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# Lu-Hf systematics of 4.0 – 2.3 Ga old zircons from the Turee Creek Group (Pilbara Craton, W. Australia): Implications on the rise of atmospheric oxygen and global glaciation during the Paleoproterozoic

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1 **Lu-Hf systematics of 4.0 – 2.3 Ga old zircons from the Turee Creek**  
2 **Group (Pilbara Craton, W. Australia): Implications on the rise of**  
3 **atmospheric oxygen and global glaciation during the Paleoproterozoic**

4  
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17

18 **Abstract**

19 We investigated the Hf isotopic systematics of detrital zircons in a succession of  
20 siliciclastic sediments and glacial diamictites from the early Paleoproterozoic sequence of the  
21 Boolgeeda Iron Formation (Hamersley Group) and overlying Turee Creek Group of the  
22 Pilbara Craton, Western Australia. About 400 detrital zircons yielding > 95% concordant U-  
23 Pb ages were analyzed for Hf isotopes to constrain their magmatic sources. 70% of the  
24 analyzed zircons display super-chondritic initial Hf isotopic compositions, demonstrating  
25 crystallization in mantle-derived magmas. Most of the data are comprised between model age

26 lines at ~2.5 and 3.2 Ga, which suggests a sub-continuous crust generation by extraction from  
27 the depleted mantle during this time period. A single grain yields a 4.0 Ga age, which  
28 represents the first Hadean age for a zircon from the Pilbara Craton. Our results are  
29 significantly different from zircon Hf isotope data of the Glenburgh Terrane, in the southern  
30 border of the Turee Creek Group, or older successions of the Pilbara, Kaapvaal and Superior  
31 cratons, but show overlap with some of the Yilgarn Craton. This together with the occurrence  
32 of a Hadean zircon crystal preserved in the Boolgeeda glacial diamictite with similar Hf  
33 isotopic signature than the Jack Hills zircons makes the Yilgarn Craton a possible source  
34 material for the Boolgeeda glacial horizon. Alternatively, the majority of the zircons analyzed  
35 show ages which are consistent with those of the underlying 2.45 – 2.78 Ga Hamersley and  
36 Fortescue groups, formed by sedimentary successions interleaved with thick subaerial  
37 volcanic sequences associated with the emplacement of Large Igneous Provinces. Such  
38 subaerial volcanic rocks could account for the relatively juvenile character of the zircon  
39 analyzed. A local provenance of the siliciclastic material delivered to the Turee Creek Basin  
40 would support the role of large subaerial magmatic provinces as triggers of the rise of  
41 atmospheric oxygen and the onset of glaciations at the beginning of the Proterozoic.

42

### 43 *Keywords*

44 Zircon; Hafnium isotopes; Turee Creek Group; Great Oxidation Event; Paleoproterozoic  
45 glaciations; Hadean.

46

## 47 **1. Introduction**

48 The Archean-Proterozoic transition was marked by important redox and climatic  
49 changes, specifically the rise of atmospheric oxygen, the so-called Great Oxidation Event  
50 (GOE) (Holland, 2002), and several Paleoproterozoic glacial events (e.g., Rasmussen *et al.*,

51 2013; Caquineau *et al.*, 2018). The causal relationships between these events remain largely  
52 debated (e.g., Kopp *et al.*, 2005; Teitler *et al.*, 2014). One model proposed by Kump and  
53 Barley (2007) explained the rise of atmospheric oxygen by a change in the eruptive dynamic  
54 of the Earth at ca. 2.5 Ga that shifted the main style of volcanism from marine to subaerial.  
55 This increase in the subaerial proportion of Large Igneous Provinces (LIPs) would have  
56 buffered the greenhouse gases content of the atmosphere due to carbon dioxide consumption  
57 through enhanced silicate weathering, thus enabling atmospheric oxygen to build up and  
58 surface temperature to cool down, leading to global glaciation (Cox *et al.*, 2016). In addition,  
59 subaerial eruptions are known to produce more oxidized lavas and gases (Holland *et al.*, 2002;  
60 Gaillard *et al.*, 2011), which could also have contributed further to reduce oxygen sinks.

61 Zircon is a very resistant mineral, of which Hf is a major constituent (~ 1wt%), that  
62 represents the only witness of eroded and unknown source protoliths (Iizuka *et al.*, 2017). The  
63 incorporation of significant amounts of mantle-derived volcanic material within the upper  
64 continental crust can generate a large increase of juvenile Hf isotopic signatures in the zircons  
65 from the sediments. Glacial diamictites are considered to record the average chemical  
66 composition of the upper continental crust from which they formed by erosion (Gaschnig *et*  
67 *al.*, 2014). Consequently, U-Pb-Hf systematics in zircons sampled in these sediments appears  
68 as a valuable strategy to test the possible occurrence of materials derived from mantle sources.  
69 Here, we performed a Lu-Hf isotopic study of detrital zircons previously analyzed for U-Pb  
70 geochronology (Caquineau *et al.*, 2018) from a suite of glaciogenic and non-glaciogenic  
71 sediments of Paleoproterozoic age that spans the GOE. The rocks investigated are from the  
72 Boolgeeda Iron Formation, which represents the uppermost succession of the Hamersley  
73 Group, and the Turee Creek Group (TCG) in the Hamersley Basin at the southern margin of  
74 the Pilbara Craton, Western Australia (Fig. 1). The main objective of this study is to (i)  
75 identify the major sources and further constrain the formation's model of this sedimentary

76 sequence and (ii) to test the hypothesis of massive subaerial LIPs emplacement in the Pilbara  
77 Craton and its implications on the GOE and the Paleoproterozoic glaciations.

