

# Annually resolved sediments in the classic Clarkia lacustrine deposits (Idaho, USA) during the middle Miocene Climate Optimum

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## ABSTRACT

The world-renowned Miocene Clarkia paleolake in northern Idaho (USA) is closely associated with Columbia River Basalt Group volcanism. The flood basalt dammed a local drainage system to form the paleolake, which preserved a plant fossil *Lagerstätte* in its deposits. However, the precise age and temporal duration of the lake remain unsettled. We present the first unequivocal U-Pb zircon ages from interbedded volcanic ashes at the P-33 type location, constraining the deposition to  $15.78 \pm 0.039$  Ma. Using micro-X-ray fluorescence and petrographic and spectral analyses, we establish the annual characteristics of laminations throughout the stratigraphic profile using the distribution of elemental ratios, mineral assemblages, and grain-size structures, as well as organic and fossil contents. Consequently, the ~7.5-m-thick varved deposit at the type location P-33 represents ~840 yr of deposition, coincident with the end of the main phase of Columbia River Basalt Group eruptions during the Miocene Climate Optimum. The timing and temporal resolution of the deposit offer a unique opportunity to study climate change in unprecedented detail during global warming associated with carbon-cycle perturbations.

## INTRODUCTION

Lava flows from the Columbia River Basalt Group dammed the proto-Saint Maries River in present-day northern Idaho, USA (Fig. 1A), creating the Clarkia paleolake in a steep-sided narrow valley (Fig. 1B; Smiley and Rember, 1985). Despite the Clarkia deposit being studied for almost 50 years, for its exquisitely preserved fossil biota, biomolecules, and isotope signals (Yang and Huang, 2003, and references therein), a precise temporal framework of this classic Cenozoic lacustrine deposit remains elusive due to the lack of direct radiometric ages and unresolved sedimentation rates. Early paleobotanical studies suggested that the Clarkia floras are early to middle Miocene (Smiley and Rember, 1981, 1985). The type locality of the deposit, site P-33 of Smiley and Rember (1981;  $46^{\circ}59'29.5''N$ ,  $116^{\circ}16'35.2''W$ ; Fig. 1C), contains ash layers tephrochronologically correlated to other ash

beds in the Pacific Northwest dated between 16 and 15.4 Ma (Nash and Perkins, 2012; Ladderdud et al., 2015). These ages correspond to the Miocene Climate Optimum (MCO), a global warming event marked by the maximum  $\delta^{13}C$  and minimum  $\delta^{18}O$  benthic foraminiferal values over the past 23 m.y. (Zachos et al., 2001). The MCO had high atmospheric  $CO_2$  levels attributed by some to volcanic outgassing in the Columbia River Basalt Group (Hodell and Woodruff, 1994; Foster et al., 2012; Zhang et al., 2013; Kasbohm and Schoene, 2018). An initial debate regarding whether the laminated sedimentary structures represent rapid deposition due to frequent storm events (Smiley and Rember, 1981) or reflect seasonal variations (Smith and Elder, 1985) has precluded the establishment of a time frame for the Clarkia deposits.

Precise sedimentation rates for Clarkia deposits coupled with radiometric dating on their volcanic ashes can constrain the sedimentary-volcanic-climatic relations within the Columbia Plateau. They offer a chronological framework

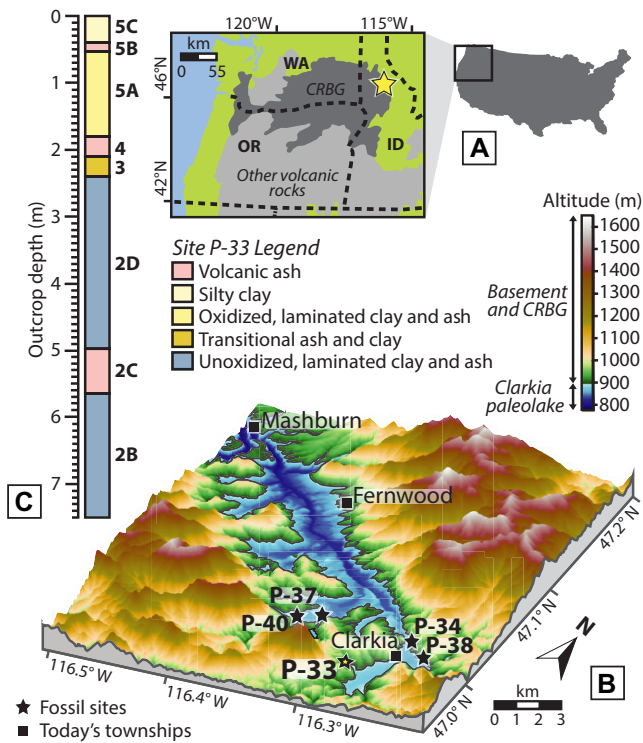
for investigating global climate and carbonate-cycle perturbations during the warmest phase of the entire Neogene. Also, establishing an age model based on the sedimentation rates at site P-33 can provide insights into the development and preservation of the world-renowned fossil *Lagerstätte* that has served as both a source of material for the discovery of ancient biomolecules and a test bed for the application of new technology over several decades (e.g., Yang and Huang, 2003; Wang et al., 2017).

