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# ORIGINAL ARTICLE



# A sedimentary record of the evolution of the global marine phosphorus cycle

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# **Abstract**

Phosphorus (P) is typically considered to be the ultimate limiting nutrient for Earth's biosphere on geologic timescales. As P is monoisotopic, its sedimentary enrichment can provide some insights into how the marine P cycle has changed through time. A previous compilation of shale P enrichments argued for a significant change in P cycling during the Ediacaran Period (635-541 Ma). Here, using an updated P compilation—with more than twice the number of samples—we bolster the case that there was a significant transition in P cycling moving from the Precambrian into the Phanerozoic. However, our analysis suggests this state change may have occurred earlier than previously suggested. Specifically in the updated database, there is evidence for a transition ~35 million years before the onset of the Sturtian Snowball Earth glaciation in the Visingsö Group, potentially divorcing the climatic upheavals of the Neoproterozoic from changes in the Earth's P cycle. We attribute the transition in Earth's sedimentary P record to the onset of a more modern-like Earth system state characterized by less reducing marine conditions, higher marine P concentrations, and a greater predominance of eukaryotic organisms encompassing both primary producers and consumers. This view is consistent with organic biomarker evidence for a significant eukaryotic contribution to the preserved sedimentary organic matter in this succession and other contemporaneous Tonian marine sedimentary rocks. However, we stress that, even with an expanded dataset, we are likely far from pinpointing exactly when this transition occurred or whether Earth's history is characterized by a single or multiple transitions in the P cycle.

# 1 | INTRODUCTION

It is generally accepted that Earth's surface phosphorus (P) cycle has changed dramatically through time, in concert with fundamental changes to ocean–atmosphere chemistry, increasing biological complexity and the ability of the biosphere to transform the environment

of our planet (Knoll, 2003). Earth's earliest history was characterized by a pervasively reducing ocean-atmosphere system, likely with limited phosphorus availability, as evidenced most conspicuously by a complete lack of sedimentary phosphate deposits known as phosphorites (Figure 1; Holland, 2005). Sedimentary phosphorite deposition and P enrichments in marginal marine sediments begin

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FIGURE 1 Summary of the geochemical record of phosphorus against the backdrop of Earth system evolution. Shown in (a) is the evolution of atmospheric  $O_2$  through time, with shaded boxes showing approximate ranges based on geochemical proxies and the red curve showing one possible trajectory through time (from Reinhard et al., 2017). Shown in (b) are sedimentary phosphorite occurrences scaled through time. (c) displays sterane to hopane ratios through time adapted from Brocks (2018) and Isson et al. (2018), with data for the Visingsö formation (green) from Zumberge et al., *in press*. Displayed in (d) are phosphorus concentrations through time in siliciclastic marine sediments, updated from the compilation presented in Reinhard et al. (2017), with values for the Visingsö Group from this study overlain. Inset to (D) shows the bootstrap-resampled mean phosphorus content of pre-753.3 ma (blue) and post-753.3 ma (red) samples (n = 10,000). In (c and d), colors correspond to the following age groups: >753.3 ma (blue) and <753.3 ma (red). Data from the Visingsö Group (753.3 ma) are highlighted in green. Data in Tables S1–S3.

to sporadically occur in the rock record following the initial oxygenation of Earth's atmosphere at ~2.3 Ga (Lyons et al., 2014). Some of these deposits accumulated in the aftermath of the Great Oxidation Event (GOE) and may be related to a protracted, but ultimately transient, interval of relatively high atmosphere O2 levels known as the Lomagundi Event (e.g., Bekker & Holland, 2012; Kipp et al., 2020; Melezhik et al., 2015). Nevertheless, phosphorite deposits remain rare and are stratigraphically limited until the latest part of the Proterozoic Eon. Consistent with observations from the phosphorite record, a large compilation of P enrichment data from shallow and intermediate water depths was recently used to argue for a significant increase in P enrichments and marine P cycling during the Ediacaran Period (635-541 Ma), the latest part of the Neoproterozoic Era (Reinhard et al., 2017). With similar analysis, Laakso et al., 2020 argued that while the frequency of P-rich sedimentary units increased at the Ediacaran, the average P enrichment in shales did not change. They suggest that shift in the P cycle was initiated by Proterozoic orogenesis and maintained by biospheric feedbacks. However, they focused only on the past 800 million years-rather than looking throughout complete Precambrian and Phanerozoic compilation.

