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Supplemental Material

U-Pb dating reveals multiple Paleoproterozoic orogenic events (Hamersley orogenic cycle) along southern Pilbara margin spanning onset of atmospheric oxygenation

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SHRIMP Analytical Techniques and Data Processing

Xenotime and monazite were analysed for U-Pb dating with the Sensitive High-Resolution Ion MicroProbe (SHRIMP-II) instrument at the John De Laeter Centre, Perth, Western Australia. Grains were identified in polished thin sections, and 3 mm diameter plugs were extracted from the thin sections with a hollow-core rotary drill and mounted in 25 mm diameter epoxy discs. The mounts were cleaned and gold coated before each analytical session. Xenotime and monazite standards were set into separate mounts and gold coated simultaneously with sample mounts. Standard and sample mounts were loaded together into the SHRIMP for concurrent analysis during each of the four analytical sessions.

Instrument setup followed protocols for small-spot, in-situ analysis of xenotime and monazite developed in Rasmussen et al. (2001), Fletcher et al. (2000; 2004; 2010). A primary beam of O_2^- ions was focused through a 50 µm Kohler aperture to produce an oval 10 µm wide spot on the sample surface with a current of 0.3 nA. The secondary ion system was focused through a 100 µm collector slit onto an electron multiplier to produce mass peaks with flat tops and a mass resolution of >5400 in all sessions. Background counts from scattered ions were reduced using a flight retardation lens, which is known to cause slight session-dependent instrumental mass fractionation (IMF) of Pb isotopes (Rasmussen et al., 2008). IMF corrections were applied to all analyses.

Data were collected in sets of 8 scans, with standard xenotime or monazite analysed every 4-6 sample analyses. Count times per scan for Pb isotopes 204, background position 204.045, 206, 207 and 208 were 10s, 10s, 10s, 30s and 10s, respectively. Monazite was analysed with a 13-peak run table as defined in Fletcher et al. (2010), which includes mass stations for the estimation of La, Ce and Nd ($REEPO_2^+$), and Y ($YCeO^+$). Measurements on monazite standards FRENCH, Z2234 and Z2908 (see Fletcher et al., 2010 for

details) were done concurrently for Pb/U and Pb/Th calibration (FRENCH), 204 a.m.u. isobar corrections, IMF corrections and matrix corrections required for variable U, Th, Y and Nd contents.

Raw data were processed using a customised task file for the SQUID2 (v. 2.50.12.03.08) add-in for Excel 2003 (Ludwig, 2009), and plotted using the ISOPLOT (v. 3.76.12.02.24) add-in (Ludwig, 2008). Common Pb corrections were made from ^{204}Pb measurements corrected for the 204 a.m.u isobar, following the procedure in Fletcher et al. (2010). Common Pb composition was assumed to be that of Broken Hill lead, a common environmental contaminant in Australia, with a composition equivalent to that at 1.6 Ga in the two-stage evolution model of Stacey and Kramers (1975).

Xenotime was analysed with a 9-peak run table following analytical protocols in Fletcher et al. (2000, 2004). Pb/U calibrations and matrix corrections for U and Th contents were based on concurrent measurements of the standards MG-1 (Fletcher et al., 2004) and z6413 ("Xeno1"; Stern and Rayner, 2003). Pb/Th was determined indirectly, using a fixed Th/U calibration (Fletcher et al., 2004). Matrix corrections for REE assumed the samples to have REE abundances similar to Xeno1.

Data are presented in table with 1σ errors. Individual analysis errors are quoted in text at 1σ, and pooled ages are quoted in text and figures at 95% confidence levels. A minimum spot-to-spot external precision of 1% (1σ) was assumed for standard analyses in all sessions and propagated to U-Pb measurements on all samples of both monazite and xenotime.

