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# Steady decline in mean annual air temperatures in the first 30 k.y. after the Cretaceous-Paleogene boundary

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1                   **Steady decline in mean annual air temperatures in the first 30 ka after the Cretaceous-**  
2   **Paleogene boundary**

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17  
18   **ABSTRACT**

19                   **The Cretaceous-Paleogene (K-Pg) boundary marks one of the five major mass extinctions of**  
20   **the Phanerozoic. How the climate system responded to a bolide impact and extensive volcanism at**  
21   **this time over different timescales is highly debated. Here we use the distribution of branched**  
22   **tetraether lipids (brGDGT) from fossil peats at two sites in Saskatchewan, Canada (paleolatitude ~55**  
23   **°N), to generate a high-resolution (millennial) record of mean annual air temperature (MAAT)**  
24   **spanning the last ~4 ka of the Cretaceous and first ~30 ka of the Paleogene. Our study shows that**  
25   **MAATs ranged from 16–29°C, with the highest value in the first millennia of the Paleogene. The**  
26   **earliest Paleogene averaged ~25°C—maintaining or enhancing warmth of the latest Cretaceous,**

27 followed by a general cooling to ~20°C over the following ~30 ka. No abrupt post-boundary cooling  
28 (e.g., an “impact winter”) or warming are evident in our data, implying that if such phenomena  
29 occurred, their duration was relatively short-lived, (i.e., sub-millennial). Further, no long-term  
30 impact- or volcanism-driven warming is evident. As such, hypothesized mechanisms of millennial-  
31 scale temperature change at the K-Pg boundary must be reassessed in light of our new high-  
32 resolution data. The range of temperature change observed is considerably greater than that  
33 derived from marine proxy records over the same time interval. Our findings therefore more  
34 properly place bounds on the magnitudes and durations of temperature change on land during this  
35 interval of time—the main setting in which the demise of the dinosaurs and the rise of mammals  
36 occurred.

37

## 38 INTRODUCTION

39 The Cretaceous-Paleogene (K-Pg) boundary marks one of the five major mass extinctions of  
40 the Phanerozoic. Climate change triggered by bolide impact on the Yucatán Peninsula and Deccan Trap  
41 volcanism is implicated in the mass extinction, but how different parts of the climate system  
42 responded to these triggers and at what timescales remains highly debated (e.g., Schoene et al. 2019;  
43 Sprain et al. 2019; Hull et al. 2020). Both the impact and volcanism have spurred numerous models of  
44 their effect on climate, but tests of these hypotheses are hampered, in part, by a lack of sufficiently  
45 resolved temperature constraints close to the boundary itself, especially from the terrestrial realm.

46 Bolide impact models suggest an intense heat pulse in the first minutes to hours after impact  
47 caused by the return flux of larger ejecta and flash heating of the atmosphere (Lewis et al. 1982;  
48 Melosh et al. 1990); an “impact winter” lasting months to millennia, due to atmospheric loading of  
49 dust, soot, and sulfate aerosols (Pope, et al. 1994; Bardeen et al. 2017; Brugger et al., 2017); and  
50 greenhouse heating caused by CO<sub>2</sub> from impact-volatilized carbonates (and wildfires) beginning 10<sup>3</sup>  
51 years after impact (O'Keefe and Ahrens 1989). Establishing a relationship between Deccan volcanism  
52 and climate change at the K-Pg boundary is limited by difficulties in dating the lava flows (Schoene et

53 al. 2019; Sprain et al. 2019) and constraining the amount and rate of associated CO<sub>2</sub> and SO<sub>2</sub> release  
54 (Self et al. 2006; Schmidt et al. 2016). Nevertheless, a compilation of global temperature records  
55 across the K-Pg boundary (Hull et al. 2020) shows: (1) that an episode of warming (~2°C) between 350  
56 and 200 ka before the K-Pg boundary was coincident with the onset of volcanism; (2) a subsequent  
57 decline in temperatures lasting until the boundary itself, likely driven by absorption of CO<sub>2</sub> into the  
58 ocean following the cessation of outgassing; and (3) that the first 1 Ma of the Paleocene was  
59 characterized by warming of ~1°C. Discrete reconstructions of temperature change at the K-Pg  
60 boundary at higher temporal resolution are confined to shallow-marine records (e.g., Vellekoop et al.  
61 2016). Because it is not known whether the atmosphere-ocean climate system was coupled at this  
62 time, high-resolution air temperature data are required to develop a holistic understanding of the  
63 global response to events at the K-Pg boundary, and to contextualize climate change on land—the  
64 main setting for the demise of the dinosaurs and rise of mammals.

65 In this study, we apply a lipid biomarker paleotemperature proxy (MBT<sup>5Me</sup>; Weijers et al.  
66 2007; Naafs et al. 2017) to fossil peats (coals) to reconstruct mean annual air temperatures (MAAT) at  
67 a millennial resolution across the K-Pg boundary. The coals are from two sites located 45 km apart—  
68 Wood Mountain Creek and Rock Creek West (Saskatchewan, Canada)—which lay at a palaeolatitude  
69 of 54–56°N at the time of deposition (van Hinsbergen et al. 2015; Fig. 1). Both coals contain the  
70 distinctive Ir-enriched claystone, palynological extinction, and fern-spore spike that globally mark the  
71 boundary (Sweet and Braman 1992). The Ir-anomaly claystone and δ<sup>13</sup>C<sub>org</sub> stratigraphies of the coals  
72 are used to correlate the two sites and produce a composite MAAT record. These data allow the  
73 testing, for the first time, of hypothesized terrestrial climate change at the K-Pg boundary at the  
74 timescales of 10<sup>3</sup> to 10<sup>4</sup> years.

75

## 76 **APPROACH AND METHODS**

77 The MBT<sup>5Me</sup> proxy is based on branched glycerol dialkyl glycerol tetraethers (brGDGTs), which  
78 are membrane lipids produced by bacteria. The degree of methylation of these branched tetraethers

79 (MBT) is dependent on the temperature at which the molecule was metabolized, allowing for their  
80 use as a MAAT proxy as far back as the Paleogene (e.g., Naafs et al. 2018).

81 Coal seams from Wood Mountain Creek (49°25'20"N 106°19'50"W) and Rock Creek West  
82 (49°02'20"N 106°34'00"W) were sampled contiguously (Fig. 2), and freeze-dried and powdered for  
83 geochemical analysis (Supplementary Material). Samples were solvent extracted using a MARS6  
84 microwave extraction system. The total lipid extracts (Wood Mountain Creek) or polar fractions (Rock  
85 Creek West) were dissolved in a hexane-isopropanol mixture (99:1, v/v), filtered (0.45 µm PTFE) using  
86 hexane:isopropanol (99:1, v/v), and analyzed using high performance liquid chromatography-  
87 atmospheric pressure chemical ionization-mass spectrometry (HPLC/APCI-MS) on a ThermoFisher  
88 Scientific Accela Quantum Access triple quadrupole (Wood Mountain Creek, University of Bristol) and  
89 a TSQ Quantum Access Orbitrap HPLC-MS (Rock Creek West, University of Plymouth; Vickers et al.  
90 2020). The apolar fractions were then analyzed using an Agilent 5975C gas chromatograph with an  
91 Agilent 5975C MSD mass spectrometer. Bulk organic  $\delta^{13}\text{C}$  analyses were conducted at Plymouth  
92 University using an Isoprime mass spectrometer connected to an Isoprime Microcube elemental  
93 analyzer.