We present the first U-Pb zircon ages of volcanic ash layers interbedded in the site P-33 deposit. By applying micro-X-ray fluorescence ( $\mu$ -XRF) and spectral analysis of elemental distribution, we also tie the laminated deposit to annual- to centennial-scale sedimentary processes during the MCO. Our data contextualize the Clarkia paleolake in a millennial time frame at the end of the primary phase of Columbia River Basalt Group eruptions (Kasbohm and Schoene, 2018) with elevated greenhouse gas levels and global warming (Hodell and Woodruff, 1994; Armstrong McKay et al., 2014; Kasbohm and Schoene, 2018; Sossian et al., 2020).

## METHODS

Uranium-lead (U-Pb) zircon ages of the ash layers at site P-33 (Units 2C, 4, and 5B; Fig. 1C) were obtained via laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) and chemical abrasion-isotope dilution-thermal ionization mass spectrometry (CA-ID-TIMS) at three different laboratories (University of Arizona, Tucson, Arizona, USA; Texas A&M University, College Station, Texas, USA; and China University of Geosciences, Wuhan, China). Sedimentation rates were determined using sediment block samples from Units 2B, 2D, and

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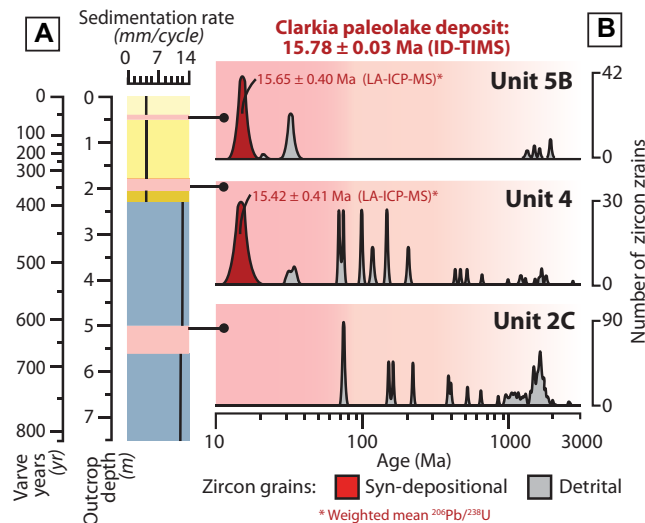
5A (Fig. 1C). Mineralogical and textural aspects of the laminated sediments were assessed via thin-section petrography and paired with  $\mu$ -XRF data. The latter were processed using spectral analysis to detect changes in the grain-size and elemental distribution along with the laminated structures. Detailed methodology and analytical procedures are provided in the Supplemental Material<sup>1</sup>.

### RADIOMETRIC AGES OF THE SITE P-33 VOLCANIC ASH LAYERS

The LA-ICP-MS U-Pb zircon ages from the tephra layers 2C, 4, and 5B are geochronological markers for site P-33 (Fig. 2; see the Supplemental Material). Concordant detrital ages range from  $74.0 \pm 1.3$  Ma to  $2533.5 \pm 17$  Ma (Unit 2C),  $14.0 \pm 1$  Ma to  $2704.5$  Ma (Unit 4), and  $15.07 \pm 0.46$  Ma to  $1914.7 \pm 14.2$  Ma (Unit 5B). Miocene volcanogenic populations, identified in Units 4 and 5B, have a weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  age of  $15.42 \pm 0.41$  Ma (mean squared weighted deviation [MSWD] = 1.8;  $n = 20$ ) and  $15.65 \pm 0.40$  Ma (MSWD = 1.9;  $n = 23$ ), respectively, which are interpreted as the time of zircon crystallization (Fig. S1 in the Supplemental Material). Zircon crystals identified as belonging to the Miocene population in Unit 5B were removed from the LA-ICP-

<sup>1</sup>Supplemental Material. Detailed methodology, supplemental figures, and separate data files containing raw U-Pb zircon data,  $\mu$ -XRF elemental ratios, and color intensity variation. Please visit <https://doi.org/10.1130/GEOL.S.14346857> to access the supplemental material, and contact [editing@geosociety.org](mailto:editing@geosociety.org) with any questions.