SHRIMP Analytical Results

Sample MG366, 271.58 m (Mount BR21-05 monazite, plugs G, J)

A total of 15 analyses were acquired from two monazite grains in plugs G and J. The grains low to moderate U (50-189 ppm, mean 108 ppm) and variable Th (25-10050 ppm, mean 2235 ppm) (Table S1). Three analyses record >10% discordant data and are disregarded in age interpretation. The 12 concordant or near-concordant analyses (incl. two that required >1% common Pb correction) yielded ^{207}Pb/^{206}Pb dates ranging from 2378 ± 25 Ma to 2243 ± 60 Ma, with a weighted mean of 2345 ± 25 Ma. Considering the large MSWD value of 2.4, the age population were divided into two subgroups at 2365 ± 17 Ma (n = 6, MSWD = 0.30) and 2295 ± 27 Ma (n = 6, MSWD = 1.17) (Fig. S1), which are comparable to the older and younger monazite age components recorded by samples from MG368 (see below).

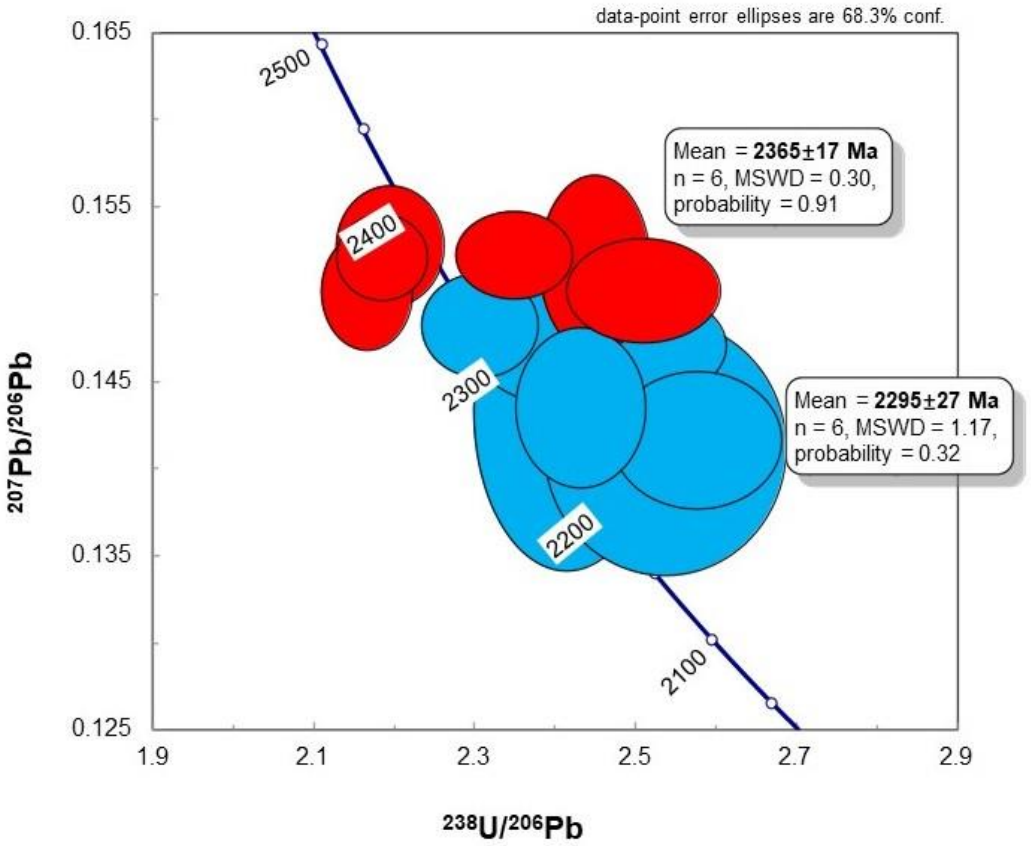

Figure S1. U–Pb concordia plot for monazite from sample MG366 (271.8 m).