94

#### 95 **TEMPERATURE DATA, CORRELATION, AND AGE MODEL**

96 The brGDGT records from both sites show similar absolute values, translating to MAATs (Naafs  
97 et al. 2017) ranging from 16.7–27.9 ± 4.7°C at Rock Creek West and 14.4–29.1 ± 4.7°C at Wood  
98 Mountain Creek (Fig. S2). The similarity in the two records, in both absolute values and trends,  
99 provides confidence in the fidelity of the individual records.  $\delta^{13}\text{C}$  values at both sites are similar (-25.5  
100 to -21.1 ‰ at Rock Creek West and -27.2 to -21.9 ‰ at Wood Mountain Creek), and show recognizable  
101 excursions with similar shapes and magnitudes that allow for reliable correlation between the sites  
102 (Fig. 2). The Ir-enriched claystone is used as the primary datum for correlation between the two sites.  
103 The inflexion points on the  $\delta^{13}\text{C}$  curves are used as secondary, independent, tie points to produce the  
104 composite temperature record (Fig. 3).

105 Notwithstanding likely differences in rates of peat accumulation and/or compaction between  
106 the two sites, it is possible to place bounds on the duration of time represented by the composite  
107 record. Absolute age determinations of two tuffs by Renne et al. (2013) at a contemporaneous coal-  
108 bearing K-Pg site (Hell Creek Marina Road, Montana), located 170 km SSW of Rock Creek West (Fig.  
109 1), yield for 1 m of coal a most likely duration of ~40 ka (with a maximum duration of up to 175 ka,  
110 and an unlikely minimum duration that equates to instantaneous deposition; Supplementary  
111 Material). However, modelling of the timing of bolide impact debris fall-out (Goderis et al., 2021), and  
112 estimates of the duration of time represented by the fern-spore spike (Clyde et al. 2016) contained  
113 within the Ir-enriched impact claystone (Sweet and Braman 1992), imply that the first MAAT datapoint  
114 above it must represent at least ~1 ka after bolide impact at the K-Pg boundary (Fig. 3). We use the  
115 value for the most likely duration (1 m of coal = 40 ka) to estimate time at our two sites, but continue  
116 to acknowledge the uncertainty outlined above (Fig. 3).

117 Given the difficulty in constraining age in such a high-resolution section, and the large  
118 paleotemperature calibration error, we treat detail in our MAAT record cautiously: we focus on the  
119 most pronounced trends and have binned the data into 10-ka intervals. Our combined record (Fig. 3)  
120 shows general warmth in the latest Cretaceous, albeit with scatter (avg. = 24.5°C ±2.7, n = 7), which  
121 increased to the highest MAATs (avg. = 25.2°C ±2.0, n = 21) in the first 10 ka after the K-Pg boundary  
122 (p = 0.13). The subsequent 10 ka interval experienced a general decline in temperatures (avg. = 22.8°C  
123 ±1.8, n = 22, p = 0.00), followed by a continued decline to 20.4°C (±2.7, n = 19, p = 0.00) over the  
124 remaining 10 ka of our record.

125

## 126 **DISCUSSION**

127 The absolute MAATs (17–29°C) and binned averages (20–25°C) are more than 10°C higher than  
128 modern MAATs at the equivalent latitude in the western interior of North America. However, these  
129 data agree with general circulation models (e.g., Sellwood and Valdes 2006; Tabor et al. 2016) and  
130 other terrestrial proxy data (Zhang et al. 2019) that indicate temperatures of 20°C in the Arctic to 30°C

131 at the southern margin of the North American western interior during the K-Pg. These new data  
132 support hypotheses of a reduced latitudinal temperature gradients at this time (Zhang et al. 2019).

133 Several observations stand out from the Saskatchewan record. Firstly, neither abrupt warming  
134 nor cooling are observed immediately post-impact. Latest Cretaceous warmth was weakly enhanced  
135 by 0.7°C between the last ~4 ka of the Cretaceous and first ~10 ka of the Paleogene (Fig. 3). Climate  
136 models suggest that the impact winter lasted only years to decades (Pope et al. 1994; Tabor et al.  
137 2016; Bardeen et al. 2017), which is below the resolution of our record. Our data therefore do not  
138 preclude an episode of post-impact cooling at the timescales indicated by these models. However, our  
139 data do not support the notion of an impact winter lasting more than a thousand years, as interpreted  
140 from the shallow-marine record of Vellekoop et al. (2014). The “winter” observed in this record occurs  
141 within the interpreted fall-out of the waning tail of a tsunami, which, irrespective of linear time-  
142 interpolation models, represents a geologically instantaneous event bed. Further, the origin of organic  
143 matter within such deposits should also be treated with caution, given the potential of such flows to  
144 entrain and transport sediment from marine and terrestrial environments.

145 There is no evidence for abrupt global warming and sustained over  $10^4$ – $10^5$  years as suggested  
146 by some models (O’Keefe and Ahrens 1989), and purportedly supported by marine (e.g., MacLeod et  
147 al. 2018; Taylor et al. 2018) and terrestrial (Lyson et al. 2019) proxy data. Though the highest individual  
148 MAAT datapoints of our record occur in within the first 10 ka of the Paleogene, our data suggest this  
149 interval was preceded by general warming in the last 5 ka of the Cretaceous (Fig. 3). This trend is also  
150 evident in the relatively high-resolution benthic marine record of Barnet et al. 2017). The “abrupt” 3–  
151 5°C of warming in existing records (MacLeod et al. 2018; Taylor et al. 2018; Lyson et al. 2019) may be  
152 an artifact of low-resolution spot-sampling. In those studies, a there is a temporal gap of no less than  
153 ~3 ka before the last Cretaceous datapoint and the K-Pg boundary itself. The data from these studies  
154 are consistent with our results, insofar as there is a temperature increase across the K-Pg boundary,  
155 but we posit that these values represent the culmination of a longer-term warming trend—pre-dating  
156 the bolide impact—and cannot be considered “abrupt”. Although bolide impact is inextricably linked

157 to the mass extinction, its effect on terrestrial temperatures appears to have been modest at most;  
158 either weakly enhancing an already existing latest Cretaceous warmth or failing to interrupt pre-  
159 existing trend of latest Cretaceous warming.

