#### SUPPLEMENTARY INFORMATION

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# Globally asynchronous sulphur isotope signals require re-definition of the Great Oxidation Event

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### 9 Supplementary Note 1 - Geological setting

10 The Turee Creek group stratigraphy has been described as a broad shallowing-upward profile from 11 deep-water banded iron formation of the Boolgeeda Iron Formation, through fine-grained siliciclastic 12 deposits of the Kungarra Formation, to fluvial and shallow marine strata of the Koolbye and Kazput formations<sup>1,2</sup>. Diamictites of the Meteorite Bore Member (MBM) and a second<sup>3</sup> or possibly third<sup>4</sup> 13 14 recently discovered units of glacial diamictites were interpreted to be deposited from a floating ice 15 sheet over a marine basin<sup>5</sup>. Sedimentation of the Turee Creek Group is considered to have occurred in an asymmetric basin, called the McGrath Trough, which reflects flexural subsidence driven by the 16 migration to the northeast of a geanticline or a thrust-fold belt system<sup>1,5</sup>. More recently, Van 17 Kranendonk et al. (2015)<sup>6</sup> proposed that the Turee Creek Group was deposited in an intracratonic 18 19 basin that deepened to the northwest, with terrigenous input sourced from erosion of uplifted bedrock 20 in the southeast.

21 The chronology of the Turee Creek Group and broad correlation to timing of the Great Oxidation 22 Event and to other Paleoproterozoic basins is constrained by: (i) detailed stratigraphic reconstruction 23 from the Hardey Syncline (~4,000 m-thick), Deepdale (~2,500 m thick) and Boundary Ridge (7 mthick) sedimentary successions (Supplementary Fig. 1); (ii) three U-Pb zircon ages of  $2,450 \pm 3 \text{ Ma}^7$ . 24  $2,340 \pm 22$  Ma<sup>8</sup> and  $2,209 \pm 15$  Ma<sup>2</sup> at the base of the underlying Boolgeeda Iron Formation 25 26 (Woongarra rhyolites), at the base of the Meteorite Bore Member diamicites, and at the base of the 27 Wyloo Group (Cheela Springs Basalt), respectively; and (iii) sulphur-isotope data obtained on the Boundary Ridge area<sup>9,10</sup> located about 50 km north west of the Hardey Syncline. The occurrence of 28 29 thin (1 to 3 meters thick) glaciogenic diamictites at the Boundary Ridge and Deepdale sections are 30 significant in terms of interpreted lithostratigraphy. Martin et al. (1999)<sup>1</sup> interpreted the glaciogenic 31 diamictites at Deepdale to be part of the Boolgeeda BIFs, and therefore that the glacial diamictites 32 were deposited as part of the Boolgeeda Iron Formation in a deep water part of the basin. In contrast, Van Kranendonk (2010)<sup>11</sup> and Van Kranendonk et al. (2015)<sup>6</sup>, argued that the glaciogenic horizon at 33 the Boundary Ridge was located at the transition between the Boolgeeda BIF and overlying Kungarra 34

35 Formation and therefore that it could be correlated with the 400 m-thick Meteorite Bore Member 50 km to the south-east. Williford et al.,  $(2013)^9$ , using this stratigraphic interpretation in combination 36 with sulphur isotope data obtained at the Boundary Ridge, suggested that the Boundary Ridge and 37 38 Meteorite Bore Member glaciogenic diamictites were deposited at the same time during the final stage of the Great Oxidation Event. In contrast, Swanner et al., (2013)<sup>10</sup>, using a different set of sulphur 39 isotope data obtained at the Boundary Ridge, proposed that the Boundary Ridge diamictite was a 40 41 temporally distinct, older, unit relative to the Meteorite Bore Member, which is in agreement with the 42 stratigraphic interpretation of Martin et al  $(1999)^{1}$ .

