

## 1 Supplemental Information

2 An expanded Supplemental Information section is provided here as a reference for: **(1)**  
3 methodologies used during geochemical analyses; **(2)** results for individual zircon grain  
4 analyses; **(3)** expanded interpretation of bentonite intervals as individual events of primary  
5 ashfall (vs. reworked detrital zircon grains in volcanoclastic sediment); **(4)** references cited  
6 herein. We also provide a figure showing photographs of individual zircon grains analyzed and  
7 five data tables showing: **(1.1)** stratigraphic correlation of collected paleontological specimens;  
8 **(1.2)** stratigraphic correlation of collected bentonites; **(1.3)** stratigraphic correlation of  
9 collected geochemical samples; **(2)** single grain U-Pb isotope data for samples analyzed at the  
10 Pacific Centre for Isotopic and Geochemical Research (PCIGR) at the University of British  
11 Columbia, Canada (i.e., Table 2.1) and the Massachusetts Institute of Technology (MIT),  
12 Cambridge MA (i.e., Table 2.2); **(3)** trace element concentration data for zircon grains (sample  
13 2017GC3.8); **(4)** chondrite normalized rare earth elements, selected trace element  
14 concentrations, and Ti-in-zircon temperatures for zircons (sample 2017GC3.8); and **(5)** LA-  
15 ICPMS and CA-ID-TIMS U-Pb age comparison data for all bentonite samples in this study.

16

### 17 1. Expanded Methodology

18 We collected 70 samples for carbon isotope analysis and 30 samples for conodont analysis. We  
19 collected float and *in situ* macrofossils (ammonoids, bivalves, and hydrozoans) from 51 fossil  
20 horizons that yielded 103 specimens and sampled 4 ash beds for U-Pb CA-ID TIMS analysis.  
21 Geochemical samples were analyzed for organic carbon isotope composition ( $\delta^{13}\text{C}_{\text{org}}$ ) and  
22 whole-rock total organic carbon ( $\text{TOC}_{\text{wr}}$ ) content at Durham University, England; conodont

23 samples were processed at the Geological Survey of Canada (Vancouver); ammonoids and  
24 hydrozoans were identified at Western Michigan University, USA; bivalves were identified at  
25 the Museum für Naturkunde (Berlin, Germany) and SUNY Cortland (New York); and U-Pb CA-  
26 ID TIMS analysis took place at PCIGR and MIT. All fossils and geochemical samples were  
27 collected under National Parks Service collection and research permits (WRST-2018-SC1-0004  
28 and WRST-2018-SCI-0005) and paleontological specimens curated with the Wrangell-St Elias  
29 National Park Museum. Geochemical residues are curated and stored at the respective  
30 institutions.

31

### 32 *Carbon-isotope analysis*

33 Organic  $\delta^{13}\text{C}$  data were obtained following the methods described by Skrzypek and Debajyoti  
34 (2006) and Gröcke et al. (2009). Approximately 200 mg of sediment was decalcified using 3M  
35 HCl, left overnight at room temperature. Samples were then neutralized using deionized water  
36 and dried in an oven at 55 °C.  $\delta^{13}\text{C}_{\text{org}}$  data were acquired using a Costech Elemental Analyzer  
37 (ECS 4010) coupled to a ThermoFinnigan Delta V Advantage mass spectrometer. Carbon-  
38 isotope ratios were corrected for  $^{17}\text{O}$  contribution and reported in standard delta ( $\delta$ ) notation in  
39 per mil (‰) relative to the VPDB scale. Data accuracy was monitored through routine analyses  
40 of in-house standards, which are calibrated against international standards (USGS 40, USGS  
41 24, IAEA 600, IAEA CH6): this provides a linear range in  $\delta^{13}\text{C}$  between +2‰ and -47‰.

42 Analytical uncertainty for  $\delta^{13}\text{C}_{\text{org}}$  is typically  $\pm 0.1\text{‰}$  for replicate analyses of the

43 international standards and typically  $< 0.2\text{‰}$  on replicate sample analysis.

44

45 *CA-ID-TIMS analysis*

46 High precision U-Pb zircon analyses were carried out at the University of British Columbia  
47 (UBC) and the Massachusetts Institute of Technology (MIT). All analyses were single grain  
48 chemical abrasion isotope dilution thermal ionization spectrometry (CA-ID-TIMS) analyses.  
49 The MIT analyses followed the procedures outlined in Rioux et al. (2012) and data reduction  
50 was done using the ET Redux software package (Bowring et al., 2011; McLean et al., 2011). The  
51 UBC analyses followed procedures modified from Mundil et al. (2004), Mattinson (2005), and  
52 Scoates and Friedman (2008), and are detailed below.

