Data Repository item 2005115

“Giant iron-ore deposits of the Hamersley province related to the breakup of Paleoproterozoic Australia: New insights from in situ SHRIMP dating of baddeleyite from mafic intrusions “

by Müller et al.

SAMPLING AND PROCESSING FOR SHRIMP ANALYSIS

Field samples from the Ashburton and Hamersley provinces and drill core from the Paraburdoo mine were collected for petrographic examination to identify baddeleyite-bearing mafic intrusions, and tuffaceous rocks with primary magmatic zircon. Because few samples contained baddeleyite, selected intrusions were sampled in greater detail. After identification using transmitted light microscopy, baddeleyite crystals were imaged and analyzed on selected polished thin sections with a scanning electron microscope/microprobe JEOL 6400 SEM, suitable crystals chosen and their positions recorded. As the baddeleyite crystals were too small or fragile (plates with a maximum width of 10-20 µm) for conventional heavy mineral separation, 3 mm diameter plugs were drilled from the thin sections and mounted with standards on epoxy discs (~16 plugs on each 25 mm mount) for SHRIMP analysis.

Petrographic examination of the siltstone sample from the Mount Olympus area established a volcaniclastic origin. Relict pumice and shard textures, and angular volcanic quartz were identified. The sample was crushed, milled, sieved, washed and separated into mineral concentrates using conventional heavy liquid and magnetic gravity procedures. Zircon grains were then handpicked from the heavy-mineral concentrate and mounted with standards on epoxy discs for SHRIMP analysis.

All mounts were analyzed at the SHRIMP II facility (SHRIMP A + B) at Curtin University, Western Australia.

SAMPLE DETAILS (GPS grid used: Australian Geod 84')

Baddeleyite is rare in some of the mafic intrusions, particularly the dolerite dykes. To obtain enough data, numerous samples were taken from each intrusion and polished thin sections prepared. Two samples from mafic sill HTC were analyzed. Two samples from mafic sill ETC were also analyzed and nine samples from dolerite dyke S72-5. The data from the Paraburdoo mine were collected mainly from one drill-core intersection (DDH48, three samples). Dolerite dykes in drillholes DDH53 (two samples) and DDH59 (one sample) were also included in the analysis.

Zircon grains from the tuffaceous siltstone (DNO) were extracted from 1.24 kg of crushed sample. After milling, sieving, washing and drying, 380 g of the sample were left for heavy mineral separation procedures.

Sample coordinates:

1.) HTC (HTC2g + HTC4), mafic sill

Location: Hardey syncline, near Meteorite Bore

HTC2g at 503,056mE -7,465,727mN

HTC4 at 503,025mE –7,465,771mN

2.) ETC (ETC9 + Pf7), mafic sill

Location: Turee Creek syncline, near Balgara Well (ETC9), and Fish Pool (Pf7)

ETC9 at 631,110mE -7,433,194mN

Pf7 at 612,104mE -7,432,675mN

3.) S72-5 (S72-5a, S72-5b, MHS1, MHS2, MHS3, MHS4, MHS5, MHS6, MHS7), dolerite dyke

Location: Hardey syncline, near Shingle Creek

S72-5a + b at 485,958E -7,471066mN

MHS1 at 485,888mE -7,471,114mN

MHS2 at 485,937mE -7,471,076mN

MHS3 at 485,965mE -7,471,054mN

MHS4 at 485,985mE -7,471,037mN

MHS5 at 486,176mE -7,470,823mN

MHS6 at 486,240mE -7,470,744mN

MHS7 at 486,438mE -7,470,598mN

4.) DDH48 + (48/12, 48/9, 48/6, 53/24, 53/27, 59/41), dolerite dykes (drill core)

Location: Paraburdoo mine

!GPS grid used here: AMG C 50!

DDH48 at 561,601mE -7,429,836mN

- **48/12** from 214.65 -214.75 m
- **48/9** from 225.15 -225.35 m
- **48/6** from 227.0 -227.10 m

DDH53 at 562,090mE -7,429,695mN

- **53/24** from 317.85 -318.0 m
- **53/27** from 319.90 -320.0 m

DDH59 at 562,043mE -7,429,560mN

- **59/41** from 265.05 –265.20 m

5.) DNO, tuffaceous siltstone

Location: Mount Olympus mine, south of Mount Channar (Diligence Dome)

DNO at 598,794mE -7,408,360mN

SUMMARY OF SHRIMP Pb/Pb BADDELEYITE RESULTS (using ^{208}Pb corrected data)