MS grain mount for CA-ID-TIMS analysis, which yielded a weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  age of  $15.85 \pm 0.03$  Ma (MSWD = 4.6;  $n = 14$ ). Although the  $^{206}\text{Pb}/^{238}\text{U}$  ages are over-dispersed, all analyses are concordant, and there is no clear basis for excluding analyses at either end of the distribution. We interpret the youngest, high-resolution CA-ID-TIMS age of  $15.78 \pm 0.035/0.035/0.039$  (uncertainties are listed in the order of analytical / including tracer / including decay constants) from the Unit 5B ash (analysis 12 in Table S1) as the timing of zircon crystallization and the best estimate for the age of volcanism at this level in the Clarkia deposit at site P-33. Although Unit 5B overlies Unit 4 ( $15.42 \pm 0.41$  Ma), the proposed duration



of the entire section, <1000 yr (see below), is well outside the resolution of U-Pb dating methods. The U-Pb data presented here are mainly to place the Clarkia volcanic deposits within the temporal framework of the Columbia River Basalt Group (cf. Kasbohm and Schoene, 2018). The bottommost ash layer, Unit 2C, does not yield Miocene volcanogenic ages, and ground-mass separates of the volcanic material show few rounded zircon grains (Fig. S2) and abundant glass shards. These features are strong evidence of reworking of much older material from the region during deposition.

A previous study correlated chemical signatures of volcanic glass shards and tephra of Unit 4 with the Cold Springs tuff in Nevada volcanic fields with a known age between  $15.85 \pm 0.16$  Ma and  $15.50 \pm 0.08$  Ma ( $\pm 2\sigma$  relative to Fish Canyon sanidine recalibration by Ladderud et al., 2015; Brueske and Hart, 2008). There is also a potential connection between Unit 2C and the Bully Creek Formation's tuff in Oregon ( $15.66 \pm 0.07$  Ma; Downing and Swisher, 1993; Nash and Perkins, 2012). The  $15.78 \pm 0.039$  Ma zircon age for the Clarkia deposit agrees with a previous chronology established through paleobotanical data and tephra correlations (Nash and Perkins, 2012; Ladderud et al., 2015).

### ANNUALLY RESOLVED SEDIMENTATION RATES

Except for the ash layers, sediments show laminations of fining-upward couplets throughout the succession at site P-33. Their chemical and mineral compositions, grain-size structures, and fossil content strongly suggest that these laminations represent annual rhythmicity in depositional records (i.e., varved structures; Figs. S3A–S3B). Site P-33 laminations show intercalation of dark, fine-grained, fossil-rich layers and light, coarse-grained, fossil-barren layers.

In the lower portion of the outcrop, defined as the unoxidized zone (Units 2B and 2D; Fig. 1C), petrographic thin-section analysis revealed that couplets in the dark-gray laminated clays are mineralogically very similar but vary in grain size and proportions of detrital minerals (Fig. S3C). In this zone, well-preserved fossil leaf compressions are found only in the fine-grained lamina (Fig. S3D). The fining-upward sequence suggests that the suspended sediment brought by seasonal runoff was segregated by density in a stratified water body. Coarser, denser particles are likely to deposit in the growth season (i.e., spring and summer), and finer particles are suspended in the water column, settling at lower rates in the deciduous season (i.e., autumn and winter) along with abundant deciduous plant leaves to complete an annual depositional cycle. In the upper portion of the outcrop, the oxidized zone (Units 5A to 5C; Fig. 1C; Fig. S3E) presents less fossil content than units below, mostly as leaf impressions. The cryptocrystalline texture of the ash-fall layers (Units 2C, 4, and 5B) hindered the petrographic assessment of these samples (Fig. S3F).