Sample MG368, 320.4 m B (Mount BR10-09 monazite, plugs G, K, L)

A total of 24 analyses were performed on 3 monazite grains, including 7 analyses from a grain (plug G) below a tuff, 8 analyses from 1 grain (plug K) in the tuff, and 9 analyses from 1 grain (plug L) in a vein (Table S2). All analyses record low common ^{206}Pb (<0.5%), and are concordant (within 5% disc.) except 2 analyses (L.1-2 and L.1-4) which are near-concordant (6% disc.). The 2 near-concordant analyses are also included into the dataset and calculation given their consistence in U-Pb data. Pooled together, U-Pb data

from the 3 monazite grains can be divided into two groups: an older group consisting of 10 analyses from plug G and K yields a weighted mean $^{207}Pb/^{206}Pb$ date of 2373 ± 14 Ma (MSWD = 0.62; 95% confidence level); the remaining 5 analyses from plug G and K plus the 9 analyses (plug L) from the vein forms a younger group, which gives a weighted mean $^{207}Pb/^{206}Pb$ date of 2320 ± 10 Ma (MSWD = 0.60; 95% confidence level) (Fig. S2). Analyses of the older group have low U (41-189 ppm; mean 89 ppm) and variable Th (14-7252 ppm; mean 725 ppm) with Th/U values varying from 0.1-69, where those of the younger group have low to moderate U (57-369 ppm; mean 153 ppm) and more uniform Th (125-1167 ppm; mean 573 ppm) with Th/U between 0.6 and 10.

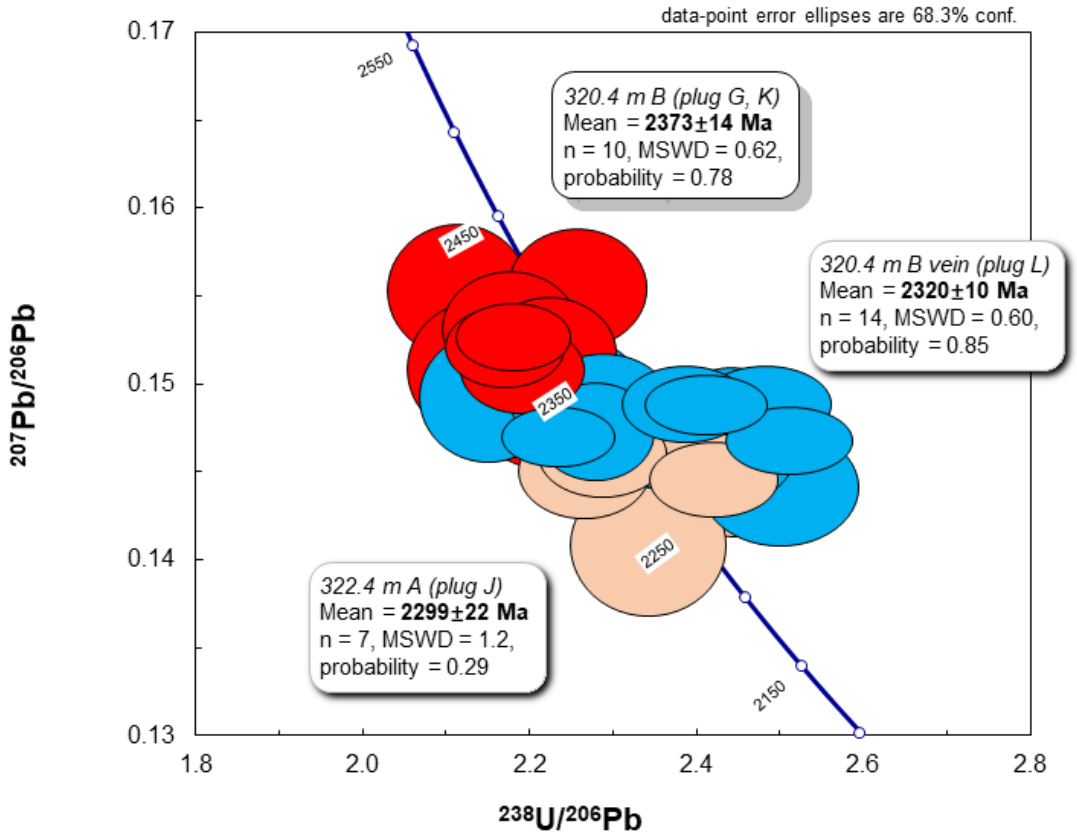

Figure S2. U–Pb concordia plot for monazite from sample MG368 (320.4 m B).

