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Revisiting K-Pg boundary with lithium isotopes: divergent marine and continental responses

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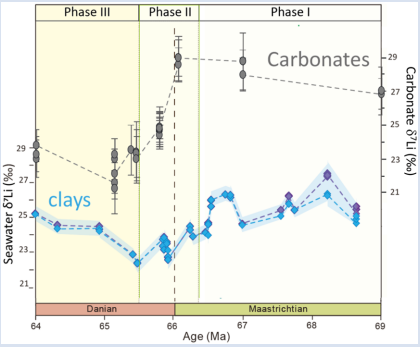
Abstract



Seawater lithium isotope records (δ^7Li) are powerful tools for investigating long term climate change and its relationship with continental silicate weathering. While most past ocean δ^7Li reconstructions rely on foraminifera-rich carbonates, we introduce a novel approach using marine authigenic clays. Our findings demonstrate that clay authigenesis is an abiotic process that fractionates Li isotopes consistently in laboratory settings and across marine sediments. We apply this method to the Cretaceous-Paleogene (K-Pg) boundary, a critical interval marked by one of Earth's five largest mass extinctions, and present a new clay derived seawater δ^7Li record (64–69 Ma). Our results, compared with those of the carbonate record, uncover a dual environmental disturbance. The clay record indicates a protracted perturbation initiated at 69 Ma, driven by gradual shifts in soils, fluvial systems, and continental weathering, which remained largely unaffected by Deccan volcanism or the meteorite impact. In contrast, abrupt fluctuations are recorded in marine carbonates at 66 ± 0.3 Ma, signaling short lived yet significant changes of oceanic carbon chemistry. These findings highlight the asynchronous behaviour of terrestrial and marine systems during this major ecological transition.

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Introduction

Reconstructing past variations in continental weathering both in rate and intensity is crucial for understanding and modelling changes in atmospheric and oceanic pCO_2 levels. Lithium isotopes (expressed as δ^7Li) in riverine and marine systems have increasingly been recognised as a robust proxy for silicate weathering intensity. Given the Li residence time in the ocean (~1.5 million years), Li isotopes are particularly well suited for investigating the global carbon cycle over extended geological periods.

Numerous studies attribute the rise in Cenozoic seawater δ^7Li values deduced from carbonates to significant changes in riverine Li flux and/or its δ^7Li composition. The riverine δ^7Li signature is primarily governed by the dissolution of silicate phases and the formation of secondary clays. A strong connection between continental silicate weathering and climate has thus been inferred from carbonate δ^7Li records (*e.g.*, Misra and Froelich, 2012; Vigier and Goddéris, 2015). On shorter time scales, such as during Oceanic Anoxic Event 2 (Pogge von Strandmann *et al.*, 2013), sedimentary carbonate records also reveal rapid δ^7Li fluctuations (≪1 Myr). These variations have been interpreted as a temperature driven acceleration of silicate weathering, subsequently impacting the carbon cycle.

At the Cretaceous-Paleogene (K-Pg) boundary (~66 Ma), δ^7Li values in foraminifera drop by ~5 ‰ in less than 400,000 years, a shift attributed to a major disruption in the marine Li budget in response to large changes in continental fluxes (Misra and Froelich, 2012). The transition from the late Maastrichtian to the Paleogene is characterised by profound environmental upheavals and one of the most devastating mass extinctions in Phanerozoic history. This catastrophic event is closely linked to two pivotal geological phenomena: the Chicxulub impact and the peak activity of the Deccan Traps volcanism (Schoene *et al.*, 2019). Both events released substantial volumes of CO_2 and SO_2 into the ocean-atmosphere system, with the impact triggering abrupt climatic shifts and the volcanism sustaining emissions over 100–150 thousand years (Schoene *et al.*, 2019; Sprain *et al.*, 2019). These processes likely induced significant changes in ocean chemistry, biogeochemical

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cycles, and global environmental conditions (Henehan *et al.*, 2019). However, the precise timing and magnitude of these events, as well as their effects, remain debated.