78

## 79 **2. Geological setting**

80         The Turee Creek Group (TCG) is a ca. 4 km-thick continuous sedimentary succession  
81 conformably overlying the Boolgeeda Iron Formation of the Hamersley Group and  
82 unconformably overlain by the Beasley River Quartzite and the Cheela Springs Basalt of the  
83 lower Wyloo Group (Figs. 1, 2; Martin, 1999; Van Kranendonk *et al.*, 2015). From bottom to  
84 top, the TCG consists of siliciclastic sedimentary rocks, minor stromatolitic carbonates and  
85 glaciogenic strata of the Kungarra Formation, quartz-rich sandstones of the Koolbye  
86 Formation, and shales and stromatolitic marine carbonates of the Kazput Formation (Fig. 2b;  
87 Trendall, 1981; Martin, 1999; Van Kranendonk *et al.*, 2015). The TCG sedimentary sequence  
88 has been described as a broad shallowing upward profile from deep-water iron formation of  
89 the Boolgeeda Iron Formation, through fine-grained siliciclastic deposits of the Kungarra  
90 Formation, to fluvial and shallow marine strata of the Koolbye and Kazput formations  
91 (Martin, 1999; Mazumder *et al.*, 2015; Van Kranendonk *et al.*, 2015). The Boolgeeda Iron  
92 Formation overlies the Woongarra Rhyolite and Weeli Wooli Formation, which have been  
93 interpreted as a subaerial bimodal mafic and felsic volcanic sequence emplaced at 2450 Ma  
94 (Barley *et al.*, 1997). This intense volcanic event would have produced large volumes of lavas  
95 ( $>30\,000\text{ km}^3$ , Barley *et al.*, 1997) covering the continental surface at the time of the glacial  
96 event recorded in the Boolgeeda Iron Formation, and possibly during the subsequent  
97 deposition of the overlying TCG. The unconformably overlying Beasley River Quartzite  
98 represents a terrestrial deposit composed of a basal fluvial conglomerate and overlain by an  
99 unit of fine-grained quartzite (Mazumder and Van Kranendonk, 2013). Metamorphism of the  
100 group did not exceed sub-greenschist facies conditions (Rasmussen *et al.*, 2005).

101 Several sections of the TCG have been described in the southwestern margin of the  
102 Hamersley Basin (Martin, 1999; Van Kranendonk *et al.*, 2015; Krapež *et al.*, 2017). These  
103 include from East to West: the Turee Creek, Hardey and Duck Creek synclines, as well as the  
104 Yeera Bluff area (Fig. 1b). Glaciogenic horizons were used as reference markers to establish  
105 stratigraphic correlations among these different sections. The main glacial body is the ~400m  
106 thick Meteorite Bore Member (MBM) that was originally described in the Hardey Syncline  
107 section (Trendall, 1981; Figs. 1, 2b and e). More recently, Van Kranendonk and Mazumder  
108 (2015) reported a second level of massive diamictites located above the reference Meteorite  
109 Bore Member in the Hardey Syncline; in the following, we will refer to this horizon as the  
110 ‘second Kungarra diamictite’. A third diamictite horizon associated with sandstones and  
111 siltstones was described in the Duck Creek Syncline at the Boundary Ridge locality and at  
112 Yeera Bluff located 50 and 150 km north westward from the Hardey Syncline, respectively  
113 (Fig. 1b, Martin, 1999; Van Kranendonk *et al.*, 2015). These diamictites, associated with deep  
114 marine sediment of the Boolgeeda Iron Formation, were interpreted as distal equivalents of  
115 the Meteorite Bore Member formed as sediment gravity flows and rain-out from rare icebergs  
116 (Martin, 1999; Van Kranendonk *et al.*, 2015). However, Caquineau *et al.* (2018) and Philippot  
117 *et al.* (2018) recently reported the occurrence of a 2m-thick diamictite horizon within the  
118 underlying Boolgeeda Iron Formation in the Hardey Syncline (drill core TCDP1, see below  
119 and Fig. 2d). Because this diamictite layer is stratigraphically located ~1500 meters below the  
120 Meteorite Bore Member, it thus represents a different and older glacial event. Based on new  
121 geochronological (see below) and stratigraphic constraints, Caquineau *et al.* (2018) and  
122 Philippot *et al.* (2018) showed that this diamictite layer can be correlated with the diamictites  
123 horizons reported in the Boolgeeda Iron Formation at Yeera Bluff and the Boundary Ridge  
124 locality. This glacial horizon is named hereafter the ‘Boolgeeda diamictite’.

125 Time constraints on the deposition of the sedimentary sequence of interest include: (i)  
126 a U-Pb zircon age of  $2450 \pm 3$  Ma for the underlying Woongarra Rhyolite (Figs. 1, 2a and b;  
127 Trendall *et al.*, 2004), (ii) a U-Pb detrital zircon maximum age of  $2460 \pm 9$  Ma for the  
128 Boolgeeda diamictite (Caquineau *et al.*, 2018), (iii) a Re-Os age of  $2312 \pm 6$  Ma obtained on  
129 diagenetic pyrite from the Meteorite Bore Member diamictite (Philipipot *et al.*, 2018) in  
130 agreement with a U-Pb maximum age on detrital zircon of  $2340 \pm 22$  Ma (Caquineau *et al.*,  
131 2018) for this glacial formation, and (iv) a U-Pb zircon age of  $2209 \pm 15$  Ma for the overlying  
132 Cheela Springs Basalt (Martin *et al.*, 1998). A baddeleyite U-Pb age of  $2208 \pm 10$  Ma was  
133 obtained on dolerite sills that intrude the TCG and are truncated by the unconformably  
134 overlying Beasley River Quartzite (Müller *et al.*, 2005). Thus, the significance of the age of  
135 the Cheela Springs Basalt has been questioned (Müller *et al.*, 2005). Accordingly, the age of  
136 the Beasley River Quartzite remains loosely bracketed between  $\sim 2210$  Ma and  $2031 \pm 6$  Ma  
137 (Fig. 2a, Martin *et al.*, 1998; Müller *et al.*, 2005).

138 The sedimentation of the TCG is considered to have occurred in the McGrath Trough,  
139 an asymmetric basin structurally linked with the migration to the northeast of a geanticline or  
140 a thrust-fold belt system (e.g., Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015). This basin  
141 has been interpreted either as a retroarc foreland basin (Martin *et al.*, 2000; Müller *et al.*,  
142 2005; Krapež *et al.*, 2017) or as an intracontinental basin (Van Kranendonk *et al.*, 2015). It is  
143 generally accepted that the Turee Creek Group formed in a long-lived basin between  $\sim 2.45$   
144 and 2.2 Ga (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015). An alternative view (Krapež *et*  
145 *al.*, 2017), which proposes that the TCG formed in a short-lived foreland basin that closed by  
146  $\sim 2.43$  Ga or shortly after, can be ruled out by recent geochronological constraints on the  
147 MBM diamictites mentioned above (Caquineau *et al.*, 2018; Philipipot *et al.*, 2018). The  
148 provenance of the sediments forming the TCG is subject of debate, mainly because it relies on  
149 controversial paleocurrent data, which established that the detritus was principally derived