Spectral analysis of elemental distribution in the unoxidized and oxidized zones shows rhythmic cycles coherent with the varve structures observed in the block samples and thin sections (Fig. 3; Figs. S4–S5). Element ratios traditionally attributed to grain-size variation

(K/Ti and Rb/Zr) concur with changes in the lamination thickness, commonly seen in varves (e.g., Palmer et al., 2019). Titanium (Ti) and zirconium (Zr) represent sand-sized material input, enriched in heavy minerals, while potassium (K) and rubidium (Rb) reflect clay minerals and micas in the fine-grained fractions (Dypvik and Harris, 2001; Chen et al., 2006; Hennekam and de Lange, 2012). Power spectra of red-color intensity transecting across the varves (Color\* in Fig. 3) represent color changes in scanned images from varve structures. Organic content is detected by the Compton and Rayleigh counts (Inc/Coh in Fig. 3), in which the augmented intensity of incoherent scatter at an energy level is lower than the tube-anode radiation (Thomson et al., 2006). Higher Inc/Coh ratios correspond to the fine-grained, fossil-rich layers in the varved sediments (Fig. 3; Figs. S4–S5). These matching changes of grain size, color, and fossil content occur at every ~1 cm in the unoxidized zone and 0.5 cm in the oxidized zone (Fig. 3; Figs. S4–S5), a variation of varve thickness that is within the range of documented varved sediments (Zolitschka et al., 2015).

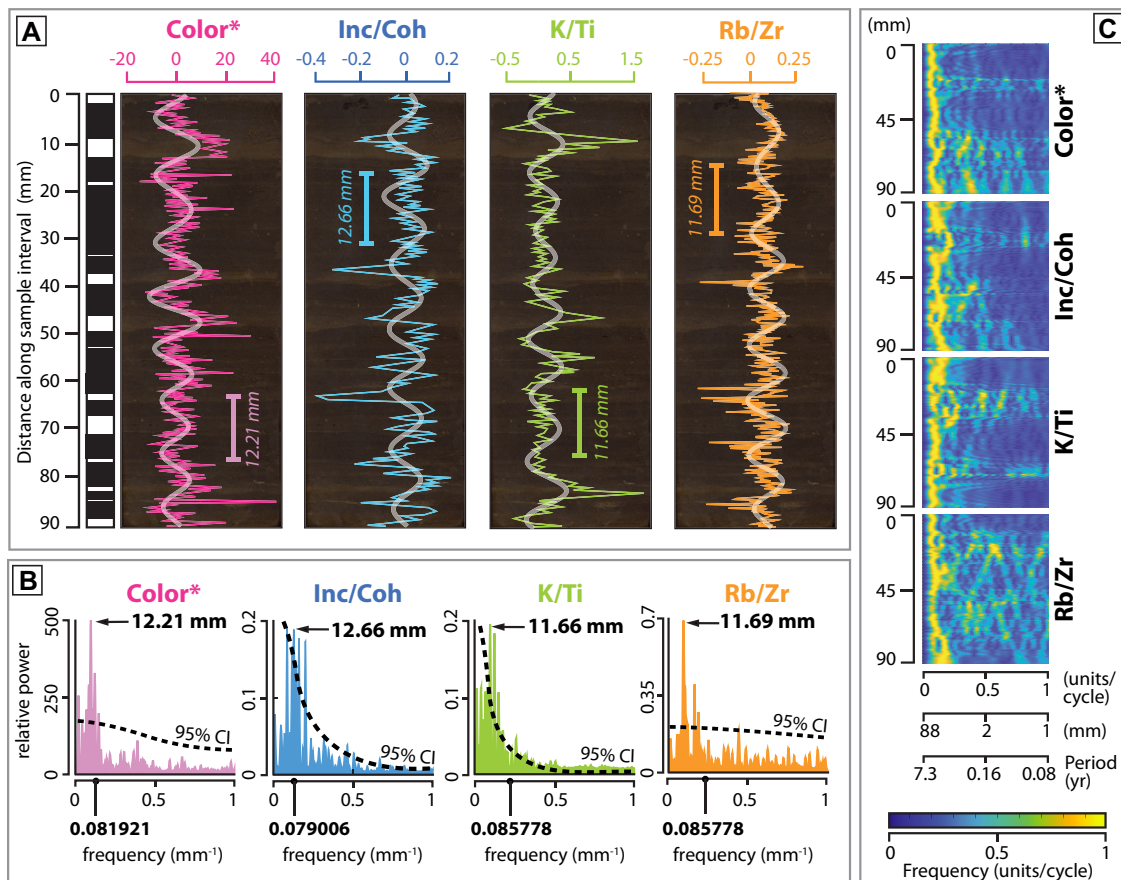
The Clarkia plant fossil assemblage, characterized by deciduous species reflecting a mixed mesophytic flora developed in a warm temperate climate with strong seasonality (Smiley and Rember, 1985), indicates a climate regime that favored deposits with seasonal rhythms. The ex-

cellent preservation of plant fossils in laminated Clarkia sediments (Smiley and Rember, 1985) and the absence of bioturbation (Smith and Elder, 1985) attest to sustained lake stratification with anoxic hypolimnion conditions (Fig. 4).