Table DR1 HTC (Sill in the Hardey syncline); UWA mounts C-91 and C-92

Spot name	^{238}U (ppm)	^{232}Th (ppm)	Th/U	f_{206} (%)	^{207}Pb/^{206}Pb (± %)	Apparent age (Ma) ^{207}Pb/^{206}Pb (1σ)
First session						
Htc4.18-a	5724	432	0.08	0.215	0.1399 ± 1.1	2226 ± 18
Htc4.18-b	6121	494	0.08	0.000	0.1401 ± 1.1	2229 ± 18
Htc4.16-a	4321	385	0.09	0.200	0.1370 ± 1.7	2189 ± 29
Htc4.13-b*	2137	221	0.11	0.417	0.1376 ± 1.5	2197 ± 26
Htc4.13-a	3205	421	0.14	0.154	0.1371 ± 1.3	2191 ± 22
Htc2.18-a	3712	529	0.15	0.780	0.1432 ± 1.3	2267 ± 23
Htc2.9-b	5286	898	0.18	0.243	0.1394 ± 1.0	2219 ± 17
Htc2.9-a	6181	1167	0.20	0.405	0.1394 ± 1.0	2220 ± 18
Excluded (see below)						
Htc2.12-a	6307	1755	0.29	2.398	0.1442 ± 1.5	2279 ± 25
Htc2.3-a	5948	1719	0.30	8.612	0.1383 ± 7.4	2206 ± 128
Htc2.10-a	1679	492	0.30	2.380	0.1659 ± 1.8	2517 ± 31
Htc2.20-a	4927	1485	0.31	3.053	0.1382 ± 2.7	2204 ± 48
Htc4.13-c*	7177	2371	0.34	0.673	0.1522 ± 3.5	2370 ± 60
Htc2.18-b	3861	1282	0.34	1.608	0.1529 ± 3.3	2378 ± 56
Htc2.5-a	7260	2605	0.37	6.541	0.1549 ± 4.0	2401 ± 67
Htc2.1-a	2148	1428	0.69	7.388	0.1928 ± 2.4	2766 ± 39
Htc4.18-c*	5413	5110	0.98	0.187	0.2263 ± 3.6	3026 ± 57
Htc4.18-d*	7827	9954	1.31	0.000	0.2600 ± 4.4	3247 ± 70
Htc4.16-b	4932	523	0.11	0.189	0.1337 ± 1.0	2147 ± 18
Htc4.16-c*	5412	689	0.13	0.314	0.1269 ± 1.5	2055 ± 26
Second session		**(after re-polish)**				
Htc4.6-a	5826	389	0.07	-0.063	0.1387 ± 0.9	2212 ± 15
Htc4.6-b	6533	453	0.07	0.102	0.1376 ± 0.8	2198 ± 14
Htc2.10-c	2593	183	0.07	0.429	0.1382 ± 1.3	2205 ± 22
Htc2.10-b	2009	149	0.08	0.736	0.1351 ± 1.3	2165 ± 23
Htc4.18-e	6911	526	0.08	0.060	0.1372 ± 0.8	2193 ± 14
Htc4.18-f	8655	742	0.09	0.067	0.1383 ± 0.7	2205 ± 11
Htc4.13-d	1708	169	0.10	0.319	0.1366 ± 1.5	2185 ± 26
Htc4.13-e	2109	221	0.11	0.378	0.1351 ± 1.7	2165 ± 29
Htc2.9-d	2677	373	0.14	0.439	0.1406 ± 1.4	2235 ± 23
Htc2.9-c	3425	543	0.16	0.441	0.1400 ± 1.3	2228 ± 23
Excluded (see below)						
Htc2.18-c	7161	1953	0.28	0.915	0.1525 ± 1.0	2374 ± 17
Htc4.18-g*	5420	10067	1.92	0.613	0.3280 ± 5.4	3608 ± 82

Htc4.16-d	6478	795	0.13	0.160	0.1297 ± 1.3	2094 ± 23
	Third session					
	Excluded (see below)					
Htc2.20-c	3260	875	0.28	13.163	0.1255 ± 15.6	2037 ± 277
Htc2.20-b	3286	1282	0.40	11.236	0.1607 ± 4.7	2463 ± 80
Htc2.5-b	3850	2170	0.58	18.380	0.1529 ± 9.8	2378 ± 166

All data with a Th/U ratio >0.2 were excluded from the age calculation. Common ^{206}Pb was low in the HTC baddeleyite crystals and the highest value of the analyses used in the age calculation was 0.78%. Analyses Htc4.16-b, Htc4.16-c* and Htc4.16-d were excluded as obvious outliers compared to the overall dataset. The 2σ error of the mean of the standard U/Pb ratio was 1.74% (n = 9) in the first session, 3.89% (n = 5) in the second session and 1.48% (n = 7) in the third session. A 30 µm Kohler aperture was used for all SHRIMP analyses of baddeleyite (including data below).

