

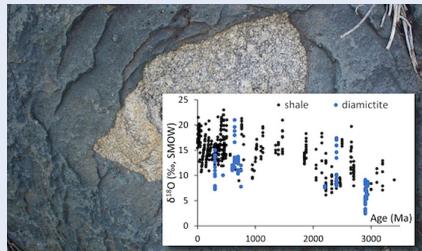
■ Earth's first glaciation at 2.9 Ga revealed by triple oxygen isotopes

A. Hofmann^{1*}, I.N. Bindeman²

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Abstract



We here report the lowest (~3 ‰ VSMOW) $\delta^{18}\text{O}$ values for any weathering-related sedimentary rock in Earth's history, from shales and diamictites of the Mesoarchaean Pongola Supergroup of South Africa. This volcano-sedimentary succession was deposited in a shallow epeiric sea on continental crust of the Kaapvaal Craton and includes the record of the Earth's oldest surface glaciation. Oxygen isotope data of shales of the Mozaan Group indicate gradual climatic cooling of the surface environments that culminated in glacial conditions at ~2.90 Ga. Mathematical inversion of measured $\Delta^{17}\text{O}$ and $\delta^{18}\text{O}$ values results in $\delta^{18}\text{O}$ values around -20 ‰ for weathering waters, suggesting cold climate conditions. These observations suggest continental weathering of the Kaapvaal Craton involving low $\delta^{18}\text{O}$ meteoric waters, possibly in a near-polar position.

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Introduction

The Pongola Supergroup is the tectonically least disturbed Mesoarchaean volcano-sedimentary sequence on Earth (Luskin *et al.*, 2019). Its deposition started at 3.0 Ga in an epicontinental sea that flooded the Kaapvaal Craton, which had become a stable continent ~100 Myr earlier, but experienced extension and subsidence (Paprika *et al.*, 2021). The Pongola Supergroup contains the oldest purported glacial deposits (von Brunn and Gold, 1993; Young *et al.*, 1998). Mesoarchaean diamictites of possible glacial origin were first described from the time-equivalent West Rand Group of the Witwatersrand Supergroup (Wiebols, 1955), although formation modes other than glacial processes have been proposed, such as by cohesive debris flows (Martin *et al.*, 1989). Despite their importance for the evolution of Earth's climate through time, a glacial setting for the Pongola diamictites remains a matter of debate. As the oxygen isotopic composition of surface materials is strongly dependent on latitude and climate, we have applied triple oxygen isotope analysis to scrutinise their origin. We report $\delta^{18}\text{O}$ values of shales and diamictites lower than any siliciclastic deposits analysed so far, supporting glacial conditions during deposition of the Pongola Supergroup.

Geological Setting

The Archaean Pongola Supergroup is a volcano-sedimentary succession that was deposited between 2.99 and 2.87 Ga on continental crust of the south-eastern part of the Kaapvaal Craton (Fig. 1a; Gumsley *et al.*, 2015; Luskin *et al.*, 2019). It is lithologically and stratigraphically similar to the Witwatersrand

Supergroup and underlying Dominion Group, and together they form the oldest preserved cratonic cover succession (Beukes and Cairncross, 1991; Paprika *et al.*, 2021). Two groups comprise the Pongola Supergroup: the lower Nsuze Group, dominated by volcanic rocks ranging from flood basalt to rhyolite, and the upper Mozaan Group, dominated by shallow marine siliciclastic sedimentary rocks (Luskin *et al.*, 2019). Chemical sedimentary rocks, including banded iron formation (BIF) and stromatolitic carbonates, Mn-rich shales, and palaeosols provide an exceptional record of surface processes in the Mesoarchaean and indicate shallow-marine oxygen oases under an anoxic atmosphere (Siahi *et al.*, 2016; Eickmann *et al.*, 2018; Ossa *et al.*, 2018; Heard *et al.*, 2021).