160 Early Paleogene warmth cannot be said to be “sustained” at the timescales implied by models  
161 of global warming caused by impact-volatilization (O’Keefe and Ahrens, 1989) in our data. Irrespective  
162 of the most likely (30 ka) or longest possible (130 ka) duration for our earliest Paleogene record, we  
163 document a steady decline in MAAT in the tens of thousands of years that follow the K-Pg boundary,  
164 most likely driven by the sequestration of carbon by biomass recovery in the wake of the mass  
165 extinction (e.g., Lyson et al. 2019). The marine  $\delta^{18}\text{O}$  records of Barnett et al. (2019) from the South  
166 Atlantic and MacLeod et al. (2018) from the Tethyan K-Pg type section at El Kef (Tunisia) document 1-  
167 2°C of cooling over ~30 Ka, and up to 5°C of cooling over up to 200 ka respectively. The timescales of  
168 cooling in these studies are within the error margin of the this study and one-another. From the  
169 terrestrial realm, lower-resolution qualitative palaeobotanical studies from the western interior of  
170 North America that indicate lower temperature relative to the latest Cretaceous mean within the first  
171 ~100 ka of the Paleogene (Davies-Vollum and Wing 1998; Wilf et al. 2003; Peppe 2010; Lyson, et al.  
172 2019). Overall, data converge on the notion of temperature maxima immediately following the K-Pg  
173 boundary, followed by several tens of thousands of years, at least, of cooling in the earliest Paleogene.  
174 Mechanistically, MAAT decline over these timescales were most likely driven by the sequestration of  
175 carbon linked to the rapid recovery of biomass at this time (e.g. Lyson et al., 2019), irrespective of the  
176 source (bolide impact-associated volatilization and wildfires or Deccan volcanism) of  $\text{CO}_2$ .

177 Finally, relative to comparable marine records, the magnitude (up to 5°C in the binned data)  
178 of MAAT change in our data is greater than that of Barnett et al. (2017), but similar to that of MacLeod  
179 et al. (2018). Owing to the different thermal masses of land and ocean, differences in the magnitude  
180 and duration of temperature change in response to external drivers might be expected (Sutton et al.  
181 2007). Understanding and quantifying these differences is limited by the error margins of age models  
182 and temporal resolution of existing data. As such, the generation of directly comparable, time-



183 calibrated marine and terrestrial archives is critical to a holistic understanding of the entire ocean-  
184 atmosphere system.

185

## 186 **CONCLUSION**

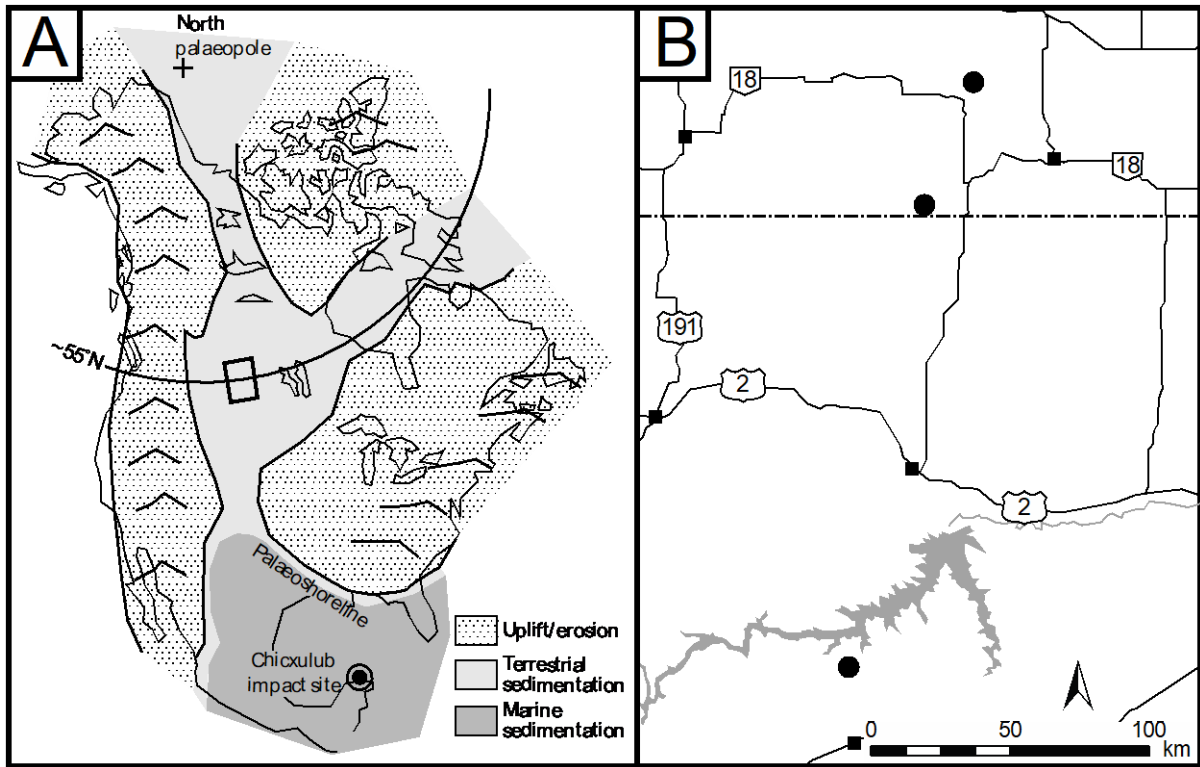
187 We observe similar absolute values and trends of terrestrial temperature change across the  
188 K-Pg boundary at two closely spaced fossil peats that were located at ~55°N in what is now southern  
189 Saskatchewan. This similarity has permitted the generation of a millennial-scale, composite MAAT  
190 record through this critical time interval. MAATs peaked at ~25°C within the first ~10 ka of the  
191 Paleogene, which we tentatively interpret to represent the weak enhancement of warmth from the  
192 last ~4 ka of the Cretaceous. Peak warmth was followed by ~5°C cooling over the following 20 ka, most  
193 likely driven by the sequestration of carbon by biomass recovery in the wake of the mass extinction.  
194 We observe no abrupt “impact winter” nor a spike in temperature immediately following the  
195 boundary. If such phenomena occurred, their duration was below the resolution of our record: ~1 ka.  
196 Our data highlight the value of peat as a sensitive, high-resolution palaeotemperature archive, and  
197 place new bounds on the magnitude and rate of millennial-scale MAAT change in the terrestrial  
198 realm—the main setting for the demise of the dinosaurs and the rise of mammals.

199

## 200 **ACKNOWLEDGEMENTS**

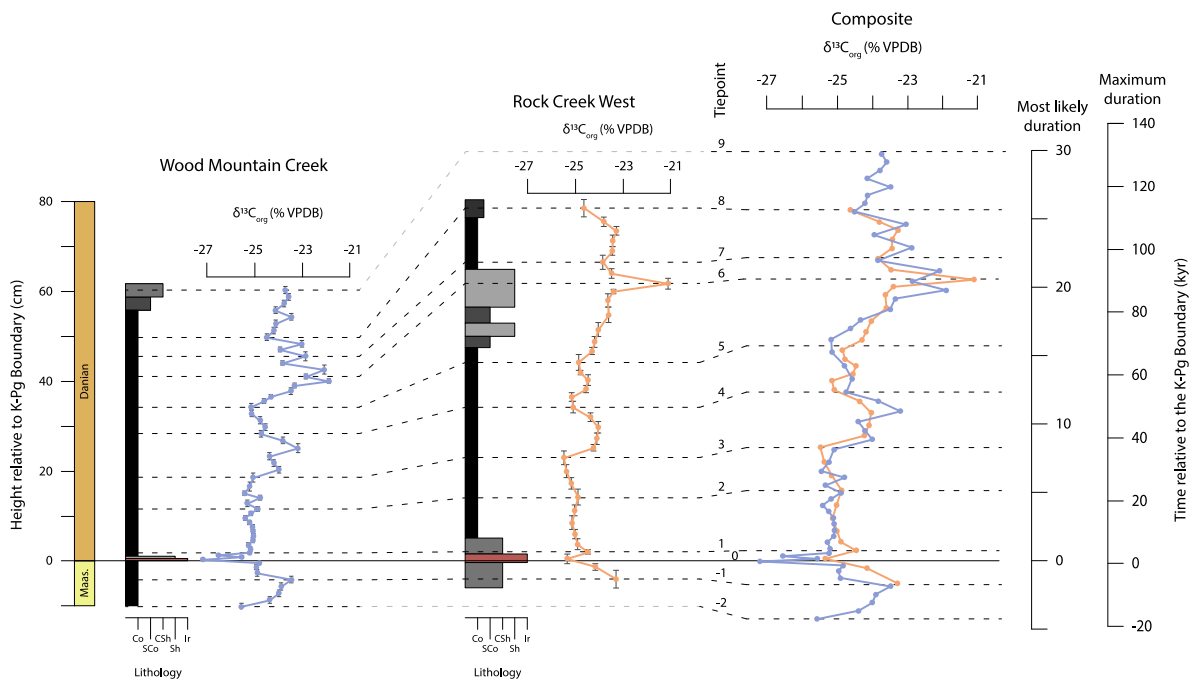
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206 and Ian Bull (University of Bristol) for technical assistance.