Throughout the Phanerozoic, marine δ^7Li records have largely relied on fossil calcium carbonates, such as foraminiferal shells, dolostones, and shallow water marine carbonates (Misra and Froelich, 2012; Kalderon-Asael *et al.*, 2021; Liu *et al.*, 2023). A fundamental assumption is that they faithfully reflect the seawater δ^7Li at the time of their formation. However, biogenic carbonates represent a negligible Li sink, and biological or environmental factors may influence their δ^7Li values (Vigier *et al.*, 2015; Chen *et al.*, 2023; Poet *et al.*, 2023). In contrast, marine clays represent the dominant oceanic Li sink. Authigenic smectite can form extensively under hydrothermal or low temperature conditions, through the alteration of volcanic glass or biogenic opal (Zhao *et al.*, 2025). Marine clays are typically Li-rich compared to any other authigenic phases and they preferentially incorporate the lighter isotope (^{6}Li). This inorganic process explains the modern open ocean δ^7Li value of 31.1 ‰ ± 0.6 ‰ (Jouini *et al.*, 2026), significantly higher than that of its primary sources (riverine δ^7Li: 23 ‰; hydrothermal fluids: 8 ‰).

In this study, we explore the potential of marine clays to serve as a robust proxy for reconstructing seawater δ^7Li values at the time of their formation. We present a new high resolution δ^7Li record spanning the K-Pg transition and discuss its implications for environmental changes during this critical period.

A Single Temperature Dependent Li Isotopic Fractionation Law

To establish marine authigenic clays as reliable proxies for palaeo-seawater δ^7Li reconstruction, we first establish the seawater-clay Li isotopic fractionation factor at temperatures representative of early marine diagenesis (T < 90 °C). We then validate this approach by comparing results with modern seawater and recent carbonate shell records (0–11 Ma), a time frame particularly relevant to the long Li residence time in the ocean.

Previous experimental studies have quantified Li isotope fractionation during smectite formation at temperatures ranging from 90 °C to 250 °C, revealing significant enrichment of ^{6}Li in clays (Vigier *et al.*, 2008; Wunder *et al.*, 2010; Hindshaw *et al.*, 2019). The clay-solution Li isotopic fractionation factor is temperature dependent. However, experiments conducted below 90 °C have yielded inconclusive results. These challenges stem from lower crystallinity and the persistent presence of exchangeable Li (Vigier *et al.*, 2008). We therefore developed an optimised protocol for low temperature conditions. This protocol includes extended experiment durations to enhance crystallinity and more efficient removal of exchangeable Li, which may have a distinct isotopic composition (Yang *et al.*, 2023). Additionally, we investigated the influence of clay type and chemical composition on the fractionation factor (see the Supplementary Information for the protocol, chemical and mineralogical analyses by XRD, FTIR and TEM).

Collectively, the new and published data reveal a consistent temperature dependent trend for the clay-solution Li isotopic fractionation factor (Fig. 1). Over a range of 25 °C to 250 °C, the fractionation factor exhibits an inverse correlation with temperature, independent of solution chemistry and of the amount of Li incorporated into the crystalline structure. As a result, the δ^7Li signature of smectite is primarily governed by two key factors: (1) temperature, and (2) the δ^7Li value of the solution. This relationship provides a theoretical framework for reconstructing water δ^7Li values from clay samples. Using

