

U-Pb dating of pedogenic calcite near the Permian–Triassic boundary, Karoo Basin, South Africa

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ABSTRACT

We report U-Pb age determinations of carbonate nodules from an in situ paleosol horizon in the Upper Permian Balfour Formation and from several horizons of pedogenic nodule conglomerate (PNC) in the Triassic Katberg Formation, Karoo Basin, South Africa, using laser ablation–inductively coupled plasma–mass spectrometry (LA-ICP-MS). The paleosol sample yields an age of 252 ± 3 Ma, which overlaps with a previous high-precision U-Pb zircon date from a volcanic ash deposit 2 m above the paleosol. This relationship demonstrates the reliability of using LA-ICP-MS dating techniques on terrestrial pedogenic calcite. Two PNC samples collected at the base of the Katberg Formation within the same sandstone unit yield ages of 255 ± 3 Ma and 251 ± 3 Ma. The age of 251 ± 3 Ma overlaps with the high-precision U-Pb zircon date below the PNC and is a maximum age estimate of deposition for the base of the Katberg Formation. Our results show that reworked nodules in the same concentrated conglomerate lag can be of different ages, but that similarly aged nodules are spatially associated. In addition, two PNC samples collected higher in the section yield ages of 249 ± 3 Ma and 241 ± 3 Ma, providing maximum depositional ages for the lower to middle Katberg Formation for the first time. We demonstrate that pedogenic carbonate nodules can be dated with meaning-

ful precision, providing another mechanism for constraining the age of sedimentary sequences and studying events associated with the Permian–Triassic transition in the central Karoo Basin, even though the extinction boundary may not be preserved in this area.

INTRODUCTION

The end-Permian crisis represents the largest known mass extinction in Earth's history. It is reported to have affected ~90% of marine invertebrate species and is marked by the demise of conodonts, micro-gastropods, fusulinids, various brachiopods, echinoderms, molluscan clades, and other taxa (Erwin, 1994; Ogg, 2012; Shen et al., 2019; Dal Corso et al., 2022). The marine extinction event is recorded by high-resolution conodont zonation in the Permian–Triassic Global Boundary Stratotype Section and Point (GSSP) at Meishan, China, which is mainly defined by the first appearance datum of the conodont *Hindeodus parvus* (Chen et al., 2015). Here, high-precision U-Pb geochronology on zircons from several ash deposits indicates that the end-Permian extinction event occurred between 251.941 ± 0.037 Ma and 251.880 ± 0.031 Ma, which represents an interval of 60 ± 48 ka (Burgess et al., 2014). A similar age for the marine event of 251.939 ± 0.031 Ma was found at Penglaitan, South China (Shen et al., 2019).

Several hypotheses propose that climate change due to increased atmospheric pCO₂ from intense volcanism associated with the emplacement of the Siberian Traps was the main cause of biodiversity loss (Knoll et al., 1996; Brand et al., 2012; Burgess and Bowring, 2015; Burgess et al., 2017; Wu et al., 2021b). Other

proposed volcanism-related proximal causes include metal poisoning (Chu et al., 2021) and ozone depletion (Visscher et al., 2004; Black et al., 2014; Benca et al., 2018). The Siberian Traps, a large igneous province (LIP), is derived from one of the most voluminous and explosive volcanic series of events known in the Phanerozoic. It comprises a composite ~6500-m-thick sequence that was deposited over a period of less than 1 Ma (Kamo et al., 2003). U-Pb chemical abrasion–isotope dilution–thermal ionization mass spectrometric (CA-ID-TIMS) zircon ages indicate that magmatic activity at the Siberian Traps began between 252.47 ± 0.14 Ma (Davydov et al., 2021) and 252.24 ± 0.12 Ma (Burgess and Bowring, 2015), while the end of magmatic activity is estimated at 250.2 ± 0.3 Ma (Kamo et al., 2003). The timing and duration of Siberian magmatism are consistent with events of the marine record.

Several authors have proposed that terrestrial ecosystems were affected at the same time as the marine crisis and experienced a similar degree of biodiversity loss (Twitchett et al., 2001; Ward et al., 2005; Viglietti et al., 2021; Wu et al., 2021a). The response of terrestrial ecosystems, the extent to which continental floras and faunas were affected, and the timing of that response continue to be debated (Davydov et al., 2021; Nowak et al., 2019; Vajda et al., 2020; Aftabuzaman et al., 2021; Gastaldo et al., 2021; Lucas, 2021; Yang et al., 2021; Tabor et al., 2022). Although the marine Permian–Triassic boundary is well established, numerical age data that would help constrain the effects in and around the terrestrial Permian–Triassic boundary are limited and localized in just a few basins. The relative dearth of datable minerals in continental

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sedimentary rocks, especially zircon from air-fall ash deposits, as well as the fact that continental successions are notoriously incomplete and difficult to correlate, hamper these attempts.

There are several continental basins across the planet for which geochronometric ages constrain the latest Permian and earliest Triassic successions that surround the end-Permian crisis. The Karoo Basin, South Africa, is considered to contain one of the most important continental sequences spanning the Permian–Triassic boundary and served as an early model for the terrestrial extinction event. This is due to its extensive and well-preserved paleontologic record of vertebrates (Ward et al., 2005; Viglietti et al., 2016, 2021; Gastaldo et al., 2019) and, to a much lesser extent, fossil plants (Prevec et al., 2010; Gastaldo et al., 2019) and pollen-and-spore records (Barbolini et al., 2018; Gastaldo et al., 2020a). Here, geochronometric age constraints have demonstrated that diagnostic taxa previously used to define the post-extinction vertebrate fauna (Viglietti et al., 2021), and considered to be Early Triassic (Botha et al., 2020; Smith et al., 2022), are preserved in latest Changhsingian strata at 252.24 ± 0.11 Ma (Gastaldo et al., 2020a). More recently, these same taxa were demonstrated to have coexisted across the same late Changhsingian landscapes as taxa diagnostic of the pre-extinction fauna, all of which thrived in vegetation dominated by the gymnosperm *Glossopteris* (Gastaldo et al., 2021). In recent years, the response of terrestrial ecosystems in Sydney Basin, Australia, has superseded the Karoo Basin as a model for this extinction event in Gondwana. The collapse of the *Glossopteris*-dominated plant communities in eastern Australia appears to have started earlier than any perturbation in the marine realm (Fielding et al., 2019; Mays et al., 2021), beginning at ca. 252.31 ± 0.07 Ma (latest Changhsingian; Mays et al., 2020), with ecosystem disturbance in this part of the southern hemisphere extending into the Olenekian (Fielding et al., 2021; Mays et al., 2021).

The pattern of vegetational change occurring hundreds of thousands of years before the marine extinction event is not repeated at other paleolatitudinal positions, but there is ample evidence of several perturbations in the floral realms starting in the Late Permian up to the Middle Triassic (Hermann et al., 2011). For example, collapse of terrestrial and marine ecosystems in Greenland occurred synchronously, but the extinction of characteristic Permian plant taxa is phased (Looy et al., 2001; Twitchett et al., 2001). In the fully continental record outcropping in the North China Block, Bogda Mountains, China (Yang et al., 2021), vegetational turnover occurred during the early Induan (ear-

liest Triassic, 251.9–251.2 Ma), which postdates the marine event. Evidence for a continuous record of riparian vegetation and upland forests occurs in this rift-basin setting into the early Olenekian (251.2–247.2 Ma). Similarly, the fully continental succession in the Kuznetsk Basin, Russia, shows no evidence of an end-Permian vegetational extinction. Rather, this basin documents the extirpation of a humid-dominated cordaitean flora at ca. 820 ka prior to the marine event, with subsequent floristic diversification across the Permian–Triassic transition (Davydov et al., 2021).