207



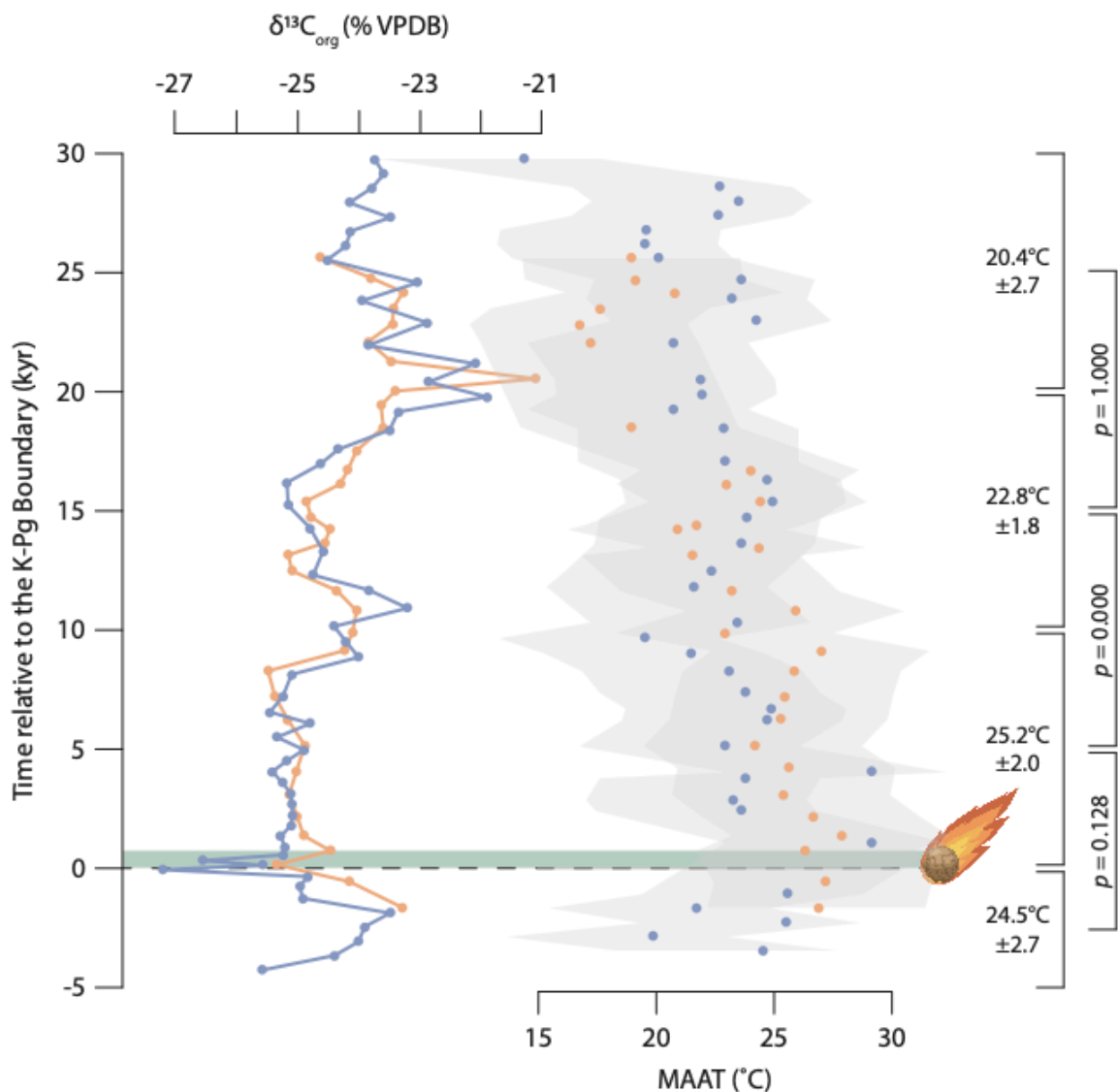
208

209 **Figure 1.** [A] Cretaceous-Paleogene paleogeography of North America (based on Smith et al. 1981 and  
 210 van Hinsbergen et al. 2015) and study area in [B] outlined by black box. [B] Inset of study area with  
 211 modern geography showing the locations of sites named in this study. We use samples from Wood  
 212 Mountain Creek and Rock Creek West.



213

214 **Figure 2.**  $\delta^{13}\text{C}_{\text{org}}$  records from Wood Mountain Creek (blue) and Rock Creek West (orange),  
 215 Saskatchewan. plotted against height above the K-Pg boundary. Vertical error bars show the  
 216 stratigraphic range of each sample. Dashed lines show chemostratigraphic ( $\delta^{13}\text{C}$ ) correlation tiepoints,  
 217 numbered 1–9 (Supplementary Table S3). Solid line shows the Ir-claystone K-Pg datum. The composite  
 218 record is plotted against time, with the most likely (40 ka/m of coal) and maximum (175 ka/m of coal)  
 219 durations shown. On the lithology log, Co = coal, ShC = shaley coal, CSh = coaly shale, Sh = shale, and  
 220 Ir = iridium-enriched claystone.



221  
 222 **Figure 3.** Composite  $\delta^{13}\text{C}_{\text{org}}$  and mean annual air temperature (MAAT) records from Wood Mountain  
 223 Creek (blue) and Rock Creek West (orange) plotted against time relative to the K-Pg boundary (using

224 *the most likely duration). The dashed line represents the K-Pg boundary. Average MAATs  $\pm$  1 standard*  
225 *deviation for each temporal bin are shown to the right with P values of t tests comparing means (Table*  
226 *S5). The green box must represent at least  $\sim$ 1 ka after bolide impact at the K-Pg boundary.*

227

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345

346

## 347 SUPPLEMENTARY MATERIAL

### 348 Site description and sampling

349 The Ferris Coal at Wood Mountain Creek (49°25'20"N 106°19'50"W) and Rock Creek West  
350 (49°02'20"N 106°34'00") has been shown to span the K-Pg boundary, based on the distinctive 1–2 cm  
351 thick pink- to buff-coloured Ir-enriched claystone contained in each section (Nichols et al. 1986; Sweet  
352 and Braman 1992). The claystone is also coincident with a palynologically defined extinction of  
353 Cretaceous flora (Sweet and Braman 1992). At the time of the K-Pg boundary, the sites were (1) peat  
354 mires accumulating in the foreland basin of the ancestral Rocky Mountains; (2) inland, approximately  
355 equidistant between the shorelines of the (proto) Gulf of Mexico and the Boreal Sea (now the Arctic  
356 Ocean; Fig. 1); (3) at a palaeolatitude of approximately 54–56°N (van Hinsbergen et al. 2015); and (4)  
357 at negligible altitude, as evidenced by the persistence of marine conditions in the Maastrichtian  
358 Bearpaw Formation some tens of metres below the K-Pg boundary (Sweet and Braman, 2001).  
359 Vitrinite reflectance data ( $R_o$ ) values for the Ferris Coal at the sites are <0.38%, indicating lignite rank  
360 (Bustin 1991).