Sample MG368, 322.4 m A (Mount BR10-09 monazite, plug J)

Seven analyses were obtained from this sample, all from one monazite grain. They record low common ^{206}Pb (<0.5%) and good concordance (within 5% disc.) (Table S3). Their U and Th concentrations are in the range of 50-180 ppm and 990-7200 ppm, respectively, and Th/U values between 8 and 95. These analyses yielded consistent $^{207}Pb/^{206}Pb$ dates that give a weighted mean at 2299 ± 22 Ma (MSWD = 1.2; 95% confidence level) (Fig. S2).

Sample MG368, 238.1 m A (Mount BR20-07 monazite, plugs C, D, E)

A total of 9 analyses were acquired from three monazite grains in plugs C, D, and E. The grains are characterized by depleted U (7-42 ppm, mean 23 ppm) and variable Th (27-12,000 ppm, mean 2407 ppm) (Table S4). As a result, Pb/Th analyses from four spots that record the highest Th are used to derive an approximate growth age for the monazite. A weighted mean $^{208}Pb/^{232}Th$ date of 2295 ± 47 Ma (MSWD = 0.005; 95% confidence level) is calculated from the four analyses.

Sample MG367, 192.4 m D (Mount BR20-06 xenotime)

Twelve analyses of a large xenotime grain in plug E yielded concordant data that have <0.3% common ^{206}Pb (Table S5). U and Th concentrations of the analyses vary from 790-1900 ppm and 1100-7900 ppm, respectively, with Th/U values in the range 1.4-5.3. All analyses form a tight cluster on the concordia plot (Fig. S3) and give a weighted mean $^{207}Pb/^{206}Pb$ date of 2291 ± 11 Ma (*n* = 12, MSWD = 1.4, 95% confidence level).

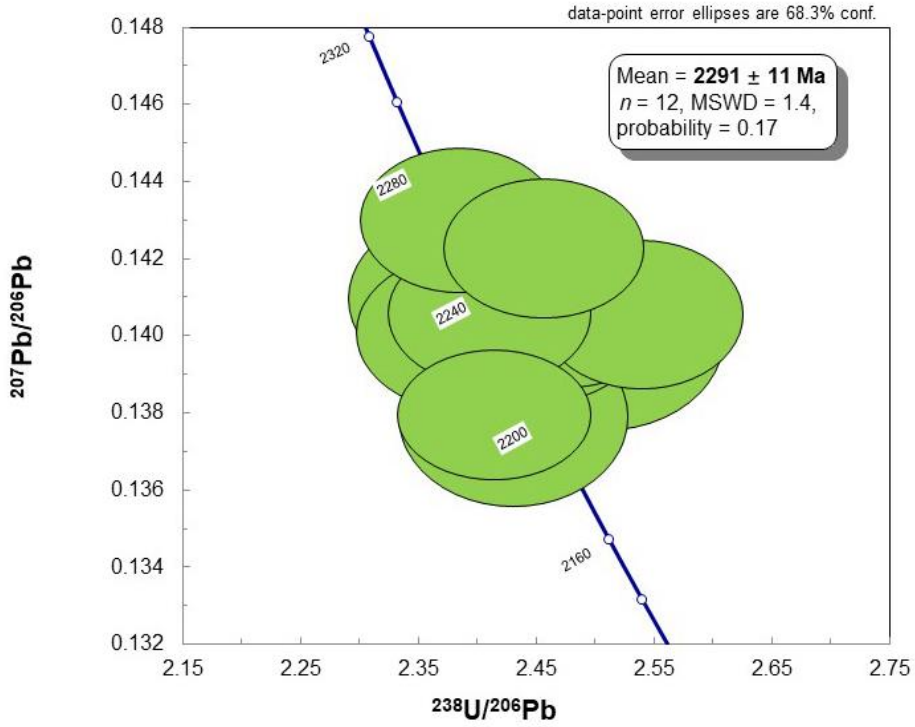

Figure S3. U–Pb concordia plot for xenotime from sample MG367 (192.6 m D).

