

1 **Constraining the Late Pleistocene history of the Laurentide Ice Sheet by dating the**  
2 **Missinaibi Formation, Hudson Bay Lowlands, Canada**

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11 **Abstract**

12 Well-dated paleorecords from periods prior to the Last Glacial Maximum (LGM) are  
13 important for validating models of ice-sheet build-up and growth. However, owing to glacial  
14 erosion, most Late Pleistocene records lie outside of the previously glaciated region, which limits  
15 their ability to inform about the dynamics of paleo-ice sheets. Here, we evaluate new and  
16 previously published chronology data from the Missinaibi Formation, a Pleistocene-aged deposit  
17 in the Hudson Bay Lowlands (HBL), Canada, located near the geographic center of the  
18 Laurentide Ice Sheet (LIS). Available radiocarbon (AMS = 44, conventional = 36), amino acid (n  
19 = 13), uranium-thorium (U-Th, n = 14), thermoluminescence (TL, n = 15) and optically  
20 stimulated luminescence (OSL, n = 5) data suggest that an ice-free HBL may have been possible  
21 during parts of Marine Isotope Stage 7 (MIS 7; ca. 243,000 to ca. 190,000 yr BP), MIS 5 (ca.  
22 130,000 to ca. 71,000 yr BP) and MIS 3 (ca. 29,000 to ca. 57,000). While MIS 7 and MIS 5 are

23 well-documented interglacial periods, the development of peat, forest bed and fluvial deposits  
24 dating to MIS 3 (n = 20 radiocarbon dates; 4 TL dates, 3 OSL dates), suggests that the LIS  
25 retreated and remained beyond, or somewhere within, the boundaries of the HBL during this  
26 interstadial. Ice sheet models approximate the margin of the LIS to Southern Ontario during this  
27 time, which is 700 km south of the HBL. Therefore, if correct, our data help constrain a  
28 significantly different configuration and dynamicity for the LIS than previously modelled. We  
29 can find no chronological basis to discount the MIS 3 age assignments. However, since most  
30 data originate from radiocarbon dates lying close to the reliable limit of this geochronometer,  
31 future work on dating the Missinaibi Formation using other geochronological methods (e.g. U-  
32 Th, OSL) is necessary in order to confirm the age estimates and strengthen the boundaries of the  
33 LIS during this period.

#### 34 **Keywords**

35 MIS 3, MIS 5, interstadial, pre-LGM, mid-Wisconsin, land-based verification, marine incursion,  
36 meta-analysis, Canadian quartz

#### 37 **Highlights**

- 38 • Synthesis of pre-LGM chronology data from the central region of the LIS
- 39 • Data consist of previously published (n=88) and new contributions (n=39)
- 40 • Results suggest an ice-free HBL during parts of MIS 7, MIS 5 and MIS 3
- 41 • Radiocarbon, OSL and TL ages form the basis for the MIS 3 assignment
- 42 • Implies more dynamicity for the LIS than previously modelled for MIS 3

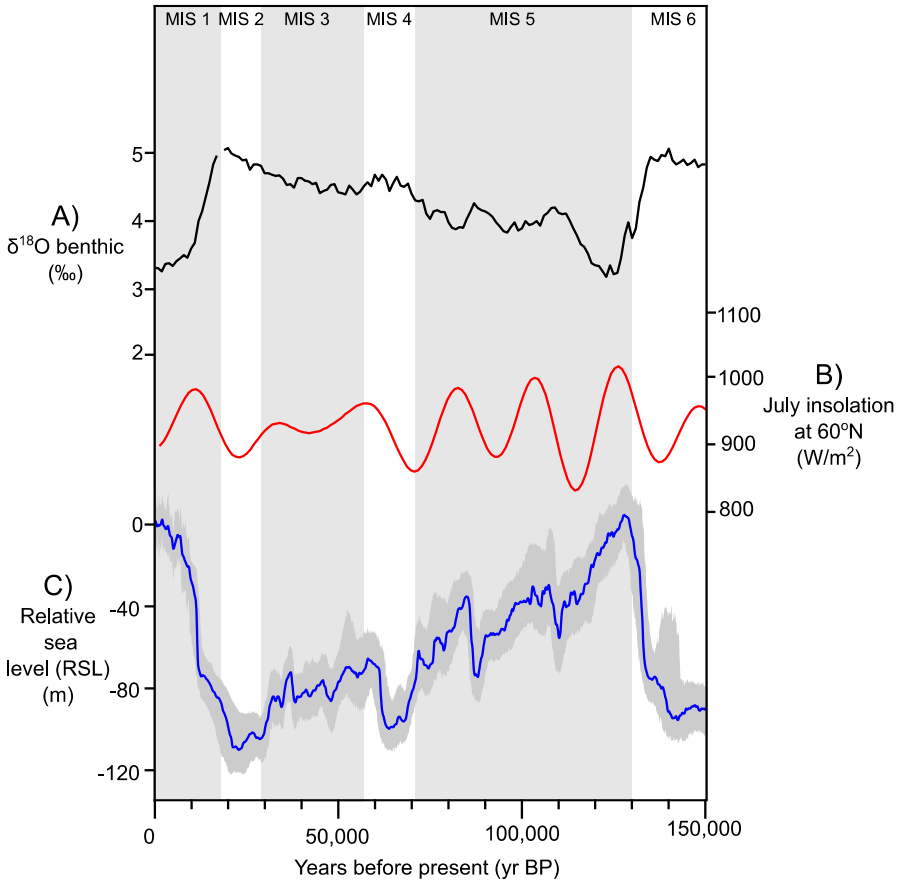
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## 44 **1. Introduction**

45 Understanding the quantitative relations amongst the biosphere, cryosphere and atmosphere  
46 is critically important towards formulating accurate predictions for future climates; and the  
47 growth and decay of ice sheets in the Late Pleistocene provides boundary conditions for testing  
48 Earth System Models (Kleinen et al., 2015; Loutre and Berger, 2003). To make such climate  
49 predictions, these models require empirically derived boundary conditions including the duration  
50 and dynamics of previous glaciations. To that end, the recent deglaciation sequence of the  
51 Laurentide Ice Sheet (LIS) from the Last Glacial Maximum (LGM) to the present-day is well  
52 understood owing to well constrained models of isostatic rebound (Peltier et al., 2015) and a  
53 plethora of radiocarbon ages (Dyke, 2004). However, because of glacial erosion, we have a  
54 highly incomplete understanding of the period prior to the LGM (Kleman et al., 2010).

55 Records of relative sea level (RSL) and the  $\delta^{18}\text{O}$  from benthic foraminifera are important  
56 tools for approximating the volume of continental ice during the Pleistocene. For example, a  
57 decrease in RSL to -100 m (compared to present-day) (Grant et al., 2014), paired with an  
58 increase in the  $\delta^{18}\text{O}$  from benthic foraminifera (Lisiecki and Raymo, 2005) from ca. 68,000 to  
59 63,000 years before present (yr BP), implies moderate glaciation over North America at that time  
60 (Fig. 1). Immediately following this stadial was a partial deglaciation of the continent as shown  
61 by a rapid rise in RSL, maintaining a level between -70 m and -80 m until 40,000 yr BP (Grant et  
62 al., 2014), and a slight decrease in the  $\delta^{18}\text{O}$  from benthic foraminifera (Lisiecki and Raymo,  
63 2005). This period of implied partial continental glaciation corresponds broadly to the early part  
64 of Marine Isotope Stage 3 (MIS 3; ca. 57,000 to ca. 29,000 yr BP; Lisiecki and Raymo (2005)),  
65 where summer insolation was stable and higher than today at 60° N (Berger and Loutre, 1991).

66



67

68 **Fig. 1** (*single-column figure*) Climate proxies for the most recent 150,000 years. (A)  $\delta^{18}\text{O}$  record  
 69 from benthic foraminifera (Lisiecki and Raymo, 2005); (B) July insolation at  $60^\circ\text{N}$  (Berger and  
 70 Loutre, 1991); (C) Relative sea level from the Red Sea (Grant et al., 2014).