361 At both sites, the entire coal seam was contiguously sampled. Wood Mountain Creek was  
362 sampled in 2011 (56 samples). Sampling methodology and  $\delta^{13}C_{org}$  analyses were previously published  
363 in Jerrett et al. (2015) and the latter are re-reported in Supplementary Table S1. Remaining material  
364 from the Wood Mountain Creek site were used for brGDGT analysis in this study. Rock Creek West  
365 was sampled in 2019 (36 samples), using the method described in Jerrett et al. (2015).

366

### 367 Geochemical analysis and brGDGT method validation

368 All samples were freeze-dried and finely ground using a granite pestle and mortar prior to  
369 geochemical analyses.

370

371 *Wood Mountain Creek*

372 Approximately 0.5 g of sediment was solvent-extracted using ~20mL of DCM/methanol (9:1,  
373 v/v) in a Milestone Ethos Ex microwave extraction system at the Organic Geochemistry Unit (OGU) at  
374 the University of Bristol. Temperature in the microwave was programmed to increase linearly from  
375 room temperature to 70°C over 10 min at which it was held for 10 min, and then cooled to 25°C over  
376 20 minutes. The total lipid extract (TLE) was dissolved in hexane:propanol (99:1, v/v) and filtered  
377 (0.45 µm PTFE) prior to analysis.

378 Samples were analysed for their GDGT content by high-performance liquid  
379 chromatography/atmospheric pressure chemical ionization–mass spectrometry (HPLC/APCI-MS) at  
380 the University of Bristol using a ThermoFisher Scientific Accela Quantum Access. Normal-phase  
381 separation was achieved using an Alltech Prevail Cyano column (150 mm x 2.1 mm; 3 µm internal  
382 diameter). Isocratic elution for 5 min using hexane:isopropanol 99:1 (v/v) was followed by a linear  
383 gradient to 1.8% isopropanol (45 min). Selective ion monitoring (SIM) was used to detect the [M+H]<sup>+</sup>  
384 ions of the following masses: *m/z* 1050, 1048, 1046, 1036, 1034, 1032, 1022, 1020, 1018, 744). The  
385 average standard deviation for duplicate MBT' measurements of an in-house standard was 0.026  
386 units. The data are reported in Supplementary Table S1. The MBT'5Me index was calculated following  
387 De Jonge et al. (2014):

$$388 \quad \text{MBT}'5\text{Me} = \frac{[\text{Ia}] + [\text{Ib}] + [\text{Ic}]}{[\text{Ia}] + [\text{Ib}] + [\text{Ic}] + [\text{IIa}] + [\text{IIb}] + [\text{IIc}] + [\text{IIIa}]}$$

389 The peat specific calibration ( $\text{MAAT}_{\text{peat}}$ , Naafs et al. 2017) was used to convert MBT'5Me values into  
390 mean annual air temperatures:

$$391 \quad \text{MAAT}_{\text{peat}} (\text{°C}) = 52.18(\text{MBT}'5\text{Me}) - 23.05$$

392

393 *Rock Creek West*

394 For  $\delta^{13}\text{C}_{\text{org}}$  analysis, samples were oven dried (30°C, 24 h), and decarbonated using hydrochloric  
395 acid (10% v/v) until any visible reaction had ceased. The samples were then repeatedly washed with

396 deionised water until a neutral solution was obtained, and oven dried again (30°C, 24 h). Carbon  
397 isotope analyses were conducted at the University of Plymouth using a Thermo Scientific Delta V  
398 Advantage. Carbon-isotope ratios are expressed using the internationally accepted per mil (‰)  
399 standard notation relative to the Vienna Peedee belemnite (VPDB) standard (Supplementary Table  
400 S1). Instrument calibration was achieved using three international standards: USGS 40 (l-glutamic acid,  
401  $\delta^{13}\text{C} = -26.389\text{‰}$ ), USGS 24 (graphite,  $\delta^{13}\text{C} = -16.049\text{‰}$ ), and IAEA CH-7 (polyethylene,  $\delta^{13}\text{C} = -$   
402  $32.151\text{‰}$ ). The standard deviation on replicates in run analyses of the USGS 40 standard was  $\pm 0.12\text{‰}$ .

403         Approximately 1 g of sediment was solvent-extracted using 20mL of DCM/methanol (9:1, v/v)  
404 in a microwave assisted reactor system (MARS 6, CEM) at the University of Manchester following the  
405 same protocol as for Wood Mountain Creek. The TLE was separated into polar and apolar fractions by  
406 column chromatography, using hexane/DCM (9:1, v/v) and DCM/methanol (1:1, v/v) respectively as  
407 the eluents, and  $\text{Al}_2\text{O}_3$  as the stationary phase. The polar fraction was re-dissolved in hexane/propanol  
408 (99:1, v/v) and filtered using a 0.45  $\mu\text{m}$  PTFE filter prior to analysis.

409         Analysis of GDGTs was carried out using a Dionex LPG-U3400(SDN) UHPLC liquid  
410 chromatography system and Thermo Scientific Q Exactive Focus mass spectrometer with Atmospheric  
411 Pressure Chemical Ionisation (APCI). For the determination of MBT'5Me, the method of Hopmans et  
412 al. (2016) was adapted to this high-performance instrument employing a single Waters Acquity UPLC,  
413 BEH HILIC 1.7  $\mu\text{m}$  (150 x 2.1 mm, 1.7  $\mu\text{m}$ ) column and precolumn at 40°C. Using solvents A (hexane)  
414 and B (9:1 hexane:isopropanol v/v) and a flow of 600  $\mu\text{l min}^{-1}$ , the mix started at 5% B (isocratic from  
415 0–3 min), rising to 18 % B at 5 min (isocratic 5–10 min), rising to 35% B at 15 min and 100% B at 17.4  
416 min, with 2.6 min re-equilibration time, which slightly shortened the analysis time. APCI was carried  
417 out in positive polarisation mode, at a capillary temperature of 275°C. Masses were scanned from  $m/z$   
418 200 to 2000, and resolved to 70,000 at  $m/z$  200, with mass calibration carried out externally in electro  
419 spray ionisation mode, using auto-calibration Pierce LTQ Velos ESI positive ion calibration solution (n-  
420 butylamine, caffeine, MRFA, and Ultramark 1621). GDGTs were identified based on retention times  
421 and accurate masses: using 1022.00967 (brGDGT-Ia), 1019.99402 (brGDGT-Ib), 1017.97837 (brGDGT-

422 lc), 1036.02532 (brGDGT-IIa+IIa'), 1034.00967 (brGDGT-IIb+IIb'), 1031.99402 (brGDGT-IIc+IIc'),  
423 1050.04097 (brGDGT-IIIa+IIIa'), 1048.02532 (brGDGT-IIIb+IIIb'), 1046.00967 (brGDGT-IIIc+IIIc'), and  
424 1292.24442 (crenarchaeol). Integration was carried out using Xcalibur 4.2 using QuanBrowser  
425 integration and data management. The data are reported in Supplementary Table S1.