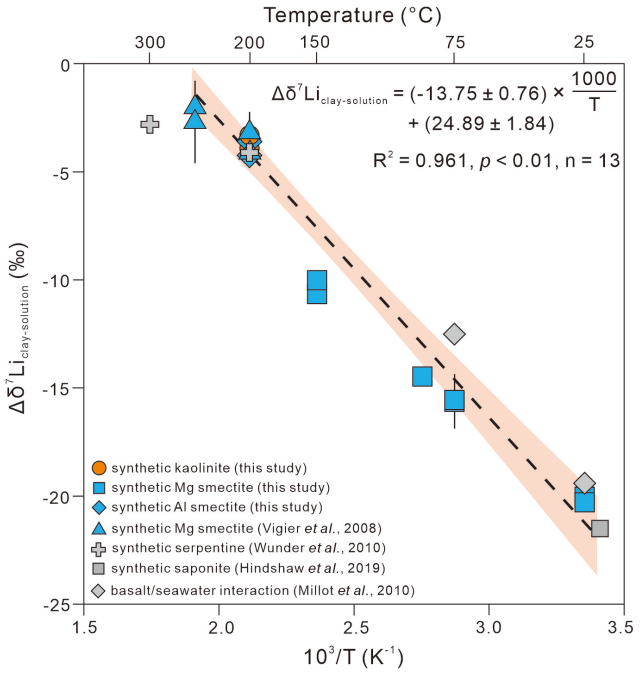

Figure 1 Temperature dependent Li isotope fractionation during clay formation ($\Delta\delta^7Li_{clay\text{-}solution} = \delta^7Li_{clay} - \delta^7Li_{solution} = 1000\ln(\alpha)$, with α denoting isotopic fractionation factor). The linear fitted line with a 95 % confidence interval is drawn based on data from synthetic smectite (blue symbols) from this study and Vigier *et al.* (2008). Data for synthetic kaolinite (this study), serpentine (grey plus), saponite (grey square), and basalt/seawater interaction (grey diamonds) are shown for comparison (Wunder *et al.*, 2010; Millot *et al.*, 2010; Hindshaw *et al.*, 2019).

weighted linear regression that accounts for analytical uncertainties, the empirical relationship derived from experimental studies can be expressed by the equation:

$$\Delta\delta^7Li_{smectite\text{-}solution} = (-13.75 \pm 0.76) \times \frac{1000}{T} + (24.89 \pm 1.84)$$

where T is in Kelvin. Although this relationship is primarily established for Mg smectite, the δ^7Li values of kaolinite and Al smectite (formed at 200 °C) with distinct chemical compositions (see Table S-1) also adhere to this trend. Additionally, published data for various secondary phases and clay mixtures are also consistent with our new experiments (Fig. 1). This unity is explained by theoretical modelling (Dupuis *et al.*, 2017), which identifies the strength of the Li-O bond and its corresponding vibrational energy as the primary factor controlling the preferential incorporation of ^{6}Li into the clay structure. The Li-O bonding dynamics can overshadow potential isotopic effects arising from variations in solution or clay chemistry. This consistent linear correlation suggests that the Li isotopic fractionation during Li incorporation into clay octahedral sites is largely independent of clay type (kaolinite or smectite) and its chemical composition. Instead, temperature emerges as the dominant control.

Authigenic Clays: a New Proxy of Past Seawater δ^7Li Variations

To assess the applicability of the relationship in Figure 1 for reconstructing palaeo-seawater δ^7Li variations, we studied smectite-rich sediments collected through international ocean drilling programmes (DSDP, ODP). These samples represent both modern (core top sediments) and "recent" (<11 Myr)

formation. Modern seawater δ^7Li values, extensively documented in the literature, are close to homogeneous (δ^7Li = 31.1 ± 0.6 ‰; Jouini *et al.*, 2026). Given the Li residence time in the ocean, seawater Li concentration and δ^7Li values can be considered stable over the past 4–5 million years. Furthermore, palaeo-environmental studies using beryllium 10 (Lenard *et al.*, 2020) also indicate steady erosion rates during the late Cenozoic, implying negligible disturbances in riverine Li flux and only minor variations in the oceanic Li budget during this period. Thus, our analysis of recent and sub-recent authigenic clays from open ocean environments provides a robust framework to verify whether the solution δ^7Li values derived from the α-T relationship in Figure 1 are valid.

The core top smectite-rich clays from Valdivia VA13/2 96BL drill core yield an average δ^7Li value of 5.6 ± 0.6 ‰ (see Table S-7). Using the equation given above and a temperature typical of the Central Pacific abyssal hill (3 °C), this corresponds to a seawater δ^7Li value of 30.5 ± 0.6 ‰ that is consistent with that of modern seawater (δ^7Li = 31.1 ± 0.6 ‰; Jouini *et al.*, 2026). Over the 0–11 Ma period, δ^7Li records from foraminifera, brachiopod, bulk carbonates and dolostones document a roughly consistent trend (Misra and Froelich, 2012; Washington *et al.*, 2020; Liu *et al.*, 2023). Note that some of these data had been corrected for isotopic vital (biological) effects during shell growth. We analysed smectite-rich marine sediments from sites 800, 1218, and 1219 in the Tropical Pacific Ocean to reconstruct seawater δ^7Li variations over the past 11 million years (see Table S-7). These samples were thoroughly characterised using scanning electron microscopy (SEM), laser ablation spectroscopy (LAS), and X-ray diffraction (XRD). Sediments with >80 % smectite content and no detectable opal or quartz were selected to minimise detrital inputs (see Supplementary Information).