The Mozaan Group overlies the Nsuze Group disconformably, the latter as young as 2954 ± 8 Ma, the youngest age of the correlative Dominion Group (Paprika *et al.*, 2021). The Mozaan Group is ~5 km thick and consists largely of shallow marine sandstone and shale deposited in an intracratonic sea subjected to sea-level oscillations (Beukes and Cairncross, 1991). Despite regional metamorphism to lower greenschist facies grade, rocks are extremely well preserved, not requiring the usage of terminology for metamorphic rocks. Following Luskin *et al.* (2019) and references therein, it is subdivided into ten formations (Fig. 1b). At the base is the Sinqeni Formation, consisting of two fluvial to shallow-marine sandstone units separated by a unit of shale and BIF. The Ntombé Formation is a transgressive sequence of ferruginous shale, siltstone, and sandstone and is overlain by compositionally similar marine highstand deposits of the Thalu Formation. Both formations contain shales enriched in Mn-carbonate, derived diagenetically from Mn(IV)-oxyhydroxides that precipitated in oxygenated

1. Department of Geology, University of Johannesburg, South Africa

2. Department of Earth Sciences, 1272 University of Oregon, Eugene OR 97403, USA

* Corresponding author (email: ahofmann@uj.ac.za)



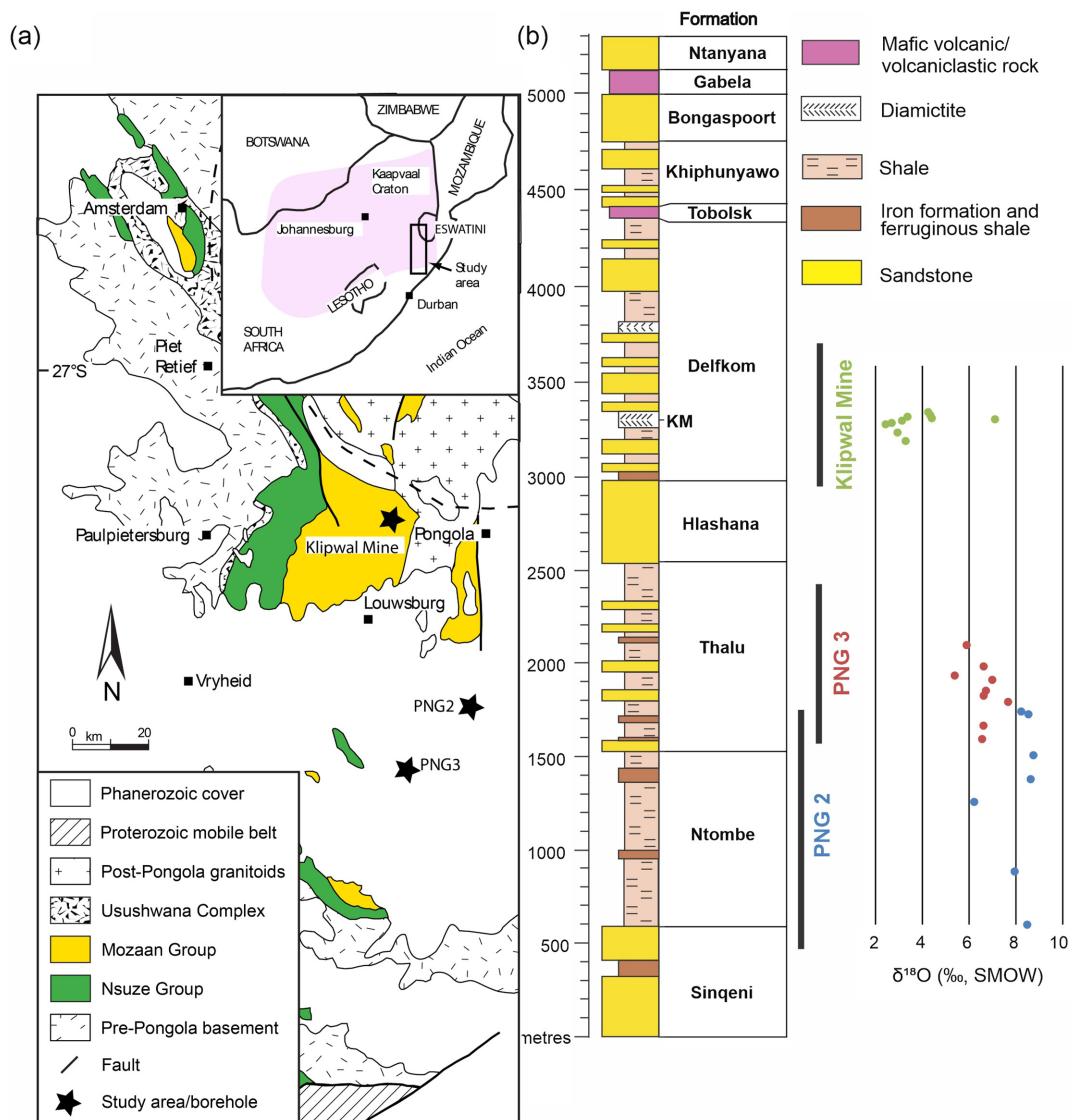


Figure 1 (a) Simplified geological map showing the distribution of the Nsuze and Mozaan groups of the Pongola Supergroup and its location within the Kaapvaal Craton. (b) Stratigraphy of the Mozaan Group of the Pongola Supergroup (Luskin *et al.*, 2019). Distribution of $\delta^{18}\text{O}$ in shales and diamictites with depth in PNG cores and surface samples from Klipwal Mine area. KM, Klipwal Member.

surface waters (Ossa *et al.*, 2018). The Hlashana Formation is dominated by cross-bedded sandstone of a current-dominated shelf sea environment and is overlain by the Delfkom Formation, composed of sandstone, shale, and diamictite, the focus of this study. The youngest concordant detrital zircon date of 2903 ± 14 Ma from a sandstone of this formation provides a maximum depositional age (Zeh and Wilson, 2022). For the description of the units higher up in the Mozaan Group stratigraphy, the reader is referred to Luskin *et al.* (2019). A dolerite sill dated at 2869 ± 5 Ma provides a minimum age for Mozaan Group deposition (Gumsley *et al.*, 2015). The Delfkom Formation and its diamictites were thus deposited between 2.90 and 2.87 Ga.