150 from the south and the southwest (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015; Krapež  
151 *et al.*, 2017). The major source terranes include the Pilbara Craton, the Hamersley Province to  
152 the north (Martin *et al.*, 2000; Van Kranendonk *et al.*, 2015), and the Glenburgh Terrane to  
153 the south (Krapež *et al.*, 2017), a continental exotic fragment that is thought to have collided  
154 with the southwestern margin of the Pilbara Craton ca. 2.2 Ga ago (Johnson *et al.*, 2011;  
155 2013). The occurrence of three discrete glacial diamictite horizons, and new age constraints of  
156 the TCG led Caquineau *et al.* (2018) to propose that the ca. 2.45 Ga-old Boolgeeda diamictite  
157 can be correlated with (1) the pre-2.43 Ga Makganyene Formation of the South African  
158 Transvaal Supergroup (Gumsley *et al.*, 2017), (2) the basal glaciogenic horizon of the Ramsay  
159 Lake Formation of the North American Huronian Supergroup (Rasmussen *et al.*, 2013), and  
160 the ca. 2.43 Ga old Fennoscandian glacial diamictites (Melezhik *et al.*, 2013). This early  
161 glacial event at about 2.45 Ga recognized in five different cratons could therefore have been  
162 global in extent (i.e., Snowball Earth). Caquineau *et al.* (2018) further proposed that the two  
163 diamictites of the Kungarra Formation (the MBM and the second Kungarra diamictite) can be  
164 correlated with the pre-2.31 Ga Gowganda Formation (North America) and upper Deutschland  
165 (South Africa) glacial diamictites (Rasmussen *et al.*, 2013), and the ca. 2.26 Ga old upper  
166 Timeball Hill (North America) and Ditojana (South Africa) diamictites (Rasmussen *et al.*,  
167 2013), as well as the Enchantment Lake Formation (North America).

168

### 169 **3. Samples**

170 The samples analyzed in this study have been described in detail in Caquineau *et al.*  
171 (2018). These include drill core samples collected in the course of the Turee Creek Drilling  
172 Project (TCDP1 and TCDP2) obtained at the base and intermediate level of the Turee Creek  
173 Group, respectively, as well as surface samples collected in lithostratigraphic levels not  
174 intercepted by the drill cores (Fig. 2). Six samples were selected from different stratigraphic



175 levels in drill cores TCDP 1 and TCDP 2 and eight complementary samples were collected in  
176 the field (Fig. 2; drill core samples are noted T1-X or T2-X, with X referring to the depth;  
177 surface samples are labelled with Pi- followed by the sampling year and the sample number).  
178 These include from bottom to top: Three glaciogenic dropstone-bearing sandstones of the  
179 Boolgeeda glacial diamictite (T1-152 and Pi-09.07 and Pi-09.08 of the Boundary Ridge  
180 Locality), a sandstone layer at the base of TCDP 2 (T2-332) and four samples of the main  
181 glacial diamictite deposit of the Meteorite Bore Member (T2-272, T2-205, T2-169, T2-130),  
182 three samples of the second Kungarra glacial diamictites (Pi-15.15, Pi-15.16, Pi-15.17), two  
183 samples from meter-scale massive sandstone bars of the upper part of the Kazput Formation  
184 (Pi 15.41, Pi-15.42), and one sample of the Beasley River Quartzite (Pi-15.02).

185 The Hf isotopic composition was measured in 382 detrital zircon grains from  
186 diamictites, sandstones and quartzites that previously gave > 95% concordant U-Pb dates  
187 (Fig. 2 and Table 2). These include: (1) 82 zircons from the Boolgeeda diamictite both from  
188 the Hardey (drill core sample T1-152) and Duck Creek (surface samples Pi 09.07 and Pi  
189 09.08) synclines (Fig. 2c and d), (2) 108 zircons from the Meteorite Bore Member diamictite  
190 (drillcore samples T2-130, T2-169, T2-205 and T2-272) and an underlying sandstone bar (T2-  
191 332) (Fig. 2e), (3) 101 zircons from the second Kungarra diamictite (surface samples Pi  
192 15.15, Pi15.16, Pi 15.17), (4) 59 zircons from two sandstone layers of the upper part of the  
193 Kazput formation (surface samples Pi 15.41, Pi 15.42) (Fig. 2f), and (5) 32 zircons from the  
194 Beasley River Quartzite (surface sample Pi 15.02).

195

#### 196 **4. Methods**

197 The cathodoluminescence image (CL) of the Hadean mounted zircon grain was  
198 generated using a Zeiss Sigma FEG-SEM equipped with a detector operating at 15kV current.  
199 Mineral separation involved crushing and sieving at the threshold of 300  $\mu\text{m}$ , Wilfley table,

200 Frantz magnetic separator and heavy liquids. U-Th-Pb isotopic data on zircons were obtained  
201 by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) at the  
202 Laboratoire Magmas & Volcans (LMV, Clermont-Ferrand, France). Detailed analytical  
203 techniques are reported in Hurai *et al.* (2010), Paquette *et al.* (2014) and Caquineau *et al.*  
204 (2018).

205 Hafnium isotope analyses were performed with a Thermo Scientific Neptune Plus  
206 Multicollector (MC) ICPMS coupled to a Resonetics M50E 193nm Excimer laser system at  
207 the LMV (Clermont-Ferrand). The operating conditions and instrument settings are described  
208 in Moyen *et al.* (2017) and Paquette *et al.* (2017) and detailed in S1. Repeated analyses of  
209 both natural (91500, GJ-1 and Mud-Tank) and synthetic Hf and REE-doped (Fischer *et al.*,  
210 2011) zircon reference materials are reported in Table S1. More than 50 replicates of 91500,  
211 GJ-1 and Mud Tank zircon reference materials yielded  $^{176}\text{Hf}/^{177}\text{Hf}$  of  $0.282307 \pm 16$ ,  
212  $0.282011 \pm 15$  and  $0.282520 \pm 12$ , respectively, which well agree with reference values (eg.  
213 Hu *et al.*, 2012). Mean and two standard deviation values obtained on MUNZirc 0-2a  
214 ( $^{176}\text{Hf}/^{177}\text{Hf} = 0.28213 \pm 3$ ), MUNZirc 1-2b ( $0.28214 \pm 2$ ); MUNZirc 3-2b ( $0.28215 \pm 2$ ) and  
215 MUNZirc 4-2b ( $0.28216 \pm 3$ ) are fully consistent with reference values (Fisher *et al.*, 2011).  
216 The  $\epsilon_{\text{Hf}}$  values are calculated as parts per 10,000 deviation of the  $^{176}\text{Hf}/^{177}\text{Hf}$  of the zircons at  
217 the time of their formation relative to the Chondritic Uniform Reservoir (CHUR) defined by  
218 the  $^{176}\text{Lu}$  decay constant of  $1.867 \times 10^{-11}\text{yr}^{-1}$  (Söderlund *et al.*, 2004) and the CHUR  
219 parameters ( $^{176}\text{Hf}/^{177}\text{Hf} = 0.282793$ ;  $^{176}\text{Lu}/^{177}\text{Hf} = 0.0338$ ; Iizuka *et al.*, 2015). A  $^{176}\text{Lu}/^{177}\text{Hf}$   
220 ratio of 0.015 typical of bulk continental crust (Griffin *et al.*, 2004) has been used for  
221 representing model age lines.