Summary

Collectively, authigenic monazite from MG366 (271.58 m A) and MG368 (322.4 m A and 238.1 m A) appear to record two growth episodes with combined weighted mean ages of 2370 ± 11 Ma (*n* = 16, MSWD = 0.50) and 2312 ± 8 Ma (*n* = 31, MSWD = 0.95). Xenotime from MG367 (192.5 m D) yielded a younger age at 2291 ± 11 Ma (*n* = 12, MSWD = 1.4).

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Table S1. SHRIMP U-Pb data for monazite in Roy Hill Shale Member from drill-hole MG366, 271.58 m

Analysis No.	U (ppm)	Th (ppm)	Th/U	f_{206} (%)	Total $^{238}U/^{206}Pb$	±1σ	Total $^{207}Pb/^{206}Pb$	±1σ	$^{238}U/^{206}Pb^*$	±1σ	$^{207}Pb^*/^{206}Pb^*$	±1σ	Disc. (%)	$^{207}Pb^*/^{206}Pb^*$ Age (Ma)	±1σ
Older group (2365 ± 17 Ma, n = 6, MSWD = 0.30)															
2105J.1-2	103	8378	81	0.39	2.18	0.04	0.1533	0.0017	2.19	0.04	0.1528	0.0022	-2	2378	25
2105G.1-5	189	1024	5	-0.13	2.35	0.05	0.1481	0.0013	2.35	0.05	0.1523	0.0016	4	2372	18
2105G.1-6	174	383	2	-0.05	2.18	0.04	0.1488	0.0014	2.18	0.04	0.1522	0.0015	-3	2371	17
2105G.1-1	147	360	2	0.44	2.44	0.04	0.1527	0.0030	2.45	0.05	0.1518	0.0033	7	2366	37
2105J.1-6	96	609	6	0.02	2.51	0.06	0.1475	0.0018	2.51	0.06	0.1503	0.0019	8	2349	22
2105G.1-2	78	3441	44	-0.03	2.16	0.04	0.1470	0.0020	2.16	0.04	0.1502	0.0021	-4	2349	24
Younger group (2295 ± 27 Ma, n = 6, MSWD = 1.17)															
2105G.1-7	180	1278	7	0.50	2.29	0.05	0.1498	0.0013	2.30	0.05	0.1483	0.0019	0	2326	22
2105J.1-4	92	1105	12	0.67	2.44	0.10	0.1501	0.0018	2.45	0.10	0.1471	0.0027	5	2313	32
2105G.1-3	111	1455	13	0.95	2.41	0.05	0.1492	0.0018	2.43	0.05	0.1436	0.0030	2	2270	36
2105G.1-4	50	3876	78	2.18	2.36	0.07	0.1598	0.0027	2.41	0.08	0.1433	0.0061	1	2268	73
2105J.1-3	84	432	5	0.48	2.57	0.07	0.1432	0.0018	2.57	0.07	0.1417	0.0026	6	2248	32
2105G.1-8	53	10042	189	1.38	2.49	0.10	0.1507	0.0026	2.53	0.10	0.1413	0.0049	4	2243	60
Rejected															
2105J.1-5	93	645	7	0.53	2.64	0.07	0.1518	0.0037	2.66	0.07	0.1500	0.0043	12	2346	49
2105G.1-9	50	499	10	0.13	2.51	0.09	0.1565	0.0028	2.50	0.09	0.1585	0.0031	11	2440	33
2105J.1-1	126	25	0	0.00	2.78	0.04	0.1491	0.0019	2.78	0.04	0.1521	0.0020	16	2369	23

f_{206} is the proportion of common Pb in ^{206}Pb, determined using the measured $^{204}Pb/^{206}Pb$ and a common Pb composition from the Stacey-Kramers (1975) model at the approximate age of the sample.
Pb^* represents radiogenic Pb.
Disc. is apparent discordance, as $100 \cdot (t[^{207}Pb/^{206}Pb] - t[^{206}Pb/^{238}U]) / t[^{207}Pb/^{206}Pb]$.