Only precise geochronology makes it possible to understand the patterns preserved in the stratigraphic record, and high-precision numerical age data, thus far, have depended on the presence of zircon in ash-fall deposits. Deposition of such material requires nearby volcanism and, once deposited, the probability for preservation of these mostly fine-grained fall deposits is low due to continual reworking (e.g., phyto- and bioturbation in soils and ongoing fluvial and erosional processes), which is common in terrestrial systems (Rasmussen et al., 2021). Unlike ash-fall deposits, carbonate material is abundant in both marine and continental depositional environments, precipitating either as primary and/or secondary mineral phases, and is preserved throughout an extensive geologic record. On land, pedogenic (soil-precipitated) carbonate minerals form mostly in regions of seasonally dry climate (Railsback, 2021), but they are also reported from humid environments (Sheldon and Tabor, 2009) and, since the early Cenozoic, from grasslands (Retallack et al., 2002). Pedogenic carbonates have been used to reconstruct deep-time paleoclimate. Using geochemical properties, including stable carbon and oxygen isotopes (Leone et al., 2000; Retallack, 2005), it is possible to determine whether a paleosol developed under seasonally wet-and-cool or warm-and-dry conditions (Tabor et al., 2022; Sheldon and Tabor, 2009; Gastaldo et al., 2020b). In addition, it is possible to derive estimates of atmospheric $p\text{CO}_2$ at the time of calcite precipitation from calcite-cemented soil nodules (Gastaldo et al., 2014; Joachimski et al., 2022).

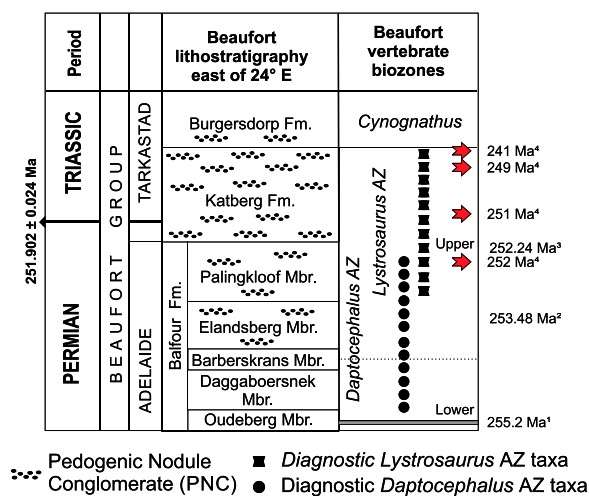
Calcite-bearing paleosols incorporate uranium at the time of mineral precipitation (Jahn and Cuvelier, 1994; Rasbury and Cole, 2009). Therefore, a U–Pb age can be calculated for the time of soil formation provided that the ratios of U to initial common Pb concentrations are high enough and the carbonate was not affected by late diagenetic alteration. Secondary calcite normally precipitates with a low-U content and a significant common Pb component, making it a challenge to obtain accurate and precise U–Pb ages. However, recent advances in laser abla-

tion–inductively coupled plasma–mass spectrometry (LA–ICP–MS) and Bayesian statistical analysis extend the range of samples that can be usefully dated (Roberts et al., 2020; Davis and Rochín-Bañaga, 2021). We present results of the application of these techniques to pedogenic calcite nodules obtained from one paleosol and several pedogenic nodule conglomerates (PNC) from the Balfour and Katberg formations. The paleosol interval has been inferred to straddle the Permian–Triassic extinction boundary in the Karoo Basin, South Africa. Currently, our understanding of the timing and duration of this critical interval in this basin has been limited by the dearth of volcanic deposits. However, U–Pb LA–ICP–MS dating of pedogenic calcite has the potential to provide direct timing of the events surrounding the Permian–Triassic extinction, even when this boundary may not be preserved in the central part of the Karoo Basin.

GEOLOGIC BACKGROUND

The Karoo Basin is considered to represent a large subsiding retroarc foreland basin (Caturanu et al., 2005; Smith et al., 2020) that contains a succession of upper Paleozoic deglacial sedimentary rocks overlain mainly by fully continental deposits known as the Karoo Supergroup. Sedimentary rocks resulting from deglaciation are found in the basal Dwyka (upper Carboniferous) and Eccra (Lower–middle Permian) groups, with a transition to fully continental deposits in the Beaufort (Permian–Triassic) and Stormberg groups (Triassic–Jurassic). The Beaufort Group is subdivided into two subgroups: the lower Adelaide and upper Tarkastad. The depositional setting of the Balfour Formation (Fig. 1), the uppermost unit of the Adelaide subgroup that is postulated to span the Permian–Triassic transition, is interpreted as fluvial-lacustrine with intervening paleosols having various weathering characteristics (Gastaldo et al., 2014, 2020b). Paleosols from the uppermost exposures of this unit formed under conditions that fluctuated from relatively humid (Ward et al., 2000) and cool to seasonally dry and warm (Gastaldo et al., 2020c).

The overlying Tarkastad subgroup is subdivided into the arenaceous Katberg and argillaceous Burgersdorp formations. The Katberg Formation is traditionally interpreted to represent fluvial systems deposited under more seasonally dry to semiarid conditions (e.g., Ward et al., 2000; Pace et al., 2009). It consists of a monotonous succession of fine- to very fine-grained feldspathic wacke fining upward into mottled mudrock, and has been the focus of different paleontologic studies because of its extensive fossil record. Therapsid fossils, including *Lystrosaurus murrayi* and *L. declivis*, are abun-



the Permian–Triassic Boundary in the Katberg Formation after Gastaldo et al. (2020a, 2015, 2017); and (4) age constraints for the Katberg Formation as exposed on Farm Nooitgedacht presented herein. The uncertainty surrounding whether a boundary exists between the traditional lower and upper *Daptocephalus* AZ (Viglietti et al., 2016) and an overlying *Lystrosaurus declivis* AZ (Botha and Smith, 2020) follows Gastaldo et al. (2021).

dant and well preserved throughout the formation (Smith and Botha-Brink, 2014; Smith et al., 2022). These fossils were previously used as diagnostic taxa to delimit the *L. declivis* Assemblage Zone (AZ; Botha and Smith, 2020) and, in part, the Katberg Formation itself. Recently, it was demonstrated empirically that diagnostic taxa reported to have their first appearance datum in the Triassic coexisted with the diagnostic taxa of the underlying *Daptocephalus* AZ in glossopterid-dominated latest Permian landscapes (Gastaldo et al., 2021). Based on a combination of geochronologic, magnetic polarity stratigraphic, and palynological evidence, the Permian–Triassic transition has been placed in the Katberg Formation (Gastaldo et al., 2020a, 2021).

MATERIALS AND METHODS

Locality and Samples

The sampling locality is located ~20 km north-northwest of the town of Bethulie in the southern Free State Province (S 30.32530°, E 025.93132°), South Africa, on Farm Nooitgedacht 68 (Fig. 2). Fieldwork focused on the koppie (butte) Loskop (Fig. 3A), which rises ~100 m above a plain with its talus-covered flanks consisting of horizontal beds of blocky fine–very fine feldspathic wacke, weathered and friable mudrock varying in color from greenish-gray (5GY 6/1) to grayish-brown (5 YR 4/2), along with scattered colluvium, including pedogenic nodules and vertebrate bone fragments. Measured sections are facilitated by a series of

erosional scars (dongas) with typically excellent exposure (Gastaldo et al., 2020a), and resistant sandstone bodies that traverse the koppie and are used to constrain depositional units (Fig. 3B). Lenses of PNC lag deposits appear sporadically

Figure 1. Generalized stratigraphy of the Karoo Basin with a focus on the Upper Permian to Middle Triassic succession. Geochronometric age constraints for stage boundaries follow the International Chronostratigraphic Chart v2022/10 (www.stratigraphy.org) with (1) the base of the *Daptocephalus* Assemblage Zone (AZ) after Rubidge et al. (2013); (2) the age constraint on the Elandsberg Member (Mbr.) after Gastaldo et al. (2015, 2018); (3) the age on the upper Palingkloof Member after Gastaldo et al. (2020a); the placement of

at the bases of channel sandstone bodies exposed in the upper part of the koppie (Fig. 3B). These lag deposits mark erosional basal unconformities in the succession, with the calcite-cemented pedogenic nodules representing residuum from landscape degradational processes (Gastaldo and Demko, 2011). Lags, which also contain abundant bone fragments, have concentrated these recalcitrant soil nodules from eroded paleosols, including vertisols (Gastaldo et al., 2020b, 2020c). All PNC-bearing sandstones exposed across the Loskop and Spitskop koppies (Fig. 2B) are assigned to the Katberg Formation (Botha-Brink et al., 2014, 2020; Gastaldo et al., 2020a).