### THE CLARKIA AGE MODEL AND IMPLICATIONS

Evidence of varved structures found throughout the site P-33 and their sedimentation rate variation is crucial to reconstruct an age model associated with the change of depositional environments for the Clarkia deposit (Fig. 2A). Assuming similar sedimentation rates for the transitional Unit 3 and instantaneous deposition of autochthonous ash layers (e.g., Units 4 and 5B), the ~7.5 m sedimentary sequence at site P-33 corresponds to ~840 varve years at the end of the primary phase of Columbia River Basalt Group eruptions (Fig. 2). The U-Pb ages from the ash layers, although not precise enough to calibrate the age model, do help to place the Clarkia deposits into the overall temporal framework of Columbia River Basalt Group eruption. The age model has strong implications for Clarkia paleolake's evolutionary history, the exceptional preservation of fossil material, and the middle Miocene CO<sub>2</sub> pulse.

The interpretation of laminated Clarkia sediments as recording seasonality is favored over the storm hypothesis due to the tight coupling



**Figure 3. (A)** Frequency analysis of Color\*, Inc/Coh, K/Ti, and Rb/Zr reveals the interval of depositional cycles in Unit 2B of Site P-33 at the Clarkia deposit in Idaho, USA. All data are detrended and filtered using a bandpass. Bars represent the sedimentation cycles detected by each ratio. (B) Signals of depositional cycles stand out above the 95% confidence interval (CI) in power spectra. Arrows represent the most dominant depositional signal. (C) Fourier transform processing also demonstrates the frequency of the strongest depositional signal (light-colored vertical bands).