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44 The 2 m-thick diamictite horizon located within the Boolgeeda Iron Formation at about 20 meters 45 below the contact with the overlying Kungarra Formation of our T1 drill core (Supplementary Fig. 2 46 d,e,f) shows the same textural characteristics as the one identified at the Boundary Ridge locality 47 (Supplementary Fig. 2 g,h). The evidence comes from several  $\sim$ 5 cm pebbles and several cm-scale 48 pebbles isolated in a siltstone matrix in a meter-scale layer. This clearly demonstrates that the 49 Boundary Ridge diamictite represents a distinct glacial event separated from the overlying MBM by 50 nearly 1500 metres of the Kungarra shales. Our new Re-Os sulphide age of ~2.31 Ga (Supplementary 51 Fig. 9) for the base of the MBM supports this interpretation, namely that the deep basin glaciomarine 52 horizon identified in the Boolgeeda IF is much older and most likely correlative with the first 53 Huronion glacial event (Ramsay Lake) at about 2.45 Ga. This interpretation is further confirmed by a 54 new U-Pb age of  $2.454 \pm 23$  Ma obtained on detrital zircons extracted from the diamictite horizons of 55 the Boundary Ridge and T1 core (Hardey Syncline)<sup>9</sup>.

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#### 57 Supplementary Note 2 - Drilling and samples

58 Drilling was performed in the Hardey Syncline area, which represents the most complete exposures of 59 the Turee Creek Group (Supplementary Fig. 1a). Geological mapping of surface outcrops was 60 undertaken in order to best assess where to locate the drill sites, in reasonably accessible areas, away 61 from faulting, and where representative stratigraphic sections were preserved. Several geological 62 sections were made through the syncline at various places along strike. Three drill sites were chosen in 63 the northwestern (T1), and southern (T2 and T3) parts of the Hardey Syncline (Supplementary Fig. 1b). The first hole (T1, 22°48'31.00"S-116°47'15.90"E) was drilled with RC hammer to a depth of 64 65 98.8 m and diamond drilling with NQ core (47.6 mm diameter) from 98.8.0 to 130.9 m through the 66 base of the Kungarra Formation and from 130.9 to 174.9 m through the top of Boolgeeda Iron 67 Formation. The second hole (T2, 22°50'49.70"S - 116°52'27.70"E) intercepted the base of the 68 Meteorite Bore Member diamictites. Drilling was performed with RC hammer to a depth of 117.7 m 69 and diamond drilling with NQ core from 117.7 to 281.7 m through the MBM diamictites and from 70 281.7 to 384.9 m through the underlying mudstone, siltstone, sandstone and carbonate stromatolite of 71 the Kungara Formation. The third hole (T3, 22°52'15.90"S - 116°56'46.40"E) was drilled with RC 72 hammer to a depth of 75.8 m and diamond drilling with NQ core from 75.8 to 182.4 m through the 73 base of the Kazput Formation and from 182.4 to 189.3 m through the top of Koolbye Formation 74 quartzites. The three drillcores were analysed for their major and trace element compositions using X-75 ray core scanning at high stratigraphic resolution (2 cm step). This compositional dataset has permitted 76 robust localisation of sedimentological contacts, both sharp and transitional as well as critical zones of 77 elemental enrichments, revealing shifts in clastic input (Al, Ti, Zr) and enrichment of redox-sensitive 78 elements (S, Fe, Mn, Cr) (work in preparation). The main lithologies exposed at the surface are shown 79 in Supplementary Fig. 3 together with their drill core equivalent collected at depths. The 135 samples 80 from the three drill cores were collected to reconstruct a chemostratigraphic record of sulphur isotopes 81 throughout the Turee Creek Group. In addition, 5 samples were selected at the base of the Meteorite 82 Bore Member diamictites to perform Re-Os chronology.