Five PNC samples and one paleosol were collected and analyzed in this study (Fig. 4). The paleosol sample (NGT9-6) was collected in situ from a red siltstone unit ~2–3 m below the stratigraphic position from which the 252.24 ± 0.11 Ma zircon age is reported (Fig. 3C; Gastaldo et al., 2020a; their fig. 2).

Two samples, NGT9-7 and NGT9-8, originate from the lowest PNC-bearing sandstone bed in the section, which is ~7 m above the thin ash-fall deposit yielding the 252.24 ± 0.11 Ma zircon age (Gastaldo et al., 2020a). NGT9-7 and NGT9-8 were collected in situ, and each sample comes from a different part of the same lag

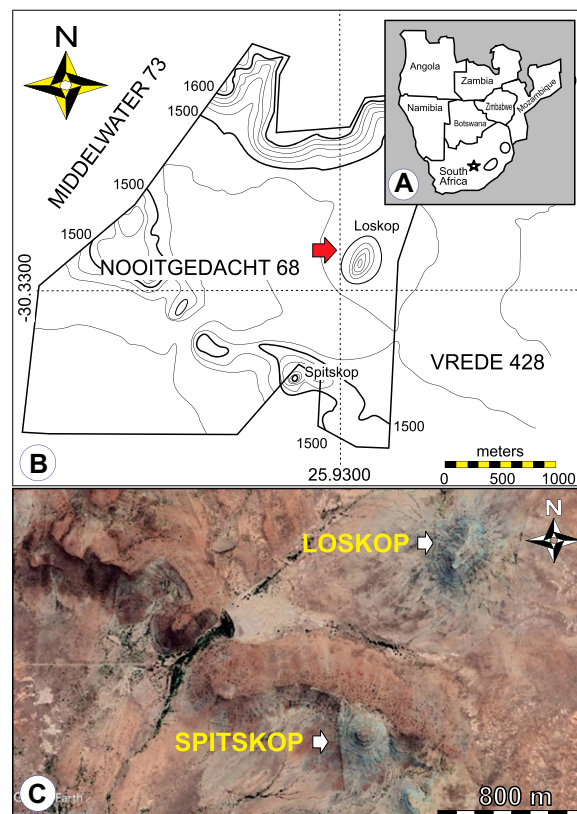


Figure 2. Location of Farm Nooitgedacht, Free State Province, South Africa, where pedogenic nodules were sampled from intraformational conglomerate deposits occurring as fluvial lags in the Katberg Formation. (A) Map of southern Africa; a star indicates the location of the farm. (B) Contour map of Farm Nooitgedacht (S 30.32530°, E 025.93132°; after Gastaldo et al., 2020a) showing exposed uppermost Permian and Lower Triassic intraformational conglomerate, sandstone, and mudstone on two small buttes (koppie). Devitrified claystone (ash-fall deposit) from which a concordant U-Pb chemical abrasion–isotope dilution–thermal ionization mass spectrometric (CA-ID-TIMS) single crystal zircon age of 252.24 ± 0.11 Ma was obtained crops out on Loskop. Pedogenic nodule conglomerate was collected from Loskop (red arrow). (C) Google Earth image of Farm Nooitgedacht. Scales are in meters.

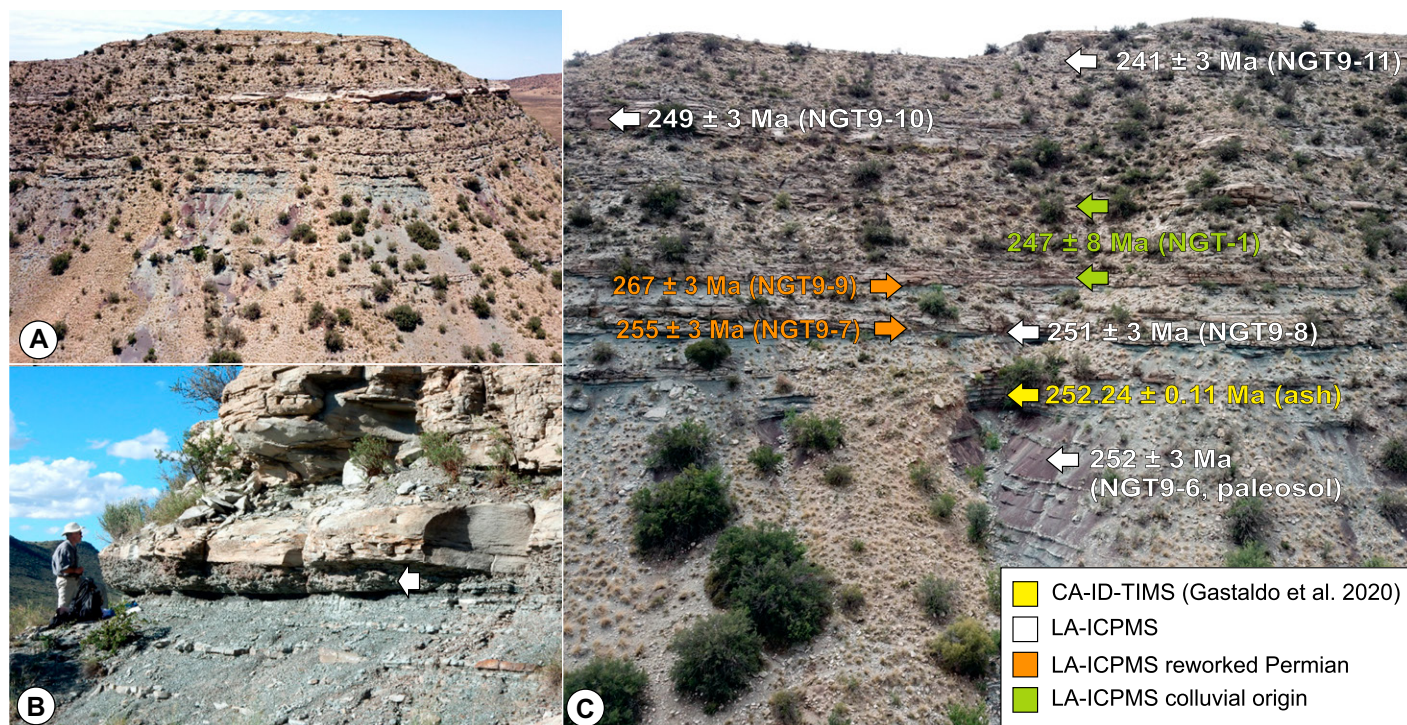


Figure 3. Loskop koppie on Farm Nooitgedacht. (A) Drone image of western exposure where intraformational pedogenic nodule conglomerate (PNC) occurs as intermittent lag deposits at the base of sandstone bodies. Here, the Katberg Formation is composed of thin sheet sandstones with one thick channel-form body attaining a thickness of <8 m. (B) Image of lowest exposed PNC lag deposit (samples NGT9-7 and NGT9-8) positioned ~1 m stratigraphically above the devitrified volcanogenic ash-fall deposit from which a concordant U-Pb chemical abrasion–isotope dilution–thermal ionization mass spectrometric (CA-ID-TIMS) age of 252.24 ± 0.11 Ma has been reported (Gastaldo et al., 2020a). (C) Stratigraphic positions and laser ablation–inductively coupled plasma–mass spectrometric (LA-ICP-MS) age estimates of calcite nodules from a paleosol and PNC lag deposits.

accumulation at the base of this channel body; hence, these are lateral equivalents (Fig. 3C). In contrast, NGT-1 was collected from colluvium at the base of the Loskop cliff face below where the thickest sandstone body is exposed (Fig. 3C). Calcitic nodules in these samples range from ~2 mm to 6 mm in diameter, vary in shape from rounded to subrounded, and are enclosed in a fine-grained sand matrix in which fragmented bone, rip-up mudclasts, and mud aggregates also occur (Pace et al., 2009; Gastaldo et al., 2013). The PNC sample NGT9-9 was collected from a 1.5-m-thick greenish-gray siltstone, whereas samples NGT9-10 and NGT9-11 were collected higher, from PNC lenses bounding fine-grained sandstone benches (Fig. 3C; Table 1).