53

54 Following standard mineral separation procedures, zircons were handpicked in alcohol. The  
55 clearest, crack- and inclusion-free grains are selected, photographed, and then annealed in  
56 quartz glass crucibles at 900 °C for 60 hours. Annealed grains were transferred into 3.5 mL PFA  
57 screwtop beakers, ultrapure HF (up to 50% strength, 500 mL) and HNO<sub>3</sub> (up to 14N, 50 mL)  
58 were added and caps were closed finger tight. The beakers were placed in 125 mL PTFE liners  
59 (up to four per liner) and ~2 mL HF and 0.2 mL HNO<sub>3</sub> of the same strength as acid within  
60 beakers was added to the liners. The liners were then slid into stainless steel Parr™  
61 high-pressure dissolution devices, which were sealed and brought up to a maximum of  
62 200 °C for 8–16 hours (typically 175 °C for 12 hours). Beakers were removed from liners  
63 and zircon separated from leachate. Zircons were rinsed with >18 MΩ.cm water and

64 subboiled acetone. Then 2 mL of subboiled 6N HCl was added and beakers were set on  
65 a hotplate at 80–130 °C for 30 minutes and again rinsed with water and acetone.

66 Masses were estimated from the dimensions (volumes) of grains. Single grains were  
67 transferred into clean 300 mL PFA microcapsules (crucibles), and 50 mL 50% HF and  
68 5 mL 14N was added. Each grain was spiked with a  $^{233-235}\text{U}$ - $^{205}\text{Pb}$  tracer solution (EARTHTIME  
69 ET535), capped, and again placed in a Parr liner (8–15 microcapsules per liner). HF and nitric  
70 acids in a 10:1 ratio, respectively, were added to the liner, which was then placed in Parr high-  
71 pressure device and dissolution was achieved at 240 °C for 40 hours. The resulting solutions  
72 were dried on a hotplate at 130 °C, 50 mL 6N. HCl was then added to the microcapsules, and  
73 fluorides were dissolved in high-pressure Parr devices for 12 hours at 210 °C. HCl solutions  
74 were transferred into clean 7 mL PFA beakers and dried with 2 mL of 0.5N  $\text{H}_3\text{PO}_4$ . Samples  
75 were then loaded onto degassed, zone-refined Re filaments in 2 mL of silicic acid emitter  
76 (Gerstenberger and Haase, 1997).

77  
78 Isotopic ratios were measured using a modified single collector VG-54R or 354S (with Sector 54  
79 electronics) thermal ionization mass spectrometer equipped with analog Daly  
80 photomultipliers. Measurements were done in peak-switching mode on the Daly detector.  
81 Estimated U analytical blanks are 0.2 pg and common Pb ranged from 0.15–0.57 pg per grain in  
82 this study. U fractionation was determined directly on individual runs using the  $^{233-235}\text{U}$  tracer,  
83 and Pb isotopic ratios were corrected for fractionation of 0.23%/amu, based on replicate

84 analyses of the NBS-982 Pb reference material and the values recommended by Thirlwall  
85 (2000). Data reduction employed the excel-based program of Schmitz and Schoene (2007).  
86 Standard concordia diagrams were constructed and regression intercepts, weighted averages  
87 were calculated with Isoplot (Ludwig, 2003). Unless otherwise noted, all errors are quoted at  
88 the 2 sigma or 95% level of confidence. Isotopic dates were calculated the decay constants  
89  $\lambda_{238}=1.55125\text{E-}10$  and  $\lambda_{235}=9.8485\text{E-}10$  (Jaffey et al. 1971). EARTHTIME U-Pb synthetic  
90 solutions are analyzed on an ongoing basis in the UBC lab to monitor the accuracy of results.