Table S2. SHRIMP U-Pb data for monazite in Roy Hill Shale Member from drill-hole MG368, 320.4 m

Analysis No.	U (ppm)	Th (ppm)	Th/U	f_{206} (%)	Total $^{238}U/^{206}Pb$	±1σ	Total $^{207}Pb/^{206}Pb$	±1σ	$^{238}U/^{206}Pb^*$	±1σ	$^{207}Pb^*/^{206}Pb^*$	±1σ	Disc. (%)	$^{207}Pb^*/^{206}Pb^*$ Age (Ma)	±1σ
Older group (2373 ± 14 Ma, n = 10, MSWD = 0.62)															
1009G.1-1	62	1354	22	-0.17	2.28	0.06	0.1538	0.0019	2.26	0.05	0.1555	0.0023	2	2407	25
1009G.1-2	64	389	6	0.35	2.14	0.05	0.1539	0.0020	2.13	0.05	0.1509	0.0025	-5	2357	29
1009G.1-3	50	380	8	0.43	2.21	0.06	0.1546	0.0020	2.22	0.06	0.1509	0.0027	-2	2356	30
1009G.1-5	64	4466	69	0.47	2.21	0.05	0.1543	0.0028	2.20	0.05	0.1503	0.0033	-3	2349	37
1009G.1-6	41	321	8	-0.18	2.14	0.06	0.1536	0.0022	2.11	0.06	0.1554	0.0026	-4	2406	28
1009K.1-1	62	133	2	0.07	2.19	0.06	0.1536	0.0020	2.18	0.05	0.1532	0.0022	-2	2382	24
1009K.1-2	124	14	0	0.10	2.18	0.05	0.1529	0.0013	2.17	0.05	0.1522	0.0016	-3	2371	18
1009K.1-4	93	57	1	0.21	2.21	0.05	0.1535	0.0017	2.22	0.05	0.1518	0.0021	-1	2367	23
1009K.1-6	189	55	0	0.04	2.18	0.05	0.1529	0.0011	2.18	0.05	0.1527	0.0013	-2	2377	15
1009K.1-7	137	82	1	0.08	2.18	0.05	0.1513	0.0014	2.19	0.05	0.1508	0.0017	-3	2355	19
Younger group (2320 ± 10 Ma, n = 14, MSWD = 0.60)															
1009G.1-4	188	934	5	0.012	2.31	0.05	0.1489	0.0018	2.29	0.05	0.1490	0.0019	0	2334	21
1009G.1-7	82	530	6	0.466	2.27	0.05	0.1531	0.0017	2.26	0.05	0.1492	0.0023	-1	2336	26
1009K.1-3	59	125	2	0.283	2.14	0.05	0.1517	0.0020	2.15	0.05	0.1493	0.0024	-5	2338	28
1009K.1-5	264	475	2	0.046	2.25	0.05	0.1473	0.0009	2.23	0.04	0.1471	0.0012	-3	2312	13
1009K.1-8	186	823	4	0.013	2.30	0.05	0.1473	0.0018	2.28	0.05	0.1474	0.0019	-1	2316	22
1009L.1-1	71	131	2	0.054	2.32	0.06	0.1472	0.0017	2.30	0.05	0.1469	0.0019	-1	2310	22
1009L.1-2	369	208	1	0.00	2.54	0.05	0.1466	0.0009	2.51	0.05	0.1468	0.0013	6	2309	15
1009L.1-3	263	532	2	0.014	2.44	0.05	0.1488	0.0010	2.41	0.05	0.1489	0.0012	4	2333	13
1009L.1-4	167	482	3	0.085	2.51	0.05	0.1495	0.0013	2.48	0.05	0.1489	0.0015	6	2333	17
1009L.1-5	102	825	8	0.114	2.36	0.07	0.1476	0.0015	2.36	0.07	0.1467	0.0017	1	2308	20
1009L.1-6	57	144	3	0.076	2.51	0.06	0.1447	0.0020	2.50	0.06	0.1442	0.0022	5	2278	26
1009L.1-7	112	1167	10	0.022	2.45	0.06	0.1471	0.0025	2.44	0.05	0.1471	0.0026	4	2312	30
1009L.1-8	87	779	9	0.167	2.40	0.06	0.1479	0.0016	2.38	0.05	0.1466	0.0019	2	2306	22
1009L.1-9	138	872	6	-0.07	2.42	0.05	0.1481	0.0012	2.39	0.05	0.1489	0.0015	3	2333	17