Using the experimental α-T relationship (Fig. 1) and deep ocean temperature (Meckler *et al.*, 2022), we calculated the δ^7Li values of seawater in equilibrium with clays during their formation. Over the past 5 million years, smectite-rich clay fractions yield seawater δ^7Li values ranging from 29.7 ± 0.3 ‰ to 30.5 ± 0.6 ‰, consistent with the relatively stable values observed in carbonate records (Fig. 2). From 5 to 10.6 million years ago, clay derived δ^7Li values gradually decline to 28.1 ± 0.5 ‰, a trend that matches the general pattern established from carbonates. Deep sea water temperature reconstructions for this period may carry an uncertainty of 3–8 °C (Meckler *et al.*, 2022). Considering this, the propagated uncertainty for seawater δ^7Li values reconstructed from marine authigenic clays is ~1.1 ‰ (see Supplementary Information). Additionally, uncertainty may arise from structural differences between the natural and experimentally synthesised clays. The time scale of authigenic clay formation in the deep sea has recently been constrained to <40 days (Zhao *et al.*, 2025), which is close to our synthesis times at low temperatures. The faster reaction rates typically result in the formation of less crystalline minerals. Nevertheless, the complete removal of exchangeable Li and the consistent $\Delta\delta^7$Li values of −20.3 ‰ and −20.0 ‰ observed at 25 °C after 4 and 8 weeks strongly suggest that Li isotopic equilibration has been achieved in our experiments (Table S-1). We acknowledge that potential differences may exist between synthetic and natural authigenic clays, and that Li isotope fractionation during natural authigenic clay formation requires further investigation.

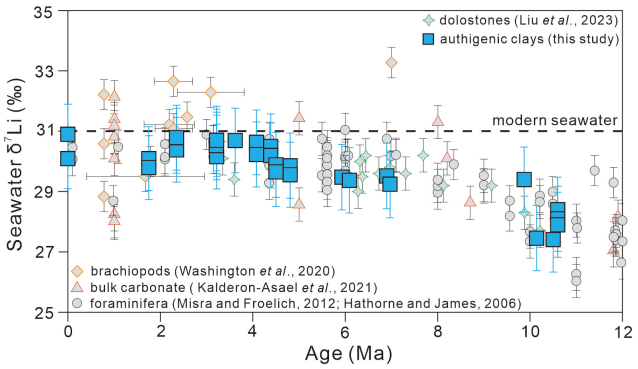

Figure 2 Record of seawater δ^7Li values over the past 11 Ma. Seawater δ^7Li values from our study are represented by blue squares. Published data for foraminifera (grey circles), brachiopods (grey diamonds with orange edges), bulk carbonate (grey triangles with red edges) and dolostones (grey stars with green edges) are shown (Hathorne and James, 2006; Misra and Froelich, 2012; Washington *et al.*, 2020; Kalderon-Asael *et al.*, 2021; Liu *et al.*, 2023).

There are advantages of using authigenic smectite-rich clays to reconstruct palaeo-oceanic δ^7Li variations. Planktonic foraminifera underwent significant biotic crises, such as observed at the Cretaceous-Paleogene (K-Pg) boundary, and the influence of vital effects on the biogenic δ^7Li values remains a subject of debate. Our clay based δ^7Li proxy opens new potentials in settings where carbonate archives are absent or ambiguous. While diagenetic effects warrant further investigation, our study indicates that their impact is minimal, likely due to the negligible influence of exchangeable Li within clays. In contrast, marine carbonates display Li concentrations several orders of magnitude lower than those in clays, suggesting that their Li isotopic compositions may be more susceptible to modification.

Ocean δ^7Li Variations across the K-Pg Boundary

During the transition from the Late Maastrichtian to the Danian, seawater δ^7Li values reconstructed from planktonic foraminifera records initially exhibit a gradual increase between 69 and 66 million years ago, followed by an abrupt and pronounced decrease of ~5 ‰ in less than 400,000 years (Fig. 3b). This rapid decline has been attributed to enhanced dissolution of continental soils and a massive increase in riverine Li flux (Misra and Froelich, 2012). However, the duration of this event is brief compared to the modern Li residence time in the ocean and the time scales required for changing the continental weathering regime. Notably, the dominance of thick lateritic soils during the Cretaceous also suggests a buffering effect against rapid changes (Vigier and Goddéris, 2015). The abrupt δ^7Li decrease in foraminifera at 66 Ma cannot be explained by a shorter oceanic Li residence time, as the expected rapid return to the preceding K-Pg values is absent. Instead, environmental disruptions like meteorite impacts or massive volcanism may have altered foraminiferal growth. For instance, temperature and pH changes can alter growth rates, which in turn affects both the entrapment of trace elements (including lithium) in crystals and the kinetics of isotopic fractionation (*e.g.*, Vigier *et al.*, 2015; Roberts *et al.*, 2018; Chen *et al.*, 2023). Species specific characteristics may have also influenced vital (isotopic) effects, as marine biodiversity underwent substantial and rapid changes during the K-Pg transition.