This Ediacaran shift in P enrichments was proposed to be linked to the progressive oxygenation of the ocean and the waning of a deep-sea inorganic P trap characteristic of anoxic and Fe (II)-rich oceans (Derry, 2015; Poulton & Canfield, 2011; Reinhard et al., 2017). Historically, local redox conditions were assumed to play a critical role in controlling local P enrichments (Ingall & Jahnke, 1994, 1997; Van Cappellen & Ingall, 1994, 1996), but there is no clear relationship between marine redox proxies and P enrichments in current shale databases (Reinhard et al., 2017). Instead, the observed increase in both the mean and variance of sedimentary P enrichments was hypothesized to reflect an increase in primary production and more P-rich organic matter associated with higher marine-dissolved phosphate concentrations, potentially coinciding with a dramatic change in the elemental composition of phytoplanktons. These changes are predicted to drive a concurrent increase in the capacity of marine sediments to form diagenetic apatite (Reinhard et al., 2017). This model builds from observations that the elemental stoichiometry of primary producers (especially cyanobacteria) may be flexible in the face of fluctuating, or depressed, nutrient levels (e.g., White et al., 2006).

Recent work suggests that the sedimentary P record can be used to piece together the history of marine P cycling (Alcott et al., 2022; Dale et al., 2016; Guilbaud et al., 2020; Kipp & Stüeken, 2017; Poulton, 2017; Reinhard et al., 2017). However, conclusions garnered from geochemical databases are only as sound as the quality of those databases and the accuracy and precision of ages that are assigned to key geological units and the data derived from them. To improve our understanding of temporal variations in the sedimentary P record, we present a new phosphorite occurrence and shale P enrichment database-more than doubling the number of occurrences and samples in the previous compilation. These new data bolster the case that there was a fundamental shift in P cycling during the Neoproterozoic, but we cast some doubt on our ability to pinpoint when this transition occurred. We generated new P data for a data-poor interval in the Neoproterozoic and found that these new analyses-limited in number relative to the overall dataset-changed when, based on statistical tests, the shift in P cycling occurred. Specifically, by coupling these observations with a new rhenium-osmium (Re-Os) radioisotopic age constraint on the P-rich Visingsö Group, we tentatively suggest a transition in marine biogeochemistry likely occurred during the latter part of the Tonian Period (1000-720 Ma), >100 million years earlier than previously thought and prior to the onset of the Sturtian glaciation.

# 2 | GEOLOGICAL SETTING AND GEOCHRONOLOGY OF THE VISINGSÖ GROUP

We targeted the mid-Neoproterozoic Visingsö Group given an obvious dearth of data in the early and middle Neoproterozoic. The Visingsö Group comprises predominantly heterolithic siliciclastics (shale, sandstone, and conglomerate) and is estimated to be ~1000 m thick (Collini, 1951; c.f., Pulsipher & Dehler, 2018). The upper member (<580 m thick) is predominantly carbonaceous shale with subordinate beds of dolomite, sandstone, and dolomite/phosphorite nodules. This succession was deposited in an intracratonic basin that was affected by extensional tectonism, possibly related to the breakup of Rodinia (Larsen & Nørgaard-Pedersen, 1988; Pulsipher & Dehler, 2018; Vidal, 1974).

Phosphorus nodules and larger phosphate grains (e.g., larger than sand sized grains), in addition to disseminated P enrichments (abundant clay- to sand-sized phosphate grains), are present in the organic-rich silt- and clayey shales (Figure 2). We determined the P content of >100 samples from the Visingsö Group that lacked evidence for macroscopic phosphates. Until now, the geochronologic age of the Visingsö Group was unconstrained—with the only direct age being a detrital zircon with a maximum depositional age of 886±9 Ma (Moczydłowska et al., 2018; Pulsipher & Dehler, 2018).

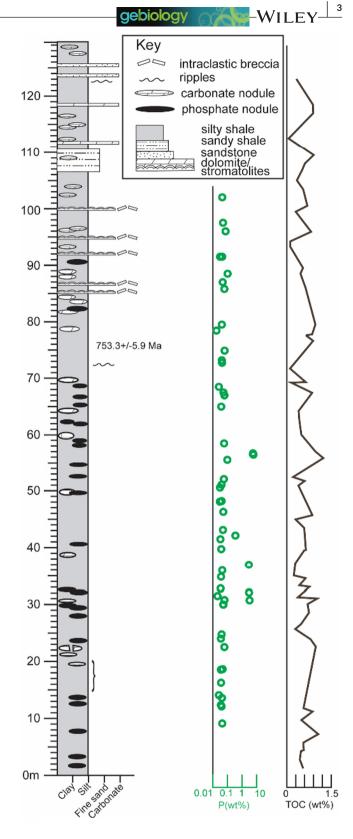
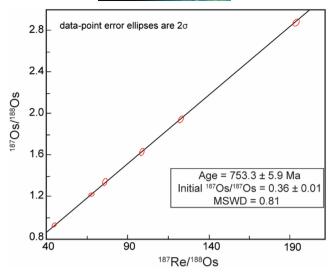