Table DR2 ETC (Sill in the Turee Creek syncline); UWA mounts C-91 and C-92

Spot name	**^{238}U** (ppm)	**^{232}Th** (ppm)	**Th/U**	**f_{206}** (%)	**$^{207}Pb/^{206}Pb$** (± %)	**Apparent age (Ma)** **$^{207}Pb/^{206}Pb$** (1σ)
	First session					
Etc9.18-b	5809	258	0.05	0.968	0.1382 ± 1.0	2205 ± 18
Etc9.18-a	6359	305	0.05	1.739	0.1381 ± 1.5	2203 ± 26
Etc9.13-b	6706	427	0.07	1.076	0.1383 ± 1.4	2206 ± 24
Etc9.14-b	7146	495	0.07	0.586	0.1385 ± 2.4	2209 ± 41
Etc9.13-a	8679	610	0.07	0.676	0.1379 ± 0.8	2201 ± 14
Etc9.14-a	9536	671	0.07	0.146	0.1384 ± 0.6	2208 ± 11
Etc9.9-b	7343	534	0.08	0.636	0.1396 ± 1.2	2223 ± 20
Etc9.9-a	6382	538	0.09	2.359	0.1369 ± 3.2	2188 ± 55
Etc9.4-a	4259	475	0.12	0.605	0.1362 ± 1.5	2179 ± 26
Pf7.a-a	11347	1319	0.12	2.048	0.1348 ± 2.1	2161 ± 37
	Excluded (see below)					
Pf7.8-a	6604	33223	5.20	2.252	0.5971 ± 7.8	4501 ± 114
Etc9.19-a	6215	372	0.06	4.443	0.1186 ± 4.9	1935 ± 88
Pf7.c-a	2567	286	0.11	0.502	0.1166 ± 4.2	1905 ± 76
	Second session		**(after repolish)**			
Etc9.18-c	4204	207	0.05	1.908	0.1342 ± 1.9	2153 ± 33
Etc9.13-e	8293	537	0.07	2.865	0.1376 ± 2.6	2197 ± 45

Pf7.c-b	3978	258	0.07	3.384	0.1344 ± 3.6	2157 ± 63
Etc9.13-d	12125	828	0.07	0.108	0.1380 ± 0.8	2202 ± 13
Etc9.13-c	11109	764	0.07	0.151	0.1392 ± 0.6	2217 ± 11
Etc9.19-d	3221	229	0.07	0.631	0.1410 ± 1.7	2239 ± 30
Etc9.19-c	2394	173	0.07	0.358	0.1399 ± 1.8	2226 ± 32
Etc9.19-b	1841	240	0.13	3.045	0.1455 ± 1.7	2294 ± 29
	Excluded (see below)					
Etc9.6-a	4995	303	0.06	4.507	0.1341 ± 4.9	2152 ± 85
	Third session					
Etc9.13-f*	4854	320	0.07	0.802	0.1384 ± 0.8	2208 ± 13
Etc9.4-b	2597	230	0.09	2.947	0.1317 ± 2.4	2121 ± 43
	Excluded (see below)					
Etc9.6-a**	3130	194	0.06	4.600	0.1317 ± 3.3	2120 ± 58
Etc9.19-a**	2658	624	0.24	2.157	0.1535 ± 2.2	2385 ± 37
Pf7.a-a**	3625	1010	0.29	1.902	0.1349 ± 1.9	2163 ± 32
Pf7.a-b**	3995	1316	0.34	2.461	0.1339 ± 2.8	2149 ± 49

All data with a Th/U ratio >0.2 were excluded from the age calculation. Analyses with common ^{206}Pb >3.5% were also not used. To increase the precision of the data, the normal cut-off value of <2% common ^{206}Pb was lifted to <3.5% with no effect on the age but with the result of significant improvement of the MSWD. Analysis Pf7.c-a was excluded as an outlier compared to the overall dataset. The 2σ error of the mean of the standard U/Pb ratio was 1.74% (n = 9) in the first session, 3.89% (n = 5) in the second session and 1.48% (n = 7) in the third session (all sessions simultaneous with HTC).

Table DR3 S72-5 (Dolerite dyke in the Hardey syncline)

Spot name	**^{238}U** (ppm)	**^{232}Th** (ppm)	**Th/U**	**f_{206}** (%)	**^{207}Pb/^{206}Pb** (± %)	**Apparent age (Ma) ^{207}Pb/^{206}Pb** (1σ)
	First session					
MHS3.P-a	778	65	0.09	0.590	0.1192 ± 1.7	1944 ± 31
MHS5.G-a	1159	176	0.16	-0.029	0.1218 ± 3.5	1983 ± 63
MHS6.H-a	2435	453	0.19	0.278	0.1219 ± 1.4	1984 ± 25