426

#### 427 *Comparison of HPLC data from University of Bristol and Plymouth University*

428 To allow comparability between the samples analysed at the University of Bristol and the  
429 University of Plymouth, in particular for 6-me separation, a set of standards and unrelated,  
430 representative samples, were analyzed at Bristol and Plymouth (marine standard and EH-8, Lengger  
431 et al. 2018). Further, as the HPLC-APCI-MS method for GDGT analyses was shortened to allow higher  
432 sample-throughput, taking advantage of the UHPLC-Orbitrap-MS system at the University of  
433 Plymouth, a subset of standards and samples were run on both methods ('long' for standard method  
434 acc. to Hopmans et al. (2016), 'short' method for 20 min method: shown are marine standard and EH-  
435 8 as described in Lengger et al. (2018); RCW-26 this work), to determine reproducibility  
436 (Supplementary Fig. S1). The 'short' method is a variation of Hopmans et al. (2016), modified to run  
437 on one HPLC column (not two), and completes a sample run in 20 minutes (described in Vickers et al.  
438 2020). As a result, the resolution is slightly compromised when compared to Hopmans et al. (2016),  
439 though the resolution of the 'short' method is comparable to the method at the University of Bristol  
440 (Supplementary Fig. S1). The results from both 'short' and 'long' methods are very similar, with similar  
441 MBT' and MBT'5Me values calculated for both (Supplementary Table S2). In addition, the 'long'  
442 method was also implemented at Plymouth and used for cross-calibration.

443

#### 444 *Fidelity of the MAAT record*

445 Where possible, all GDGT analyses were duplicated (Supplementary Table S1). A minority either could  
446 not be analysed a second time or showed a standard deviation >1 so were eliminated from the study

447 (Supplementary Table S1). Shale samples were also excluded as the proxy is not meant for non-coals.  
448 To explore the fidelity of our GDGT-reconstructed temperatures, we examined the distributions of  
449 other GDGTs and biomarkers indicative of depositional environment. Previous work has shown that  
450 major changes in depositional setting, as inferred from reconstructed pH, can bias temperature  
451 estimates (Weijers et al. 2011; Inglis et al. 2019). The  $CBT_{peat}$  index was calculated following De Jonge  
452 et al. (2014):

$$453 \quad CBT' = \log \frac{Ic + IIa' + IIb' + IIc' + IIIa' + IIIb' + IIIc'}{Ia + IIa + IIIa}$$

454 The peat specific calibration ( $CBT_{peat}$ , Naafs et al. 2017) was used to convert CBT'5 values into pH:

$$455 \quad pH = 2.29(CBT_{peat}) + 8.07$$

456 In these sections,  $CBT_{peat}$ -pH does vary (from 4.4 to 6.8; Supplementary Table S1), though at  
457 neither Rock Creek West nor Wood Mountain Creek do these changes correlate with temperature  
458 changes. At Rock Creek West there is a decline in pH from 7 to 6.5 from the basal (Cretaceous) parts  
459 to the lowermost (Paleogene) parts of the coals, and thereafter vacillates between 5 and 6. At Wood  
460 Mountain Creek, the pH is highly variable in the Cretaceous (from 4.4 to 6.3), but in the earliest  
461 Paleogene declines from 6.5, and thereafter varies between 5 and 6. The patterns are consistent with  
462 palynological (Sweet and Braman 1992) and petrographic (Sweet and Cameron 1991; Jerrett et al.  
463 2015) data, that imply that the coal at both sites represents a hydroseral succession upwards from  
464 (typically more alkaline) ponded, disconnected forested rheotrophic mires in-filling topographic  
465 hollows, to (typically more acidic) aerially expansive herbaceous, possibly ombrotrophic mires.  
466 However, we observe no correlation between pH and temperature, and no change in CBT associated  
467 with the post-boundary cooling. Although the variations in CBT-derived pH are larger than those  
468 observed in other settings (e.g., Lauretano et al. 2021), they are smaller than the previous studies  
469 where pH was inferred to bias temperature estimates (Weijers et al. 2011; Inglis et al. 2019). Some of  
470 the smaller variations in pH do coincide with changes in reconstructed temperature, so we focus on  
471 the main trends in MAAT.

472 To further explore the wider biomarker deposition at these sites, we determined the bacterial  
473 hopane isomerisation ratio; isomerisation at the C<sub>17</sub> and C<sub>21</sub> positions (from the 17β,21β(H) to the  
474 17α,21β(H) configuration) normally increases with thermal maturity, but this process is accelerated,  
475 especially for the C<sub>31</sub> hopane, under acidic conditions in wetlands, hence it is not a useful maturity  
476 indicator under such conditions (Inglis et al. 2019).

477 The apolar fractions were analysed using an Agilent 7890A gas chromatograph (GC) interfaced  
478 to an Agilent 5975C MSD mass spectrometer (MS) operated in electron ionisation scan/SIM mode  
479 (scanning range, *m/z* 50–600; SIM masses used: *m/z* 57, 66, 191 and 205; ionisation energy, 70 eV;  
480 solvent delay of 2.5 min) using helium as the carrier gas at a constant flow (1 mL/min). The GC was  
481 equipped with an Agilent 7683B auto-sampler and programmable temperature vaporization (PTV)  
482 inlet. The samples were dissolved in hexane prior to injection, injected using pulsed split-less injection  
483 (1 μL; inlet pressure of 25 psi for 0.75 min), and separated on a Zebron ZB-5MS capillary column  
484 (Phenomenex; length 30 m; 250 μm ID, 0.25 μm film thickness). The heated interface (MSD transfer  
485 line) and PTV temperatures were set to 280°C, the mass source, at 230°C and the mass spectrometer  
486 quadrupole at 150°C. The samples were injected at 50°C and the oven was programmed to ramp to  
487 130°C at 20°C/min and then to 310 °C at 6 °C/min, at which it was kept isothermally for 15 min.  
488 Compounds were identified by comparison of their molecular mass (*m/z*) with The National Institute  
489 of Standards and Technology (NIST) library. Quantitative data were determined by comparison of  
490 individual peak areas with a known concentration of the internal standard deuterated tetracosane,  
491 added prior to analyse and pH was calculated using the C<sub>31</sub> hopanes following Inglis et al. (2018) :

492 
$$\text{pH} = 5.22 \left( \frac{\beta\beta}{\alpha\beta + \beta\beta} \right) + 3.11$$

493 Overall values of the C<sub>31</sub> ββ/(ββ+αβ) hopane ratio range between 0.06 and 0.42, consistent with an  
494 acidic peat forming environment. Another unusual feature of these sections is the lack of 6'-methyl  
495 brGDGTs in nearly all samples despite reconstructed pH above 5; at such pH and especially above pH  
496 6, 6'-methyl brGDGTs do occur in modern mineral soils and peatlands (Naafs et al. 2018). This is not

497 an artefact of the analytical method, as we cross-checked with the “long” method in Plymouth  
498 (Supplementary Fig. S1). Therefore, we interpret our MAAT record cautiously, focusing primarily on  
499 the most pronounced variations which are not correlated with changes in CBT. That is, peak  
500 temperatures immediately above the K-Pg boundary, and the long-term decline in MAATs in the  
501 Paleogene part of the record.