91

#### 92 *Sample preparation and zircon trace element LA-ICP-MS analysis*

93 After rock samples have undergone standard mineral separation procedures, zircons were  
94 handpicked in alcohol and mounted in epoxy, along with reference materials. Grain mounts  
95 were then wet ground with carbide abrasive paper and polished with diamond paste. Next,  
96 cathodoluminescence (CL) imaging was carried out on a Philips XL-30 scanning electron  
97 microscope (SEM) equipped with a Bruker Quanta 200 energy-dispersion X-ray microanalysis  
98 system at the Electron Microbeam/X-Ray Diffraction Facility (EMXDF) at the University of  
99 British Columbia. An operating voltage of 15 kV was used, with a spot diameter of 6  $\mu\text{m}$  and  
100 peak count time of 30 seconds. After removal of the carbon coat the grain mount surface was  
101 washed with mild soap and rinsed with high purity water. Prior to analysis the grain mount  
102 surface was cleaned with 3N  $\text{HNO}_3$  acid and again rinsed with high purity water to remove any  
103 surficial Pb contamination that could interfere with the early portions of the spot analyses.

104

105 Analyses were conducted using a Resonetics RESOLUTION M-50-LR, which contains a Class I  
106 laser device equipped with a UV excimer laser source (Coherent COMPex Pro 110, 193 nm,  
107 pulse width of 4 ns) and a two-volume cell designed and developed by Laurin Technic Pty. Ltd.  
108 (Australia). This sample chamber allowed for the investigation of several grain mounts within  
109 one analytical session. The laser path was fluxed by N<sub>2</sub> to ensure better stability. Ablation was  
110 carried out in a cell with a volume of approximately 20 cm<sup>3</sup> and a He gas stream that ensured  
111 better signal stability (Eggins et al., 1998). The laser cell was connected via a Teflon squid to an  
112 Agilent 7700x quadrupole ICP-MS housed at PCIGR. A pre-ablation shot was used to ensure  
113 that the spot area on the grain surface was contamination-free. Samples and reference  
114 materials were analyzed for up to 34 isotopes, including: <sup>7</sup>Li, <sup>29</sup>Si, <sup>43</sup>Ca, <sup>49</sup>Ti, <sup>85</sup>Rb, <sup>88</sup>Sr, <sup>89</sup>Y, <sup>90</sup>Zr,  
115 <sup>93</sup>Nb, <sup>139</sup>La, <sup>140</sup>Ce, <sup>141</sup>Pr, <sup>146</sup>Nd, <sup>147</sup>Sm, <sup>153</sup>Eu, <sup>157</sup>Gd, <sup>159</sup>Tb, <sup>163</sup>Dy, <sup>165</sup>Ho, <sup>166</sup>Er, <sup>169</sup>Tm, <sup>172</sup>Yb, <sup>175</sup>Lu,  
116 <sup>177</sup>Hf, <sup>188</sup>Ta, <sup>202</sup>Hg, Pb (<sup>204</sup>Pb, <sup>206</sup>Pb, <sup>207</sup>Pb, <sup>208</sup>Pb), <sup>232</sup>Th, and U (<sup>235</sup>U and <sup>238</sup>U) with a dwell time  
117 of 0.02 seconds for each isotope. These isotopes were selected based on their relatively high  
118 natural abundances and absence of interferences. The settings for the laser were: spot size of  
119 34 μm with a total ablation time of 30 seconds, frequency of 5 Hz, fluence of 5 J/cm<sup>2</sup>, power of  
120 7.8 mJ after attenuation, pit depths of approximately 15 μm, He flow rate of 800 mL/min, N<sub>2</sub>  
121 flow rate of 2 mL/min, and a carrier gas (Ar) flow rate of 0.57 L/min.

122

123 NIST 612 glass was used for both drift correction and trace element calibration with sample  
124 spacing between every five to eight unknowns and <sup>90</sup>Zr was used as the internal standard  
125 assuming stoichiometric values for zircon. NIST 610 glass was analyzed after each NIST 612  
126 analysis and used as a monitor reference material for trace elements. Raw data were reduced

127 using the Lolite 3.4 extension (Paton et al., 2011) for Igor Pro™ yielding concentration values  
128 and propagated uncertainties.