	Excluded (see below)					
MHS5.I-b	1772	384	0.22	1.044	0.1107 ± 3.2	1811 ± 57
S72-5a.J-a	594	384	0.67	2.142	0.1114 ± 24.6	1823 ± 446
MHS6.H-b*	328	29	0.09	0.710	0.1153 ± 5.3	1884 ± 95
MHS5.I-a	1168	188	0.17	0.864	0.1151 ± 1.8	1881 ± 32
	Second session					
MHS3.P-b_1*	551	25	0.05	1.492	0.1222 ± 2.2	1989 ± 40
S72-5a.Q-a	1178	94	0.08	1.047	0.1225 ± 1.9	1994 ± 35
MHS4.O-a	865	77	0.09	0.629	0.1271 ± 1.4	2058 ± 25
MHS4.O-b	712	85	0.12	1.049	0.1232 ± 1.9	2003 ± 34
S72-5a.K-a	400	50	0.13	1.076	0.1235 ± 2.9	2007 ± 51
MHS1.M-a	1222	160	0.14	0.267	0.1273 ± 1.3	2061 ± 24
S72-5b.O-a	1318	189	0.15	0.581	0.1230 ± 2.2	2000 ± 39
MHS1.M-b	1451	261	0.19	0.050	0.1269 ± 2.1	2055 ± 37
MHS6.H-c	1424	272	0.20	0.611	0.1208 ± 1.2	1968 ± 21
	Excluded (see below)					
MHS5.G-b*	825	222	0.28	1.151	0.1165 ± 4.0	1904 ± 71
MHS7.D-a	2967	817	0.28	1.322	0.1117 ± 3.0	1828 ± 54
S72-5a.K-b*	262	78	0.31	1.569	0.1194 ± 5.5	1947 ± 99
MHS4.O-b**	735	328	0.46	5.673	0.1017 ± 8.9	1656 ± 164
MHS4.O-a**	801	368	0.47	5.602	0.1054 ± 8.1	1721 ± 149
S72-5a.F-a	1299	628	0.50	0.973	0.1086 ± 5.6	1776 ± 101
MHS7.L-a	807	36	0.05	6.830	0.1267 ± 6.4	2052 ± 112
MHS2.E-a	796	36	0.05	1.582	0.1148 ± 3.0	1877 ± 53
MHS3.P-b*	704	53	0.08	0.788	0.1186 ± 2.1	1935 ± 38
S72-5b.R-a	907	58	0.07	0.945	0.1169 ± 2.9	1909 ± 53
MHS6.H-d*	657	103	0.16	1.123	0.1198 ± 2.8	1953 ± 50
S72-5b.R-b	1254	142	0.12	0.588	0.1213 ± 1.9	1975 ± 34
MHS1.N-a	784	44	0.06	2.439	0.1194 ± 3.4	1947 ± 61
S72-5b.O-b	1194	165	0.14	0.454	0.1209 ± 2.2	1970 ± 39
MHS3.P-c*	676	106	0.16	3.336	0.1107 ± 6.8	1810 ± 123
MHS3.P-c_1*	815	44	0.06	1.786	0.1196 ± 3.4	1950 ± 61
	Third session		**(after repolish)**			
S72-5b.H-a	814	39	0.05	0.347	0.1230 ± 1.4	2001 ± 25
S72-5b.H-b	782	39	0.05	0.265	0.1227 ± 1.5	1996 ± 26
MHS3.P-g*	596	39	0.07	0.425	0.1229 ± 2.0	1998 ± 35
MHS3.P-e	974	67	0.07	0.845	0.1205 ± 2.4	1964 ± 43
MHS5.I-c	680	52	0.08	1.190	0.1217 ± 2.6	1981 ± 45
S72-5a.K-c	182	31	0.17	1.913	0.1185 ± 5.4	1934 ± 96
	Excluded (see below)					
MHS2.G-a	825	187	0.23	1.352	0.1141 ± 3.6	1866 ± 65
MHS4.B-a	1227	279	0.24	0.818	0.1197 ± 2.4	1952 ± 42
S72-5b.R-c	1702	393	0.24	1.027	0.1129 ± 3.3	1847 ± 60

MHS6.H-e	347	81	0.24	2.285	0.1038 ± 7.0	1693 ± 129
MHS4.B-b	1126	267	0.24	0.628	0.1214 ± 2.4	1977 ± 43
S72-5b.J-a	698	192	0.28	8.210	0.0892 ± 25.7	1408 ± 492
MHS7.D-b	2318	767	0.34	-0.071	0.1219 ± 3.2	1984 ± 58
MHS1.M-c	1285	460	0.37	-2.486	0.1504 ± 3.7	2350 ± 64
MHS2.E-b	361	20	0.06	1.902	0.1162 ± 3.7	1898 ± 67
MHS3.P-d	899	58	0.07	0.801	0.1160 ± 2.1	1895 ± 38
MHS2.E-c	476	31	0.07	1.761	0.1172 ± 3.4	1914 ± 61
MHS5.I-d	650	43	0.07	3.219	0.1137 ± 5.6	1860 ± 100
S72-5a.Q-c	946	71	0.08	0.388	0.1191 ± 1.7	1943 ± 30
S72-5a.Q-b	892	67	0.08	0.567	0.1187 ± 1.8	1936 ± 32
MHS3.P-f*	684	74	0.11	1.000	0.1169 ± 3.1	1909 ± 56
MHS2.I-a	1595	280	0.18	0.883	0.1183 ± 3.3	1931 ± 59
MHS2.G-b	838	158	0.19	1.158	0.1158 ± 2.6	1893 ± 48