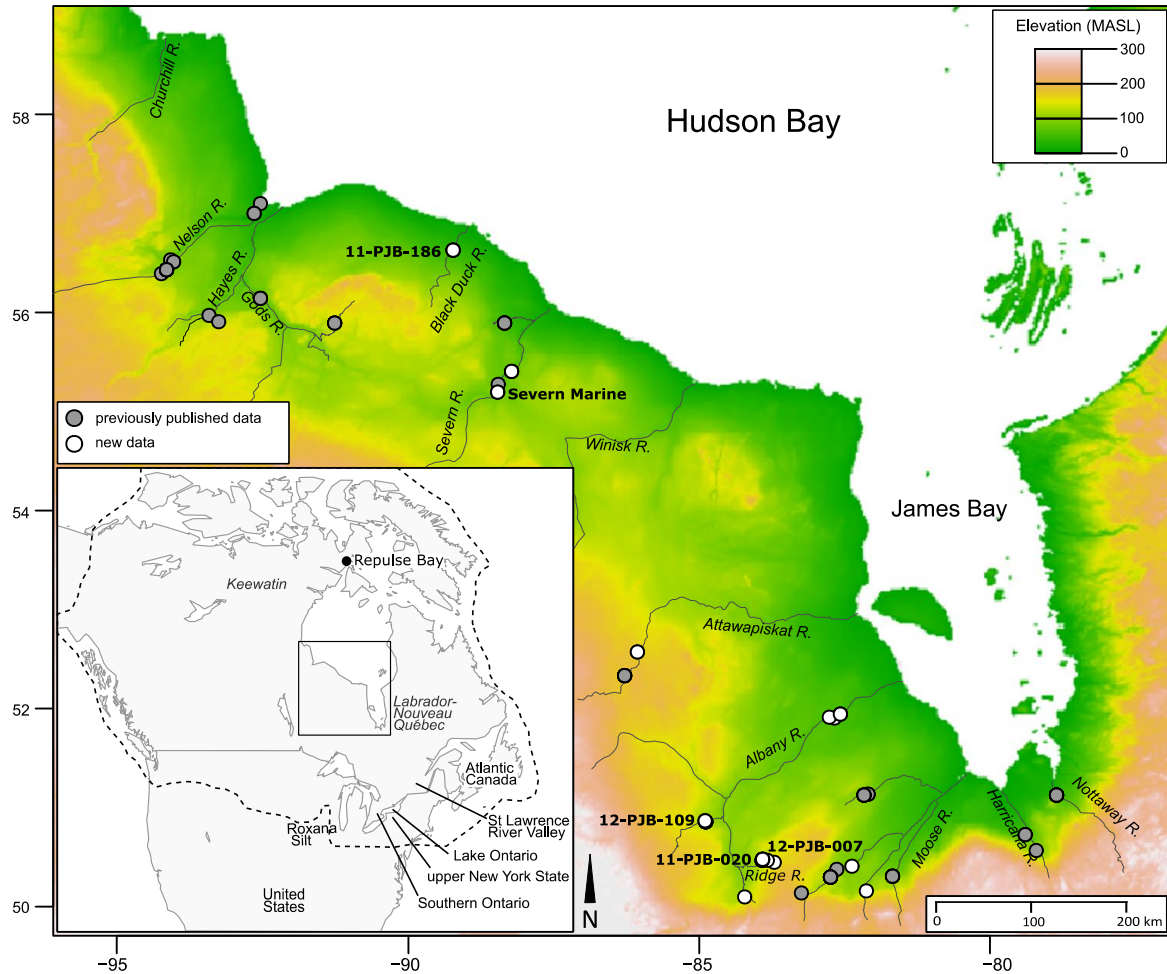
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72 Understanding the configuration of North American ice sheets during MIS 3 is important  
73 because it will help validate models which approximate ice-sheet build-up and growth for that  
74 time (e.g. Ganopolski and Calov, 2011; Ganopolski et al., 2010; Kleman et al., 2010; Stokes et  
75 al., 2012).

76 Although rare and spatially discontinuous, available paleorecords from North America  
77 suggest a dynamic and lobed margin of the LIS during MIS 3. For example, the Roxana Silt, a  
78 loess deposit dating to ca. 60,000 to ca. 30,000 yr BP, suggests that glacial activity reached the  
79 Mississippi watershed during that time (Forman and Pierson, 2002). Furthermore, several  
80 corroborative studies on sedimentological and biological records suggest that the LIS advanced  
81 into the continental United States at ca. 45,000 to ca. 42,000 yr BP, resulting in drainage  
82 southward toward the Gulf of Mexico (Hill et al., 2006; Sionneau et al., 2013; Tripsanas et al.,  
83 2007). Contrastingly, studies suggest an ice-free MIS 3 in Southern Ontario (Bajc et al., 2015;  
84 Karrow et al., 2001; Karrow and Warner, 1984; Warner et al., 1988), Atlantic Canada (Fr chet  
85 and de Vernal, 2013; R millard et al., 2013) and Repulse Bay (McMartin et al., 2015). These  
86 datasets indicate the possibility for a dynamic and regionally varied response of the ice sheet  
87 margin to MIS 3 paleoclimates. Additional terrestrial records, especially those from the  
88 previously glaciated region, are needed to further constrain the boundaries of the LIS during MIS  
89 3.

## 90 **1.1 Missinaibi Formation, Canada**

91 The Late Pleistocene history of the Hudson Bay Lowlands (HBL), Canada (Fig. 2), has been  
92 identified as an important archive for constraining the history of glaciations over North America  
93 (Dredge and Thorleifson, 1987; Kleman et al., 2010). Importantly, the HBL contains the



94  
 95 **Fig. 2** (2-column figure). Map of the Hudson Bay Lowlands (HBL) region, showing the locations  
 96 of Late Pleistocene age estimates, which are compiled for this study. The location of key sites are  
 97 noted on this map. Some sites contain several dates; details from each site are available in  
 98 Appendix A. Topographic data was compiled by Amante and Eakins (2009). Inset map shows  
 99 the HBL region (box), approximate maximum extent of the Wisconsin Glaciation (hatched lined)  
 100 (Dyke et al., 2002) and other sites/regions mentioned in the text. Names which are italicised  
 101 represent sectors of the Laurentide Ice Sheet. Further details on the creation of this map are  
 102 available in Appendix C.

103 Missinaibi Formation, a non-glacial deposit underlying till. Since the HBL is located near the  
104 geographic center of many Pleistocene ice sheets, the age of this non-glacial deposit can be used  
105 to infer the absence of regional ice sheets (e.g. Bos et al., 2009; Helmens et al., 2007; Helmens  
106 and Engels, 2010), therefore improving our understanding of the timing and spatial extent of ice-  
107 free regions during Late Pleistocene glaciations over North America. Furthermore, since this  
108 region is likely to have been a peatland for other ice-free periods in the Pleistocene (Allard et al.,  
109 2012; Terasmae and Hughes, 1960), constraining the age of this deposit will permit empirical  
110 validation of models which simulate carbon storage and potential methane release during that  
111 time (Kleinen et al., 2015).

112         Despite the importance of the Missinaibi Formation as a Pleistocene archive, there is no  
113 consensus on its age or whether the deposits are penecontemporaneous or span much of the Late  
114 Pleistocene. The inability to constrain the age of these deposits reflects that radiocarbon dating  
115 has mostly yielded infinite results and there is a scarcity of suitable material for geochronological  
116 methods such as optically stimulated luminescence (OSL) and uranium-thorium (U-Th) dating.  
117 Despite these issues, previous attempts to constrain the age of the Missinaibi Formation have  
118 resulted in the recognition of at least one MIS 5 (ca. 130,000 to 71,000 yr BP) site via U-Th and  
119 OSL dating (Allard et al., 2012; Dubé-Loubert et al., 2013), which is correlative to the  
120 penultimate interglacial period. Given the substantial glacial retreat during the MIS 5 period  
121 (Andrews and Dyke, 2007; NEEM community members, 2013), such deposits can be expected.

122         There are, however, several sites in the HBL which have yielded MIS 3 ages (Berger and  
123 Nielsen, 1990; McNeely, 2002; Wyatt, 1989). These results have ignited considerable debate,  
124 since an ice-free HBL during that time would imply a significantly different configuration of the  
125 LIS than predicted by glacial models (e.g. Stokes et al., 2012) and what was documented from

126 the LGM to present-day (Dyke, 2004). Furthermore, chronology constraints are largely based on  
127 conventional radiocarbon dates (e.g. Wyatt, 1989), or accelerator mass spectrometry (AMS)  
128 radiocarbon determinations made on peat or shell samples (e.g. McNeely, 2002), which can be  
129 subject to contamination and wide error ranges, depending on the context of samples selected for  
130 dating. As a result, evidence for an ice-free HBL during MIS 3 has been largely dismissed, with  
131 a lack of AMS dates on wood being cited as “a benchmark consideration against the possibility  
132 of Middle Wisconsinan deglaciation of the Hudson Bay Lowland” (McNeely, 2002).