222

## 223 **5. Zircon Hf isotopic compositions**

224 Hf isotopic data are presented in Table 1. The zircons from the Boolgeeda diamictite  
225 yielded a range of  $^{207}\text{Pb}/^{206}\text{Pb}$  ages between 2436 and 3216 Ma, as well as a single Hadean  
226 grain (discussed below), and initial  $\epsilon_{\text{Hf}}(t)$  values between -12.4 to +6.2 (Fig. 3a). About half  
227 of the analyzed zircons display a supra-chondritic Hf isotopic composition (i.e. > 0) reflecting  
228 the addition of material derived from partial melting of the depleted mantle at an unknown  
229 time. In contrast, the remaining zircons analyzed shows a sub-chondritic Hf initial isotopic  
230 composition (i.e. < 0) indicating that zircons parental melts recycled an evolved crust.

231 Zircons from the MBM diamictite yielded a range of  $^{207}\text{Pb}/^{206}\text{Pb}$  ages between 2359  
232 and 3000 Ma and initial  $\epsilon_{\text{Hf}}(t)$  values between -5.2 and +7.6 (Fig. 3b). The mantle-derived  
233 component is proportionally more important in the MBM diamictite than in the Boolgeeda  
234 diamictite and the time interval appears shifted toward slightly younger ages. Zircons from  
235 the second Kungarra diamictite yielded a range of  $^{207}\text{Pb}/^{206}\text{Pb}$  ages between 2430 and 3350  
236 Ma and initial  $\epsilon_{\text{Hf}}(t)$  values between -11.8 and +6.7 (Fig. 3 c). Again the addition of a mantle-  
237 derived component appears predominant as recorded by chondritic to supra-chondritic Hf  
238 initial isotopic compositions. This pattern appears very similar to that of the Booldeega  
239 diamictite. Zircons from the sandstones of the upper part of the Kazput Formation yielded a  
240 range of  $^{207}\text{Pb}/^{206}\text{Pb}$  ages between 2430 and 3230 Ma and initial  $\epsilon_{\text{Hf}}(t)$  values between -7.4  
241 and +6.4 (Fig. 3d). Most of the data display supra-chondritic Hf initial isotopic composition,  
242 which demonstrates a prevailing input of mantle-derived component. These Hf isotope data  
243 share similarities with those of the MBM diamictite (Fig. 4b).

244 Zircons from the Beasley River Quartzite yielded a range of  $^{207}\text{Pb}/^{206}\text{Pb}$  ages between  
245 2450 and 3230 Ma and initial  $\epsilon_{\text{Hf}}(t)$  values between -10.4 and +5.9 (Fig. 3e). Almost equal  
246 proportions of sub- and supra-chondritic zircon Hf isotopic composition were recorded. The  
247 results present similar ages and Hf initial isotopic signatures than the other formations.

248           When all the analyses are plotted together (Fig. 3f), two major points are evidenced:  
249 (1) the majority of the U-Pb ages range from 2.45 Ga to 3.20 Ga, and (2) more than 70% of  
250 the analyses plot in the supra-chondritic field, which demonstrates the prevailing occurrence  
251 of mantle-derived materials. Considering a  $^{176}\text{Lu}/^{177}\text{Hf}$  ratio of 0.015 typical of bulk  
252 continental crust (Griffin et al., 2004), the data can be confined between two model age lines  
253 at *c.a.* 2.54 Ga and 3.20 Ga. As discussed by Vervoort and Kemp (2016), these ages are  
254 dependent of a number of factors such as mantle source models and Lu/Hf values and  
255 therefore should not be taken at face values and only considered as indicative of the time  
256 when the source of the zircon's protoliths separated from the mantle.

257

## 258 **6. The first record of a Hadean zircon in the Pilbara Craton**

259           Four analyses of a single zircon crystal (120 x 60  $\mu\text{m}$  in size) from sample Pi 09.07 of  
260 the 2.45 Ga Boolgeeda diamictite at the Boundary Ridge locality yielded an upper intercept at  
261  $4013 \pm 69$  Ma. One of the analyses is concordant at  $3998 \pm 15$  Ma (Fig. 4a and Table 2). The  
262 Th/U ratios (0.2 to 0.8) favour a magmatic origin for the crystal. This zircon crystal represents  
263 the first record of a Hadean relict within the Pilbara Craton. The zircon shows a negative  
264 initial  $\epsilon_{\text{Hf}}(t)$  value of  $-3.3$  (Fig. 4b), which is consistent with the filtered Hf isotope dataset  
265 for Jack Hills zircons in the Yilgarn Craton after Whitehouse et al. (2017). These authors  
266 interpreted these data to represent zircon growth events during the Hadean, although the  
267 nature of these events remains unconstrained. As discussed by Kemp et al. (2010), it is  
268 therefore tempting, although speculative, to propose the crystallization of this Hadean zircon  
269 in a magma source derived from the Depleted Mantle and carrying an evolved crustal  
270 component earlier than 4.0 Ga. Depending on the nature of the crust (i.e. felsic or mafic) from  
271 which the zircon crystallized, the Hf model ages are 4.27 or 4.49 Ga (Fig. 5b), which are in

272 agreement with Nd model ages obtained on samples from the 3.5 Ga Dresser Formation in the  
273 Pilbara Craton (Tessalina *et al.*, 2010).