All data with a Th/U ratio >0.2 were excluded from the age calculation. Analyses with common ^{206}Pb >2% were also not used in age calculation. Approximately 70% of the baddeleyite crystals had a zircon overgrowth which was too small to be analyzed by SHRIMP. The overgrowth results from a later metamorphic event, possibly during the Capricorn orogeny at around ~1770 Ma. Analysis spots which overlapped the edge of the baddeleyite grain with possible analysis of metamorphic zircon were excluded from the age calculation. The 2σ error of the mean of the standard U/Pb ratio was 3.57% (n = 4) in the first session, 1.35% (n = 5) and 2.54% (n = 9) in the second session (this dataset was treated as two separate sessions due to SHRIMP failure/restart) and 3.90% (n = 7) in the third session.

Table DR4 DDH48+ (Dykes in the Paraburdoo mine); UWA mounts 03-91 and 03-92

Spot name	**^{238}U** (ppm)	**^{232}Th** (ppm)	**Th/U**	f_{206} (%)	**^{207}Pb/^{206}Pb** (± %)	**Apparent age (Ma)** **^{207}Pb/^{206}Pb** (1σ)
	Second session		**(after repolish)**			
48/9.4-c*	471	18	0.04	1.32	0.1209 ± 2.0	1970 ± 36
48/9.4-b*	445	19	0.04	0.91	0.1276 ± 2.3	2066 ± 40
48/12.32-a	408	23	0.06	0.77	0.1237 ± 1.5	2011 ± 27
48/12.32-b	392	25	0.07	0.44	0.1239 ± 1.4	2013 ± 26

48/12.25-b*	424	33	0.08	1.47	0.1227 ± 2.6	1995 ± 46
48/12.3-a	390	31	0.08	1.05	0.1263 ± 1.6	2047 ± 28
52/24.7-b	143	11	0.08	1.58	0.1189 ± 3.9	1940 ± 69
48/12.3-a***	430	38	0.09	1.13	0.1225 ± 1.7	1994 ± 30
48/12.29-a	285	31	0.11	1.18	0.1239 ± 2.1	2014 ± 38
48/12.3-c*	327	37	0.12	0.81	0.1258 ± 1.8	2040 ± 32
48/12.29-b	264	30	0.12	1.55	0.1235 ± 2.1	2008 ± 37
48/12.3-b*	392	47	0.12	0.81	0.1217 ± 1.9	1981 ± 35
53/27.8-b	510	78	0.16	1.29	0.1212 ± 1.7	1974 ± 31
53/27.8-a	522	84	0.17	0.70	0.1237 ± 1.6	2010 ± 29
48/12.3-a**	545	92	0.17	1.29	0.1254 ± 1.9	2034 ± 33
59/41.2-a	270	46	0.17	1.75	0.1206 ± 2.5	1966 ± 45
	Excluded (see below)					
48/6.1-b	189	51	0.28	1.76	0.1276 ± 3.0	2066 ± 54
48/6.1-a	213	86	0.42	2.79	0.1257 ± 3.5	2038 ± 62
48/9.31-a	91	3	0.03	7.94	0.1309 ± 7.4	2110 ± 130
53/27.28-a	122	5	0.04	5.73	0.1196 ± 5.4	1950 ± 96
53/24.7-a	145	9	0.06	4.16	0.1260 ± 4.6	2042 ± 81
48/12.30-a	212	16	0.08	18.53	0.1246 ± 13.3	2023 ± 236
48/12.15-c*	48	4	0.09	9.31	0.1311 ± 10.4	2113 ± 183
48/12.19-a	477	72	0.16	5.48	0.1219 ± 3.8	1985 ± 68
53/27.26-a	361	57	0.16	9.20	0.1202 ± 9.9	1960 ± 176
48/12.15-b	209	41	0.20	6.02	0.1139 ± 7.7	1863 ± 139
53/24.27-a	181	7	0.04	2.33	0.1246 ± 2.9	2022 ± 51
53/27.24-a	137	7	0.05	2.52	0.1211 ± 3.5	1973 ± 63
48/12.25-c*	372	22	0.06	2.07	0.1259 ± 2.7	2042 ± 47
48/12.25-a	337	20	0.06	2.60	0.1233 ± 2.2	2004 ± 38
53/27.18-a	331	20	0.06	2.77	0.1272 ± 2.9	2059 ± 50
48/9.4-a	301	20	0.07	3.03	0.1238 ± 2.9	2011 ± 52
48/12.15-a	207	37	0.18	2.45	0.1239 ± 3.6	2014 ± 63