## 133 **1.2 Objectives**

134 Here, we summarize all pre-LGM chronology data in the HBL and contribute new AMS  
135 radiocarbon, OSL and U-Th data to critically evaluate the age(s) of the Missinaibi Formation.  
136 Geochronological data originated from a range of government, academic and unpublished  
137 sources spanning several decades and covering a wide range of uncertainties and errors. To  
138 temper these uncertainties and ensure an objective research approach, we include a short  
139 discussion of all major issues inherent to dating Pleistocene deposits. This information is then  
140 used to rank the chronology data to distinguish between highly-reliable age determinations and  
141 those that have an increased chance of being erroneous. Particular attention is paid to  
142 radiocarbon age estimates, especially discussing the sample material and potential for modern-  
143 day contamination, since the MIS 3 period lies at the limit of this geochronometer. A similar  
144 approach was used by Wohlfarth (2010) to evaluate a pre-LGM chronology dataset from  
145 Sweden, by Hughes et al. (2016) for reconstructing the most recent 40,000 years of glaciation  
146 over Eurasia, and by Forman et al. (2014) for evaluating the chronology of Holocene-aged shells  
147 in Lake Turkana, Kenya.



148 **2. Regional setting**

149           The HBL is a coastal plain encompassing 325,000 km<sup>2</sup> of land, located in central Canada,  
150 and constrained by the uplands of the Canadian Shield, James Bay and Hudson Bay (Riley,  
151 2003) (Fig. 2). This remote region is dominated by ombrotrophic bogs, minerotrophic fens and  
152 permafrost along the northern coast (Riley, 2003), all of which are underlain by Paleozoic and  
153 Mesozoic sedimentary rocks. The HBL is situated a maximum of ~170 m above sea level, with a  
154 gradual decrease in elevation towards the James and Hudson bays. Several major rivers are  
155 deeply incised, but meander through this region, discharging into the James and Hudson bays. A  
156 marine incursion, the Tyrell Sea, inundated large parts of the HBL region following the post-  
157 LGM deglaciation owing to high sea levels and isostatically depressed land (Lee, 1960).

158           In the HBL, non-glacial deposits underlying till were first noted in a series of exploratory  
159 trips in the late 19<sup>th</sup> century (Bell, 1879, 1886), and are comprised of marine, fluvial, peat and  
160 forest-bed units (Skinner, 1973). The marine unit has rarely been noted in the HBL. These  
161 deposits are commonly overlain by two tills (Nguyen, 2014; Skinner, 1973), and subsequently  
162 overlain by Holocene-aged marine, lacustrine and peat deposits. This Pleistocene-aged  
163 stratigraphy is exposed along river banks and ranges in height from 10 to 30 m, with the non-  
164 glacial Missinaibi Formation commonly ranging from 1 to 5 m in thickness. The regional extent  
165 of these deposits is unknown because the occurrence is disparate, but it may be correlative with  
166 non-glacial deposits from central and southern Ontario (e.g. Bajc et al., 2015; DiLabio et al.,  
167 1988).

168           While the reason for the preservation of the Missinaibi Formation is not well understood, the  
169 relatively low topography of the HBL, in combination with the confining topographic high of the

170 Canadian Shield, may have mitigated glacial erosion in this region, thus preserving these  
171 Pleistocene-aged sediments. Furthermore, the Missinaibi Formation commonly contains fluvial  
172 sequences, which would have presumably been deposited in river valleys similar to today, and  
173 these sheltered environments may have acted to protect these deposits from glacial erosion  
174 (Barnett and Finkelstein, 2013).

### 175 **3. Critical evaluation of geochronological techniques**

176 We assembled a database (n = 127) consisting of all previously published (n = 88) and  
177 new (n = 39) geochronological data for the Missinaibi Formation (Appendix A). These data  
178 consist of AMS radiocarbon (n = 44), conventional radiocarbon (n = 36), amino acid (n = 13), U-  
179 Th (n = 14), TL (n = 15) and OSL (n = 5) methods. All chronology data was ranked on a scale of  
180 1 to 3, with '1' representing most reliable dates; '2' representing ages with somewhat more  
181 uncertainty owing to sample material or depositional context, and '3' less reliable dates. Ranks  
182 and rationales are discussed below, and available in Appendix A.

#### 183 **3.1 Radiocarbon dating**

184 Sample material, which can have a substantial bearing on the resulting data, varied  
185 widely in our database. So long as it is not reworked, wood is the ideal material for radiocarbon  
186 dating since cellulose does not exchange carbon with the atmosphere after formation (Bowman,  
187 1990). As a result, we consider wood dates to be reliable (n = 27 <sup>14</sup>C AMS of which 25 are new  
188 contributions; n = 18 <sup>14</sup>C conventional).

189 Peat (n = 8 <sup>14</sup>C AMS; n = 15 <sup>14</sup>C conventional) and shell dates (n = 9 <sup>14</sup>C AMS; n = 12  
190 <sup>14</sup>C conventional), which have unique contamination issues, are common in our database. To  
191 minimize the risk of modern-day contamination, peat samples were examined for root structures,

192 and humic acids were removed prior to radiocarbon dating. Since no root structures were  
193 identified in the samples, and peat dates have been used commonly and accepted in Holocene  
194 HBL studies (Packalen et al., 2014), we assign high confidence to our newly contributed peat  
195 dates (n = 8). If similar details on the removal of humic acids and rootlets from the samples were  
196 noted for previously published peat dates, we consider those dates to be reliable as well.

197 Radiocarbon dating of marine shells from the HBL is problematic because most shells are  
198 located in till (e.g. McNeely, 2002), meaning that they are inherently transported and may not  
199 have originated in the HBL. These shell dates are assigned low confidence because they are not  
200 considered to have been deposited *in situ*. Furthermore, the calcium carbonate component of  
201 shells is commonly subject to post-death isotope fractionation, especially from modern carbon  
202 sources, which can cause artificially young dates (Oviatt et al., 2014; Pigati, 2002). Blake (1988)  
203 attempted to circumvent this issue by dating the inner and outer fraction of an *in situ* shell, but  
204 the inner fraction resulted in an infinite determination (sample GSC-1475 inner/outer), and is  
205 therefore of limited use in our analysis. The only other *in situ* marine shells in our dataset are  
206 from McNeely (2002) (samples AA-7563, TO-2503), however there is limited information about  
207 the pre-treatment and processing of those samples. As a result, we assign lower confidence to  
208 these shell dates in our database.

209 Radiocarbon ages up to 46,401  $^{14}\text{C}$  yr BP were calibrated using the CALIB Rev 7.0.4 and  
210 the INTCAL13 curve (Reimer et al., 2013; Stuiver and Reimer, 1993). Since finite ages greater  
211 than 46,401 (n = 5) exceed the calibration curve, they were left as radiocarbon years (yr  $^{14}\text{C}$ ).  
212 Following Stuiver and Polach (1977), all dates were rounded to the nearest 100, and error values  
213 were rounded up to the nearest 50-year increment. Some ages (n = 3) were not distinguishable

214 from background (Stuiver and Polach, 1977), and were therefore considered to be the same age  
215 as background, which is ca.  $49,600 \pm 950$  yr  $^{14}\text{C}$  (Appendix B).

### 216 **3.2 U-Th dating**

217 Uranium-Thorium dating has provided a chronological constraint for the MIS 5 period in  
218 the HBL (Allard et al., 2012). This method measures the rate of decay of  $^{238}\text{U}$  into daughter  
219 isotope species and can be used to date material up to ca. 350,000 yr BP (Geyh, 2008). The main  
220 requirements for this technique are that the material must contain uranium at deposition, and that  
221 it is not affected by uranium or thorium from the surrounding environment while buried (van  
222 Calsteren and Thomas, 2006). Wood is not commonly dated using this technique because it does  
223 not naturally contain uranium, therefore any uranium uptake must have originated from the  
224 surrounding sediment shortly after burial (Vogel and Kronfeld, 1980). Because U-Th dating of  
225 wood is dependent on initial uranium contamination of the sample, several corroborative age  
226 estimates from the same stratigraphic unit are needed to definitively assign an age (e.g. Allard et  
227 al., 2012; Causse and Vincent, 1989; De Vernal et al., 1986; Mott and Grant, 1985).