274

## 275 **7. Discussion**

### 276 **7.1 Summary of the zircon Hf isotopic signatures**

277 Zircon Hf isotopic data appear relatively consistent throughout the sedimentary  
278 sequence of the Boolgeeda Iron Formation and the Turee Creek Group. About 70 % of the  
279 analyzed zircons are characterized by chondritic to supra-chondritic Hf isotopic compositions,  
280 with 30% of the analyses showing  $\epsilon_{\text{Hf}}(t)$  values higher than +3. Most of the data plot between  
281 model age lines at ~2.5 and 3.2 Ga (Fig. 3f). Although these ages are not representative of the  
282 time of extraction of juvenile magmas from the depleted mantle (Vervoort and Kemp, 2016),  
283 the lack of evidence for a crustal contribution older than 3.2 Ga indicates that the majority of  
284 the data is consistent with a sub-continuous or multi-episodic crust generation through  
285 depleted mantle differentiation after about 3.2 Ga. Eo- and Paleo-Archean crustal rocks did  
286 not contribute significantly to the sedimentary material of the Boolgeeda diamictite and TCG  
287 sediments, in agreement with zircon age peaks at 2.54, 2.68, 2.82, 2.95 and 3.15 Ga  
288 (Caquineau *et al.*, 2018). The recognition that the zircon Hf isotopic compositions of the  
289 Beasley River Quartzite (unconformably overlying the Turee Creek Group) are  
290 undistinguishable from the Boolgeeda and Turee Creek zircon Hf compositions further  
291 support the notion of a relatively homogeneous source over a substantial long period of time  
292 between ~2.45 and 2.0 Ga.

293

### 294 **7.2 Paleogeographic significance of the zircon Hf isotopic data**

295 To evaluate the main sources of the Boolgeeda diamictite and the TCG sediments we  
296 have considered our isotope data in the broader context of published radiogenic isotope data

297 for igneous rocks from several representative cratons in Australia (Glenburgh Terrane and  
298 Pilbara and Yilgarn cratons), South Africa (Kaapvaal Craton) and North America (Superior  
299 Craton).

300

### 301 *7.2.1 Glenburgh Terrane*

302 The Glenburgh Terrane has been defined as an exotic continental fragment that  
303 collided with the southwestern margin of the Pilbara Craton during the ~2.22 – 2.15 Ga  
304 Ophthalmian orogeny (Blake & Barley, 1992; Martin *et al.*, 2000; Johnson *et al.*, 2011).  
305 Zircons from the 2430–2555 Ma old Halfway Gneiss, which represents the oldest component  
306 of the Glenburgh Terrane, have a variety of Paleoproterozoic and Archean ages and a  
307 relatively narrow range of sub-chondritic Hf isotopic compositions interpreted to reflect  
308 reworking of an older 2600–2730 Ma crust (Johnson *et al.*, 2011). Some of the ages and Hf  
309 isotopic data overlap those of the Boolgeeda diamictite and TCG detrital zircons (Fig. 5a), but  
310 only a few of the zircons from the Glenburgh Terrane match the supra-chondritic values  
311 recorded in our samples. Hence, while the Glenburgh Terrane may have contributed to Turee  
312 Creek basin (Krapež *et al.*, 2017), it cannot be considered as a major source material of the  
313 Boolgeeda diamictite and TCG sediments.

314

### 315 *7.2.2 Pilbara Craton.*

316 Magmatic zircons from the Pilbara Craton mainly crystallized at about 3.2–3.3 Ga or  
317 before (Fig. 5b; Gardiner *et al.*, 2017; 2019; Petersson *et al.*, 2019 and references therein).  
318 These TTG suites are interpreted as derived from partial melting of basaltic crust with the  
319 contribution from older felsic components (e.g. xenoliths of the Strelley Pool Formation up to  
320 3.7 Ga; Gardiner *et al.*, 2019). After ca. 3.3 Ga, the trend toward more negative Hf values is  
321 interpreted by these authors as recording more extensive crust reworking. In the eastern

322 Pilbara Craton, multiple pulses of magmatism produced voluminous granitic complexes from  
323 3.5 Ga to 2.95-2.92 Ga and 2.85-2.82 Ga. This age range is much older than the one recorded  
324 for the Boolgeeda and TCG detrital zircons. As shown in figure 5b, the Hf isotope data from  
325 both central and eastern Pilbara do not overlap with those of the TCG, ruling out any  
326 significant contribution of the Pilbara Craton to this sedimentary succession.

327

### 328 *7.2.3 Northern Yilgarn Craton*

329 The Yilgarn Craton located south of the Pilbara Craton and Glenburgh Terrane  
330 consists of several distinct tectonic entities (the Narryer, Youanmi and South West terranes  
331 and the Eastern Goldfields Superterrane) that have been accreted to the amalgamated Pilbara  
332 Craton–Glenburgh Terrane during the ~1.96 – 2.0 Ga Glenburgh orogeny (Occhipinti *et al.*  
333 2004; Johnson *et al.*, 2011, 2013), about 200 Ma after deposition of the TCG (i.e. 2210 Ma,  
334 Müller *et al.*, 2005). Available Hf isotope data of magmatic zircons from the Yilgarn Craton  
335 (Griffin *et al.*, 2004) show similarities with our results. Both display supra-chondritic values  
336 during Neoproterozoic times, a quite similar range of ages and only a few zircon grains older than  
337 3.2 Ga (Fig. 5c). However, the Boolgeeda sample also contains younger grains with supra-  
338 chondritic Hf isotopic compositions down to 2.4 Ga compared to the Yilgarn zircons (2.6 Ga).  
339 Nevertheless, the strong overlap between our Hf isotope data and those of the Yilgarn Craton  
340 together with the occurrence of a Hadean zircon crystal in the Boolgeeda diamictite, similar in  
341 age and Hf isotopic signature to the Jack Hills zircons (Whitehouse *et al.*, 2017 and references  
342 therein) makes the Yilgarn Craton a possible source for the Boolgeeda glacial horizon. This  
343 conclusion is in apparent contradiction with the cratonisation history and paleogeographic  
344 reconstruction of the Yilgarn and Pilbara cratons, which are considered to be part of two  
345 different supercratons during the early Paleoproterozoic (Cheney, 1996; de Kock *et al.*, 2009;  
346 Smirnov *et al.*, 2013), and to have accreted during the ~1.96 – 2.0 Ga Glenburgh orogeny

347 (Johnson *et al.*, 2013), that is about 200 Ma later than the upper age limit of the TCG (Müller  
348 *et al.*, 2005; see chapter 7.4 below for further discussion).