502

### 503 **Age Model**

504 *Correlation:* First order correlation between the two sites is based on the occurrence of the  
505 distinctive Ir-bearing claystone that is also palynologically enriched in fern spores (Sweet and Braman,  
506 1992), and marks the base of the Paleogene. Its base is used as a horizontal datum in Figure 2 and  
507 Supplementary Figure S2. Jerrett et al. (2015) interpreted these coals to represent, at least in part,  
508 small, disconnected rheotrophic mires, readily subject to local autogenic clastic input. Consequently,  
509 a lithostratigraphic approach would not be appropriate for the generation of other  
510 chronostratigraphic tie-points in this case. Instead, secondary tiepoints are provided by inflexion  
511 points on their respective  $\delta^{13}\text{C}_{\text{org}}$  stratigraphies of the two sites (Tiepoints 1–9; Figs. 2 and Supp. Fig.  
512 S2). These can be interpreted as representing a more regional stratigraphic signal relating to changes  
513 in the carbon isotopic composition of the atmosphere the plants in the peats were metabolising (Arens  
514 and Jahren, 2000). This correlation is consistent with an independent correlation between the two  
515 sites based on petrographic criteria by Jerrett et al. (2015).

516 *Timescales:* Estimations of the duration of time represented by such short stratigraphies are  
517 difficult. Broad estimates of the duration of time represented by the two coal seams, however, can be  
518 estimated from the absolute age determinations of Renne et al. (2013) at the coal-bearing K-Pg site at  
519 Hell Creek Marina Road (Montana), located 170 km SSW of Rock Creek West (Fig. 1). The stratigraphy  
520 of the site is represented by 160 cm of coal, which directly overlies the Ir-enriched claystone marking  
521 the K-Pg boundary. The coal contains two tuffs termed Z2 and Z1, 80 cm and 120 cm above the K-Pg



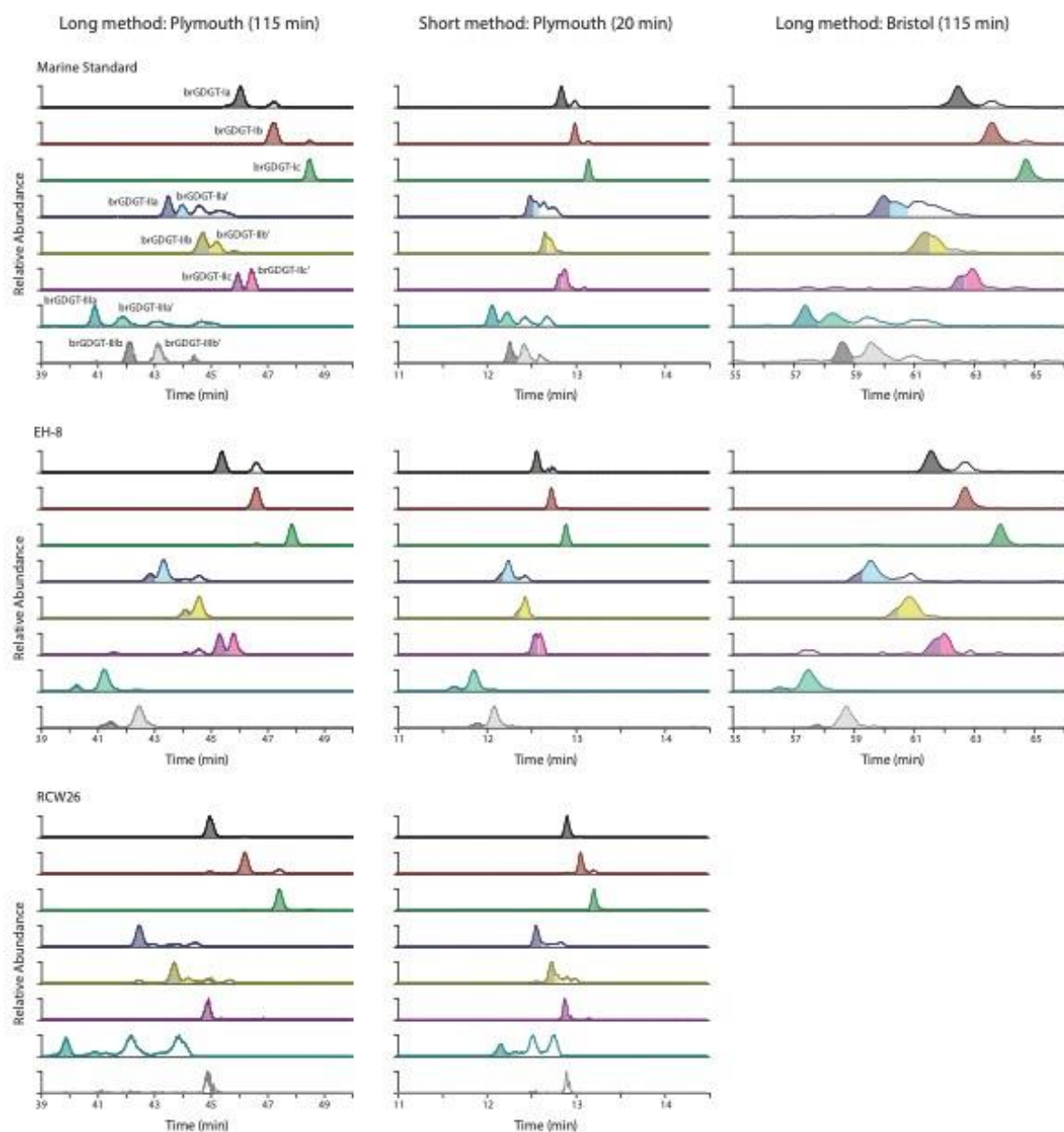
522 boundary, respectively.  $^{40}\text{Ar}/^{39}\text{Ar}$  dating of sanidines in the tuffs yield an age of  $66.019 \pm 0.021$  Ma for  
523 Z2, and  $66.003 \pm 0.033$  Ma for Z1 (Renne et al. 2013), giving a most likely duration of 16 ka for the 40  
524 cm of intervening coal (i.e., 1 m represents 40 ka). Error estimates of the ages suggest a maximum  
525 possible duration of 70 ka is possible for the 40 cm of intervening coal (i.e., 1 m represents 175 ka),  
526 and an unlikely minimum duration that equates to instantaneous deposition.