228 Wood pieces encased in clay result in limited permeability to surrounding groundwater,  
229 and are preferred for the U-Th method. Such conditions were met by Allard et al. (2012), who  
230 dated 9 wood logs from deposits underlying till along the Nottaway River. Although slightly  
231 different uranium concentrations were recorded on the outer edge of these logs, the inner, less  
232 permeable, portions yielded consistent age determinations (Allard et al., 2012), which we  
233 consider to be reliable. In the western HBL, two U-Th dates from Nielsen et al. (1986) are  
234 considered less reliable owing to the porosity of the surrounding environment (sand, silt), and

235 evidence of thorium contamination, which are suspected to have caused dissimilar isotopic  
236 measurements on wood pieces from the same stratigraphic unit.

237           We made several new attempts to date wood from two sites in the HBL. Two wood  
238 pieces were submitted from 12-PJB-109 for analysis at Geotop, Université du Québec à  
239 Montréal, for which three dates were obtained (Appendix A). However, in all three cases, the  
240 system was believed to be open with respect to uranium, owing to significantly different results  
241 from the same stratigraphic unit. This exchange may have been caused by the composition of the  
242 sediment matrix, which, although clay-rich (~35%), contained ~50 % silt and ~15% sand. This  
243 texture may have promoted water infiltration. As a result, we consider these ages to be minimum  
244 estimates. A further attempt at 12-PJB-007 showed that there was no significant uranium uptake,  
245 therefore an age assignment was not possible at this site, and these results are excluded from our  
246 dataset.

### 247 **3.3 OSL dating**

248           Given that MIS 3, our period of interest, corresponds to the limit of radiocarbon dating,  
249 OSL techniques may hold potential to improve our understanding of the age of HBL deposits.  
250 However, OSL dating can be less successful on sediments derived from the Precambrian Shield,  
251 which yields quartz grains showing low light emissions with optical stimulation (“dim quartz”)  
252 (e.g. Demuro et al., 2013). The reason for this low luminescence signal may be that newly-  
253 eroded quartz has a limited ability to store charge given a minimal number of cycles of dosing  
254 and solar resetting (Sawakuchi et al., 2011). Glacial environments associated with rapid burial  
255 and high energy settings may also result in partial resetting of electron traps (King et al., 2014;

256 Lukas et al., 2007; Rhodes, 2011). As a result, there are no previously published studies which  
257 use OSL on quartz grains from the HBL.

258 In an attempt to resolve this issue, we used OSL dating on quartz at two separate sites,  
259 12-PJB-109 as well as two samples from the Severn Marine site. An *a priori* assumption is that  
260 quartz grains in this fluvial system were not uniformly solar reset because of the short distance of  
261 transport in turbid water conditions and possible deposition during the fall and winter with  
262 sedimentation beneath ice cover. Single aliquot regeneration (SAR) protocols (Murray and  
263 Wintle, 2003; Wintle and Murray, 2006) were used to estimate the apparent equivalent dose for a  
264 different size fraction in each sample (Table 1). For 12-PJB-109, each aliquot contained  
265 approximately 10 to 30 quartz grains corresponding to a 2 mm circular diameter of grains  
266 adhered (with silicon) to a circular aluminum disc of 1-cm diameter. Such a small number of  
267 grains per aliquot was measured to isolate the youngest, full solar-reset grain population (cf.  
268 Duller, 2008). It is suspected that < 20% of grains of each aliquot emitted light, i.e. 2 to 6 quartz  
269 grains.

270 An Automated Risø TL/OSL-DA-15 system was used for SAR analyses with light from  
271 blue diodes. Optical stimulation for all samples was completed at an elevated temperature (125  
272 °C) using a heating rate of 5 °C/s. All SAR emissions were integrated over the first 0.8 s of  
273 stimulation out of 40 s of measurement, with background based on emissions for the last 30- to  
274 40-second interval. In this study, we used the threshold “fast ratio” of > 15 (cf. Durcan and  
275 Duller, 2011) to quantitatively determine aliquots that are dominated by a fast component and  
276 thus, only those aliquots are included in equivalent dose calculations. The majority of aliquots  
277 (>75%) exhibited a clear so called “fast component” (Fig. 3) which is one of the requirements of  
278 the SAR protocols (Murray and Wintle, 2003).

279 Table 1: Optically stimulated luminescence (OSL) ages on quartz grains from the sub-till Missinaibi Formation, Hudson Bay  
 280 Lowland, Canada

281

Sample/ Horizon	Laboratory number	Particle Aliquots <sup>a</sup> Size (µm)	Equivalent dose (Gray) <sup>b</sup>	Over- dispersion (%) <sup>c</sup>	U (ppm) <sup>d</sup>	Th (ppm) <sup>d</sup>	K (%) <sup>d</sup>	Cosmic Dose rate (mGray/yr) <sup>e</sup>	Dose rate (mGray/yr) <sup>f</sup>	OSL age (yr) <sup>g</sup>	
12-PJB-109	BG3800	98/67	250-150	72.27 ± 3.87	62 ± 5	1.22 ± 0.01	6.65 ± 0.01	1.31 ± 0.01	0.16 ± 0.01	1.69 ± 0.09	42,845 ± 3740
Severn Marine 84HBL022	BG3807	90/62	100-63	97.36 ± 6.23	30 ± 3	1.31 ± 0.01	5.78 ± 0.01	1.53 ± 0.01	0.10 ± 0.01	1.64 ± 0.09	52,480 ± 5055
Severn Marine 84HBL023	BG3808	50/30	64-44	85.14 ± 5.26	55 ± 7	1.38 ± 0.01	6.49 ± 0.01	1.64 ± 0.01	0.10 ± 0.01	2.02 ± 0.10	42,190 ± 4010

282

283 <sup>a</sup>Aliquots used in equivalent dose calculations versus original aliquots measured.

284 <sup>b</sup>Equivalent dose calculated on a pure quartz fraction analyzed under blue-light excitation (470 ± 20 nm) by single aliquot regeneration protocols (Murray and  
 285 Wintle, 2003; Wintle and Murray, 2006). A finite mixture model was used with overdispersion values >20% to determine the youngest equivalent dose  
 286 population, with at least 10 aliquots defining this equivalent dose population (Galbraith and Green, 1990).

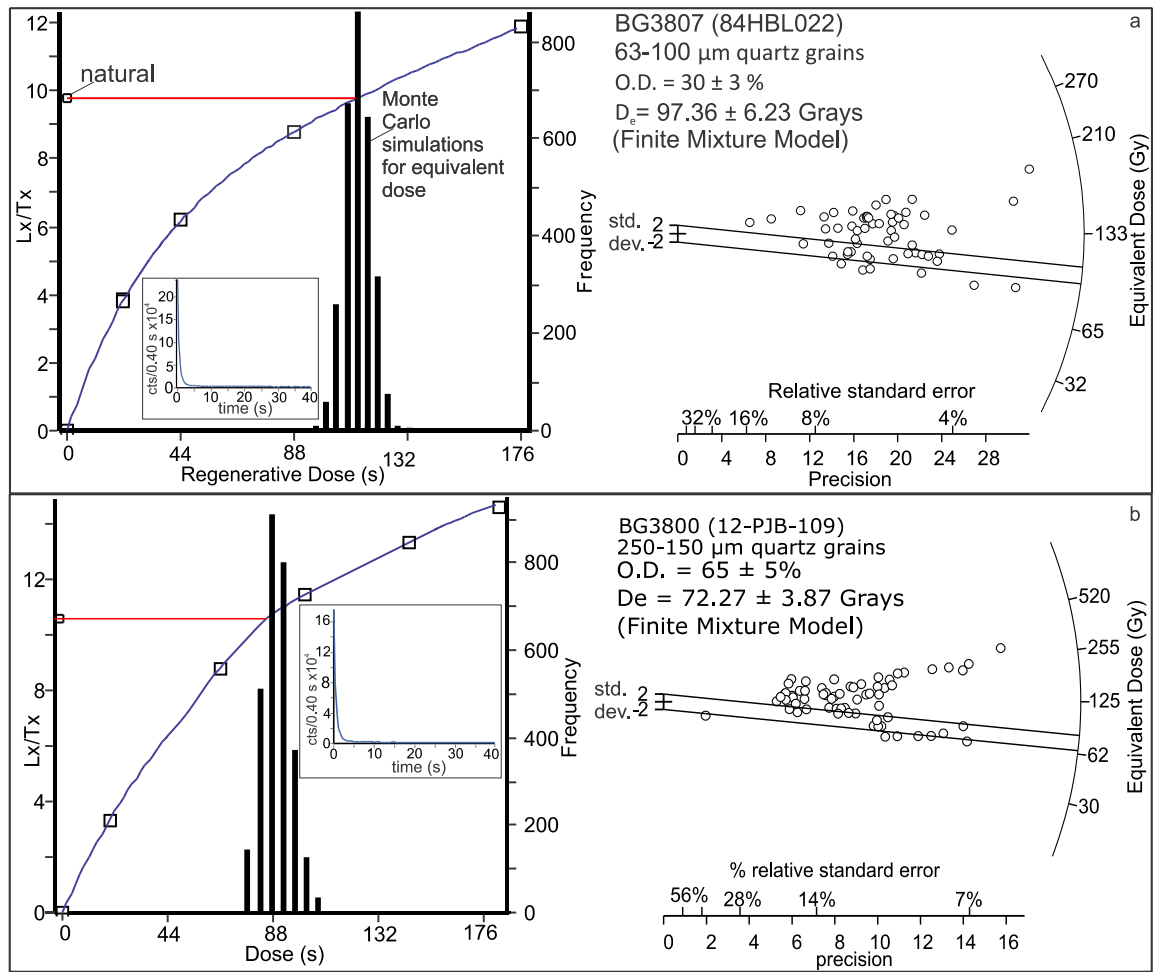
287 <sup>c</sup>Values reflects precision beyond instrumental errors; values of ≤ 20% (at 1 sigma limit) indicate low dispersion in equivalent dose values and an unimodal  
 288 distribution.