FIGURE 2 Stratigraphic column for the broken nodule section of the Visingsö Group. Presented alongside the stratigraphic column are chemostratigraphic profiles for P (wt%) and total organic carbon (TOC; wt%).



**FIGURE 3** Regression of the Re–Os isotopic composition data from samples A1509 of the broken nodule section yields a model 1 age of  $753.3 \pm 5.9$  ma (all data point error ellipses are 2 sigma and their diameters are larger than calculated error ellipses. Data are presented in Table S1).

To address the need for better age constraints, we obtained a new Re-Os age for the phosphate-rich Upper Formation in the Visingsö Group of  $753.3 \pm 5.9$  Ma (Figures 2 and 3; Table S1).

## 3 | RESULTS AND DATABASE DETAILS

An updated record of phosphorite occurrences and sediment P enrichments is provided in Figure 1. We document 326 phosphorites, which we define as sedimentary units where the mineral apatite (or carbonate fluorapatite to be more accurate) is a major component of the sedimentary rock (which, following Holland (2005), we take as roughly 10% of the rock mass as P). In contrast to previous compilations, we also provide stratigraphic thicknesses for each of the phosphorites in the database. Over 85% of the phosphorites in the compilation are Phanerozoic in age, with an additional 8% occurring in the Neoproterozoic. The vast majority of Proterozoic phosphorites (>90%) have thicknesses on the sub-decimeter scale, with only four exceptions—the Ediacaran Doushantuo Formation, the Tonian Visingsö Group, and the Paleoproterozoic Aravalli and Baraga groups (Table S2). The Paleoproterozoic occurrences are stromatolitic, while the younger Proterozoic occurrences are re-worked and diagenetic beds and nodules, more analogous to typical Phanerozoic phosphorites (Table S2).

Our new shale P database contains ~18,360 samples with P concentration data (Table S3), significantly expanding on the previous compilation of 8700 samples (Reinhard et al., 2017). We apply the same basic filters to define a 'shale' as Reinhard et al. (2017)—namely, selecting only sediments with >2 wt% Al—and exclude phosphorites from our analysis. As expected, the dataset remains heavily left skewed, given the high number of Cenozoic samples. Given unequal

sampling densities, we present bootstrap resampled means to explore if there are significant shifts between time periods. We exclude phosphorites since the sampling for these is unlikely to be comparable to routine bulk chemistry measurements-not done focusing on P content-that dominate the database. Excluding phosphorites is conservative given the main conclusion of the paper and that most phosphorites are Phanerozoic in age. Despite a significant increase in the size of the sedimentary database (from Reinhard et al., 2017) the Neoproterozoic-Phanerozoic and Archean-Neoproterozoic averages remained markedly similar (Reinhard et al., 2017:  $0.209 \pm 0.023$  and  $0.051 \pm 0.003$ , versus this study:  $0.150 \pm 0.012$  and  $0.054 \pm 0.003$ ; Figure 1). With an expansion of the database by almost 10,000 samples, the confidence intervals for the Archean-Proterozoic changed from [0.046, 0.054] to [0.051, 0.058], and for the Neoproterozoic-Phanerozoic [0.190, 0.237] to [0.140, 0.164] at the 95% level. In addition to the approximately ~2.8 fold increase in mean p values, the median values increase by ~1.6 fold (0.057 vs. 0.035). However, the timing of the transition in P cycling identified by these two databases is different. In our larger database, the most significant transition—according to change point analysis (Figure 1; Data Repository, see below)—occurs in the late Tonian as defined by data from the Visingsö Group rather than the Ediacaran (the Visingsö Group P enrichments are from new analyses generated for this study).