349

#### 350 *7.2.4 Kaapvaal Craton*

351 Magmatic zircons from the Kaapvaal Craton mainly crystallized from material that is  
352 much older than the one recorded for the Boolgeeda and TCG detrital zircons (Fig. 5e, Zeh *et*  
353 *al.*, 2009). Assuming the ‘Vaalbara’ hypothesis (Cheney, 1996; deKock *et al.*, 2009; see  
354 Eriksson *et al.*, 2011 for an alternative hypothesis), the post-2080 Ma Magaliesberg Quartzite,  
355 which is stratigraphically located at the top of the Transvaal Supergroup, could represent an  
356 analog of the TCG sediments. Two populations of zircons can be distinguished on the basis of  
357 U-Pb ages and initial  $\epsilon_{\text{Hf}}(t)$  values (Fig. 5d, Zeh *et al.*, 2016). One is characterized by Archean  
358 age and both supra- and sub-chondritic Hf isotopic signatures; the second population shows  
359 Paleoproterozoic age and sub-chondritic Hf isotopic compositions (Fig. 5d). Our data from  
360 the Boolgeeda diamictite and TCG sediments mostly plot between these two zircon  
361 populations, which argue for distinctly different sources. The zircons of Archean age  
362 (population 1) of the Magaliesberg Quartzite have been attributed to the erosion of the  
363 Pietersburg Block of the Kaapvaal Craton. The absence of major overlap with the Boolgeeda  
364 and TCG zircons indicates that this terrane was not a major source of the Boolgeeda and TCG  
365 sediments. The source of the Paleoproterozoic zircons (population 2) is unclear. The dominant  
366 crustal recycling signature indicated by the strongly negative  $\epsilon_{\text{Hf}}(t)$  values could be linked to  
367 the break-up of the Vaalbara Supercontinent between ~2.45 and 2.2 Ga. Accordingly, U-Pb  
368 geochronology, Hf isotopes and paleogeographic reconstructions all argue against the  
369 Kaapvaal Craton as a possible source for the Boolgeeda diamictite and TCG sediments.

370

#### 371 *7.2.5 Superior Craton*



372           The Paleoproterozoic glacial events also affected the Superior Craton during the  
373 deposition of the Huronian Supergroup (Young, 1970). Available zircon Hf isotopic data from  
374 the Superior Craton are mostly supra-chondritic (Corfu and Noble, 1992; Corfu and Stott,  
375 1993 and 1996; Davis *et al.*, 2005) and characterize samples older than 2.7 Ga. As shown in  
376 figure 5f, data from the Boolgeeda diamictite, the TCG and the Superior Craton are mostly  
377 different, likely due to different cratonization histories.

378

### 379 **7.3 Implications for a prevailing mantle-derived magma source**

380           The comparison of our detrital zircon  $\epsilon_{\text{Hf}}(t)$  data with those of the literature shows that  
381 the Glenburgh Terrane and the Pilbara, Kaapvaal, Zimbabwe and Superior cratons did not  
382 contribute significantly to the Boolgeeda and TCG sediments. Over 80% of the analyzed  
383 zircons yielded ages spanning those of the Hamersley and Fortescue groups between 2.45 and  
384 2.78 Ga (Arndt *et al.*, 1991; Trendall *et al.*, 2004). This supports the interpretation that the  
385 Boolgeeda Iron Formation and the TCG successions mainly derived from the erosion of the  
386 underlying Hamersley and Fortescue groups. Martin *et al.* (2000) and Van Kranendonk *et al.*,  
387 (2015), originally proposed this interpretation based on geological and stratigraphic  
388 reconstructions. As shown by their supra-chondritic Hf isotopic compositions, detrital zircons  
389 from the Boolgeeda diamictite and the TCG successions preferentially incorporated mantle-  
390 derived material. This is not typical of terrigenous sedimentation in cratonic settings, where  
391 erosion products generally record diverse lithologies of various ages reflecting the erosion of  
392 older crustal basement characterized by sub-chondritic Hf isotopic compositions. The marked  
393 differences in zircon Hf compositions between the Boolgeeda diamictite and TCG sediments,  
394 and the Pilbara Craton (Fig. 5b) and the Glenburgh Terrane (Fig. 5a) is particularly striking,  
395 as these two continental blocks are the most likely sources in addition to the Fortescue and  
396 Hamersley groups.

397 A possible explanation for the minor proportion of sub-chondritic Hf isotopic  
398 compositions in the Boolgeeda diamictite and TCG zircons can be a lack of emerged crustal  
399 blocks carrying a sub-chondritic Hf isotope record at the time of deposition. This is  
400 particularly critical for the Pilbara Craton to the north and the Glenburgh Terrane to the south,  
401 which have been considered as the main potential sources of the TCG successions (e.g.,  
402 Krapez *et al.*, 2017). The scarcity or lack of surface exposure of Pilbara Craton lithologies  
403 seems unlikely as it comprises a wide range of rocks of different ages between 2.7 and 3.5 Ga  
404 that crop out on thousands of km<sup>2</sup> (Hickman and Van Kranendonk, 2012). Additionally, the  
405 Mount Bruce Supergroup, which contains the TCG and the underlying Hamersley and  
406 Fortescue groups, was unconformably deposited on the basement of the Pilbara Craton (Arndt  
407 *et al.*, 1991). The lack of detrital zircon much older than 3.2 Ga within the TCG may also be  
408 related to the absence of efficient transport and/or erosion of Archean material. However,  
409 sandstones of the Koolbye Formation and of the Beasley River Quartzite (see Fig. 2) have  
410 been interpreted as fluvial to shallow marine, and fluvial to aeolian deposits, respectively  
411 (Mazumder and Van Kranendonk, 2013; Mazumder *et al.*, 2015), witnessing the activities of  
412 rivers and wind during the deposition of the TCG.

413 About 75% of the zircons analyzed here come from glaciogenic deposits  
414 (i.e. Boolgeeda, MBM and second Kungarra diamictites). Glaciogenic rocks typically derive  
415 from widespread physical erosion of the upper continental crust by icesheets and are therefore  
416 geographically dispersed (e.g., Gaschnig *et al.*, 2014). The supra-chondritic Hf isotopic  
417 composition recorded in the major part of the zircon populations from the Boolgeeda and  
418 Turee Creek sediments is at odd with a sub-chondritic Hf isotopic composition generally  
419 expected for the recycled upper continental crust (e.g., Condie, 2014). The Welli Wooli  
420 Formation of the Hamersley Group, comprising basalts, dolerites as well as the Woongarra  
421 Rhyolite, has been interpreted as a subaerial bimodal Large Igneous Province (LIP) emplaced