289 <sup>d</sup>U, Th and K content analyzed by inductively-coupled plasma-mass spectrometry analyzed by ALS Laboratories, Reno, NV; U content includes Rb equivalent.

290 <sup>e</sup>A cosmic dose rate calculated from parameters in Prescott and Hutton (1994)

291 <sup>f</sup>Assumes a moisture content (by weight) of 25 ± 5% for the burial period

292 <sup>g</sup>Systematic and random errors calculated in a quadrature at one standard deviation. Datum year is AD 2010



293

294

295 **Fig 3** (2 column figure). Optically stimulated luminescence data for quartz grains (BG3807 and

296 BG3800) from waterlain deposits. Inset figure is a representative shine down curve of natural

297 luminescence. Shown are regenerative growth curves, with errors by Monte Carlo simulations

298 and radial plots defining statistic parameters for equivalent dose determinations. Mean equivalent

299 dose was determined by the Finite Mixture Model (FMMM) of Galbraith and Green (1990)

300 because of high overdispersion values  $>25\%$ ; parallel lines denote the lowest significant

301 equivalent dose population defined by at least 20 aliquots.



302 Calculation of equivalent dose by the single aliquot protocols was accomplished for a  
303 majority of aliquots (Table 1). Aliquots were removed from analysis if (1) the fast ratio was <15  
304 (Durcan and Duller, 2011), (2) the recycling ratio was not between 0.90 and 1.10, (3) the zero  
305 dose was >5 % of the natural emission or (4) the error in equivalent dose determination is >10 %.  
306 Equivalent dose ( $D_e$ ) distributions are log normal, highly negatively skewed and exhibited  
307 overdispersion values of 23 % to 103 % (Table 1; Fig. 3). An overdispersion percentage of a  $D_e$   
308 distribution is an estimate of the relative standard deviation from a central  $D_e$  value in context of  
309 a statistical estimate of errors (Galbraith and Roberts, 2012; Galbraith et al., 1999). A zero  
310 overdispersion percentage indicates high internal consistency in  $D_e$  values with 95% of the  $D_e$   
311 values within  $2\sigma$  errors. Overdispersion values  $\leq 20$  % are routinely assessed for small aliquots  
312 of quartz grains that are well solar reset, like far-traveled eolian and fluvial sands (e.g. Meier et  
313 al., 2013; Olley et al., 2004; Wright et al., 2011) and this value is considered a threshold metric  
314 for calculation of a  $D_e$  value using the central age model of Galbraith et al. (1999).  
315 Overdispersion values >20 % may indicate mixing of grains of various ages or partial solar  
316 resetting of grains. The finite mixture model is an appropriate statistical treatment for such data  
317 (Galbraith and Green, 1990), and this model was used to calculate optical ages (Fig. 3; Table 1).

318 In addition to our new OSL data, Dubé-Loubert et al. (2013) dated sediments ( $n = 2$ )  
319 using feldspar grains, which can be used to date sediments back to 500,000 yr BP. However,  
320 feldspar is more commonly affected by anomalous fading, a process whereby electrons gradually  
321 vacate their traps in the absence of light or heat exposure, which can lead to underestimation of  
322 results (Huntley et al., 1985). Dubé-Loubert et al. (2013) applied an equivalent dose correction  
323 developed by Lamothe et al. (2003) to mitigate anomalous fading, and we therefore consider  
324 these data points to be reliable.

### 325 **3.4 Thermoluminescence dating**

326           Similar to OSL dating, TL dating measures the last exposure of a sediment to sunlight.  
327 However, TL dating can be impacted by anomalous fading, which can lead to underestimation of  
328 results (Huntley et al., 1985). This issue can be mitigated by introducing sample preheats or  
329 adding days to weeks of wait time to allow the laboratory-induced luminescence to pre-fade.  
330 Forman et al. (1987) dated two marine sediments samples from the Severn River in the northern  
331 HBL using this approach to mitigate the effects of anomalous fading, and Berger and Nielsen  
332 (1990) used prolonged sample storage to remove pre-fade for five samples along the Nelson  
333 River (Appendix A). Since effort was made to mitigate the issue with anomalous fading, we  
334 retained the data in our dataset and increased the error to  $2\sigma$ .

335           Eight TL samples from non-glacial intervals overlain by till from sites along the Nelson  
336 River were also analyzed by Roy (1998) to determine the extent of solar resetting and anomalous  
337 fading. Seven samples are considered to be unreliable owing to large grain sizes (150 - 250  $\mu\text{m}$ )  
338 which are suspected to have caused improper solar resetting. This insufficient solar resetting was  
339 confirmed by a Holocene-aged sample which resulted in two age estimates of ca. 50,000 yr BP  
340 (Roy, 1998). However, one sample (MOON 2C (delayed)) is more likely a close estimate to the  
341 true depositional age because the grain size is much smaller (4 - 8 $\mu\text{m}$ ), which would have  
342 allowed for prolonged sediment suspension prior to deposition, and therefore more effective  
343 solar resetting. Furthermore, this sample was corrected for anomalous fading by storing for one  
344 year prior to taking this measurement. However, Roy (1998) acknowledges that anomalous  
345 fading may have continued after the one-year delay.

346

### 347 **3.5 Amino Acid Epimerization**

348 Amino acid epimerization of allo-isoleucine to isoleucine from molluscs has provided  
349 some of the first evidence for a large-scale recession of the LIS during MIS 3 (Andrews et al.,  
350 1983). This technique measures the post-mortem changes in amino acid chirality (e.g.  
351 racemization) for molluscs, such as *Hiatella arctica* or *Mya truncata* (Miller and Brigham-  
352 Grette, 1989; Rutter et al., 1979). Such changes to amino acid configuration can be detected for  
353 up to ca. 2,000,000 years, making this method suitable for materials of Pleistocene age (Miller  
354 and Brigham-Grette, 1989).

355 A disadvantage to amino acid dating is that it is a relative dating method. In the HBL,  
356 amino acid age inferences are based on the implicit assumption that the largest ratio corresponds  
357 to a marine incursion during MIS 5e. Younger dates are assigned an age according to this  
358 assumption. Consequently, the application of this technique in the HBL has been controversial  
359 (Andrews et al., 1983; Dyke, 1984), and we assign limited confidence to these age estimates.  
360 Nevertheless, we compiled age estimates from *in situ* shells in the database. Shells from till (e.g.  
361 Andrews et al., 1983; Nielsen et al., 1986; Shilts, 1982; Wyatt, 1989) are not included in this  
362 compilation because they were incorporated and resided within the glacier for an unknown  
363 amount of time where racemization may have ceased or slowed down (Barnett, 1992).