Analytical Methods

U-Pb isotopic analyses were conducted at the University of Toronto using an Agilent 7900 ICPMS and an NWR193 excimer laser system. U-Pb data (^{238}U , ^{206}Pb , and ^{207}Pb) were collected using scan-lines over individual nodules resulting in hundreds of cycle data per line (Drost et al., 2018; Hoareau et al., 2021; Davis and

Rochín-Bañaga, 2021). ^{230}Th was also monitored to check for detrital contamination but was found to be uniformly low.

Targeted nodules show massive matrices and were selected randomly. Each scan-line analysis, 4–5 mm long, was pre-ablated at a fast scan rate, 200 $\mu\text{m/s}$, and high frequency, 20 Hz, using a larger diameter beam (150 μm) to remove surface contamination. U-Pb line analyses were conducted with a fluence of ~ 4.5 J/cm², beam diameter of 120 μm , and frequency of 10 Hz at a rate of 15 $\mu\text{m/s}$. Baselines were accumulated on each scan-line for 20 s prior to opening the laser. The depth of the scan-line is ~ 10 –20 μm . LA-ICP-MS parameters for U-Pb analyses are provided in Table S1 in the Supplemental Material¹. U decay constants are from Jaffey et al.

¹Supplemental Material. The supplemental material contains the LA-ICP-MS parameters for U-Pb analyses, the Tera-Wasserburg concordia diagrams for the calcite standards, and the Utilchron Excel files used for data processing and age regressions from individual samples. Please visit <https://doi.org/10.1130/GSAB.S.23638722> to access the supplemental material, and contact editing@geosociety.org with any questions.

(1971), and the $^{238}\text{U}/^{235}\text{U}$ ratio is taken as 137.88 (Steiger and Jäger, 1977).

Both the glass standard NIST612 and Duff Brown Tank were used to correct for mass and oxide elemental fractionation bias. The uncertainty of our U-Pb ages includes measurement errors in mass signals from the sample NIST612, and Duff Brown Tank calcite-standard measurements. The Duff Brown Tank standard was used as a matrix-matched reference. Ages have been corrected by an ablation bias factor of 1.03 based on comparison of the measured age of Duff Brown Tank of 62 ± 1 Ma (mean square of weighted deviates [MSWD] = 0.68; Y intercept fixed at 0.738) with its ID-TIMS age of 64.04 ± 0.67 Ma (Hill et al., 2016). An additional calcite standard (Walnut Canyon standard, WC-1) with an ID-TIMS age of 254.4 ± 6.4 Ma (Roberts et al., 2017) was measured to provide control on accuracy and precision. The age of WC-1 was measured at 254 ± 3 Ma (2σ , MSWD = 0.68) with a fixed $^{207}\text{Pb}/^{206}\text{Pb}$ ratio of 0.85, after correction using the factor of 1.03. Tera-Wasserburg concordia diagrams from the Duff Brown Tank and WC-1 standards can be found in the Supplemental Material.

Data Processing

Excel data sheets with results, errors, and software for regressions, as well as instructions for repeating calculations, are given in the Supplemental Material. Calcite minerals usually contain significant common as well as radiogenic Pb. Hence, age determinations are based on defining a mixing line in isotope-ratio space whose intersection with the concordia (curve showing radioactive equilibrium at a given age) shows the age of the radiogenic component while its Y ($^{207}\text{Pb}/^{206}\text{Pb}$)-axis intercept on the Tera-Wasserburg plot gives the isotopic composition of the initial common Pb component (denoted PbC). Mixing lines are defined using a data set, or multiple data sets, from samples similar in age and PbC. Data were processed as single measurement cycles to exploit the maximum U/Pb spread along the mixing line.

U-Pb regression was carried out using a Bayesian statistical program (Utlilchron) that regresses mass-count data with 1-sigma absolute errors ($1/\sqrt{N}$, where N is the total count number) in a 3-D signal space (Davis and Rochín-Bañaga, 2021). Details of data processing and regressions are given for individual samples in Utlilchron Excel files in the Supplemental Material data. These files contain detailed instructions for use. U-Pb data are also regressed using conventional least-squares regression of ratios (Isoplot; Ludwig, 2012) for comparison purposes and to determine the MSWD. U-Pb data are plotted and regressed using Tera-Wasserburg (inverse) concordia diagrams, where (1) the age represents the intersection of the best-fit line with the concordia curve and (2) PbC is given by its Y-axis intercept. Data sets consist of hundreds of measurements; as such, error ellipses are not shown but are taken into account in the calculations. Errors on age and initial $^{207}\text{Pb}/^{206}\text{Pb}$ are quoted at 95% confidence (2 sigma). Tera-Wasserburg (inverse) concordia diagrams from the pedogenic nodules are found in Figures 5 and 6.

RESULTS AND DISCUSSION

U-Pb LA-ICP-MS data from the paleosol pedogenic nodules and pedogenic nodule conglomerate lags are presented in Table 1 and Figures 5 and 6. The paleosol sample shows U and Pb content of 22 ppm and 13 ppm, respectively. The U content in nodules collected in channel lag deposits varies from 19 ppm to 63 ppm, with $^{206}\text{Pb}/^{238}\text{U}$ in the range of 0.13 and 0.52. The paleosol and PNC nodules have low-Th/U values due to the insolubility of Th in aqueous solutions, which should also be of low concentration in diagenetic fluids.



Figure 4. Calcite nodule samples from the pedogenic nodule conglomerate at Farm Nooitgedacht, Karoo Basin. Scale bar is 1 cm. White arrows indicate the nodules selected for U-Pb laser ablation-inductively coupled plasma-mass spectrometric (LA-ICP-MS) dating.

TABLE 1. LASER ABLATION-INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRIC U-Pb AGE RESULTS FROM PEDOGENIC NODULE SAMPLES FROM THE KATBERG FORMATION, FARM NOOITGEDACHT, FREE STATE PROVINCE, SOUTH AFRICA

Sample	Samples coordinates	^{238}U (ppm)	^{206}Pb (ppm)	Th/U	Age (Ma)	MSWD	$^{207}\text{Pb}/^{206}\text{Pb}$ (PbC)
NGT9-6	S 30.32603°, E 26.93248°	22.0	13.0	0.2	252 ± 2.8	2.4	0.832 ± 0.007
NGT9-7	S 30.32530°, E 25.93132°	Weighted mean age =			254 ± 2.9	0.6	
Nodule 1		30.2	5.5	0.1	253 ± 7	1.7	0.833 ± 0.013
Nodule 2		20.4	10.7	0.2	258 ± 6	1.2	0.844 ± 0.008
Nodule 3		43.6	8.0	0.1	253 ± 7	1.2	0.830 ± 0.012
Nodule 4		43.1	6.2	0.1	254 ± 5	2.4	0.838 ± 0.004
NGT9-8	S 30.32530°, E 25.93132°	Weighted mean age =			251 ± 2.6	0.5	
Nodule 1		42.3	8.7	0.1	250 ± 7	1.3	0.839 ± 0.012
Nodule 2		33.4	6.9	0.1	248 ± 7	1.3	0.820 ± 0.008
Nodule 3		62.9	11.7	0.2	252 ± 5	1.2	0.833 ± 0.013
Nodule 4		20.8	5.7	0.1	252 ± 4	1.3	0.840 ± 0.009
NGT-1							
Nodule 1		19.0	4.7	0.1	268 ± 5	2.0	0.809 ± 0.007
Nodule 2		43.9	5.7	0.0	247 ± 8	1.1	0.805 ± 0.008
Nodule 3		23.0	5.9	0.2	268 ± 12	1.3	0.849 ± 0.009
NGT9-9	S 30.32569°, E 25.93307°	30.0	9.1	0.1	267 ± 2.6	1.7	0.834 ± 0.007
NGT9-10	S 30.32592°, E 25.93287°	29.0	4.3	0.1	249 ± 2.9	1.0	0.828 ± 0.008
NGT9-11	S 30.32611°, E 25.93314°	18.1	8.3	0.2	241 ± 3.3	2.0	0.833 ± 0.007

Note: MSWD—mean square of weighted deviates; PbC— $^{207}\text{Pb}/^{206}\text{Pb}$, isotopic composition of the initial common Pb component.