Sampling and Petrography

Stratigraphically resolved sampling of diamictite and shale from the Delfkom Formation was conducted in the Klipwal Mine area (Fig. 1a; von Brunn and Gold, 1993). At this locality, four diamictite units have been described that are interbedded with

ferruginous shale, quartz arenite and conglomerate (Fig. S-1). The most prominent of the diamictite units is the Klipwal Member that has been sampled for this study together with several samples of underlying shale. The shales consist largely of silt-sized detrital quartz, detrital and diagenetic feldspar, euhedral chlorite, disseminated magnetite and some euhedral pyrite and Ti-oxide (Fig. S-2). The composition of diamictite has been described by von Brunn and Gold (1993) and Young *et al.* (1998), and its matrix is compositionally similar to the shale (Fig. S-3).

Shale samples were also obtained from two deep borehole cores (PNG2, PNG3) drilled in 1988 by the AngloGold Exploration Division (Fig. 1a). The boreholes intersected lower stratigraphic units of the Mozaan Group, including the Sinqeni, Ntombé and lower Thalu formations (Figs. 1b, S-4). A limited geochemical dataset of samples from PNG2 have been reported by Ossa *et al.* (2018). The shales are ferruginous, consisting largely of silt-sized detrital quartz in a predominantly chlorite matrix with total organic carbon contents of <1 wt. %.

Isotopic Composition

All samples were subjected to oxygen (including $\Delta^{17}\text{O}$) and hydrogen isotope analysis at the University of Oregon (Table S-1) and all data are reported relative to VSMOW. Samples from the Klipwal Mine area were also analysed for major element contents at the University of Johannesburg (Table S-2). Analytical procedures are outlined in the Supplementary Information.

Shale samples from drill core PNG2 ($n=8$) cover the Ntombé and lower Thalu formations. The $\delta^{18}\text{O}$ values are relatively constant throughout the core, averaging $8.1 \pm 0.9 \text{ ‰}$. The δD values average $-74.8 \pm 4.6 \text{ ‰}$, excluding the two stratigraphically uppermost samples that range between -119.9 and -100 ‰ .

Drill core PNG3 intersected the Thalu Formation. The $\delta^{18}\text{O}$ values of shale samples ($n=9$) from this core are lighter compared to those of PNG2, averaging $6.6 \pm 0.6 \text{ ‰}$. The δD values are relatively constant throughout the core, averaging $-56.3 \pm 7.2 \text{ ‰}$.

