

SUPPLEMENTARY MATERIAL

1. METHODOLOGY

1.1. $^{40}\text{Ar}/^{39}\text{Ar}$ METHODOLOGY

Samples ZULU-1-2150 and ZULU-1-133 were irradiated in the 8C facility of the Mac Master Nuclear Reactor (Hamilton, Ontario, Canada). Irradiation lasted 80 h with a global efficiency (J/h) of $6.755 \times 10^{-5} \text{ h}^{-1}$. The irradiation standard was sanidine TCRs ($28.608 \pm 0.033 \text{ Ma}$ according to Renne *et al.* (1998, 2010 and 2011)). Four whole rock fragments were analyzed by the $^{40}\text{Ar}/^{39}\text{Ar}$ method by step-heating with a CO_2 laser probe coupled with a MAP 215 mass spectrometer. The procedure was described by Ruffet *et al.* (1991, 1995, 1997).

Blanks were performed routinely each first or third/fourth run, and are subtracted from the subsequent sample gas fractions. Apparent age errors are plotted at the 1σ level and do not include the errors on the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio and age of the monitor and decay constant. Plateau ages were calculated if 70% or more of the $^{39}\text{Ar}_K$ was released in at least three or more contiguous steps which define apparent ages agreeing, to within 2σ , with the integrated age of the plateau segment. Pseudo-plateau ages (PPA) can be defined with less than 70% of the $^{39}\text{Ar}_K$ released. The errors on the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio and age of the monitor and decay constant are included in the final calculation of the error margins on the plateau ages.

$^{40}\text{Ar}/^{39}\text{Ar}$ ages are provided with 1σ errors. Analytical data and parameters used for calculations (*e.g.* isotopic ratios measured on pure K, Ca and Cl salts; mass discrimination; atmospheric argon ratios; J parameter; decay constants) and reference sources are available in the supplementary data repository.

2. $^{40}\text{Ar}/^{39}\text{Ar}$ RESULT

			J param eter	error J			Mass Discrim ination (1+e)	Err Discrimination							
	ZULU-1-2150 Whole Rock		5,44E- 03	1,84E- 05			1,0094 05	1,32E -03							
step	40Ar	Error 40Ar	39Ar	Error 39Ar	38Ar	Error 38Ar	37Ar	Error 37Ar	36Ar	Error 36Ar	40Ar*/ 39ArK	Error 40Ar*/39 ArK	Apparent age (My)	Error Age (My)	Delay to irradiation (day)
1	13947 ,5442	6,101 017	5,805 897	0,058 312	0,011 518	0,017 301	2,1854 87	0,032 123	47,75 6713	0,101 082	37,82 4346	13,6598 32	338,064 387	111,36 1226	141,970833
2	1516, 6245	2,183 166	3,705 993	0,056 703	0,009 085	0,017 382	1,5773 57	0,023 369	5,177 782	0,029 653	8,208 76	3,22910 4	78,9272 53	30,381 654	141,990972
3	3966, 62511	2,500 83	11,62 0303	0,039 437	0,000 001	0,018 984	3,1115 88	0,067 258	13,16 2724	0,057 14	16,19 7553	2,27198 5	152,566 809	20,531 451	142,000694
4	2533, 40396	2,148 812	11,67 2432	0,056 312	0,000 001	0,020 373	2,3244 95	0,037 907	8,182 282	0,058 727	15,92 6477	1,83528 3	150,116 615	16,611 205	142,020833
5	10726 ,7705	5,425 272	45,03 4183	0,100 496	0,000 001	0,016 589	11,596 106	0,106 11	34,34 9124	0,123 829	19,46 4004	1,43918 1	181,833 39	12,812 045	142,040972
6	4042, 35347	1,225 101	21,81 337	0,107 732	0,009 496	0,016 169	6,4809 75	0,045 846	12,60 6767	0,071 085	19,81 4972	1,31791 8	184,949 978	11,716 889	142,661111
7	8661, 53589	2,287 759	65,72 652	0,056 072	0,000 001	0,013 918	17,978 897	0,035 988	25,66 5943	0,053 087	20,14 7843	0,66570 7	187,900 898	5,9454 64	142,680556
8	996,9 85145	1,344 305	19,44 3741	0,036 301	0,000 001	0,015 522	5,0512 26	0,044 79	2,249 197	0,038 468	18,63 3238	0,61438 5	174,434 741	5,5281 91	142,690972
9	5830, 13327	3,853 757	165,9 14232	0,281 295	0,002 087	0,026 139	38,362 583	0,212 771	9,426 753	0,043 323	19,38 1758	0,14042 2	181,102 274	1,4478 17	142,730556
10	2333, 88641	0,992 268	67,24 0036	0,117 056	0,000 001	0,020 727	19,839 29	0,136 588	3,839 133	0,028 337	18,98 3644	0,16915 6	177,559 083	1,6697 55	142,750694
11	3539, 10117	3,130 017	109,6 82083	0,286 831	0,004 554	0,021 237	46,991 406	0,110 725	5,259 41	0,024 926	19,41 9636	0,13102 8	181,439 021	1,3769 59	142,770833
12	5215, 69188	1,737 048	113,5 0424	0,146 319	0,025 964	0,018 365	64,466 744	0,199 587	11,59 0196	0,026 223	17,75 7277	0,19129	166,600 661	1,8429 68	142,790972
Fusion	4582, 39111	3,004 539	115,0 19976	0,132 201	0,000 001	0,022 401	231,41 0691	0,288 736	9,498 324	0,044 58	19,94 5682	0,18911 5	186,109 307	1,8379 88	142,811111

Table 1: $^{40}\text{Ar}/^{39}\text{Ar}$ step heating result of sample ZULU-1-2150.