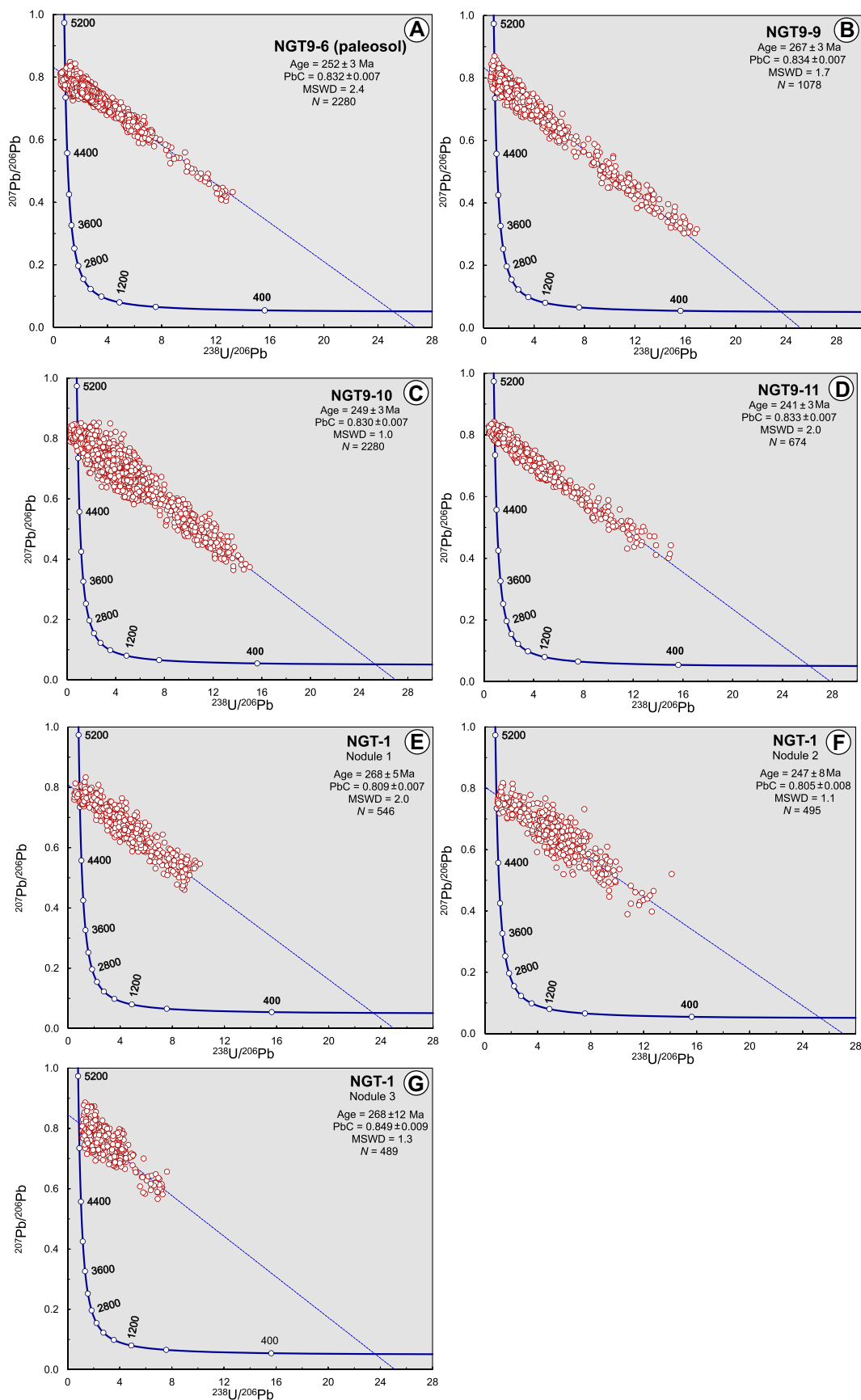


Figure 5. Tera-Wasserburg concordia diagrams from the paleosol (NGT9-6) and pedogenic nodule conglomerate (PNC) samples (NGT9-9, NGT9-10, NGT9-11, and NGT-1) as well as age results and mean age of nodules collected from two locations within the same PNC lag deposit (NGT9-7 and NGT9-8). MSWD—mean square of weighted deviates.

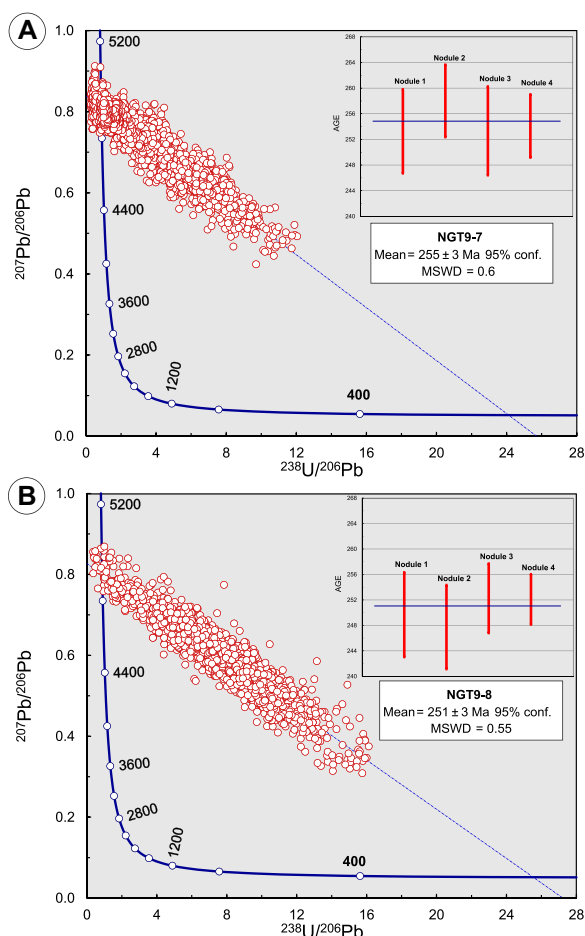


Figure 6. Tera-Wasserburg concordia diagrams and mean age of nodules collected from two locations within the same pedogenic nodule conglomerate (PNC) lag deposit (NGT9-7 and NGT9-8). MSWD—mean square of weighted deviates.

The NGT9-6 paleosol sample yields an age of 252 ± 3 Ma with a PbC of 0.832 (Fig. 5A). Individual nodules from PNC sample NGT9-7 yield overlapping ages of 253 ± 7 Ma, 258 ± 6 Ma, 253 ± 7 Ma, and 254 ± 5 Ma (Fig. 6A). The nodules from NGT9-8 within the same lag also yield overlapping ages of 250 ± 7 Ma, 248 ± 7 Ma, 252 ± 5 Ma, and 252 ± 4 Ma (Fig. 6B). All nodules show overlapping PbC values ranging from 0.820 ± 0.008 to 0.844 ± 0.008 . These values are approximately consistent with the average crustal model-based common Pb for this age (~ 0.85 ; Stacey and Kramers, 1975). The weighted mean $^{238}\text{U}/^{206}\text{Pb}$ age and 95% confidence errors for NGT9-7 are 255 ± 3 Ma (MSWD = 0.6), whereas those for NGT9-8 are 251 ± 3 Ma (95% confidence, MSWD = 0.5). These two average ages do not agree within error (MSWD = -3.1). Although the eight individual measurements yield an average with a much lower MSWD of 0.9, ages from NGT9-7 are consistently older than those from NGT9-8, and data sets from NGT9-7 are also consistently less radiogenic (Fig. 6). This suggests that the nodules from each sample are from different sources with slightly different

ages. Since the nodules are all detrital, it is possible that even those from the same sample are different.