All data with a Th/U ratio >0.2 were excluded from the age calculation. Analyses with common ^{206}Pb >2% were also not used in age calculation. Although the age did not vary significantly by including analyses with up to 3.5% of common ^{206}Pb (2011 ± 15 Ma instead of 2009 ± 16 Ma), they were excluded as this improved the MSWD. The 2σ error of the mean of the standard U/Pb ratio was 1.33% (n = 11) in the second session. The data of the first session on these samples were not used as they were collected from SHRIMP B shortly after its installation and peak centering problems were encountered. However, the reduced data gave an age very close to 2010 Ma.

SUMMARY OF SHRIMP U–Pb ZIRCON RESULTS (using ^{204}Pb corrected data)

Table DR5 DNO (Tuffaceous siltstone from Mount Olympus mine); UWA mount SM1

Spot name	U (ppm)	Th (ppm)	Th/U	f_{206} (%)	$^{238}U/^{206}Pb$	$^{207}Pb/^{235}U$ (± %)	$^{207}Pb/^{206}Pb$	Apparent age (Ma) $^{208}Pb/^{232}Th$ (1σ)	$^{207}Pb/^{206}Pb$ (1σ)	Conc. (%)
First session										
DNO.1-1	62	39	0.65	0.000	2.727 ± 0.020	6.33 ± 2.4	0.1253 ± 0.0116	2035 ± 53	2033 ± 21	99
DNO.2-1	133	96	0.74	0.131	2.704 ± 0.018	6.41 ± 2.0	0.1257 ± 0.0087	2015 ± 43	2039 ± 15	100
DNO.3-1	73	41	0.59	0.046	2.744 ± 0.021	6.30 ± 2.4	0.1254 ± 0.0111	2015 ± 54	2034 ± 20	98
DNO.4-1	125	91	0.75	0.102	2.756 ± 0.018	6.25 ± 2.0	0.1250 ± 0.0089	1971 ± 43	2029 ± 16	98
DNO.5-1	96	81	0.87	0.201	2.700 ± 0.019	6.38 ± 2.2	0.1250 ± 0.0117	1993 ± 47	2029 ± 21	100
DNO.6-1	73	44	0.63	0.253	2.798 ± 0.021	6.22 ± 2.5	0.1262 ± 0.0128	1903 ± 55	2045 ± 23	96
DNO.7-1	96	51	0.55	0.140	2.779 ± 0.019	6.15 ± 2.1	0.1240 ± 0.0100	1935 ± 48	2015 ± 18	98
DNO.8-1	61	114	1.93	0.158	2.175 ± 0.020	10.74 ± 2.3	0.1695 ± 0.0102	2426 ± 54	2552 ± 17	96
DNO.9-1	117	91	0.81	0.143	2.687 ± 0.018	6.62 ± 2.1	0.1290 ± 0.0095	1962 ± 44	2084 ± 17	98
DNO.10-1	146	103	0.73	0.181	2.773 ± 0.018	6.17 ± 2.0	0.1241 ± 0.0082	1971 ± 41	2016 ± 15	98
DNO.11-1	87	62	0.74	0.135	2.782 ± 0.019	6.24 ± 2.2	0.1259 ± 0.0110	1979 ± 50	2041 ± 20	97
DNO.12-1	112	75	0.69	0.140	2.736 ± 0.018	6.27 ± 2.0	0.1244 ± 0.0081	1941 ± 41	2020 ± 14	99
DNO.14-1	81	66	0.85	0.050	2.705 ± 0.019	6.36 ± 2.1	0.1248 ± 0.0095	1996 ± 44	2025 ± 17	100
DNO.15-1	129	95	0.76	0.088	2.736 ± 0.019	6.29 ± 2.0	0.1248 ± 0.0073	2013 ± 42	2027 ± 13	99
DNO.16-1	66	42	0.66	0.512	2.804 ± 0.020	5.96 ± 2.7	0.1212 ± 0.0172	1850 ± 68	1974 ± 31	100
DNO.18-1	131	80	0.63	0.083	2.485 ± 0.018	7.67 ± 2.0	0.1383 ± 0.0092	2097 ± 51	2206 ± 16	99
DNO.20-1	101	65	0.66	-0.162	2.693 ± 0.019	6.55 ± 2.3	0.1280 ± 0.0129	2063 ± 50	2070 ± 23	98
DNO.21-1	125	71	0.59	0.092	2.691 ± 0.018	6.40 ± 2.0	0.1250 ± 0.0082	2011 ± 44	2029 ± 15	100
DNO.22-1	49	36	0.75	-0.057	2.672 ± 0.021	6.53 ± 2.4	0.1265 ± 0.0118	2076 ± 53	2050 ± 21	100
DNO.23-1	74	51	0.71	-0.038	2.693 ± 0.019	6.31 ± 2.4	0.1233 ± 0.0098	1981 ± 59	2005 ± 17	102
DNO.24-1	56	55	1.01	-0.138	2.451 ± 0.020	7.73 ± 2.3	0.1374 ± 0.0116	2239 ± 60	2194 ± 20	101
DNO.25-1	96	62	0.66	0.054	2.666 ± 0.019	6.41 ± 2.1	0.1240 ± 0.0091	2003 ± 45	2014 ± 16	102
DNO.26-1	73	48	0.68	0.074	2.682 ± 0.019	6.50 ± 2.4	0.1263 ± 0.0139	2045 ± 62	2048 ± 25	100
DNO.27-1	48	37	0.80	0.060	2.746 ± 0.021	6.40 ± 2.4	0.1275 ± 0.0122	1937 ± 50	2064 ± 22	97
DNO.28-1	175	143	0.85	-0.003	2.733 ± 0.018	6.34 ± 1.9	0.1256 ± 0.0062	2024 ± 38	2038 ± 11	99
DNO.29-1	160	148	0.96	0.028	2.618 ± 0.019	6.65 ± 2.0	0.1263 ± 0.0065	2030 ± 41	2047 ± 11	102
DNO.31-1	130	90	0.72	0.012	2.729 ± 0.018	6.40 ± 2.6	0.1267 ± 0.0195	2042 ± 52	2053 ± 34	98
DNO.32-1	52	36	0.72	-0.294	2.669 ± 0.020	6.73 ± 2.3	0.1302 ± 0.0107	2066 ± 62	2101 ± 19	98
DNO.33-1	86	60	0.72	0.183	2.763 ± 0.019	6.24 ± 2.1	0.1250 ± 0.0100	1969 ± 45	2028 ± 18	98
DNO.34-1	84	52	0.64	0.101	2.703 ± 0.019	6.40 ± 2.2	0.1254 ± 0.0109	2046 ± 51	2035 ± 19	100
DNO.35-1	63	32	0.52	0.023	2.679 ± 0.019	6.44 ± 2.2	0.1251 ± 0.0097	2012 ± 49	2031 ± 17	101
DNO.37-1	99	72	0.76	0.230	2.145 ± 0.018	10.54 ± 2.1	0.1639 ± 0.0112	2304 ± 51	2496 ± 19	99
DNO.39-1	146	66	0.46	0.021	1.998 ± 0.018	12.73 ± 1.8	0.1845 ± 0.0050	2583 ± 59	2694 ± 8	97