We reconstructed seawater δ^7Li variations using smectite-rich sediments from DSDP Site 524 in the Southwest Atlantic across the K-Pg boundary. Samples were meticulously selected based on a comprehensive analysis of their chemical and mineralogical compositions (SEM, TEM, XRD, FTIR), which consistently demonstrated the dominance of authigenic smectite and a low degree of diagenesis (see Supplementary Information). We used the ocean bottom water temperature calculated from δ^{18}O records

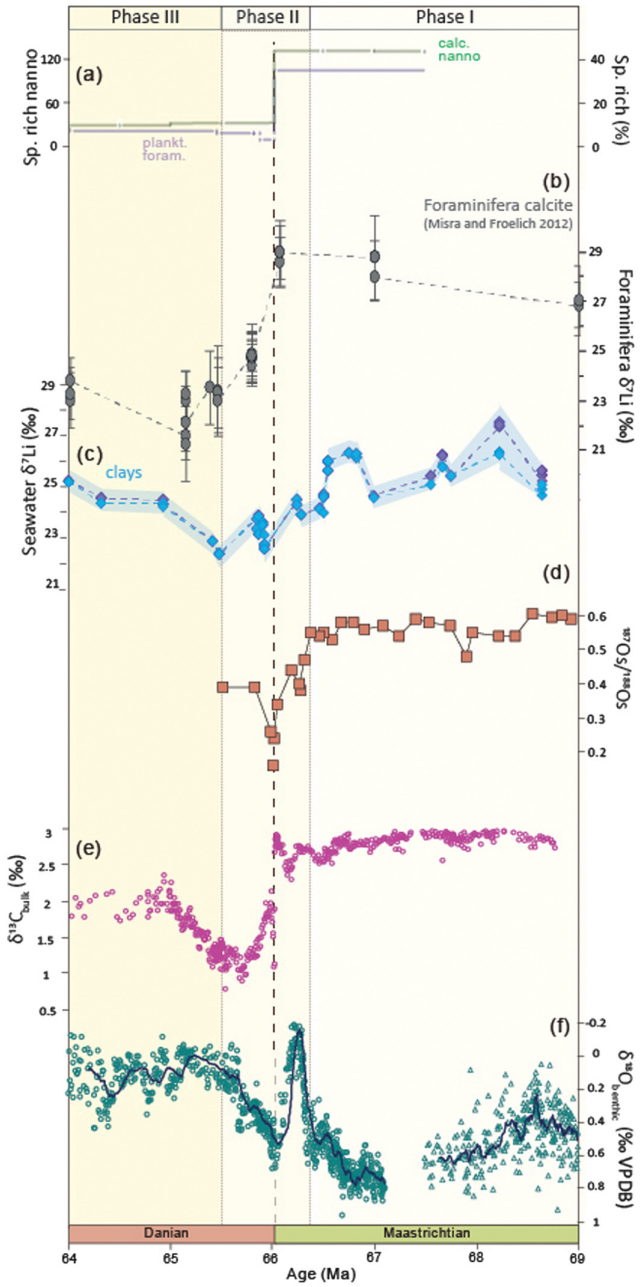

Figure 3 **(a)** Global species richness of foraminifera (pale purple) and nannofossils (green) (Hull *et al.*, 2020). **(b)** δ^7Li values of planktonic foraminifera (Misra and Froelich, 2012). **(c)** Seawater δ^7Li values reconstructed from marine clays (DSDP site 524; this study). Blue diamonds are corrected using temperatures from clumped isotopes (63–65.5 Ma; Meckler *et al.*, 2022) and bottom water δ^{18}O (65.5–68.8 Ma; Dameron *et al.*, 2017; Barnet *et al.*, 2019). Purple diamonds use bottom water δ^{18}O (63–67 Ma; Barnet *et al.*, 2019) and benthic Mg/Ca based temperatures (67–68.8 Ma; Fischer *et al.*, 2025). The shaded blue area represents external reproducibility (±0.4 ‰). **(d)** Osmium isotope record (DSDP Site 577; Ravizza and Peucker-Ehrenbrink, 2003). **(e)** Bulk carbonate δ^{13}C (IODP Site U1403; Hull *et al.*, 2020). **(f)** Benthic foraminiferal δ^{18}O records from IODP Site 1262 (green circles; Barnet *et al.*, 2019) and U1403 (green triangles; Fischer *et al.*, 2025). The K-Pg boundary is marked by a dashed line at 66.043 ± 0.040 Ma.