83 The Meteorite Bore Member represents a massive package of ~400 m thick glacial diamictite showing 84 no apparent bedding. The sequence is composed of chlorite-bearing mudstone containing randomly 85 distributed dropstones of various sizes (mm- to m-scale) and origin (carbonate, chert, gneiss, quartzite 86 and local pockets of sandstone showing convolute structure). The samples used for Re-Os dating were 87 collected throughout a ~20 meter scale interval between 252.55 and 272.46 metres depth. This section 88 consists of a uniform mudstone matrix with local dropstones one to several centimetres in size 89 (Supplementary Figure 3d, sample T2-253.3) and randomly distributed nodular aggregates of pyrites 90 interpreted to be of early diagenetic origin (see below). For the five diamictites used for bulk rock 91 dating (T2-252.55, T2-259.3, T2-264.3, T2-271.1 and T2-272.46, Supplementary Table 1), we were 92 careful to only collect the mudstone part of the sample and avoid portions with dropstones. The 93 reasons for choosing the Meteorite Bore Member diamictite for dating are as follows. First, bulk-rock 94 chemical analyses showed that the entire section is relatively static in composition with sulphur and 95 Total Organic Content (TOC) contents of about 1300 to 2300 ppm and 500 to 1000 ppm, respectively. Second, a detrital zircon U-Pb age of  $2340 \pm 22$  Ma obtained by Caquineau et al  $(2018)^8$  was obtained 96 97 in the same sedimentary interval, thus providing an independent test of evaluating the significance of the Re-Os age. Third, a detrital zircon U-Pb age of  $2.454 \pm 23$  Ma was obtained by Caquineau et al 98 99 (2018)<sup>8</sup> on the diamictite horizon of the Boolgeeda Iron Formation at the base of the T1 core (Figure 100 3). Since the Boolgeeda Iron Formation in T1 and the overlying Kungarra Formation containing the 101 MBM diamictites in T2 are in sedimentary continuity, this age constraint offered a second independent 102 mean for evaluating the significance of the Re-Os age as well as the rate of sedimentation of the 103 Kungarra Formation.

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#### 105 Supplementary Note 3 - Textures and chemical composition of sulphides

In all samples, preliminary identification of sulphides was performed by standard petrographic analyses of thin sections (Supplementary Figs. 4, 5, 6). Distinction between syngenetic/diagenetic pyrites, and detrital and epigenetic pyrites was based on sulphide chemistry, host lithology, and textural features.

110 Host lithologies that may contain detrital sulphides identified from their rounded appearance include 111 the Koolbye Formation quartzites at the base of T3 (Figs. 2 and 4) and the 5-meter thick sandstone bed 112 located beneath the Meteorite Bore Member diamictites (base of T2; Figs. 2 and 5). Only a few 113 sulphides were analysed in these rocks and special care was taken to discard rounded grains of 114 potential detrital origin, as these are not expected to preserve information on the sulphur cycle in the 115 depositional environment of the host rock. Similarly, none of the sulphides present in dropstones of 116 the Meteorite Bore Member and of the Boolgeeda Iron Formation (Supplementary Figs. 2, 3) were 117 analysed either by *in situ* or bulk rock techniques.

118 Detailed petrographic analyses indicate that the majority of the pyrites studied here show textures 119 indicative of a syngenetic or early diagenetic origin. Examples of such textures include rounded and 120 elongated zoned nodules, concretions, microcrystalline aggregates, framboidal cores, finely 121 disseminated pyrite (<10  $\mu$ m), small euhedral and subhedral crystals (< 25  $\mu$ m) aligned with bedding 122 (or conform to soft sediment deformation structures), and bands of closely spaced fined grained pyrite 123 (Supplementary Figures 4, 5, 6). The relative arrangement and proportion of these different textural 124 types in a particular horizon is variable but all forms are interpreted to originate during early 125 diagenesis or, in the case of pyrite framboids, possibly syngenetically within the water column or diagenetically below the sediment-water interface<sup>13</sup>. A few samples, however, show textures indicative 126 127 of a later generation of diagenetic pyrite. These include overgrowth of coarser euhedral to subhedral 128 pyrites as well as individual large euhedral to subhedral pyrite crystals (> 25 µm). Pyrite overgrowths 129 are regarded as diagenetic rather than from metamorphic and/or hydrothermal events as they occur 130 associated with poorly permeable lithologies (e.g., mudstone) with no evidence of pyrite 131 recrystallisation along schistosity planes. Coarser textures and overgrowth zones (see below) are 132 interpreted to be the result of local re-mobilisation of Fe and S from smaller earlier pyrite generations. 133 Of all samples studied, only one (T1-169.90) is known to record epigenetic pyrite as indicated by the 134 presence of large, inclusion-free, euhedral pyrite crystals in a mm-scale vein.