			J parameter	error J			Mass Discrimination (1+e)	Err Discrimination							
	ZULU-1-133 Whole Rock		5,28E-03	1,79E-05			1,011354	1,33E-03							
step	40Ar	Error 40Ar	39Ar	Error 39Ar	38Ar	Error 38Ar	37Ar	Error 37Ar	36Ar	Error 36Ar	40Ar*/39ArK	Error 40Ar*/39ArK	Apparent age (My)	Error Age (My)	Delay to irradiation (day)
1	1963,36063	1,636334	2,022549	0,039862	0,001287	0,013671	0,234699	0,02445	3,401642	0,028479	501,800252	11,314236	2341,08906	30,472054	238,650694
2	349,159269	1,074622	2,313371	0,037986	0,008052	0,015602	0,225727	0,031931	0,779057	0,013782	56,866368	2,120985	474,858372	15,683412	238,670833
3	1062,05041	0,884609	8,4518	0,080292	0,012057	0,013868	1,025965	0,022528	2,087852	0,022274	57,581847	1,054476	480,105125	7,93987	238,690972
4	171,883795	0,101663	3,162083	0,023161	0,000001	0,014739	0,433844	0,037101	0,377655	0,016791	22,180174	1,57834	200,29724	13,516168	238,720833
5	775,231488	1,080121	8,389772	0,080014	0,000001	0,011951	1,116611	0,029505	2,086972	0,016121	23,293821	0,741967	209,791969	6,36561	238,770833
6	579,999285	0,663776	9,99358	0,043602	0,007354	0,01028	1,416577	0,034571	1,330645	0,010569	21,999952	0,404489	198,756011	3,552338	238,800694
7	735,242418	0,556326	19,201017	0,045353	0,009299	0,011683	2,408204	0,024113	1,198927	0,014021	22,23715	0,252704	200,784217	2,30648	238,820833
8	405,435094	0,640757	17,063665	0,147096	0,000001	0,013505	2,167651	0,053093	0,354207	0,010291	19,566409	0,256349	177,81501	2,332692	238,840972
9	332,180574	0,552594	15,157971	0,041215	0,000001	0,016834	1,548994	0,028913	0,19068	0,012051	19,757376	0,245089	179,467109	2,240453	238,880556
10	322,085465	0,656611	13,990808	0,021915	0,00153	0,014043	1,40204	0,04313	0,229413	0,012542	19,750759	0,273717	179,409889	2,475934	238,900694
11	433,344501	0,770105	14,463961	0,046363	0,004405	0,013781	2,178349	0,035096	0,406314	0,01273	24,064756	0,284707	216,335709	2,564456	238,920833
12	642,24305	0,577883	10,101341	0,039862	0,000001	0,012827	3,161661	0,031046	0,594957	0,009755	51,953304	0,397594	438,412691	3,430263	238,940972
Fusion	2834,77725	1,190611	41,519315	0,060404	0,000001	0,012517	36,494625	0,107135	2,814525	0,013466	63,577181	0,31014	523,481836	2,987855	238,961111

Table 2: ⁴⁰Ar/³⁹Ar step heating result of sample ZULU-1-133.

Parameters	(36Ar/37Ar)Ca	0,000322	3	%
	(39Ar/37Ar)Ca	0,000788	4	%
	(38Ar/37Ar)Ca	0,000026	100	%
	(40Ar/37Ar)Ca	0,0006	100	%
	(40Ar/39Ar)K	0,00085	4	%
	(38A/39Ar)K	0,011	91	%
[3]	(36Cl/38Cl)	316	5	%
[1] and [1']	(40Ar/36Ar)Atm	298,56	0,104	%
[1] and [1']	(38Ar/36Ar)Atm	0,1885	0,159	%
[2]	Lambda 40	5,53E-10	1,35E-12	y-1
	Lambda 39	2,58E-03		y-1
	Lambda 37	1,98E-02		d-1
	Lambda 36Cl	2,26E-06		y-1

Table 3: $^{40}\text{Ar}/^{39}\text{Ar}$ parameters.