# 4 | CHANGE-POINT METHODS

Change-points in the P record were identified in MatLab using the findchangepts search algorithm (MathWorks, 2019) based on the root mean square statistic. Initially, change-points were identified in raw, un-binned analyses with the maximum number allowed set at 20. During this preliminary analysis, the oldest change-point identified corresponded to a sample from the 753.3 Ma Visingsö Formation. Subsequently, initial iterations decreased the maximum number of change-points allowed (15, 10, 5, 2, 1). In all cases, except for a single change point, the oldest change-point identified corresponded to an age of 753.3 Ma. When only a single change-point was identified, this corresponded to an age of 11.608 Ma, where several samples with 1–2% P were grouped together.

Following the detection of change-points in the raw data, data were binned into 50-Ma age groups for samples aged 0 to 800 Ma, a bin from 800 to 1000 Ma, and 250 Ma age bins for samples aged 1000 to 3500 Ma. A single change-point in time-binned data was identified for both the mean and median P, between bins 16 and 17, corresponding to the 750 to 800 Ma Bin and the 800 to 1000 Ma time bin. This change-point was identified through the standard deviation and root mean square of the time-binned data. A previous change-point analysis based on formation averages further identified the 753.3 Ma Visingsö formation as representing a significant shift in the mean concentration of P. As such, we consider the identification of a 753.3 Ma change point to be a robust finding.

# 5 | THE PROTEROZOIC PHOSPHORUS CYCLE

Our updated database and new age constraints yield two significant results. First, previously observed general trends in P enrichments which point to a major late Proterozoic shift in P cycling-are robustly supported by this expanded dataset. At the most basic level, this supports our case that the observed trends are robust and suggests that the fundamental shift in mean P values observed in the Neoproterozoic is unlikely to be a function of database size (i.e., biased incorporation of geologic units into the database). Second, the critical updates to the database and age constraints presented here, through one of the most poorly sampled intervals in the record, suggest that the transition to a more modern style of P cycling occurred earlier than previously identified (Reinhard et al., 2017). In our new database, this transition occurred in the late Tonian-well before the onset of the Sturtian Snowball Earth glaciations (Macdonald et al., 2010). Although there has been an understandable focus on the Ediacaran Period as the potential time for a switch in Earth system states, the transition in the marine P cycle in the late Tonian did not occur in isolation. Recent studies suggest ocean-atmosphere oxygen levels and the complexity and scope of Earth's biosphere all appear to have undergone major transformations in the late Tonian (~800-720 Ma) (e.g., Turner and Bekker, 2016). Most obviously, there is a significant increase in carbonate carbon-isotope variability by the late Tonian, marking a shift in global carbon cycle dynamics (e.g., Halverson et al., 2009). There is also putative evidence for a significant rise in marine and atmospheric O2 during this interval (see Cole et al., 2016; Lu et al., 2017). Extensive ocean oxygenation could have removed the deep ocean inorganic P trap hypothesized for most of Earth's history (e.g., Derry, 2015; Laakso & Schrag, 2014). Under anoxic conditions, iron minerals and ferruginous shales may represent a significant trap for P (e.g., Bjerrum & Canfield, 2002; Derry, 2015; Guilbaud et al., 2020), although anoxic and euxinic conditions may also promote the sedimentary and diagentic recycling of P (e.g., Alcott et al., 2019; Poulton, 2017), highlighting our need for increased knowledge regarding the mechanistic and redox controls on P recycling. Here, we propose that late Tonian marine redox shifts may provide an explanation for the observed shift in mean sedimentary p values in the later part of the Proterozoic.

In this conceptional model, median and mean P enrichment in our database reflect the strength of P sequestration in the deep sea, P entering the photic zone, and the overall strength of the marine biological pump. As the efficiency of organic carbon burial in marine sediments and attendant fluxes of  $O_2$  to the ocean-atmosphere system increases under anoxic conditions, it is difficult to maintain low ocean-atmospheric  $O_2$  levels without limiting globally integrated marine productivity (Derry, 2015; Laakso & Schrag, 2014; Lasaga & Ohmoto, 2002). In other words, there is growing consensus (Derry, 2015; Laakso & Schrag, 2014; Ozaki et al., 2019) that with near modern rates of primary productivity and nutrient cycling, it is difficult to maintain a low oxygen ocean-atmosphere system. Recent estimates suggest atmospheric oxygen levels were well below those