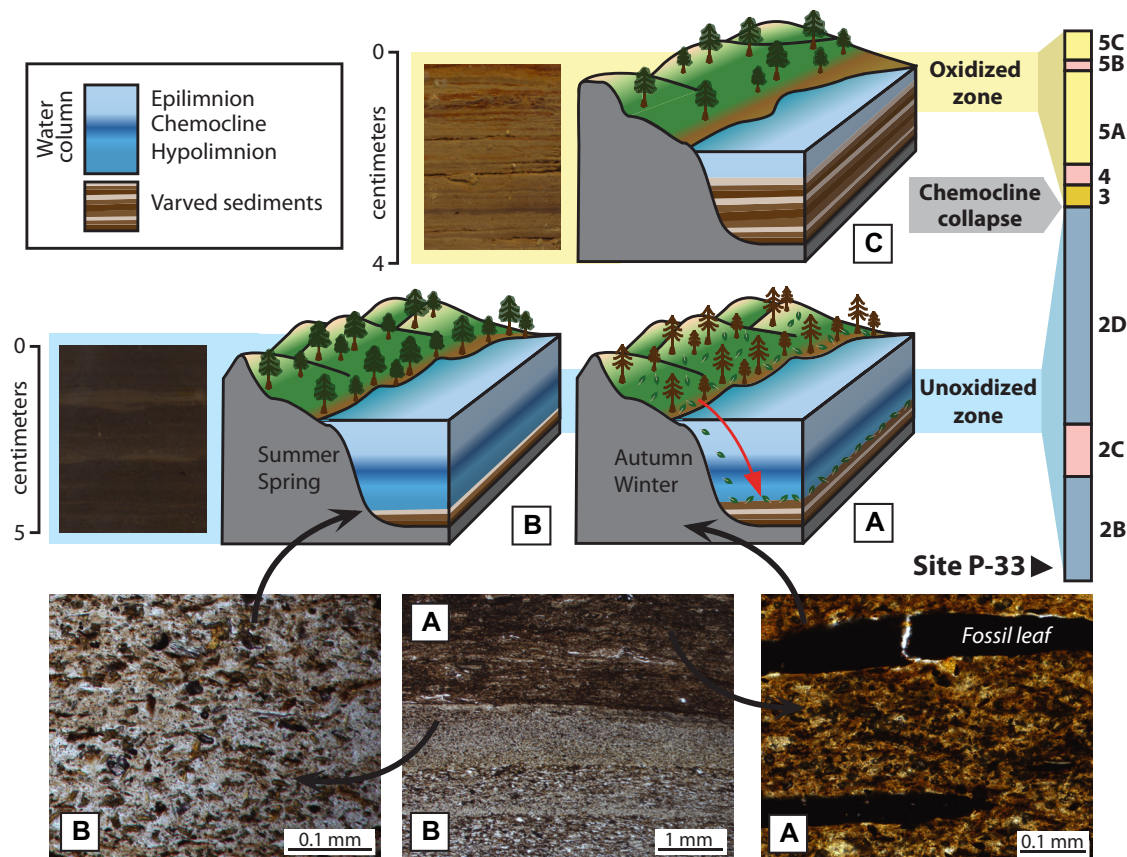


Figure 4. Evolution model of the Clarkia deposit (Idaho, USA) at site P-33. In the open-lake phase, during autumn and winter, leaves shed from surrounding trees are deposited and preserved in dark, fine-grained layers (A). These layers were interleaved with fossil-barren, coarse-grained layers deposited during spring and summer (B). As the drainage system changed (Unit 3), likely by rupture of a basalt dam holding the lake reservoir, the chemocline collapsed, leading to shallow and oxidized environment with reduced depositional rates (C). Microphotographs were taken from samples of Unit 2B. Legend for column at right is the same as in Figure 1.

between the grain-size change and fossil abundance. Abundant deciduous leaves within the fine-grained autumn sediments are consistent with rhythmic seasonal deposition of varves. While both storm deposition and dammed lakes may result in high sedimentation rates (Glenn and Kelts, 1991), irregular storms and floods tend to create turbulence that disturbs lake stratification and destroys laminations and fossil material, which contradicts the existing evidence.

Rapid burial of ancient organisms in stratified lakes is a critical controlling factor for the exceptional preservation of organic fossils (Seilacher et al., 1985; Briggs, 2003). The accumulation rate in the unoxidized zone ( $\sim 12$  mm/yr) is considered fast for varve formation in a stratified lake (Zolitschka et al., 2015), preventing the decay of organic matter and forming high-quality leaf compressions with intact tissues and biomolecules (*Konservat-Lagerstätte*). Shifts in sedimentation rates at site P-33 detected via spectral analysis of  $\mu$ -XRF data (Fig. 3; Figs. S4–S5) are consistent with the proposed sudden changes in the drainage system leading to shallowing of the Clarkia paleolake, ending the lake stratification, and transforming it into a polymictic mixing regime (Fig. 4C; Wang et al., 2017). After the reconfiguration of the lake, the sedimentation rate became much lower ( $\sim 6$  mm/yr) in an oxidized, shallow, and ventilated water column that favored the preservation of leaf

impressions (*Konzentrat-Lagerstätte*) with the same vegetation input (Wang et al., 2017).

Our new Miocene U-Pb zircon ages obtained from the Clarkia paleolake are synchronous with the Priest Rapids Member from the Wanapum Basalt of the Columbia River Basalt Group ( $15.895 \pm 0.019$  Ma; Kasbohm and Schoene, 2018). The Priest Rapids Member was formed after the peak of Columbia River Basalt Group eruption which produced a volume of basalt of  $12,175$  km<sup>3</sup> (Kasbohm and Schoene, 2018) associated with some 240–280 Pg of carbon release to the atmosphere. Vigorous volcanism is also a potential driver for the positive excursions of benthic  $\delta^{13}\text{C}$  during the MCO (Armstrong McKay et al., 2014; Kasbohm and Schoene, 2018; Sosdian et al., 2020). Fluctuations of CO<sub>2</sub> on an orbital time scale during the Miocene are large,  $>100$ – $200$  ppm, as documented by boron isotope records (Greenop et al., 2014). We speculate that some of these high-amplitude changes could be attributed to volcanic CO<sub>2</sub> release and its subsequent consumption. The better-constrained age and temporal resolution of the Clarkia deposits will aid future tests of this hypothesis. Our results provide the first absolute age constraint for the Clarkia *Lagerstätte* that confined the exceptional biomolecular preservation in annually resolved sediments at site P-33 within a millennium during the MCO, framing a unique Neogene window for future research on the

interactions of volcanism, climate, and terrestrial ecosystem.

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