527 In Supplementary Table S3, the most likely (1 m = 40 ka), minimum (1 m = 0 ka), and maximum  
528 (1 m = 175 ka) duration of time represented by the coal is used to date Tiepoints 1–9, relative to the  
529 K-Pg boundary (Fig. 2 and Fig. S2). Differences in thicknesses of coal between the tiepoints (i.e.,  
530 differences in thicknesses of the  $\delta^{13}\text{C}_{\text{org}}$  excursion stratigraphies) at Rock Creek West and Wood  
531 Mountain Creek (Fig. 2 and Fig. S2) clearly imply that there were differences in synchronous peat  
532 accumulation rates and/or post-depositional compaction of the peat at the two sites (and also  
533 between these sites and the Hell Creek Marina Road site of Renne et al. 2013). This highlights  
534 uncertainties, but the approach places clear absolute errors in the timescales of MAAT change  
535 interpreted in this study. The ages relative to the K-Pg boundary that we use for Tiepoints 1–9 are  
536 based on the average determined for Rock Creek West, and Wood Mountain Creek (Supplementary  
537 Table S3).

538 Having established the best-estimate ages of Tiepoints 1–9 (Supplementary Table S3), the  
539 midpoint of each sample was linearly extrapolated between the ages of the Tiepoints to ascribe each  
540 an individual age relative to the K-Pg boundary (Supplementary Table S4). MAAT for each datapoint  
541 were plotted against these ages to make Figure 3.

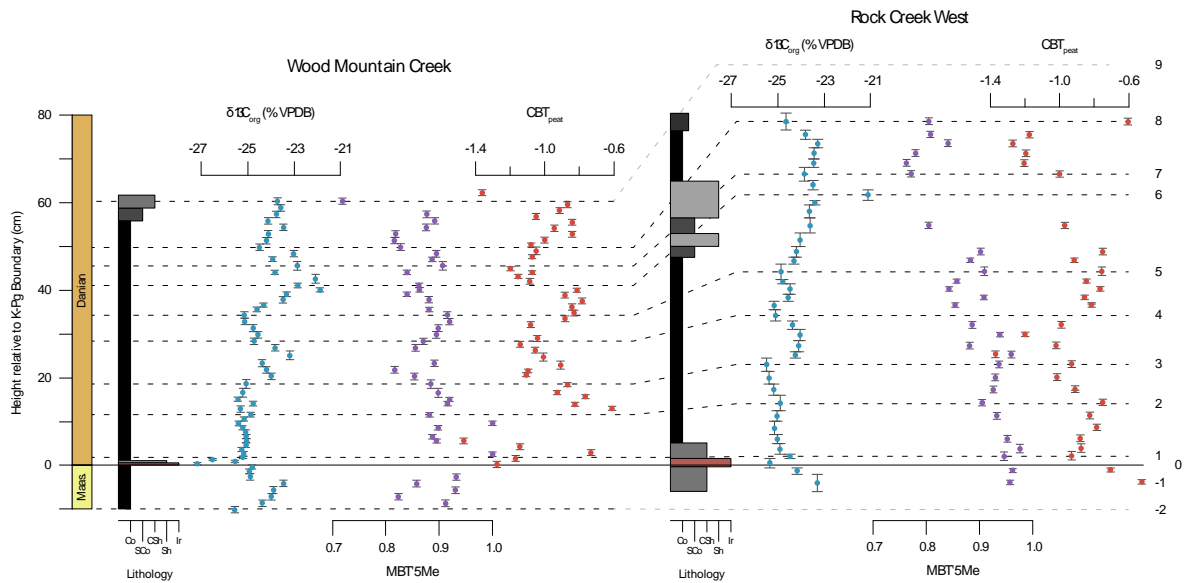
542

543 **FIGURE AND TABLE CAPTIONS**



544

545 **Supplementary figure S1.** Chromatograms showing the difference in resolution of individual brGDGTs  
 546 and their isomers in the 'Long' method at Plymouth University (A), the 'Short' method at Plymouth  
 547 University (B), and the 'Long' method at University of Bristol. 6-methyl-GDGTs are indicated with '.



548

549 **Supplementary figure S2.**  $\delta^{13}C_{org}$  (blue), brGDGT mean annual air temperature (MAAT; purple),  $CBT_{peat}$ -  
 550 pH (red) records from Wood Mountain Creek and Rock Creek West, Saskatchewan, plotted against  
 551 height relative to the K-Pg boundary. Vertical error bars show the stratigraphic range of each sample.  
 552 Dashed lines show chemostratigraphic ( $\delta^{13}C_{org}$ ) correlation tiepoints. Solid line shows the Ir-claystone  
 553 K-Pg datum. On the lithology log, Co = coal, ShC = shaley coal, CSh = coaly shale, Sh = shale, and Ir =  
 554 iridium-enriched claystone.

555 **Supplementary Table S1.** Stratigraphic and lithological data, GDGT abundances, proxy calculations  
 556 (MBT'5Me, MAAT, CBT, pH, and hopane ratio), and  $\delta^{13}C_{org}$  for all samples reported in this study.  
 557 MBT'5Me (Columns X, AO) calculated from De Jonge et al. (2015). MAAT (Columns Y, AP) calculated  
 558 from Naafs et al. (2017).  $CBT_{peat}$  (Columns Z, AQ) calculated from Naafs et al. (2018). pH (Columns  
 559 AA, AR) calculated from Naafs et al. (2018). Hopane ratio (Column AV) calculated from Inglis et al.  
 560 (2018). Blank cells (columns K–W, AB–AN) denote below the detection limit/unquantifiable GDGTs. The  
 561  $\delta^{13}C_{org}$  values (columns AY–BF) for Wood Mountain Creek are from Jerrett et al. (2015). All other data  
 562 was generated for this study. Where the standard deviation of MAAT > 1, this sample was omitted  
 563 from the study. Where polar fraction analysis not undertaken or duplicated, this sample was omitted  
 564 from the study.

565 **Supplementary Table S2.** Comparison of MBT, MBT'5Me proxy values calculated from the 'Short' and  
566 'Long' HPLC-APCI-MS methods at Plymouth University and the 'long' method at the University of  
567 Bristol. (std= standard, RCW= Rock Creek West, SD= standard deviation).

568 **Supplementary Table S3.** Age of Tiepoints 1–9 (Fig. S2) at Rock Creek West and Wood Mountain Creek  
569 relative to the K-Pg boundary (columns C–E and G–I respectively), applying the minimum (1 m = 0 ka),  
570 most likely (1 m = 40 ka), and maximum (1 m = 175 ka) duration of time represented by the coal from  
571 Renne et al. (2013). Also shown are the average relative ages of the tiepoints from Rock Creek West  
572 and Wood Mountain Creek (columns L–N). The most likely duration of the average tiepoint ages  
573 (column M) are the values used to generate the ages for each datapoint in Supplementary Table S4.

574 **Supplementary Table S4.** All samples, and their assigned ages generated through linear interpolation  
575 of the position of their midpoint between tiepoints. The ages of the tiepoints are from column M  
576 (Supplementary Table S3). Also shown are the MAAT (°C) and mean  $\delta^{13}\text{C}_{\text{org}}$  (‰) values from  
577 Supplementary Table S1. These data are used to plot Figure 3.

578 **Supplementary Table S5.** Statistical analysis of MAAT data. MAAT data were separated into four  
579 temporal bins: pre-K-Pg boundary (-5–0 ka), 0–10 ka, 10–20 ka, and 20–30 ka. These bins were then  
580 analysed using two-sample, equal variances t-tests to determine the statistical significance of  
581 temperature trends. These data are included in Figure 3.