of the modern Earth for much of the Proterozoic (e.g., Planavsky et al., 2018), although the exact levels remain debated. There is also tentative empirical evidence for more limited biospheric productivity in the Mesoproterozoic from sulfate mass-independent O isotope fractionations (e.g., Crockford et al., 2018). If primary productivity was dramatically reduced relative to the modern Earth, the required N fixation rates to support this level of productivity would also have been much lower, helping alleviate N (or trace element) limitation on marine productivity in anoxic oceans and maintaining a P-limited biosphere (Laakso & Schrag, 2018; Reinhard et al., 2017). In this light, Earth's progressive oxygenation requires a shift in the global P cycle, and we suggest that the sedimentary P record presented here provides evidence for just such a marked change in global biogeochemical cycling during the late Proterozoic. In contrast, it has also been suggested that an increase in sedimentary P recycling at the Archean-Paleoproterozoic boundary, the result of an influx of sulfate generated through oxidative sulfide weathering, played a fundamental role in the onset of the Great Oxidation Event (Alcott et al., 2022). In either case, the rise of oxygen in Earth's surface environments seems to be intimately associated with changes in the cycling of P.

Just as there is evidence for dramatic shifts in primary productivity throughout Earth's history, there is evidence that the composition of primary producers in the oceans has changed dramatically. Eukaryotes evolved relatively early in Earth's history (in the Paleoproterozoic or earlier); however, there is a lack of evidence for eukaryotes playing an important role in marine ecosystems until ~800-750 Ma (Brocks et al., 2017; Isson et al., 2018; Knoll, 2014; Zumberge et al., in press). The first sedimentary successions that host detectable amounts of regular steranes (a tracer for eukaryotes, particularly eukaryotic algae) are from the late Tonian Bitter Springs Group (ca. 800 Ma), with a stronger sterane signal first appearing later in the Chuar and Visingsö groups (ca. 750 Ma; Figure 1; Brocks et al., 2017; Zumberge et al., 2020). Although there are a myriad of factors that control pelagic ecosystems, in the modern oceans, marine biogeography and the structure of marine communities can be accurately estimated by making a fairly limited number of assumptions. One of these 'biological rules' is that the smallest cells have greater surface area-to-volume ratios, and thereby the highest nutrient uptake affinities, making them capable of the most rapid growth (e.g., Edwards et al., 2012; Hein et al., 1995). Therefore, from a growth and nutrient utilization perspective, smaller cells should outcompete larger cells under extreme nutrient depletion. For instance, cyanobacteria dominate the smallest size class of primary producers that thrive in the most nutrient-depleted region of the modern oceans (Moore et al., 2013). In this light, the potential for a shift in marine P levels in the late Tonian (Figure 1) was likely a key factor that led to the diversification of larger size classes of primary producers-e.g., the eukaryotic algae.

Although multiple proxies tentatively point to a mid-Tonian shift in biogeochemical cycling, this study highlights the difficulty in robustly pinpointing transitions with large geochemical datasets. Particularly, it demonstrates the need for more robust and more precise geochronological constraints and better statistical support in

determining the presence of a significant transition. This study suggests that the most significant shift in P cycling occurred before the Cryogenian Period but does not exclude the possibility that future work may subsequently identify that this transition occurred deeper in time as new data become available and better geochronological constraints are placed on Neoproterozoic successions. It should be noted that there is particularly poor sample coverage in our (and all current) sedimentary geochemical databases from 1000 to 800 Ma (Figure 1; Tables S1–S3). This low sampling density could potentially be driving the apparent late Tonian transitions and presents a key target for future work.

# 6 | CONCLUSIONS

We present updated phosphorite occurrence and shale P enrichment records spanning Earth's history. Despite more than doubling the size of previous compilations, we find that the general structure of previously reported averages in sedimentary P enrichments remains robust. This provides critical support that current shale databases faithfully capture basic aspects of the rock record and that there was a fundamental change in P cycling during the late Proterozoic. However, we provide new insights into the prospective timing of this transition—our work suggests this shift is likely to have occurred near the end of the Tonian Period and prior to the onset of the earliest period of the Sturtian Snowball Earth glaciation. Specifically, statistical tests suggest a significant transition in the Visingsö Group. Interestingly, the Visingsö Group and roughly coeval units also record a dramatic rise in eukaryotic abundance relative to bacteria from lipid biomarker assemblages (from a substantial increase in sterane/hopane ratios), supporting a link between P cycling and algal proliferation. This is broadly consistent with a model that links changes in ocean-atmosphere oxygenation to the intensity of nutrient cycling through Earth's marine biosphere.

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#### CONFLICT OF INTEREST

There is no conflict of interest.

## DATA AVAILABILITY STATEMENT

All analytical data generated or analysed during this study are included in this published article (and the Supplementary Information).

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#### SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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