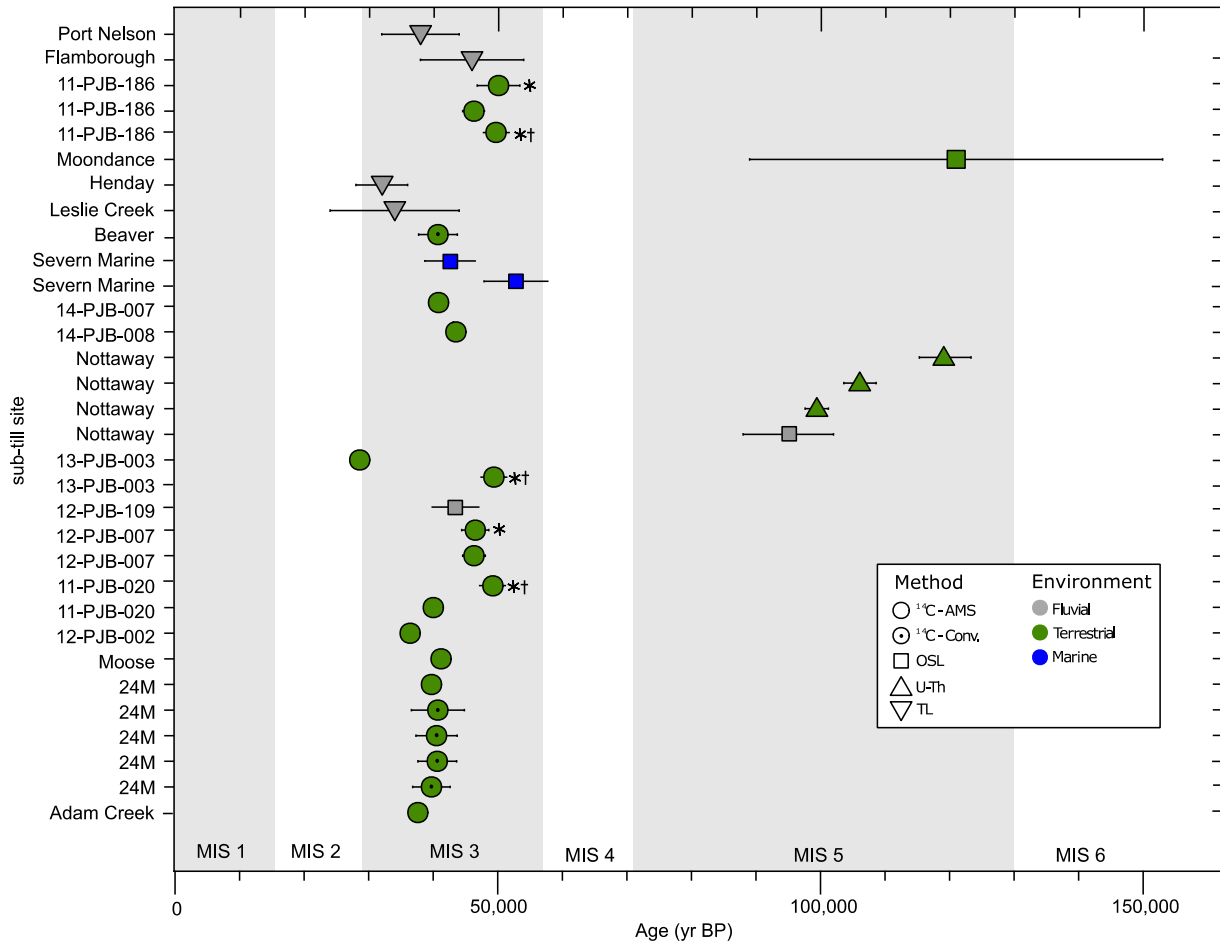
## 364 **4. Results**

365 Geochronological data for the Missinaibi Formation is largely confined to the most recent  
366 130,000 yr BP, with one exception being an OSL date suggesting a fluvial deposit at ca. 211,000  
367  $\pm$  16,000 yr BP from the Harricana River, published by Dubé-Loubert et al. (2013) (sample  
368 06HA30). This data point represents the oldest age estimate in the HBL region, and aligns with

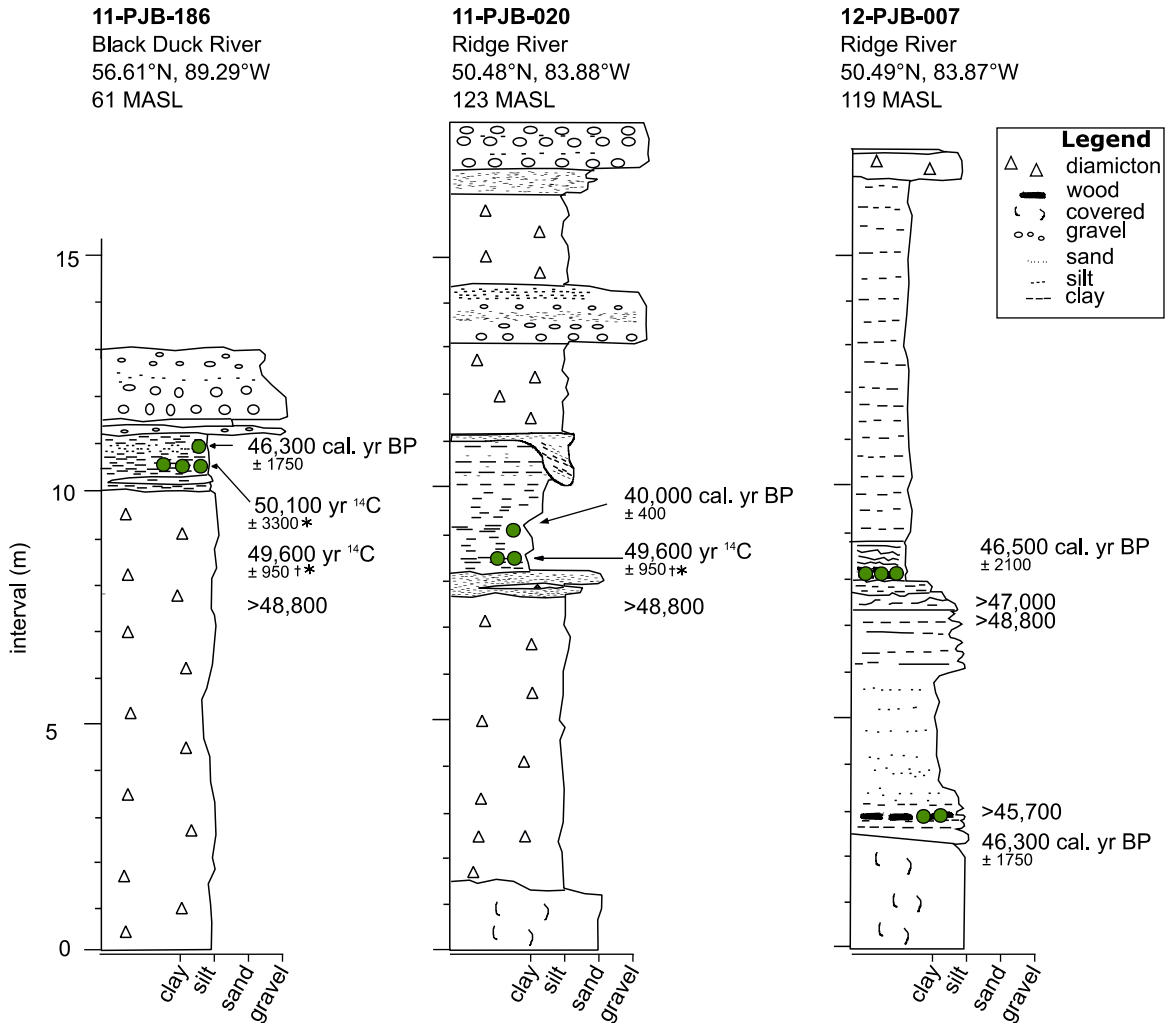
369 the interglaciation of MIS 7 (ca. 243,000 to ca. 190,000 yr BP) (Dubé-Loubert et al., 2013).  
370 Deposits dating to MIS 5 are situated along the Nottaway and Nelson Rivers, and have been  
371 described by Allard et al. (2012), Dubé-Loubert et al. (2013) and Roy (1998) (Fig. 4).

