). Both sections show intervals affected by lightning strikes and are shown as gray bars. See Figure 4 for sequence correlation. Measured sections from Wild (2005). Closed (open) squares represent directions isolated using lines (arcs).

Fig. 10. Equal area diagrams displaying the a) mean overprint directions (Components A and B) and the b) reverse ChRM direction (Component C) of the Lower Waterford (Kookfontein) Formation at Pienaarsfontein. Up (down) pointing paleomagnetic directions are indicated by open (closed) squares in the stereoplots. Arc-constraints for samples that do not reach stable endpoints were calculated with the method of McFadden and McElhinny (1988), and are shown with circle symbols. Red and blue ovals represent the cone of 95% confidence about the mean direction.

Fig. 11. Equal area diagrams displaying the a) mean overprint direction (Component B) and the b) reverse ChRM direction (Component C) of the upper Waterford Formation at SL1. Up (down) pointing paleomagnetic directions are indicated by open (closed) squares in the stereoplots. Arc-constraints for samples that do not reach stable endpoints are calculated with the method of McFadden and McElhinny (1988), and are shown with circle symbols. Red and blue ovals represent the cone of 95% confidence about the mean direction.

Fig. 12. Representative Zijdeveld and intensity decay diagrams for the Skoorsteenberg Formation from the OR1 core (vertical orientation data). The Jurassic overprint direction is removed by 400°C while the reverse polarity ChRM is isolated from 450-550°C. Demagnetization curves are indicative of magnetite as the main magnetic phase. Solid (open) squares represent projections on the horizontal (vertical) plane.

Fig. 13. Magnetostratigraphy of the Skoorsteenberg and Tierberg Formations from the OR1 core (see Fig. 1 for location). ChRM inclination (polarity) data are plotted against sampling level, U-Pb SHRIMP ages, lithology (Hodgson et al., 2011), and magnetic susceptibility measurements (SI units). Closed (open) squares represent directions isolated using lines (arcs). The section is restricted to reverse polarity (white), with one potential normal subchron (black). The age presented here is a weighted mean $^{206}\text{Pb}/^{238}\text{U}$ age. Corresponding Wetherill diagrams are shown only for reference. Pink arrows indicate the stratigraphic levels of the dated ash beds.

Fig. 14. Principal axes of magnetic susceptibility for the Eccia Group at a) Pienaarsfontein and b) SL1. Top: Equal-area plots of the directions of the principal axes of AMS with $K_1 = K_{\text{max}}$, $K_2 = K_{\text{int}}$, $K_3 = K_{\text{min}}$. Ovals represent the confidence ellipses for the mean direction. Bottom: Jelinek diagram displaying the general shape of the AMS ellipsoids with P = degree of anisotropy and T = shape factor.

Fig. 15. Lithostratigraphy, corresponding U-Pb ages, and magnetostratigraphic composite of the Permian Eccia Group and Lower Abrahamskraal Formation (Beaufort Group) of the Tanqua depocentre. GPTS* after Hounslow and Balabanov (2016) with geologic stage boundaries after Ogg et al. (2016) and lithology after Hodgson et al. (2011). Published ages shown in blue are incompatible with the reported magnetostratigraphy. *Ouberg Pass = Lanci et al. (2013).

Fig. 16. Components B (overprint) and C (ChRM) plotted alongside the recommended Gondwana APWP of Torsvik et al. (2012) with an orthogonal projection centered at 40°S, 60°E. Blue = present study; Green = Paleomagnetic results from the Karoo LIP at ca.183 Ma (Moulin et al. 2017).

Figure 1

Figure 2

Figure 3

Period	Series	Stage	Magnetic Polarity		
Triassic	Middle	Ladinian	Illawara Mixed		
		Anisian			
Permian	Early	Olenekian			
	Lopingian	Induan			
		Changhsingian			
		Wuchiapingian			
	Guadalupian	Capitanian			
		Wordian			
		Roadian			
	Cisuralian	Kungurian			
		Artinskian			
		Sakmarian			
Asselian					
Gzhelian					
Carboniferous	Pennsylvanian	Kasimovian	Kiaman Reverse Superchron		
		Moscovian			
		Bashkirian			
	Mississippian	Serpukhovian		Carboniferous Mixed	
		Visean			
	Early	Tournaisian			Carboniferous Mixed
	359.2				
	299.0				
251.9					

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

Figure 13

Figure 14

Figure 15

Figure 16