135 Sulphur isotopic signatures provide another line of evidence for a sedimentary (syngenetic/diagenetic) 136 origin of the pyrites. A syngenetic origin is particularly clear for pyrites in the 1.5 m-thick green 137 siltstone horizon of the Boolgeeda IF, showing strong enrichment in sulphur content (up to 5 weight %), together with strongly negative  $\delta^{34}$ S values (down to -35%) and  $\Delta^{33}$ S = 0% (Fig. 3). In 138 139 these samples, sulphide occurs in association with quartz forming millimetre-scale layers parallel to 140 the bedding in a matrix mainly composed of Fe-chlorite, Fe-oxides (magnetite and hematite) and 141 apatite (Supplementary Figs. 5c, d and 6). Such layering cannot be formed by secondary processes, but 142 instead reflects periodic delivery of non-anomalous sulphur component to the water column. Finally,

- 143 authigenic sulphides that incorporate sulphur from the environment of deposition can display ranges of 144  $\delta^{34}$ S and  $\Delta^{33}$ S values, which are indicative of different sources and processes (photolitic, biological 145 and/or nonbiological redox fractionation reactions at the time of deposition).
- 146 Further support for the syngenetic/diagenetic nature of sulphides is given by inter-element ratios, mostly Co/Ni but also Cu/Ni and Zn/Ni, determined by LA-ICP-MS. Previous studies<sup>14-17</sup> have shown 147 148 that Co/Ni ratio is an effective chemical indicator for the environment of pyrite formation. 149 Volcanogenic-hydrothermal pyrite is generally characterized by a high Co content with a high Co/Ni ratio<sup>18-20</sup>. On the other hand, high Ni and Co/Ni  $\leq$  2 have been observed in pyrite that forms in organic 150 matter-rich environments<sup>21,22</sup>. Large et al. (2014)<sup>16</sup> used Co/Ni ratios as a chemical screening for 151 152 sedimentary pyrites. On a Co versus Ni plot, samples which showed a linear array parallel to Co/Ni = 1 and with Co/Ni < 2 were selected as suitable indicators of a diagenetic origin<sup>16</sup>. In a recent study. 153 Gregory et al.,  $(2015)^{17}$  have defined several inter-element ratios that can be used as chemical proxies 154 155 for sedimentary pyrites. Characteristic values and composition limits of sedimentary pyrites were 156 determined to be: 0.01 < Co/Ni <2, 0.01 < Cu/Ni < 10, 0.01 < Zn/Ni < 10, 0.1 < As/Ni < 10, Ag/Au>2, 157 1 < Te/Au < 1000, Bi/Au > 1, Sb/Au > 100, and As/Au > 200.
- 158 Here, we applied to our dataset only the Ni-based ratios as discriminants as they are more robust 159 indicators due to their generally high abundance in pyrite. In addition, Co and Ni have similar ionic 160 radii to Fe and tend to be incorporated into the structure of pyrite. The behaviour of other elements, 161 like As and Au, is more complex due to their mobility over a wide range of redox conditions. Forty-162 two (42) samples have been selected for trace element analysis based on pyrite texture, lithology, and 163 stratigraphic unit. The Co/Ni ratios measured show that the majority of the samples (40 out of 42) 164 have Co/Ni  $\leq 2$  within errors and all samples have Cu/Ni and Zn/Ni  $\leq 10$  (Supplementary data 1, 165 Supplementary Figs. 7 and 8). Some samples show very low trace element abundances, which can be 166 explained by the low trace element abundance observed in the host rocks. Several studies have 167 highlighted that the trace element composition of sedimentary pyrites is highly dependent on the water 168 chemistry as such the trace element abundance variations observed along the stratigraphic profiles 169 (Supplementary Fig. 7) could be explained by changes on water chemistry and redox conditions (this 170 aspect will be discussed in a separate paper). Variations of trace element composition do not show 171 correlation with the different textural pyrite types described previously.
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Supplementary Figure 1. a, Geological map of the Turee Creek Group showing the location of the
Hardey Syncline, Boundary Ridge and Deepdale locations discussed in the text. b, Geological map of
the Hardey Syncline modified after Martin et al. (2000)<sup>5</sup> and Van Kranendonk et al., (2015)<sup>6</sup>. The
location of the three drill cores T1, T2 and T3 of the Turee Creek Drilling Project are indicated.