	Discor dant									
DNO.13-1	300	138	0.47	0.100	3.105 ± 0.017	6.11 ± 1.8	0.1376 ± 0.0057	2070 ± 42	2197 ± 10	82
DNO.17-1	313	231	0.76	0.522	6.107 ± 0.017	3.81 ± 2.0	0.1687 ± 0.0089	1043 ± 26	2545 ± 15	38
DNO.19-1	235	45	0.20	0.388	3.406 ± 0.018	5.08 ± 2.0	0.1255 ± 0.0095	1796 ± 83	2036 ± 17	82
DNO.30-1	208	57	0.29	0.062	2.364 ± 0.018	9.35 ± 2.0	0.1603 ± 0.0092	2241 ± 66	2459 ± 16	92
DNO.36-1	179	134	0.77	0.288	3.923 ± 0.030	4.38 ± 3.4	0.1247 ± 0.0154	1408 ± 51	2024 ± 27	72
DNO.38-1	81	54	0.69	0.168	2.897 ± 0.019	5.94 ± 2.2	0.1248 ± 0.0105	1878 ± 45	2027 ± 19	94
	Second session									
DNO.40-1	80	52	0.67	0.324	2.684 ± 0.018	6.37 ± 2.3	0.1240 ± 0.0134	1980 ± 57	2014 ± 24	101
DNO.41-1	103	79	0.79	0.305	2.722 ± 0.017	6.25 ± 2.1	0.1234 ± 0.0111	1948 ± 44	2006 ± 20	101
DNO.42-1	104	70	0.69	0.227	2.687 ± 0.017	6.36 ± 2.0	0.1239 ± 0.0107	2006 ± 46	2014 ± 19	101
DNO.43-1	102	73	0.74	0.362	2.716 ± 0.017	6.34 ± 2.2	0.1249 ± 0.0131	1972 ± 50	2027 ± 23	100
DNO.44-1	100	68	0.70	0.269	2.696 ± 0.017	6.27 ± 2.1	0.1226 ± 0.0121	2028 ± 49	1994 ± 21	102
DNO.45-1	71	43	0.62	0.269	2.723 ± 0.019	6.28 ± 2.5	0.1239 ± 0.0163	1961 ± 64	2013 ± 29	100
DNO.46-1	93	91	1.02	0.019	2.714 ± 0.018	6.44 ± 2.1	0.1267 ± 0.0107	2031 ± 43	2053 ± 19	99
DNO.47-1	75	46	0.64	0.297	2.627 ± 0.018	6.52 ± 2.3	0.1243 ± 0.0142	2057 ± 59	2019 ± 25	103
DNO.48-1	113	78	0.71	0.257	2.692 ± 0.017	6.32 ± 2.1	0.1234 ± 0.0115	2005 ± 47	2005 ± 20	102
DNO.49-1	138	88	0.66	0.112	2.090 ± 0.017	10.63 ± 1.8	0.1612 ± 0.0065	2542 ± 49	2468 ± 11	102
DNO.50-1	170	144	0.87	0.000	2.701 ± 0.016	6.41 ± 1.8	0.1256 ± 0.0068	2049 ± 37	2038 ± 12	100
DNO.51-1	185	156	0.87	0.054	2.104 ± 0.016	11.02 ± 1.7	0.1682 ± 0.0061	2534 ± 45	2540 ± 10	99
DNO.52-1	214	108	0.52	0.356	5.682 ± 0.017	1.81 ± 2.7	0.0745 ± 0.0214	1101 ± 31	1055 ± 43	99
DNO.53-1	97	76	0.81	-0.008	2.096 ± 0.017	11.17 ± 1.9	0.1697 ± 0.0070	2554 ± 51	2555 ± 12	98