129

## 130 **2. Expanded Results**

### 131 *Sample 2017GC3.8*

132 Zircons from sample 2017GC3.8 (collected at 0 m in the Grotto Creek section) comprise a  
133 homogenous population of elongate, euhedral to subhedral prisms 200–500 µm in length;  
134 containing elongate inclusions up to 300 µm in length that are aligned with their long  
135 dimensions roughly parallel to the length of grains (SI Fig. 1A). Eighteen grains from the  
136 sample were mounted, sectioned, and polished for cathodoluminescence (CL) imaging (SI Fig.  
137 1C); showing weak oscillatory zoning in most grains, often overprinted by broad simple tabular  
138 and sector zoning. Inclusions are mantled by material that is unzoned or has a mottled,  
139 disrupted appearance and cross-cuts oscillatory, tabular, and sector zoning. Zircons from  
140 2017GC3.8 are interpreted as magmatic grains with minor inclusions and well-developed  
141 zoning patterns. The elongate euhedral to subhedral crystal shape in conjunction with the tight  
142 age cluster of analyzed grains is indicative of a primary magmatic age with no sedimentary  
143 evidence for reworking or abrasion of zircon grains. Large inclusions are interpreted as late-  
144 magmatic, resorption-related features that likely formed via melt inclusions. Laser ablation  
145 ICP-MS trace element data were acquired for some of the mounted zircon grains (SI Fig. 1D;  
146 Tables 2, 3, 5). A plot of normalized rare earth elements (REE) shows the consistency of  
147 analyses with relative light REE depletion, Ce and Eu anomalies, and heavy REE enrichment (SI  
148 Fig 1D).

149

150 *Sample 2017GC14.9*

151 Zircons separated from ash 2017GC14.9 (collected at 11.07 m in the Grotto Creek section) are  
152 shown in photomicrograph (SI Fig. 1B). Grains are elongate, euhedral to subhedral in shape  
153 measuring 100–300  $\mu\text{m}$  in length, with some grains presenting a broken and stubby  
154 appearance. Rare inclusions are similar in appearance to those in sample 2017GC3.8. Due to  
155 the modest number of grains recovered zircons were not mounted.

156

157 *Sample Grot-124*

158 Eleven single-grain analyses from sample Grot-124 yielded overlapping Th-corrected  
159  $^{206}\text{Pb}/^{238}\text{U}$  dates from  $210.10 \pm 0.16$  to  $209.73 \pm 0.25$  Ma, with a weighted mean of  $209.92 \pm$   
160  $0.043$  Ma (MSWD = 1.6). Many of the analyzed grains were acicular—with some containing  
161 apparent melt inclusions parallel to the c-axis—consistent with a volcanic origin. We interpret  
162 the weighted mean date as the eruption age of this sample.

163

164 *Sample Grot-1*

165 Ten single-grain analyses from sample Grot-1 yielded a range of Th-corrected  $^{206}\text{Pb}/^{238}\text{U}$  dates  
166 from  $245.8 \pm 2.0$  to  $213.2 \pm 1.6$  Ma (excluding a single low precision analysis, z27). The analyzed  
167 zircons from this sample were small, equant, and had relatively low U and Pb contents, leading  
168 to less precise results. Eight of the 10 analyses overlap within uncertainty with a Th-corrected  
169 weighted mean  $^{206}\text{Pb}/^{238}\text{U}$  date of  $214.36 \pm 0.19$  Ma (MSWD = 1.2). The weighted mean is  
170 controlled by four higher-precision analyses that yield overlapping dates of  $214.98 \pm 0.65$  to



171 214.28 ± 0.25 Ma, with a weighted mean of 214.39 ± 0.19 Ma (MSWD = 1.7), in close agreement  
172 with the mean of the larger population. We interpret the weighted mean date of the eight  
173 overlapping analyses as the eruption age of the sample, while the two older grains (245.8–  
174 221.3 Ma) are likely inherited xenocrysts. The mean square of the weighted deviates (MSWD)  
175 of the weighted means for Grot-124 and Grot-50 are within the 95% confidence interval of the  
176 expected value Wendt and Carl (1991).

177

### 178 **3. Zircon ages: primary airfall ash vs reworked detrital grains**

179 The bentonite beds that were sampled in the Grotto Creek section represent four separate  
180 volcanic events and associated settling of volcanic ash through the water column onto the  
181 seafloor below storm wave-base under low oxygen to anoxic conditions. In such an  
182 environment, primary ash layers often reside on the seafloor for extended periods of time with  
183 no/little reworking. Low sedimentation rate(s) may lead to amalgamation of multiple ash-fall  
184 events or mixing of ash with detrital sediments (e.g., ver Straeten, 2004).