from the Atlantic Ocean and from Mg/Ca ratios of benthic foraminifera (Hull *et al.*, 2020; Fischer *et al.*, 2025).

From 69 to 64 million years ago, the clay derived seawater δ^7Li record can be divided into three distinct phases (Phases I, II, and III in Fig. 3). During Phase I (69–66.5 Ma), the seawater δ^7Li trend closely follows the benthic foraminifera δ^{18}O record, suggesting that the surface Li cycle was influenced by temperature. This phase is characterised by an initial increase in δ^7Li, followed by a gradual decline over 2 million years. Phase II (66.5–65.5 Ma) coincides with the peak of Deccan volcanism, as well as the Ir-rich layer and the osmium isotope decline, which are often interpreted as evidence of the Chicxulub impact (though this remains debated). This period also includes rapid and pronounced δ^{18}O fluctuations. Recent studies indicate that the main phase of Deccan volcanism lasted ~1 million years and can be further divided into four distinct mega-pulses (*e.g.*, ~66.1–66.0 Ma and ~65.9–65.8 Ma; Schoene *et al.*, 2019; Sprain *et al.* 2019). Despite these volcanic and impact related events, seawater δ^7Li derived from the clay record remains low, and only small variations are observed throughout this interval (Fig. 3). Finally, in Phase III (65.5–64 Ma), following the cessation of major Deccan volcanic activity, clay data show that the seawater δ^7Li value gradually returns to its mid-Maastrichtian level (~25 ‰).

During Phase I (69–66.5 Ma), the parallel trends in δ^{18}O and δ^7Li records suggest a long term climatic influence on continental silicate weathering, likely due to progressive soil evolution (*e.g.*, Vigier and Goddéris, 2015). Fischer *et al.* (2025) attributed the temperature decline after 68.5 Ma to reduced CO_2 levels linked to variations in continental weathering and volcanic activity. They consider that Ninetyeast Ridge volcanism triggered a warming event at ~69.2 Ma and the Mid-Maastrichtian Event, followed by cooling due to enhanced silicate weathering. The decrease in seawater δ^7Li aligns with this interpretation.

In Phase II (66.5–65.5 Ma), clay derived seawater δ^7Li reaches its lowest values with minimal fluctuation, indicating that the Deccan Traps volcanic pulses and the Chicxulub impact did not significantly perturb the riverine flux and the marine Li budget. Conversely, planktonic foraminifera exhibit a sharp δ^7Li decline (~5 ‰), likely due to vital effects influenced by changes in dissolved inorganic carbon (DIC) or pH (Vigier *et al.*, 2015; Roberts *et al.*, 2018; Poet *et al.*, 2023). Supporting this, calcium isotopes reveal significant $\delta^{44/40}$Ca fluctuations in foraminifera at the K-Pg boundary (Jouini *et al.*, 2023), tied to disruptions in ocean carbonate chemistry (CO_3^{2-}, DIC, Ω) and associated biological responses. Boron isotopes also demonstrate a rapid increase in seawater pH by ~0.4 units after the K-Pg boundary (Henehan *et al.*, 2019). Based on the proposed pH-δ^7Li relationship (Roberts *et al.*, 2018), this change is sufficient to drive the observed δ^7Li decline in foraminifera.

Overall, these findings highlight the asynchronous behaviour of terrestrial and marine systems during this major ecological transition. The contrasting records between clays and foraminifera illustrate the role of long term climate change on continental weathering. At the boundary, rapid changes in carbonates δ^7Li are consistent with large atmospheric carbon fluctuations profoundly impacting calcifier biology, with minimal impact on continental fluxes.

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Additional Information

Supplementary Information accompanies this letter at https://www.geochemicalperspectivesletters.org/article2628.

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