All 53 zircon grains were analyzed, with concordance <95% being the only criterion for data exclusion from the age determination. U–Th–Pb ratios and absolute abundances were determined relative to the CZ3 zircon standard (564 Ma: $^{206}Pb/^{238}U$ = 0.0914). Grain DNO.52-1 was not included in the concordia plot as it had experienced considerable secondary Pb loss. Also discordant data were not included in the plot. The 2σ error of the mean of the standard U/Pb ratio was 1.08% (n = 11) for the first session and 1.13% (n = 9) for the second session. A 50 µm Kohler aperture was used to produce a SHRIMP spot size with a 15-20 µm diameter necessary for the relatively small zircon grains. The primary beam intensity was ~2.5 nA in the first session and ~5.5 nA in the second session.

80% of the concordant analyses belong to one population of ca. 2030 Ma, 2% belong to a ca. 2200 Ma population and 18% to a ca. 2500–2600 Ma age group.

<u>Labeling of spot analyses:</u>

Examples: **MHS3.P-a, Htc4.18-a** or **DNO.43-1**

MHS3 (**Htc4; DNO**) is the sample, **P-…** (**18-…; 43-…**) is the grain on the plug P (18; 43), and **a (1)** the first spot analysis.

MHS3.P-b = second analysis on same spot

MHS3.P-c* = second spot analysis on same grain

MHS3.P-d* = second analysis on second spot on same grain

MHS3.P-a** = first spot analysis on second grain on plug

MHS3.P-b** second analysis on same spot of second grain (and so on)

MHS3.P-e = first spot analysis after re-polishing the mount (and so on)

Figure DR1 Concordia plot for DNO tuffaceous siltstone

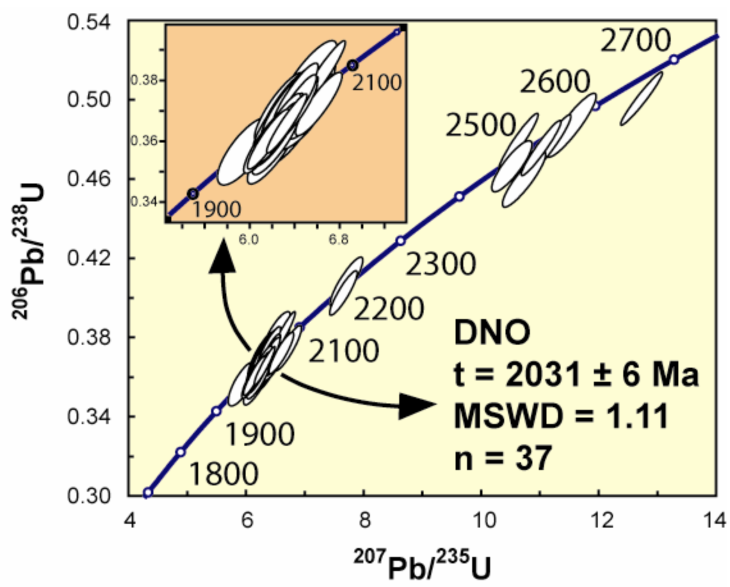

Fig. DR1. Concordia diagram showing SHRIMP U–Pb in zircon data (concordant >95%) with isolated youngest population. Sample DNO, tuffaceous siltstone from Mount Olympus.