185

186 The former (i.e., settling of volcanic ash through the water column) is most likely the case at  
187 Grotto Creek. This is due to a number of sedimentological and morphological factors including:  
188 (i) the sampled lithology is a thin light gray to yellow-weathering sticky claystone lacking  
189 induration or coarser clasts, consistent with diagenetically altered smectite clays derived from  
190 devitrified ash—more difficult to rework (e.g., ver Straeten, 2004); (ii) the dominance of  
191 needle-shaped zircons in samples 2017GC3.8, 2017GC14.9 and Grot-124 is indicative of a  
192 volcanic origin, rather than detrital grain morphologies indicative of recycling from

193 nonvolcanic sources; (iii) the euhedral morphology of the sampled zircons, some pristine (i.e.,  
194 not abraded subrounded to rounded zircon morphologies), is inconsistent with reworking or  
195 contamination by older detrital zircons; (iv) no sedimentary evidence for sediment reworking  
196 (i.e., no sedimentary structures, such as cross-stratification, early post-depositional soft  
197 sediment deformation, contorted/convolute laminae, ball/pillow structures, trace fossils, etc.);  
198 (v) no multiple graded layers or fossiliferous zones within the sampled bentonite horizons  
199 indicative of complex depositional histories that could mix ash with older/younger detritus; (vi)  
200 no glauconite or scattered phosphatic pebbles (hardgrounds) directly upon or within the  
201 sampled bentonites indicative of significant sediment starvation and condensation that might  
202 promote mixing of the ash with younger sediments containing older detrital zircons; and (vii)  
203 only minor variation in zircon ages recorded throughout the section (i.e., Fig. 7).

204

205 To expand on point (vii), there is no geochronologic evidence of pre-Triassic detrital ages in  
206 this study (i.e., Fig. 7, SI Table 2). All zircon ages cluster within Late Triassic and younger  
207 (upsection) rocks and are consistent with biostratigraphy. To better emphasize this point, age  
208 comparison data for all bentonite samples in the Grotto Creek section were generated for LA-  
209 ICPMS and CA-ID-TIMS U-Pb analyses. Data presented in Figure 7B show  $^{206}\text{Pb}/^{238}\text{U}$  age  
210 distributions that are in-line with crystals from a primary ash bed rather than a volcanoclastic  
211 sandstone containing population(s) of significantly older zircon grains.

212

213 **4. References (Supplemental Information)**

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290

## 291 **Captions (Supplemental Information)**

292

293 **Table 1:** Correlation tables for samples and specimens collected from the Grotto Creek section  
294 presented on figure 3 (main text). **(I)** paleontological specimens. **(II)** bentonite samples for (CA-  
295 ID) TIMS analysis. **(III)** samples for geochemical (i.e., organic carbon isotope and total organic  
296 carbon) analyses.

297

298 **Table 2:** Single grain U-Pb isotope data for samples analyzed at the Pacific Centre for Isotopic  
299 and Geochemical Research (PCIGR) at the University of British Columbia, Canada (i.e., table  
300 2.1) and the Massachusetts Institute of Technology (MIT), Cambridge MA (i.e., table 2.2).

301

302 **Table 3:** Trace element concentration data for zircons from ash sample 2017GC3.8.

303

304 **Table 4:** Chondrite normalized rare earth elements, selected trace element concentrations,  
305 and Ti-in-zircon temperatures for zircons from ash sample 2017GC3.8.

306

307 **Table 5:** LA-ICPMS and CA-ID-TIMS U-Pb age comparison data for all bentonite samples  
308 presented in this study.

309

310 **Figure 1:** Images for analyzed zircon grain analysis from the Grotto Creek Section. **(A)**

311 Photomicrograph of zircons from ash sample 2017GC3.8 in plane-polarized light. **(B)**

312 Photomicrograph of zircons from ash sample 2017GC14.9 in plane-polarized light. Grain IDs  
313 correspond to those that appear in CA-ID-TIMS U-Pb analytical data table. All 5 grains were

314 processed for CA-ID-TIMS with grains D and E lost in processing.

315 Laser Ablation techniques are listed in SI methodology (above). **(C)** Cathodoluminescence (CL)

316 images of zircons from ash sample 2017GC3.8. Grain IDs correspond to those in tables 2-5 and

317 CA-ID-TIMS U-Pb analytical data table. **(D)** Plot of chondrite normalized rare earth elements

318 for 2017GC3.8 zircons normalized to chondrite values of McDonough and Sun (1995).