243 Supplementary Figure 2. Different types of glaciogenic diamictites. a-c, Photomicrographs (a, b) 244 and drill core sample (c, T2) of the Meteorite Bore Member at the Hardey Syncline locality. d-f, 245 Photomicrographs (c, d) and drill core sample (f, T1) of the diamictite horizon within the Boolgeeda 246 Iron Formation at the Hardey Syncline locality. g,h, Surface exposure (g, 10 cm-long knife for scale) 247 and photomicrograph of surface sample Pi-15-16 (h) of the diamictite horizon in the Boolgeeda Iron 248 Formation at the Boundary Ridge locality. Note the similar texture and occurrence of carbonate (carb) 249 and chert dropstones in the Boolgeeda BIF glaciogenic horizons at Hardey Syncline and Boundary 250 Ridge.



253 Supplementary Figure 3. Photographs of drill core samples and corresponding surface 254 exposures. Numbers refer to depth of drilling. Cores are 5 cm large. a, Iron formation (T1, Boolgeeda 255 Iron Fm). b, Chert layer marking the contact between the Boolgeeda IF and overlying Kungarra Fm 256 (T1). c, Finely laminated siltstone with cross laminations (T1, Kungarra Fm). d, Diamictite of the 257 Meteorite Bore Member (T2). The two arrows point to sulphides in the rock matrix (analysed for their 258 S-isotope composition) and in pebbles (not analysed). e, Five to 10 metres thick sand bar located 259 beneath the MBM diamictites (T2, Kungarra Fm). f, Carbonate stromatolite located beneath the MBM 260 diamictites (T2, Kungarra Fm). g, Carbonate of the Kazput Fm (T3). h, Finely laminated siltstone with 261 cross laminations (T3, Kazput Fm.).

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Supplementary Figure 4. Backscattered electron (BSE) images of syngenetic and diagenetic
pyrites analysed in this study. a, inclusion-rich nodular pyrite aggregate (sample T1-99.48), b,
clusters of microcrystalline pyrites in green mudstone (sample T1-146.0), c, clusters of elongated
anhedral pyrite crystals typically aligned with bedding observed in mudstone (sample T2-341.28), d,
compacted inclusion-rich pyrite nodule in laminated mudstone (sample T2-365.23), e and f, inclusionrich nodular aggregates in mudstone (samples T3-172.95 and T3-182.90).



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274 Supplementary Figure 5. Backscattered electron (BSE) images of syngenetic and diagenetic 275 pyrites analysed in this study. a, inclusion-free overgrowth around syngenetic to early diagenetic 276 pyrite framboids observed in green mudstone (sample T1-130.39), b, finely disseminated euhedral to 277 subhedral pyrite crystals (sample T1-152.18), **c**, small euhedral to subhedral pyrite crystals (< 25  $\mu$ m) 278 aligned with bedding in green mudstone (sample T1-159.30), d, small euhedral to subhedral pyrite 279 crystals (< 25 µm) conform to soft sediment deformation structures observed in green mudstone 280 (sample T1-159.47), e, band of densely packed microcrystalline pyrite aggregates (sample T1-172.1), f, 281 anhedral overgrowth around large euhedral pyrite crystal in carbonate (sample T3-90.75).