A suggestion that this may be so is that regressing all four data sets from NGT9-8 gives a best-fit age of 246 ± 3 Ma, which is notably younger than the average of the four ages. The hypothesis used in regression is that all data scatter around a line defined by a single age and PbC value. Otherwise, results may be biased. For example, suppose that one were averaging two sets of data from samples having the same age but different PbC values. If one set is less radiogenic than the other and has a lower PbC value, then the average isochron through the more radiogenic data will be biased toward a lower Y intercept, which will cause the age to appear too young. Therefore, one should not combine data sets from different samples unless there is good evidence that they are uniform in age and PbC. It is better instead to average the ages from the separate regressions. The average ages from NGT9-7 and NGT9-8 within the same lag deposit, although not identical, are close and appear to be closer for the four nodules within each sample. This suggests a short transport dis-

tance with little opportunity for mixing between nodules from different horizons.

The detrital nodule from the higher level PNC sample NGT9-9 yields an age of 267 ± 3 Ma with a PbC value of 0.834 ± 0.007 (Fig. 5B). This age is older than the succession exposed at Nooitgedacht and likely represents the reworking of older sediment into the Katberg Formation. Samples NGT9-10 and NGT9-11 represent the highest samples from the section (Fig. 3C), and their nodules give the youngest ages of 249 ± 3 Ma and 241 ± 3 Ma, respectively, with PbC values of ~ 0.83 (Figs. 5C and 5D).

Three nodules from the colluvium sample NGT-1 yield ages of 268 ± 5 Ma, 247 ± 8 Ma, and 268 ± 12 Ma with statistically distinct PbC values of 0.809 ± 0.007 , 0.805 ± 0.008 , and 0.849 ± 0.009 , respectively (Figs. 5E–5G). Since they were eroded from the thick sedimentary section exposed in the adjacent scarp, it is not surprising that they represent different generations with different ages and fluid sources.

PNC lags are common across the Balfour and Katberg formations (Pace et al., 2009; Viglietti et al., 2017; Gastaldo et al., 2018) and were once considered as a diagnostic criterion for identifying the latter in the Karoo Basin (e.g., Ward et al., 2000, 2005; Smith and Botha-Brink, 2014). The base of each intraformational nodule conglomerate represents an erosional contact of a newly established channel system associated with landscape degradation (Gastaldo and Demko, 2011). The establishment of new channel systems and associated interfluvial deposits throughout the Late Permian and Early Triassic was a consequence of changes in the fluvial gradient in the basin associated with climatic oscillations and tectonism. Each channel regime and the intervening paleosols that developed across the floodplain mark the initiation of an aggradational phase of deposition (Gastaldo et al., 2020a, 2020b) in which primarily entisols and vertisols formed. As aggradation slowed and landscapes reached stasis, vertisols developed in which calcite-cemented nodules could have precipitated. The stable carbon and oxygen isotopic composition of the calcite cement reflects whether mineralization occurred under closed- (Tabor et al., 2007) or open-system (Gastaldo et al., 2014) conditions. With the initiation of a subsequent degradational phase and erosion of the landscape, fine-grained detritus (sand and mud) was re-entrained in rivers and transported farther basinward to a different depocenter. In contrast, coarser detritus—granule, pebble, and cobble-sized pedogenic nodules—was retained as bedload lag deposits at the bases of channels and represents the incompetent sediment load associated with these river systems. Hence, calcite-cemented nodules recovered from chan-

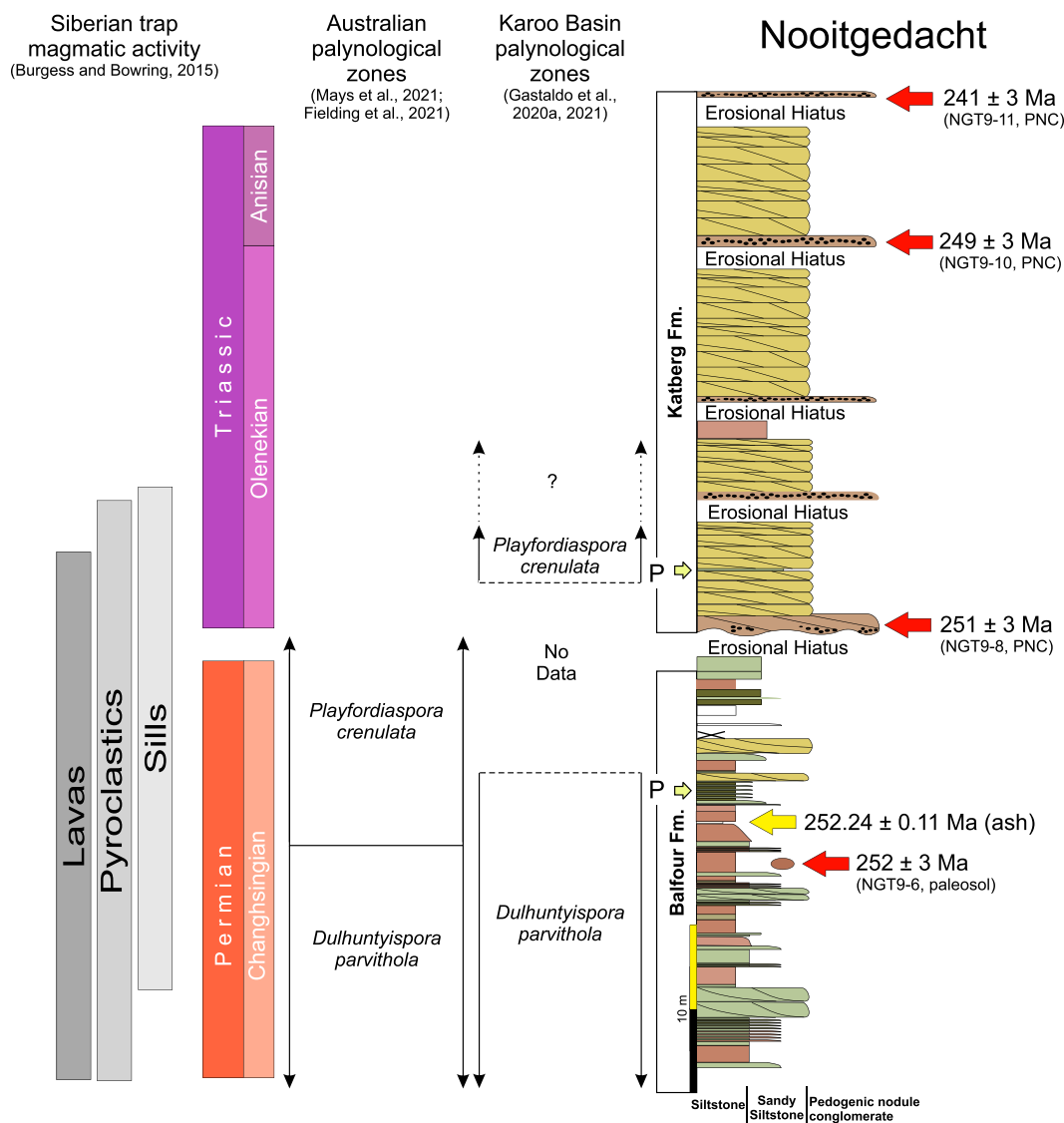


Figure 7. Geochronometric constraints of lithostratigraphic units on Farm Nooitgedacht. Geochronometric constraints are plotted against the palynological zones of the southern hemisphere in the Karoo Basin (Gastaldo et al., 2020a, 2021) and eastern Australia (Fielding et al., 2021; Mays et al., 2021). Correlation of the lithostratigraphy outcropping on Farm Nooitgedacht with the Geologic Time Scale (v2022/10). Permian-aged rocks are constrained by an age date of 252.24 ± 0.11 Ma in the upper Balfour Formation, which is latest Changhsingian. The assigned age overlying Triassic (Induan; red arrow) rocks is constrained by our U-Pb laser ablation–inductively coupled plasma–mass spectrometric (LA-ICP-MS) maximum depositional age from calcite-cemented pedogenic nodules overlying an erosional unconformity above the devitrified volcanogenic bed (yellow arrow). Due to the uncertainty of the original stratigraphic position of sample NGT-1, two possible positions of the maximum age assignment for Olenekian deposits are shown. See Figure 3 for horizons from which Olenekian and Ladinian age estimates were obtained. The age