Table S3. SHRIMP U-Pb data for monazite in Roy Hill Shale Member from drill-hole MG368, 322.4 m

Analysis No.	U (ppm)	Th (ppm)	Th/U	f_{206} (%)	Total $^{238}U/^{206}Pb$	±1σ	Total $^{207}Pb/^{206}Pb$	±1σ	$^{238}U/^{206}Pb^*$	±1σ	$^{207}Pb^*/^{206}Pb^*$	±1σ	Disc. (%)	$^{207}Pb^*/^{206}Pb^*$ Age (Ma)	±1σ
Mean age (2299 ± 22 Ma, n = 7, MSWD = 1.2)															
1009J.1-1	76	7200	95	0.406	2.45	0.06	0.1489	0.0021	2.45	0.06	0.1455	0.0028	4	2294	33
1009J.1-2	129	992	8	0.024	2.41	0.05	0.1481	0.0014	2.39	0.05	0.1481	0.0016	3	2324	18
1009J.1-3	130	1384	11	0.062	2.31	0.05	0.1464	0.0015	2.29	0.05	0.1461	0.0017	-2	2300	20
1009J.1-4	178	1011	6	0.021	2.46	0.05	0.1446	0.0013	2.42	0.05	0.1446	0.0015	2	2283	17
1009J.1-5	51	2792	55	0.283	2.36	0.06	0.1432	0.0022	2.34	0.06	0.1409	0.0027	-2	2238	33
1009J.1-6	101	2384	24	0.058	2.30	0.05	0.1454	0.0016	2.26	0.05	0.1451	0.0018	-3	2289	21
1009J.1-7	116	2064	18	0.125	2.46	0.06	0.1486	0.0015	2.44	0.05	0.1477	0.0017	4	2320	20

Footnote as in Table S1.

Table S4. SHRIMP U-Pb data for monazite in Marra Mamba Iron Formation from drill-hole MG368, 238.1 m