Supplementary Figure 6. X-ray maps of sulphide layers of drill core sample T1-159.47. a, b,
Photo-micrographs of the petrographic thin section analysed. The red boxes represent zoom in views.
Soft sediment deformations are locally preserved in this sample (Supplementary Figure 6d). BS,
Electron backscattered image, Fe-S-P, composite X-ray map showing Fe-oxide (red), pyrite (purple)
and apatite (green). Other X-ray maps correspond to sulphur (S), silicon (Si), iron (Fe) and
phosphorus (P). Note that the sulphide-bearing layers are mainly composed of sulphide and silica.



Supplementary Figure 7. Co/Ni, Zn/Ni, and Cu/Ni depth profiles of pyrite from Turee Creek
drill cores (T1, T2, and T3). Red circles correspond to individual (spot) analyses carried out with

292 LA-ICP-MS. The grey shaded areas represent the composition limits defined for sedimentary pyrites

 $293 \qquad (0.01 < Co/Ni < 2, \ 0.01 < Cu/Ni < 10, \ 0.01 < Zn/Ni < 10). \ Despite the large range observed on Co/Ni,$ 

294 Zn/Ni, and Cu/Ni ratios measured, most of the samples analysed show inter-element ratios within the

295 range of sedimentary pyrite $^{17,22}$ .



Supplementary Figure 8. Binary plots for Co, Cu, and Zn vs Ni for pyrites from Turee Creek drill cores (T1, T2, and T3). Red circles correspond to individual (spot) analyses carried out with LA-ICP-MS. Cobalt correlates moderately well with Ni, with most of the samples from drill cores T1 (a), T2 (b), and T3 (c) plotting along the Co/Ni = 1 line or on a linear array parallel to Co/Ni =1. A moderate to weak correlation is observed for Cu vs Ni (d, e, f) and Zn vs Ni (g, h, i), probably due to the scatter of the analytical data, which could be related to different trace element incorporation mechanisms into pyrite (i.e., trace element held within the pyrite structure or as nano-inclusions<sup>16,17</sup>.

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309 Supplementary Figure 9. Re-Os age. Re-Os isochron plots for (a) all samples (two pyrite separates

and 5 bulk rock samples). (b) Plot showing only Re and Os data for diamictite samples collected

311 within a 20 meters depth range using  $CrO_3 - H_2SO_4$  digestion medium. Error bars are  $\pm 2 \sigma$ .

- 312 Regression based on Isoplot<sup>12</sup>.
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Supplementary Figure 10. Photomicrograph of a sulphide analysed for its S-isotope composition
using the SHRIMP SI (ablation crater of the Cs beam) and its major and trace element composition
using SEM (analysis 004 showing the two main peaks of S and Fe characteristic of pyrite).

320 321 322 Supplementary Table 1. Re-Os concentrations and Os isotopic compositions for diamictites and pyrite separates.

Sample	Lithology	Re ppb	<b>Total Os</b>	<sup>187</sup> Re/ <sup>188</sup> Os	$\pm 2\sigma$	<sup>187</sup> Os/ <sup>188</sup>	$\pm 2\sigma$	Rho*
			ppt			Os		
T2 – 252.55	Diamictite	3.70	350	69.02	0.41	2.855	0.018	0.35
T2 - 259.3	Diamictite	3.68	629	33.03	0.17	1.450	0.002	0.47
T2 - 264.3	Diamictite	4.32	391	73.84	0.41	3.048	0.012	0.53
T2 - 271.1	Diamictite	3.59	870	22.22	0.12	1.022	0.002	0.40
T2 - 272.46	Diamictite	4.05	418	54.20	0.31	2.288	0.005	0.46
T2-272.46	Py	2.89	109	865.93	3.30	34.090	0.010	0.54
T2 - 272.46	Py	3.58	206	147.14	0.52	5.940	0.004	0.57

323 324 Rho\* stands for error correlation<sup>23</sup>.