ranges of Siberian Trap mafic lavas, pyroclastic deposits, and related mafic sills are based on Burgess and Bowring (2015). Fm.—formation; PNC—pedogenic nodule conglomerate. P marks the palynological assemblages.

nel lag deposits represent, with a few exceptions, the only remnants of the “ghost” landscapes of the Karoo (Gastaldo et al., 2020c). These lag deposits provide the only insight into processes that operated across the landscape for which no stratigraphic record is preserved.

The age estimate of 252 ± 3 Ma from the in situ paleosol nodule is in good agreement with the ash-fall deposit dated by Gastaldo et al. (2020a) at 252.24 ± 0.11 Ma (Fig. 7). This suggests that the U-Pb system in the paleosol remained stable and unaffected by later depositional and diagenetic processes. The PNC lag at the base of the lowest channel sandstone body at Farm Nooitgedacht, assigned to the Katberg Formation, was sampled ~ 7 m above the ash-fall deposit (Fig. 7). The average age estimate of

251 ± 3 Ma for the group of four nodules from NGT9-8 represents a maximum age estimate for genesis of the nodules and their deposition in this PNC lag deposit. This Late Permian–Early Triassic age estimate agrees with that of the in situ paleosol as well as the inferred stratigraphic age assigned to the Katberg Formation based on the palynological record (Gastaldo et al., 2020a; Mays et al., 2021). Most importantly, it agrees with the high-precision U-Pb zircon date from the subjacent ash deposit, which demonstrates the integrity of the U-Pb dating technique (Fig. 7).

Although it lacks a well-constrained stratigraphic position, colluvial sample NGT-1 could only have originated from one of the sandstone bodies high on the Loskop escarpment (Fig. 3A),

all of which overlie the thin ash-fall deposit that yielded the high-precision U-Pb CA-ID-TIMS late Changhsingian age estimate. Age estimates derived from the three clasts range from Roadian to Anisian (Table 1). The oldest nodules from the colluvium sample give Roadian ages, which are consistent with the age of 267 ± 3 Ma from sample NGT9-9. Remnants of middle Permian lakeshore deposits (Prevec et al., 2022), paleosols, and PNC lag deposits (Katsiaficas et al., 2010) are preserved in the Karoo Basin and could have served as a source of the reworked Wordian–Roadian nodules at the Farm Nooitgedacht section.

The oldest age from sample NGT-1 (268 ± 5 Ma), combined with the youngest age measured from sample NGT9-11 (241 ± 3 Ma),

imply that paleosols with calcite-cemented nodules formed over a period of at least 19 m.y. in the Karoo Basin. Hence, their presence in the uppermost Permian and lowermost Triassic strata in the basin cannot be relied upon to infer a unidirectional trend in aridification, as suggested by some authors (Smith and Botha-Brink, 2014; Botha et al., 2020; Viglietti et al., 2021), nor any other climatic cyclicity (Gastaldo et al., 2020c) without first establishing the ages of individual nodules. In addition, the age of 247 ± 8 Ma from the colluvium sample NGT-1, although imprecise, is consistent with the determination of ages of 251 ± 3 Ma for NGT9-8 and the PNCs higher in the section (Fig. 3C).

Vertebrate biostratigraphy continues to form the basis on which lithostratigraphic units of the Balfour and Katberg formations have been assigned stratigraphic ages (Smith et al., 2020). Geochronometric constraints have been published for only several units augmented by zircon U-Pb geochronology. Rubidge et al. (2013) provided an age estimate of <255.2 Ma for the base of the *Dicynodon* (= *Daptocephalus*) AZ from Farm Doornplaats (S 32.035021° , E 024.31327°), 32 km northwest of Graaff-Reinet and 55 km from the classic exposures of Upper Permian strata at Old Lootsberg Pass. Here, three detrital zircons, recovered from an ash of unspecified height above the Oudeberg Sandstone Member in the *Cistecephalus* AZ (Fig. 1), yielded U-Pb CA-ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ Wuchiapingian ages of 256.32 ± 0.36 Ma, and overlapping ages of 255.24 ± 0.16 Ma and 255.22 ± 0.16 Ma. Other analyses of detrital zircon indicate late Paleozoic ages ranging from Mississippian ($N = 1$; 330.60 ± 0.55 Ma; Serpukhovian), Roadian ($N = 1$; 272.11 ± 0.24 Ma), to Capitanian ($N = 2$; 259.66 ± 0.32 Ma, 259.08 ± 0.15 Ma).

A concordant CA-ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ Changhsingian zircon age estimate of 253.48 ± 0.15 Ma was reported from the Elandsberg Member at Old Lootsberg Pass, which was considered then to be in the *Dicynodon* AZ (= lower *Daptocephalus* AZ; Gastaldo et al., 2015). At the time, this horizon was considered to lie ~ 60 m below the vertebrate biozone boundary. A late Changhsingian CA-ID-TIMS $^{206}\text{Pb}/^{238}\text{U}$ zircon age of 252.24 ± 0.11 Ma was reported by Gastaldo et al. (2020a) from the Palingkloof Member of the succession at Farm Nooitgedacht near the base of the overlying *L. declivis* AZ, as then defined by Botha et al. (2020) and Botha and Smith (2020). Farm Nooitgedacht, the locality from which we report new age constraints, is located in a more distal basal setting, ~ 195 km to the northeast of Old Lootsberg Pass. Since then, the diagnostic taxa of what were considered to be two discrete biozones were shown to have overlapping stratigraphic ranges (Fig. 1;

Gastaldo et al., 2021). In contrast, Botha et al. (2020) reported a maximum depositional age of 251.7 ± 0.3 Ma (Induan) based on detrital zircon recovered from a mudstone on the Spitskop koppie (1.5 km SSW of Loskop; Fig. 2) on Farm Nooitgedacht. This is ~ 6.5 m below their biostratigraphic boundary, which they use to infer a latest Changhsingian age (Botha et al., 2020; their fig. 9) for the uppermost *Daptocephalus* AZ in support of their end-Permian extinction model (but see Gastaldo et al., 2021). To date, no maximum depositional age has been reported for any interval higher in the Beaufort Group, particularly in the Katberg Formation (Fig. 1). Our age results from the PNC deposits provide maximum depositional ages for the Katberg Formation, tightening the stratigraphic ages from Olenekian to Ladinian (Fig. 7). The ages of 249 ± 3 Ma (NGT9-10) and 241 ± 3 Ma (NGT9-11) derived from PNCs near the top of the section imply that the top of the *Lystrosaurus declivis* AZ, traditionally assigned to the early Olenekian (Lucas, 1998; Botha and Smith, 2020), should be extended to the Ladinian, which indicates the presence of long depositional gaps in the Katberg Formation. Similarly, the overlying *Cynognathus* Assemblage Zone, generally considered to range from the Olenekian to the late Anisian or early Ladinian (Liu et al., 2017; Hancox et al., 2020), is of much younger age, a hypothesis first advanced by Ottone et al. (2014).