422 at 2450 Ma (Barley *et al.*, 1997). This intense volcanic event would have produced large  
423 volumes of lavas ( $>30\,000\text{ km}^3$ , Barley *et al.*, 1997) covering the continental surface at the  
424 time of the glacial event recorded in the Boolgeeda Iron Formation, and possibly during the  
425 subsequent deposition of the overlying TCG. Thus, it is suggested that the erosion of these  
426 volcanic materials by ice sheets and their incorporation as terrigenous detritus within the  
427 glacial diamictites of the Boolgeeda and Turee Creek sedimentary successions could account  
428 for the supra-chondritic Hf isotopic compositions of many analyzed zircons (Fig. 3).  
429 Similarly, zircons corresponding to age peaks at 2540 and 2680 Ma could have been produced  
430 by previous volcanic eruptions attending deposition of the Fortescue and Hamersley groups  
431 and preserved at the continental surface. It is noteworthy that the Hf isotopic compositions of  
432 zircons from non-glacial sediments, i.e the sandstones of the Kazput Formation and the  
433 Beasley River Quartzite, do not exhibit different  $\epsilon_{\text{Hf}}(t)$  vs. time patterns than the zircons from  
434 the glacial diamictites (Fig. 3d and 3e). This suggests that these sandstones and quartzites may  
435 represent the erosional products of the same kind of LIP-derived volcanic material involved in  
436 the formation of the glacial diamictites.

437         The hypothesis that the Earth's surface was covered by juvenile lavas during the  
438 Archean-Proterozoic transition, at least on the Pilbara Craton, may also have important  
439 implications on the concomitant rise of atmospheric oxygen (the GOE) and drastic cooling of  
440 the Earth's surface. As shown by Kump and Barley (2007), a rapid increase of the subaerial  
441 proportion of LIPs at ca. 2.5 Ga would have abruptly and permanently diminished the  
442 predominant sink for oxygen (submarine volcanism) and increased the sink for carbon dioxide  
443 (silicate weathering). This in turn would have enabled oxygen to build up into the atmosphere  
444 and triggered a glacial period (Cox *et al.*, 2016).

445

#### 446 **7.4 Geodynamic implications of a Hadean zircon in the Pilbara Craton**

447 Several studies have suggested that the Pilbara Craton developed on a Hadean  
448 continental substrate (Smithies *et al.*, 2003, 2007; Champion, 2013; Tessalina *et al.*, 2010).  
449 Such ancient crust has, however, never been identified in outcrop. The oldest rock reported so  
450 far is the metamorphosed Mount Webber Gabbro located in the western part of the Shaw  
451 Granitic Complex, which formed at 3.59–3.58 Ga (Petersson *et al.*, 2019), and therefore  
452 predates the oldest known volcanic activity of the 3.53–3.23 Ga Pilbara Supergroup within  
453 the East Pilbara Terrane. Many Hadean zircons identified in Western Australia are from the  
454 Jack Hills meta-conglomerate of the Narryer Formation in the neighboring Yilgarn Craton  
455 (Wilde *et al.*, 2001, see above). The significant overlap between our Hf isotope data and those  
456 of the Yilgarn Craton, together with the occurrence of an Hadean zircon crystal preserved in  
457 the Boolgeeda diamictite with similar age and Hf isotopic signature than the Jack Hills  
458 zircons (Whitehouse *et al.*, 2017 and references therein) makes the Yilgarn Craton a possible  
459 source material for the Boolgeeda glacial horizon. Available paleogeographic reconstructions  
460 based on paleomagnetic data and cratonization history suggest that during the  
461 Paleoproterozoic, the Yilgarn and Pilbara cratons were part of two different supercratons, the  
462 Zimgarn comprising the Zimbabwe and Yilgarn cratons (Smirnov *et al.*, 2013), and the  
463 Vaalbara composed of the Kaapvaal and Pilbara cratons (Cheney, 1996; de Kock *et al.*, 2009).  
464 This argues against Yilgarn and Zimbabwe cratons as a source for the Turee Creek and  
465 Boolgeeda zircons (Fig. 5c and d). However, Gumsley *et al.* (2017) recently proposed that  
466 despite their dissimilar cratonization history, the Zimbabwe and Yilgarn cratons may have  
467 been part of a much larger supercraton, the Supervaalbara, which comprised Superior,  
468 Wyoming, Hearne, Kola, Karelia, Kaapvaal, and Pilbara. Considering that the 2.45 Ga old  
469 Boolgeeda glacial diamictite has been correlated with several glacial horizons in South Africa  
470 (pre-2.43 Ga Makganyene Formation of the Griqualand West region), North America (2.45  
471 Ga Ramsay Lake Formation of the Superior Province) and Arctic Europe (2.43 Ga

472 Urkkavaara and Polisarka Formations of Fennoscandian Craton), Caquineau et al., (2018)  
473 proposed that it could represent a global glacial event. Accordingly, although the Yilgarn  
474 craton may have been located at a latitude very different from that of the Turee Creek Basin  
475 during the Paleoproterozoic, it cannot be excluded that the Hadean zircon preserved in the  
476 Boolgeeda diamictite has been transported by icesheets over significant distances.

477

## 478 **8. Conclusions**

479 Our zircon U-Pb-Hf isotopic study indicates that Neoproterozoic (to Mesoproterozoic)  
480 mantle-derived material was the major detrital component delivered to the Hamersley basin  
481 during deposition of the glacial and non-glacial sediments of the Boolgeeda Iron Formation  
482 and the Turee Creek Group, as well as the overlying Beasley River Quartzite. In this study,  
483 we discovered the first occurrence of a Hadean zircon grain in the Pilbara Craton. Rare  
484 materials older than 3.2 Ga were recycled within the Turee Creek basin. Our data highlight a  
485 much larger proportion of mantle-derived source materials than in comparable cratons and  
486 basins, for which zircon Hf isotopic data are available. The predominantly supra-chondritic  
487 Hf isotopic signatures can be explained by a relatively large proportion of subaerial volcanic  
488 rocks during deposition of the underlying 2.45 – 2.78 Ga Hamersley and Fortescue groups. A  
489 local origin of the material delivered to the Turee Creek basin is in agreement with an  
490 intracontinental depositional setting. The massive emplacement of subaerial LIPs in the  
491 Hamersley and Fortescue groups during the Neoproterozoic followed by subsequent erosion and  
492 alteration attending the formation of the Turee Creek basin may have triggered the rise of  
493 atmospheric oxygen and associated Paleoproterozoic glacial events.