372         Much of our newly contributed data suggests the possibility of an ice-free MIS 3 in the  
373 HBL. Firstly, wood from 11-PJB-186, an organic-rich sequence overlain by post-glacial marine  
374 sediments along the Black Duck River, suggests that organic accumulation began around 50,100  
375  $\pm 3300$   $^{14}\text{C}$  yr BP (sample ISGS A1995) and 49,600  $\pm 950$  yr  $^{14}\text{C}$  (sample UOC-0587), while the  
376 upper part of the unit dates to 46,300  $\pm 1750$  cal. yr BP (sample ISGS A1656) (Fig. 5). Similarly,  
377 two sites located in close proximity ( $\sim 1.3$  km) along the Ridge River, 11-PJB-020 and 12-PJB-  
378 007 have yielded radiocarbon dates of 40,000  $\pm 400$  cal. yr BP (sample UOC-0591), 49,600  $\pm$   
379 950 yr  $^{14}\text{C}$  (sample UOC-0592; Appendix B), 46,300  $\pm 1750$  cal. yr BP (sample UOC-0590) and  
380 ca. 46,500  $\pm 2100$   $^{14}\text{C}$  yr BP (sample ISGS A2424) (Fig. 5). Both sites along the Ridge River are  
381 overlain and underlain by diamicton. At the Severn Marine site, our re-evaluation of TL samples  
382 using OSL techniques have yielded ages of 52,480  $\pm 5055$  (sample BG3807) and 42,190  $\pm 4010$   
383 (sample BG3808) (Fig. 3).

384         Data from the western region of the HBL also suggests an ice-free MIS 3, where Berger  
385 and Nielsen (1990) published a suite of TL data from fluviolacustrine sediments along a  $\sim 100$   
386 km stretch of the Nelson River. Another purported MIS 3 site is 24M, which is considered to be  
387 the type location for the Missinaibi Formation (Skinner, 1973; Terasmae and Hughes, 1960). Our  
388 AMS radiocarbon result of ca. 39,700  $\pm 800$  cal. yr BP (sample TO-1753) corresponds well with  
389 other finite determinations in the range of 39,000 to 41,000 yr BP (Olson and Broecker, 1957,  
390 1959), however is in contrast with several infinite determinations,



391 **Fig. 4** (2 column figure) Summary of chronology data for Pleistocene-aged sites in the Hudson  
 392 Bay Lowlands (HBL), Canada. Sites are arranged from north to south. Asterix (\*) symbol  
 393 represents radiocarbon dates which could not be calibrated because of exceeding the calibration  
 394 curve. Cross (†) symbol represents finite ages which are not statistically distinguishable from  
 395 background, and are therefore considered to be the same age as background. Infinite  
 396 determinations, ages exceeding 150,000 yr BP (n = 1) and those with a high chance of being  
 397 erroneous (rank 3) were excluded from this figure. See Appendix A for more details.



398

399 **Fig. 5** (2-column figure). Detailed stratigraphy of three pre-LGM sites from the Hudson Bay  
 400 Lowlands, Canada, which have the best evidence of being MIS 3 deposits. All chronology data  
 401 presented in this figure are new. Asterix (\*) symbol represents radiocarbon dates which could not  
 402 be calibrated because of exceeding the calibration curve. Cross (†) symbol represents finite ages  
 403 which are not statistically distinguishable from background, and are therefore considered to be  
 404 the same age as background.

405

406 which suggest an older age (MacDonald, 1971; Olson and Broecker, 1959; Preston et al., 1955;  
407 Stuiver et al., 1978; Vogel and Waterbolk, 1972). We therefore consider the age of the 24M site  
408 to be unresolved.

409 In addition to the data listed above, there are several sites for which only one finite age  
410 estimate is available. Although not described in detail here, these samples are all included in  
411 Appendix A as well as plotted in Fig. 4.

## 412 **5. Discussion**

413 Our synthesis of available age estimates for non-glacial materials suggests that the HBL  
414 was ice-free during MIS 7 (Dubé-Loubert et al., 2013), MIS 5 (Allard et al., 2012; Roy, 1998)  
415 and possibly during MIS 3. Deposits dating to MIS 7 or MIS 5 are not surprising given that the  
416 LIS was thought to have retreated considerably at those times. However, if age estimations from  
417 the HBL are correct, deposits dating to MIS 3 imply significant reconfiguration of the LIS.

### 418 **5.1 The validity of >40,000 yr BP radiocarbon dates**

419 A major limitation of our results and subsequent interpretations is that radiocarbon dates  
420 are largely used to constrain the purported ice-free period during MIS 3. This is problematic  
421 because radiocarbon dates in the range of 40,000 to 50,000 yr BP have lost the majority of  $^{14}\text{C}$ ,  
422 and contamination by small amounts of modern carbon can cause otherwise infinite materials to  
423 appear finite (Andrews and Dyke, 2007; Beukens, 1990). For example, 0.2 % modern-day  
424 carbon contamination will cause a 45,000 year old sample to yield an age of 40,000 years  
425 (Olsson and Eriksson, 1972). It is therefore possible that modern or re-worked carbon is  
426 influencing our radiocarbon dates, thus erroneously suggesting an ice-free MIS 3 in the HBL.

427           There is no way to determine whether a single sample has been contaminated by modern-  
428 day carbon. Only repeated measurements showing a high degree of precision can increase  
429 confidence that a true representation of the material's age has been obtained (Scott, 2007). For  
430 example, Bajc et al. (2015) investigated a purported MIS 3 site in Southern Ontario, re-dating  
431 wood pieces using three different cellulose extraction techniques, resulting in age estimate of ca.  
432 42,000 to ca. 50,000 <sup>14</sup>C years BP, therefore strengthening a MIS 3 age assignment at that site. A  
433 similar approach was used at a Late Pleistocene site from Atlantic Canada by Rémillard et al.  
434 (2013), where both peat and wood consistently yielded ages of ca. 47,100 to ca. 50,100 yr BP, all  
435 of which overlap at 1 $\sigma$ , thus supporting the MIS 3 interpretation.

436           In addition to repeated dating of samples, the stratigraphy of age determinations can help  
437 determine whether modern contamination is responsible for finite age estimates. For example, at  
438 the Pilgrimstad site in Sweden, radiocarbon estimates from ca. 40,000 to ca. 50,000 cal. yr BP  
439 were older at the bottom of the sequence and gradually became younger towards the top  
440 (Wohlfarth, 2010 and references therein). If modern-day carbon contamination had influenced  
441 these age estimates, we would expect all determinations to be artificially finite, as well as  
442 possible age reversals in the stratigraphic sequence. Since age estimates largely follow  
443 stratigraphic order, it re-enforces the MIS 3 age assignment.

444           Following the techniques outlined above, to strengthen age estimates for the Missinaibi  
445 Formation, we made an effort to (1) sample several intervals at a given site to determine if the  
446 resulting age estimates follow stratigraphic order, and (2) date samples multiple times, and at  
447 different radiocarbon laboratories, to test the precision and reproducibility of each age  
448 assignment. These efforts were focussed on three purported MIS 3 sites, 11-PJB-186, 11-PJB-  
449 020 and 12-PJB-007 and the results can be seen in Fig. 5 and Appendix A. Although some re-



450 dating attempts were limited because of low material availability, chronology data at these sites  
451 largely follows stratigraphic order, and samples which have been dated multiple times show  
452 significant reproducibility. Such an agreement would not be expected if these finite estimates  
453 were the result of modern carbon contamination. Therefore, on the basis of radiocarbon dating,  
454 we find no reason to discount the chronology at these three sites. Results from OSL dating  
455 further support the MIS 3 interpretation (Fig 2). However, fluvial and/or marine sediments were  
456 often missing from the sub-till sites, thus a direct comparison of OSL and radiocarbon dates from  
457 the same site has not yet been done. Nevertheless, both radiocarbon and OSL results suggest an  
458 ice-free HBL during MIS 3. The discovery of new sub-till sites to perform OSL dating may hold  
459 potential to significantly improve our understanding of the age of the Missinaibi Formation.

460 The abundance of infinite radiocarbon dates ( $n = 47$ ) is also worth considering, although the  
461 interpretation is challenging. Given that the MIS 3 period corresponds to ca. 29,000 to ca. 57,000  
462 yr BP, radiocarbon dating should be able to capture any deposit up to ca. 50,000 yr BP, only  
463 missing those that lie at the lower boundary for MIS 3. It is possible that some of these infinite  
464 age estimates may be from that time. It is equally possible that these infinite dates represent  
465 multiple non-glacial intervals from earlier in the Pleistocene, perhaps correlative with the late  
466 MIS 5 ages from the Nottaway River (Allard et al., 2012). Based solely on chronological  
467 evidence, we do not consider the presence of infinite radiocarbon dates to be evidence in favor or  
468 against any particular age assignment for the Missinaibi Formation.