Shale ($n=5$) and diamictite ($n=5$) samples obtained from fresh outcrop around Klipwal Mine show even lighter $\delta^{18}\text{O}$ values, with shale averaging $2.9 \pm 0.3 \text{ ‰}$ and diamictite averaging $4.7 \pm 1.4 \text{ ‰}$. The δD values show some scatter, with shale of the Delfkom Formation averaging $-73.2 \pm 16.4 \text{ ‰}$ and diamictite averaging $-83.1 \pm 14.5 \text{ ‰}$. There are moderate positive correlations between $\delta^{18}\text{O}$ values vs. SiO_2 ($R^2=0.42$) and K_2O ($R^2=0.41$). δD values neither correlate with major element contents nor with $\delta^{18}\text{O}$ values (Fig. S-5).

Discussion

Diamictites of the Mozaan Group were first described by von Brunn and Gold (1993). They reported the presence of a highly varied suite of extra-basinal clasts, some of which being striated and faceted, and argued for their emplacement by gravity flows derived from highland glaciers. Young *et al.* (1998) noted

moderate CIA values (Chemical Index of Alteration, 66.8 on average) and high Fe-oxide contents in diamictites and interbedded shales. This was regarded in support of a glacial origin of the diamictites, by analogy with Neoproterozoic glaciogenic diamictites. They further reported the rare presence of dropstones, indicative of clast-charged floating glacier ice at the time of deposition.

The oxygen isotope composition of shales (and other mud-dominated rocks such as diamictites) largely reflects weathering conditions, specifically the isotopic composition of the meteoric water during formation of clays at weathering temperature, generally leading to higher than crustal $\delta^{18}\text{O}$ values, and well above the mantle value of 5.7 ‰ (Bindeman *et al.*, 2016 and references therein). Values of $\delta^{18}\text{O}$ broadly increase through Earth history from $\sim 10 \text{ ‰}$ in the Archaean to $\sim 15 \text{ ‰}$ in the Phanerozoic (Fig. 2), linked to the combined effects of crustal differentiation and incorporation of isotopically heavy weathering products into the crust over time. Strong downward shifts of $\delta^{18}\text{O}$ values by several per mille in the Palaeoproterozoic and Neoproterozoic (Fig. 2) have generally been attributed to glaciations, as meteoric waters in glacial settings have very low $\delta^{18}\text{O}$ values, and glacial rock flour contains less weathered clastic materials (Nesbitt and Young, 1982) lower in $\delta^{18}\text{O}$.

Average $\delta^{18}\text{O}$ values for Mesoarchaean (3.2–3.0 Ga) shales have been reported as $9.7 \pm 2.4 \text{ ‰}$ (Bindeman *et al.*, 2016). Our values for shale samples from PNG2 from the lower part of the Mozaan Group fall within this average. Stratigraphically upwards, shales from PNG3 show lower values, and this decrease culminates in $\delta^{18}\text{O}$ values for shales and diamictites of the Delfkom Formation lower than mantle. These data indicate weathering of Thalu and Delfkom formations source materials by meteoric waters with progressively decreasing $\delta^{18}\text{O}$ values, potentially culminating in weathering by ultra-low $\delta^{18}\text{O}$ waters. Thus, there is strong evidence for climatic cooling during deposition of the lower Mozaan Group, eventually giving rise to a glacial environment during Delfkom Formation deposition. We note that diamictites of the Mozaan Group and Afriander Formation of the West Rand studied by