The turnover in the vertebrate-fossil record of the Karoo Basin, from the last appearance data of diagnostic taxa assigned to the *Daptocephalus* to the first appearance data of diagnostic taxa assigned to the *Lystrosaurus declivis* AZ, has been used to mark the end-Permian extinction event (Botha et al., 2020, and references therein) and has been applied to other continents in both the southern and northern paleohemispheres (Dal Corso et al., 2022). When Gastaldo et al. (2020a) reported a U-Pb zircon age of 252.24 ± 0.11 Ma at Farm Nooitgedacht, the volcanic ash-fall deposit was considered to lie in the *Lystrosaurus declivis* AZ based on the paradigm prevailing at that time. Since then, the diagnostic taxa used to delimit each vertebrate biozone as unique were shown to have been coeval during the Changhsingian (Gastaldo et al., 2021). The presence of a palynoassemblage assigned to the *Playfordiaspora crenulata* biozone from a mudstone in the lowest Katberg sandstone unit on Loskop (Fig. 7; Gastaldo et al., 2020a) is consistent with the Olenekian age estimate from the basal PNC lag.

Recent palynologic studies in Australia, constrained by geochronology, interpret the initial collapse of the glossopterid biome on that landmass to have taken place some 330 k.y. prior to the marine mass extinction event

(Fielding et al., 2019; Mays et al., 2020). These authors suggest that terrestrial ecosystems were impacted earlier than the zenith of marine extinction at 251.902 ± 0.024 Ma (Shen et al., 2019). The collapse of the *Glossopteris* flora is believed to mark the beginning of the terrestrial crisis in southern Gondwana and is associated with a marked reduction in primary productivity (Mays et al., 2020). The turnover from a *Glossopteris*-dominated landscape to that of a depauperate flora, dominated by lycopods and peltasperms, is marked by the transition from the *Dulhuntyispora parvithola* palynozone to the *Playfordiaspora crenulata* palynozone (Fig. 7; Mays et al., 2021). The boundary between the two biozones is in the latest Changhsingian at 252.31 ± 0.07 Ma (Fielding et al., 2019; Mays et al., 2020). Fielding et al. (2019) indicated two alternate positions for the Permian–Triassic boundary, namely the lower part of the *P. crenulata* zone based on preliminary CA-ID-TIMS dates and the base of the *Lunatisporites pellucides* palynozone (which overlies the *P. crenulata* and the *Protohaploxyppinus microcorpus* palynozones). Mays et al. (2020) give the same estimated chronostratigraphic range of the *P. crenulata* zone in the zone description but, in one of their figures, placed the top of the biozone coincident with the marine Permian–Triassic boundary (Mays et al., 2020; their fig. 1). Vajda et al. (2020; their fig. 2), however, reported an uncertainty about the age of this palynozone, ascribing it neither wholly to the Permian or Triassic, with McLoughlin et al. (2021) placing the Frazier Beach Member, which lies above the extinction crisis, solely in the latest Permian based on geochronological constraints. At the time, the base of the *Protohaploxyppinus microcorpus* was considered to be mid-Induan (Griesbachian, Early Triassic) in age.

More recently, Mays et al. (2021) extended the upper boundary of the *P. crenulata* palynozone equivalent with the Griesbachian–Dienarian stage boundary (250.4 Ma), with the overlying *P. microcorpus* palynozone shifted into the younger Dienarian stage (250.4–249.7 Ma). However, Karoo pollen attributable to *Glossopteris* are present in the *P. crenulata* palynozone (Fig. 6), which may suggest the temporary persistence of the lineage into the Early Triassic in this basin (Gastaldo et al., 2021). In contrast, the *P. crenulata* Zone in Eastern Australia is latest Permian, starting at 252.31 ± 0.07 Ma (Fielding et al., 2019), and older than the occurrence of *P. crenulata* at Farm Nooitgedacht (Gastaldo et al., 2020a). Here, the *P. crenulata* palynozone occurs in the sandstone body above the PNC horizon. Hence, our result of 251 ± 3 Ma provides a maximum age esti-

mate for the first occurrence of the *P. crenulata* palynozone in this part of the Karoo Basin, and the age overlaps with the interpreted age of the floristic turnover recorded in Eastern Australia (Fig. 7).

The U-Pb date of 251 ± 3 Ma reported for the youngest nodules of the lowermost PNC indicates that the Permian–Triassic boundary at Farm Nooitgedacht may be preserved in strata in the short stratigraphic succession between the ash-fall bed, which was dated at 252.24 ± 0.11 Ma by Gastaldo et al. (2020a), and the base of the overlying sandstone channel (Fig. 7). It is unknown whether these nodules might have formed during the transition between the Permian and Triassic and represent conditions in the basin during the extinction event. However, because these clasts were reworked from calcic vertisols into the PNC during landscape degradation, it is more likely that the Permian–Triassic boundary interval at Farm Nooitgedacht is not preserved.

U-Pb age errors from LA-ICP-MS analyses of nodules can be as low as 1%. This error does not include error in the standard for the same reason that errors in decay constants are normally not included in zircon U-Pb age errors. If everyone uses the same standard (or constants in the case of zircon), the same results should be obtained even though they may not reflect the true accuracy. A great deal more work needs to be done to study factors controlling reproducibility in dating calcites, but this is beyond the scope of this paper. The fact that we have shown that we can reproduce ages of four nodules from each of two hand samples provides some evidence as to the reliability of our quoted precisions.

The results of this study demonstrate that U-Pb dating of pedogenic nodules has the potential to greatly improve our understanding of mass extinction in the terrestrial environment. A 1% age error is an order of magnitude greater than for zircon, but pedogenic carbonate nodules are much more abundant than zircon-bearing tuff horizons. Even detrital nodules are useful since they are unlikely to have been transported over large distances. The application of ID-TIMS dating can significantly improve age precision. Furthermore, it has been shown that the relationship between isotopic fractionation of organic and inorganic carbon can serve as a proxy for the partial pressure of CO_2 in the atmosphere at the time of pedogenic carbonate formation (Gastaldo et al., 2014). The application of clumped O-isotope measurements may also allow for estimates of temperature (Methner et al., 2016), thus giving a detailed record of atmospheric changes during critical periods of Earth's history.

CONCLUSIONS

The calcite-cemented carbonate nodules derived from a paleosol horizon and pedogenic nodules collected from intraformational conglomerate lag deposits at Farm Nooitgedacht show unusually high-U contents (20–60 ppm), which are often around 1 ppm or less in carbonates (e.g., Li et al., 2014; Roberts et al., 2020). This allows age determinations by LA-ICP-MS to be measured with errors down to $\sim 1\%$, despite the presence of significant common Pb. Although such errors are much higher than those associated with analyses of zircon, which yields highly radiogenic Pb, they are low enough to be geologically useful, particularly when zircon dating is absent.

The age estimate of 252 ± 3 Ma derived from an in situ paleosol nodule located below a volcanogenic ash-fall deposit is consistent with the high-precision U-Pb CA-ID-TIMS zircon age of 252.24 ± 0.11 Ma (Gastaldo et al., 2020a) of that ash deposit. U-Pb dating of PNC units constrains the age range for the Katberg Formation at Farm Nooitgedacht from Olenekian to Ladinian and implies the need for a reevaluation of the age assigned to the overlying Burgersdorp Formation. U-Pb ages for multiple nodules extracted from two samples collected from different locations within the same PNC lag deposit at the base of the Katberg Formation yield overlapping ages (Wuchiapingian–Olenekian). This result suggests that the pedogenic nodule conglomerate consists of sediment derived from a proximal source. The age of 251 ± 3 Ma for the youngest group of nodules represents a maximum age estimate for genesis of the nodules and their deposition as a PNC lag deposit. These results also provide a maximum age estimate for the first occurrence of the *P. crenulata* palynozone in the Karoo Basin, which has been reported to mark the end-Permian extinction event at Sydney Basin, Australia (Mays et al., 2021).

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