494

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501

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698

### 699 **Figure captions**

700 Figure 1: Localization of the Pilbara Craton in Western Australia (a). Geological maps of (b)  
701 the Pilbara Craton and Hamersley Basin in Western Australia with Turee Creek Group  
702 outcrops in yellow and (c) the Hardey Syncline area with locations of the TCDP drill cores  
703 and surface samples (showed with numbers from 1 to 5) analyzed in this study (Modified  
704 after Martin et al. (2000) and Van Kranendonk et al. (2015).

705

706 Figure 2: Stratigraphic logs of (a) the Mount Bruce Supergroup, which comprises the  
707 Fortescue, Hamersley, and Turee Creek groups, and the overlying lower Wyloo Group with  
708 geochronological constraints, (b) the complete section of Turee Creek Group in the Hardey  
709 Syncline, (c) the Turee Creek Group section at the Boundary Ridge locality, (d) drill core  
710 TCDP1, (e) drill core TCDP2, and (f) the upper Kazput cross section (see Fig. 1 b). (a)  
711 Modified after Trendall *et al.* (2004) and Philippot *et al.* (2018), (b, d, e) after Philippot *et al.*  
712 (2018) and (c, f) after Caquineau *et al.* (2018). Samples analyzed for their Hf isotope  
713 compositions include from bottom to top: (1) Boolgeeda diamictite (T1-152, Pi-09.07, Pi-  
714 09.08), (2) a sandstone layer of the Kungarra Formation (T2-332) and diamictites of the  
715 Meteorite Bore Member (T2-272, T2-205, T2-169 and T2-130), (3) second Kungarra  
716 diamictite MBM2 (Pi-15.41, Pi-15.42) (4) two massive sandstones of the upper Kazput  
717 section (Pi-15.15, Pi-15.16, Pi-15.17) and (5) Beasley River Quartzite (Pi-15.02).

718 Abbreviations: WoD, Woolly Dolomite, CSB, Cheela Springs Basalt, BRQ, Beasley River  
719 Quartzite, Ka, Kazput Formation, Kb, Koolbye Formation, MBM, Meteorite Bore Member,  
720 Ku, Kungarra Formation, TCG, Turee Creek Group, Bo, Boolgeeda Iron Formation, WR,  
721 Woongarra Rhyolite, WW, Weeli Wolli Formation, B IF, Brockman Iron Formation, MSW,  
722 Mount Mac Rae Shale – Mt. Sylvia Formation – Wittenoon Formation, M IF, Marra Mamba  
723 Iron Formation, Jee, Jeerinah Formation, Mad/Bu, Maddina Basalt/Bunjinah Formation,  
724 Tu/Py, Tumbiana Formation/Pyradie Formation, Ky/Bo, Kylena Formation/Boongal  
725 Formation, MRB, Mount Roe Basalt, Be, Bellary Formation. References for the  
726 geochronological constraints are: (1) Müller *et al.* (2005), (2) Martin *et al.*(1998), (3)  
727 Philippot *et al.* (2018), (4) Caquineau *et al.* (2018), (5) Trendall *et al.* (2004), (6) Arndt *et al.*  
728 (1991).

729

730 Figure 3:  $\epsilon_{Hf}(t)$  vs age diagrams for each sample: (a) Boolgeeda diamictite, (b) MBM  
731 diamictite, (c) second Kungarra diamictite, (d) upper Kazput sandstones, (e) Beasley River  
732 Quartzite, (f) all data. CHUR, Chondritic Uniform Reservoir, DM, Depleted Mantle. The DM  
733 evolution line corresponds to a  $^{176}\text{Lu}/^{177}\text{Hf}$  ratio of 0.03933 (Blichert-Toft and Puchtel, 2010)  
734 evolving from the chondritic initial composition starting at 4567 Ma (Iizuka *et al.*, 2015).The  
735 data can be confined between two model age lines at *c.a.* 2.54 Ga and 3.20 Ga. Hf model age  
736 lines are represented for a  $^{176}\text{Lu}/^{177}\text{Hf}$  ratio of 0.015 typical of bulk continental crust (Griffin  
737 *et al.*, 2004).

738

739 Figure 4: A Hadean zircon in the Pilbara Craton. (a) U-Pb Concordia plot of zircon analyses  
740 showing an upper intercept at  $4013 \pm 69$  Ma and a Concordia date of  $3998 \pm 15$  Ma. Inset:  
741 cathodoluminescence image of the grain with laser spots indicated. (b)  $\epsilon_{Hf}(t)$  vs. age  
742 diagrams of the Hadean zircon with model age lines for two different natures of crust, a felsic

743 crust with a  $^{176}\text{Lu}/^{177}\text{Hf}$  ratio of 0.015 (Griffin *et al.*, 2004), and a mafic crust with a  
744  $^{176}\text{Lu}/^{177}\text{Hf}$  of 0.026 (Blichert-Toft and Albarède, 2008). The filtered domain of Jack Hills  
745 zircon analyses after Whitehouse *et al.* (2017) is represented by the yellow box.

746

747 Figure 5: Comparison of  $\varepsilon_{\text{Hf}}(t)$  data obtained in this study with literature data. The Boolgeeda  
748 diamictite and Turee Creek Group data are represented as grey squares in all diagrams. (a)  
749 Halfway Gneiss of the Glenburgh Terrane, (Johnson *et al.*, 2011), (b) Pilbara Craton, (Kemp  
750 *et al.*, 2015; Gardiner *et al.*, 2017 and 2019; Petersson *et al.*, 2019), (c) The northern part of  
751 the Yilgarn Craton, (Griffin *et al.*, 2004). (d,) South Africa: Magaliesberg Quartzite, (Zeh *et*  
752 *al.*, 2016), Kalahari Craton (Zeh *et al.*, 2009). (e) Superior craton (Corfu and Noble, 1992;  
753 Corfu and Stott, 1993 and 1996; Davis *et al.*, 2005).

754

## 755 **Tables Content**

756 **Table1.** Zircon Lu-Hf isotope data obtained by in *situ* Laser Ablation MC-ICP-MS.

757

758 **Table 2.** Zircon U-Th-Pb data from the Hadean zircon crystal obtained by in situ Laser  
759 Ablation ICP-MS.

760

761 **Supplementary Table S1.** Detail of analytical conditions for Lu-Hf isotope analysis by *in*  
762 *situ* Laser Ablation MC-ICP-MS. Lu-Hf isotope data on natural GJ-1, 91500 and Mud Tank  
763 zircon reference materials and synthetic zircons doped with hafnium and rare earth elements  
764 (Fischer *et al.*, 2011b) are included.