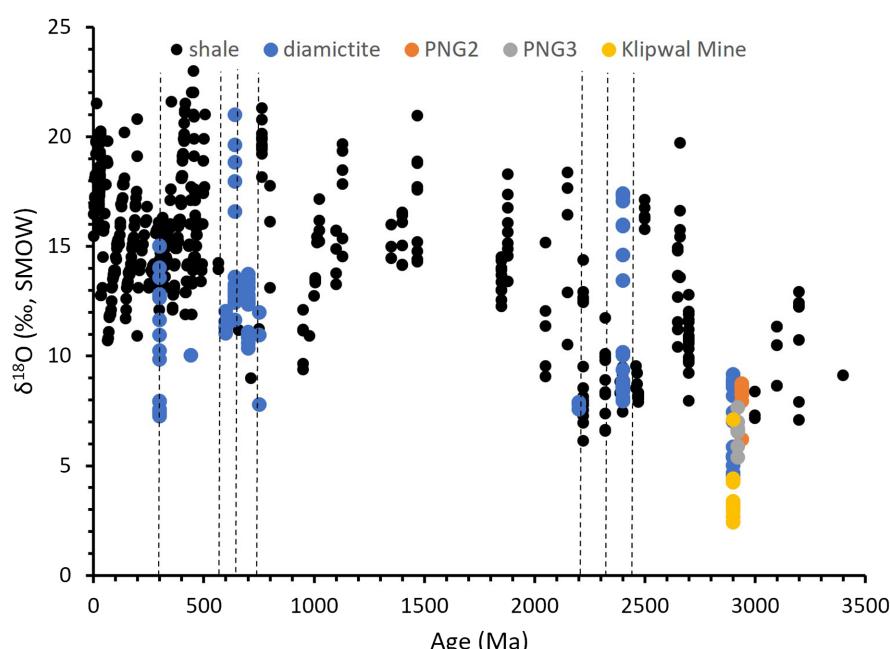


Figure 2 Oxygen isotope evolution of shales (Bindeman *et al.*, 2016) and diamictites (Gaschnig *et al.*, 2016) through time, and data from this study. Stippled lines point to Palaeo- and Neoproterozoic as well as Permo-Carboniferous glaciations.

Gaschnig *et al.* (2016) also contain low (<6 ‰) $\delta^{18}\text{O}$ values overlapping with our data (Fig. 2).

Positive correlations of $\delta^{18}\text{O}$ values with SiO_2 and K_2O in Delfkom Formation shales and diamictites indicate higher $\delta^{18}\text{O}$ values for detrital quartz and K-feldspar compared to the matrix. The δD values of our samples are similar to those of average modern shales and crustal fluids (Sheppard and Gilg, 1996). The lack of correlation between $\delta^{18}\text{O}$ and δD values (Fig. S-5) suggests isotopic re-equilibration with diagenetic and/or metamorphic fluids during recrystallisation of the chlorite precursor material and chlorite itself. No weathering history of the sediment source is thus preserved in the δD values of the Mozaan Group.

The CIA values of our samples (Table S-2) are not unlike pre- and post-2.9 Ga shales (Bindeman *et al.*, 2016). They range from 60, translating to ~20 % clay weathering product and ~80 % unweathered silicates, to 97, representing almost pure clay. The average CIA value is 69.9 for diamictite and 75.2 for shale. This makes sense as diamictites contain unweathered clasts of quartz and feldspar, as also indicated by slightly higher $\delta^{18}\text{O}$ values compared to shales. *Via* mass balance, we can derive the “weathering product” in equilibrium with weathering waters, by subtracting 0–80 % of unweathered siliciclastic detritus with an average $\delta^{18}\text{O}$ crustal value of +6.5 ‰ (Table S-3). Such a procedure was used by Bindeman *et al.* (2018) in their global shale inversion. Then we employed a mathematical inversion of measured $\delta^{18}\text{O}$ and $\delta^{17}\text{O}$ values of our samples and computed a weathering product (Fig. 3, Table S-3), by using the modern meteoric water line (MWL) equation $\delta^{17}\text{O} = 0.528 \times \delta^{18}\text{O} + 0.033$. As we have two fractionation equations for $1000\ln\alpha^{18}\text{O}_{\text{shale-water}}$, $1000\ln\alpha^{17}\text{O}_{\text{shale-water}}$, and the MWL equation, we are able to obtain solutions for $\delta^{18}\text{O}_{\text{water}}$, $\delta^{17}\text{O}_{\text{water}}$ and temperature (see Table S-3 for further details of these computations). We obtain $\delta^{18}\text{O}$ values of weathering waters of -18.9 ± 2.3 ‰ (bulk sample) and -20.9 ± 4.8 ‰ (weathering product), values found in polar regions today. Computed temperatures are 37–41 °C.