## 469 **5.2 Ice Sheet dynamics during MIS 5**

470 Based on available age estimates, the warmest part of the penultimate interglacial, MIS 5e  
471 (peak: ca. 123,000 yr BP), which has been identified elsewhere in Canada (e.g. Fréchet and de

472 Vernal, 2013; Karrow et al., 2001), is not preserved in the non-glacial sediments of the HBL at  
473 sites presented here. Instead, OSL data from the Nottaway River, and one TL age from the  
474 Nelson River correspond to the latter part of the MIS 5 interglaciation (Allard et al., 2012; Dubé-  
475 Loubert et al., 2013; Roy, 1998).

### 476 **5.3 Laurentide Ice Sheet during MIS 3**

477 Our data suggests that the HBL may have been deglaciated during ca. 50,000 to 40,000 yr  
478 BP, which, according to RSL and  $\delta^{18}\text{O}$  from benthic foraminifera (Grant et al., 2014; Lisiecki  
479 and Raymo, 2005), corresponds to a time of partial deglaciation of the North American  
480 continent. If correct, data from the HBL constrains the ice-free eastern lobe of the LIS by 700 km  
481 westward and northward than what is suggested by most other Late Pleistocene sites. In Southern  
482 Ontario, these sites include conventional radiocarbon dates on sub-till material from a borehole  
483 and creek exposure (Karrow and Warner, 1984; Warner et al., 1988), three finite AMS dates on  
484 bone and peat samples from a sub-till site exposed along a railroad cut (Karrow et al., 2001) and  
485 six finite AMS dates on sub-till wood fragments from a quarry (Bajc et al., 2015). In Atlantic  
486 Canada, Rémillard et al. (2013) documented four finite AMS ages on sub-till peat, which  
487 suggests that this region may have also been deglaciated during MIS 3. Fréchette and de Vernal  
488 (2013) also infer a deglaciation in Atlantic Canada during MIS 3, but no geochronological data  
489 was available at that site, and instead, age control was based on the stratigraphic position of the  
490 sub-till deposits.

491 Radiocarbon data from Repulse Bay, northwest of the HBL, may provide corroborative  
492 evidence for a very significant glacial recession during MIS 3. Recently-obtained radiocarbon  
493 data suggests that this region was ice-free for several thousand years during MIS 3 (McMartin et

494 al., 2015). However, notably, these data were based on marine shells, which may have associated  
495 uncertainties (see Section 3.1). Nevertheless, duplicate samples analyzed by different  
496 laboratories produced the same interpretation at that site (McMartin et al., 2015), which  
497 strengthens the interpretation. Together with data from the HBL, there seems to be a growing  
498 amount of evidence suggesting that large parts of eastern and central North America may have  
499 been ice-free during MIS 3.

500 If evidence for a significant glacial recession during MIS 3 is correct, other parts of North  
501 America must have been fully glaciated to compensate for the relatively low sea level during that  
502 time (Grant et al., 2014). It may be possible that the mid- and western regions of North America  
503 were glaciated. For example, TL, and radiocarbon data from the Roxana Silt suggest the  
504 presence of the LIS in the mid-continent during MIS 3 (Forman, 1992; Forman and Pierson,  
505 2002). Records from the Gulf of Mexico, most of which are dated using a series of AMS dates  
506 on foraminifera, also suggest that the LIS spanned into the continental United States for large  
507 parts of MIS 3 (Hill et al., 2006; Sionneau et al., 2013; Tripsanas et al., 2007).

508 Based on available age estimates of the Missinaibi Formation, it seems that the western  
509 sector of the LIS (Keewatin) was highly active, and the eastern sector (Labrador-Nouveau  
510 Québec) may have experienced restricted growth following MIS 5 and into MIS 3. Expansion of  
511 the eastern sector may have been preferentially eastward onto the expanding continental shelf as  
512 RSL fell. Its southern extension may have been affected by the isostatically depressed St  
513 Lawrence River valley, slowing expansion into the lower Great Lakes. This eastern sector of the  
514 LIS may have only reached the western end of Lake Ontario during MIS 3. In this scenario, it is  
515 possible for parts of the HBL to have remained unglaciated.

516 The lack of a marine unit at the base of most dated MIS 3 sites may provide supportive  
517 evidence for a MIS 3 age assignment. In the HBL, marine incursions can be expected  
518 immediately following deglaciation as a result of isostatic depression of the land and the close  
519 proximity to Hudson Bay (e.g. Tyrell Sea; Lee, 1960). To account for this missing marine unit,  
520 the Missinaibi Formation could have been deposited at a time when ice had recently receded  
521 beyond the boundaries of the HBL, but when significant parts of the continent remained  
522 glaciated to maintain low RSL, thus preventing a large-scale marine incursion. The early part of  
523 MIS 3 is the only time during the Late Pleistocene when what may have been an extensive  
524 deglaciation is not followed by a substantial rise in sea level to levels similar to present-day  
525 (Grant et al., 2014). We would expect such conditions to prevent a large-scale marine incursion  
526 in the HBL, allowing instead the growth of peat, forest bed and fluvial deposits directly  
527 overlying till, corresponding to the observed Missinaibi Formation. However, two newly-  
528 contributed OSL dates from the Severn River suggest that a marine incursion may have  
529 inundated the outer region of the HBL during this time (Fig. 3, 4).

530 Irrespective of the configuration of the LIS during ca. 50,000 to ca. 42,000 yr BP, there is  
531 a general consensus of substantial continental glaciation between ca. 42,000 to ca. 35,000 yr BP  
532 which would likely have covered the entire HBL region. Karig and Miller (2013) document a  
533 proglacial lake in upper New York state from ca. 37,000 to ca. 34,000 yr BP, and Berger and  
534 Eyles (1994) document till in Southern Ontario at ca. 41,000 yr BP, indicating the proximal  
535 presence of a glacial lobe during that time. Furthermore, sedimentological evidence from the  
536 Gulf of Mexico suggests that the eastern lobe of the LIS was extended beyond Lake Ontario at  
537 that time (Sionneau et al., 2013; Tripsanas et al., 2007), and radiocarbon and OSL dating of cave

538 sediments indicates that the LIS may have grown to almost the LGM limit between ca. 40,000 to  
539 ca. 30,000 yr BP (Wood et al., 2010).

540 Taken together, evidence suggests that the LIS covered large parts of North America  
541 from ca. 42,000 to ca. 35,000 yr BP, which would have undoubtedly glaciated all of the HBL  
542 during that time. Our shortage of age estimates from this time period may be taken as indirect  
543 evidence for a fully glaciated HBL, since this time period is well within the acceptable range of  
544 most geochronological methods. After this purported glaciation, there may have been a brief  
545 retreat of the LIS at ca. 30,000 yr BP (Dyke et al., 2002), followed by a rapid build-up of the ice  
546 sheet towards the LGM (Dyke et al., 2002; Lambeck et al., 2014).

## 547 **6. Conclusions**

548 Our review of chronology data from the HBL, Canada, helps to constrain the boundaries  
549 of the LIS for periods prior to the LGM, which can help validate important models of ice sheet  
550 extent, build-up and growth (Ganopolski and Calov, 2011; Ganopolski et al., 2010; Kleman et  
551 al., 2010; Stokes et al., 2012). Chronology data suggests that the HBL was ice-free during parts  
552 of MIS 7, MIS 5 and possibly during parts of MIS 3. While glacial retreats at MIS 7 and MIS 5  
553 are well-documented, evidence for a ice-free central region of the LIS during MIS 3 is  
554 noteworthy, since these data extend the ice-free eastern lobe of the LIS by at least 700 km  
555 westward and northward from what is suggested by existing Late Pleistocene sites in Southern  
556 Ontario and Atlantic Canada (Bajc et al., 2015; Rémillard et al., 2013).

557 Although largely based on radiocarbon determinations, evidence for an ice-free HBL  
558 during the MIS 3 period is reinforced by (1) our successful efforts to re-date purported MIS 3  
559 sites and test the reliability of radiocarbon dating at the limit of this geochronometer, (2)

560 paleorecords from Atlantic Canada and Southern Ontario suggesting largely ice-free conditions  
561 during MIS 3 (e.g. Bajc et al., 2009; Bajc et al., 2015; Rémillard et al., 2013), and for which the  
562 western extent is unknown, and (3) a strong agreement between low RSL during MIS 3 and the  
563 lack of marine deposits in the Missinaibi Formation. Future iterations of relevant Earth system  
564 models should include land-based information of the layout and configuration of previous ice  
565 sheets, along with results from till correlations (Dubé-Loubert et al., 2013; Kaszycki et al., 2008;  
566 Nguyen, 2014), geomorphic evidence of ice flow regimes (Kleman et al., 2010; Veillette et al.,  
567 1999) and models of ice volume (Peltier et al., 2015).

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576

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