Analysis No.	U (ppm)	Th (ppm)	Th/ U	f_{206} (%)	f_{208} (%)	Total $^{238}U/^{206}Pb$	±1σ	Total $^{208}Pb/^{232}U$	±1σ	$^{238}U/^{206}Pb^*$	±1σ	$^{208}Pb^*/^{232}U$	±1σ	$^{238}U/^{206}Pb^*$ Age (Ma)	±1σ	$^{208}Pb^*/^{232}Th$ Age (Ma)	±1σ
Mean age (2295 ± 47 Ma, n = 4, MSWD = 0.005)																	
2007C.1-3	31	2588	84	-1.35	-0.14	2.31	0.07	0.1169	0.0026	2.27	0.07	0.1203	0.0026	2356	63	**2296**	**47**
2007D.1-3	42	11909	286	0.85	0.03	2.19	0.06	0.1160	0.0030	2.20	0.06	0.1199	0.0030	2416	53	**2289**	**54**
2007D.1-2	9	1141	134	5.10	0.34	2.18	0.12	0.1173	0.0026	2.28	0.14	0.1202	0.0026	2348	123	**2295**	**47**
2007E.1-2	31	3733	121	-0.04	0.00	2.24	0.09	0.1176	0.0025	2.22	0.13	0.1204	0.0025	2394	113	**2297**	**46**
Rejected																	
2007C.1-1	28	27	1	-1.16	-10.34	2.25	0.09	0.1169	0.0086	2.21	0.09	0.1324	0.0128	2408	79	2514	226
2007C.1-2	7	823	111	-0.18	-0.02	2.14	0.14	0.1087	0.0029	2.12	0.39	0.1118	0.0034	2492	373	2141	62
2007C.1-4	33	111	3	-0.95	-2.36	2.17	0.06	0.1283	0.0043	2.14	0.06	0.1350	0.0047	2474	59	2559	83
2007D.1-1	16	800	49	1.23	0.22	2.25	0.09	0.1193	0.0027	2.26	0.09	0.1225	0.0027	2362	79	2336	48
2007E.1-1	12	530	45	3.62	0.75	2.12	0.09	0.1137	0.0026	2.19	0.10	0.1160	0.0026	2428	90	2218	48

Footnote as in Table S1.

Table S5. SHRIMP U-Pb data for xenotime in Roy Hill Shale Member from drill-hole MG367, 192.4 m

Analysis No.	U (ppm)	Th (ppm)	Th/U	f_{206} (%)	Total $^{238}U/^{206}Pb$	±1σ	Total $^{207}Pb/^{206}Pb$	±1σ	$^{238}U/^{206}Pb^{*}$	±1σ	$^{207}Pb^{*}/^{206}Pb^{*}$	±1σ	Disc. (%)§	$^{207}Pb^{*}/^{206}Pb^{*}$ Age (Ma)	±1σ
Mean age (2291 ± 11 Ma, n = 12, MSWD = 1.4)															
2006E.1-1	886	4173	4.7	0.07	2.35	0.06	0.1429	0.0014	2.43	0.06	0.1426	0.0015	-1	2259	18
2006E.1-1a	913	3839	4.2	0.00	2.38	0.06	0.1451	0.0015	2.45	0.06	0.1453	0.0015	1	2292	17
2006E.1-2	919	4855	5.3	0.13	2.35	0.06	0.1469	0.0014	2.43	0.06	0.1460	0.0016	1	2300	18
2006E.1-3	1358	2120	1.6	-0.08	2.31	0.05	0.1468	0.0011	2.39	0.05	0.1477	0.0012	0	2320	13
2006E.1-4	1629	2451	1.5	0.22	2.45	0.05	0.1470	0.0010	2.54	0.06	0.1453	0.0012	4	2291	14
2006E.1-6	1284	2334	1.8	0.13	2.40	0.05	0.1462	0.0011	2.48	0.06	0.1453	0.0012	2	2291	14
2006E.1-7	1000	2497	2.5	0.05	2.31	0.05	0.1449	0.0012	2.39	0.06	0.1447	0.0012	-1	2284	15
2006E.1-8	1269	3885	3.1	-0.06	2.38	0.05	0.1464	0.0011	2.46	0.05	0.1472	0.0011	2	2313	13
2006E.1-9	1196	1754	1.5	0.08	2.34	0.05	0.1456	0.0011	2.41	0.06	0.1452	0.0012	0	2290	14
2006E.1-10	809	1100	1.4	0.14	2.43	0.06	0.1453	0.0014	2.51	0.07	0.1444	0.0015	3	2281	18
2006E.1-11	794	2355	3.0	0.00	2.32	0.06	0.1453	0.0014	2.39	0.06	0.1455	0.0014	-1	2294	17
2006E.1-12	1891	7899	4.2	-0.03	2.33	0.05	0.1428	0.0010	2.41	0.05	0.1433	0.0010	-2	2268	12

Footnote as in Table S1.