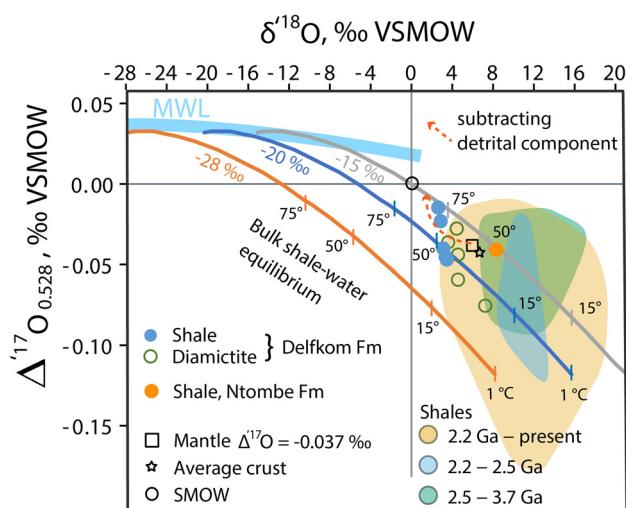


Figure 3 Triple oxygen isotope diagram showing shale-water fractionation lines connected to various parental meteoric waters (MWL, present day meteoric water line; Surma *et al.*, 2021) that participated in weathering. Note that Delfkom Formation shales and diamictites plot around -18 ± 2.3 ‰ meteoric water and have $\delta^{18}\text{O}$ values lower than any other shales in the geologic record (data of Bindeman *et al.*, 2018). Curved arrow indicates the effect of subtracting a detrital component in the samples (with crustal $\delta^{18}\text{O}$ of +6.5) to compute a “pure weathering product” (Table S-3).

As Archaean seawater may have been lower in $\delta^{18}\text{O}$ (e.g., Bindeman, 2021), we additionally performed computations assuming Archaean seawater with $\delta^{18}\text{O}$ of -5 ‰, generating a MWL equation of $\delta^{17}\text{O} = 0.528 \times \delta^{18}\text{O} + 0.078$, having higher $\Delta^{17}\text{O}$ values *per* lower $\delta^{18}\text{O}$ in seawater (e.g., Sengupta and Pack, 2018). Under this assumption, with a meteoric water line higher in $\Delta^{17}\text{O}$, $\delta^{18}\text{O}$ values of weathering waters are -26 to -28 ‰ at 6–8 °C.

The computed temperatures reflect the conditions during isotopic closure, during the last equilibrium between the weathering waters and sediment, possibly at the time of expulsion of pore waters during compaction, leading to a reduction in porosity and hydrologic impermeability (Bindeman, 2021). We consider it was then that most oxygen atoms from the hydrosphere were taken up by the bulk of the silicates and their precursors analysed here. The rocks carry no textural evidence of percolation of post-diagenetic fluids, however the effects of subsequent metamorphism and tectonic uplift, namely dehydration of rocks to potential re-hydration, on bulk isotope values of rocks are considered in the *Supplementary Information* and produce <1 ‰ shifts in $\delta^{18}\text{O}$ values but affect δD and $[\text{H}_2\text{O}]$ significantly, potentially explaining “reset” δD values.

Assuming no major change between 2.95 and 2.90 Ga in the degree of continentality and elevation of the sedimentary source terrain, the isotopic data are consistent with deposition of the Delfkom Formation under cool climatic conditions with continental weathering involving low $\delta^{18}\text{O}$ meteoric waters. Palaeomagnetic data constrain the Kaapvaal Craton to mid to high latitudes at the time of deposition of the Pongola Supergroup (de Kock *et al.*, 2021). Climatic cooling may be linked to drift of the Kaapvaal Craton towards a pole. Alternatively, climatic cooling was a global phenomenon pending verification on different crustal blocks. Stabilisation of the Singhbhum and Kaapvaal cratons at ~3.1 Ga (Hofmann *et al.*, 2022) and the Pilbara Craton at ~2.9 Ga (Hickman, 2023) allowed for subaerial emergence, enhanced continental weathering, and the drawdown of atmospheric CO_2 , providing suitable conditions for the development of continental glaciers. In addition, muted S-MIF signatures reported from Pongola strata may hint to atmospheric oxidation and destabilisation of greenhouse methane (Ono *et al.*, 2006). However, there is no intracratonic record for the 2.90 to 2.87 Ga time interval apart from the Kaapvaal Craton, necessitating a search for glacial deposits in Archaean greenstone successions that formed around that time, but under deeper water conditions. The presence of continental ice sheets may explain well developed sedimentary cyclicity recorded in the Pongola Supergroup (Beukes and Cairncross, 1991) due to glacio-eustatic sea-level changes.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2319>.



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