2.1. ISOTOPIC ANALYSES

		Rb	Sr	Th	U	Pb	⁸⁷ Sr/ ⁸⁶ Sr	2σ	²⁰⁶ Pb/ ²⁰ Pb	2σ	²⁰⁷ Pb/ ²⁰ Pb	2σ	²⁰⁸ Pb/ ²⁰ Pb	2σ	⁸⁷ Sr/ ⁸⁶ Sr	²⁰⁶ Pb/ ²⁰ Pb	²⁰⁷ Pb/ ²⁰ Pb	²⁰⁸ Pb/ ²⁰ Pb
							Sr		⁴ Pb		⁴ Pb		⁴ Pb		Sr _i	⁴ Pb _i	⁴ Pb _i	⁴ Pb _i
Funhaluro	N°1 4	0,1 8	72,4 2	1,3 7	0,3 8	4,2 8	0,705 597	6E- 06	17,939 5	0,00 07	15,507 6	0,00 07	38,167	0,0 03	0,705 58	17,784	15,500	37,984
Funhaluro	N°1 9	0,1 4	101, 45	1,7 6	0,4 4	3,9 4	0,705 566	4E- 06	17,820 4	0,00 12	15,478 1	0,00 08	38,062	0,0 03	0,705 56	17,626	15,468	37,806
Funhaluro	N°2 7	0,2 1	114, 41	1,7 6	0,4 3	2,4 1	0,705 398	4E- 06	17,965 1	0,00 08	15,499 8	0,00 10	38,186	0,0 04	0,705 39	17,654	15,484	37,767
Nhachengue	N°4 7	5,7 7	734, 09	1,6 3	0,3 7	1,2 5	0,704 163	8E- 06	18,527 7	0,00 10	15,541 8	0,00 09	38,636	0,0 03	0,704 12	18,116	15,522	38,044
Nhachengue	N°4 9	7,5 6	476, 76	1,4 6	0,3 6	1,2 0	0,704 008	4E- 06	18,514 6	0,00 09	15,538 2	0,00 08	38,609	0,0 03	0,703 92	18,097	15,518	38,054
Nhachengue	N°5 2	26, 89	524, 40	1,8 0	0,4 3	3,1 8	0,704 389	6E- 06	18,324 5	0,00 08	15,550 5	0,00 06	38,488	0,0 02	0,704 09	18,138	15,541	38,231
Nhachengue	N°5 5	17, 97	288, 13	1,1 5	0,4 6	3,3 9	0,704 616	4E- 06	18,328 2	0,00 10	15,555 2	0,00 11	38,447	0,0 03	0,704 26	18,141	15,546	38,294
Nhachengue	N°5 9	29, 23	565, 94	1,3 6	0,3 6	6,6 3	0,704 327	6E- 06	18,236 0	0,00 07	15,556 9	0,00 06	38,460	0,0 02	0,704 03	18,162	15,553	38,367

Nhamur a	N°1 15	6,1 7	147, 74	3,0 0	0,5 1	4,8 0	0,717 174	6E- 06	17,921 5	0,00 08	15,669 4	0,00 08	39,143	0,0 03	0,716 87	17,734	15,660	38,778
Nhamur a	N°1 18	40, 68	351, 49	2,1 2	0,5 6	2,8 1	0,710 485	4E- 06	18,065 4	0,00 09	15,597 0	0,00 07	38,766	0,0 02	0,709 63	17,709	15,579	38,326
Nhamur a	N°1 22	29, 10	297, 41	2,2 7	0,5 7	3,1 1	0,708 690	4E- 06	18,011 2	0,00 07	15,594 7	0,00 07	38,702	0,0 02	0,707 97	17,690	15,579	38,278
Nhamur a	N°1 26	27, 77	276, 20	2,1 7	0,6 0	2,7 9	0,708 271	6E- 06	18,056 8	0,00 06	15,594 4	0,00 07	38,751	0,0 02	0,707 53	17,677	15,575	38,299
Zululand	N°1 28	3,9 2	295, 73	0,6 9	0,1 6	1,5 0	0,703 925	6E- 06	17,900 4	0,00 10	15,513 0	0,00 07	38,173	0,0 02	0,703 83	17,717	15,504	37,906
Zululand	N°1 33	5,6 0	269, 50	0,7 3	0,1 5	1,4 5	0,704 015	6E- 06	17,807 1	0,00 13	15,496 6	0,00 10	38,180	0,0 03	0,703 86	17,623	15,487	37,892
Zululand	N°1 41	2,0 0	232, 00	0,6 6	0,1 4	2,3 1	0,703 841	6E- 06	17,673 6	0,00 09	15,477 5	0,00 07	38,050	0,0 02	0,704 04	17,568	15,472	37,886
Zululand duplicate	N°1 41						0,703 850	6E- 06	17,583 5	0,00 20	15,464 9	0,00 15	38,023	0,0 04	0,703 79	17,478	15,460	37,859

Table 4: Rb, Sr, Th, U and Pb concentrations in ppm. Initial values were calculated at 180 Ma.

3. REFERENCES

- [1] Lee, JY, Marti, K, Severinghaus, JP, Kawamura, K, Yoo, HS, Lee, JB, Kim, JS (2006). A redetermination of the isotopic abundances of atmospheric Ar. *Geochimica Cosmochimica Acta*, 70, 4507-4512.
- [1'] Mark, DF, Stuart, FM, De Podesta, M (2011). New high-precision measurements of the isotopic composition of atmospheric argon. *Geochimica Cosmochimica Acta*, 75, 7494-7501.
- [2] Renne, PR, Balco, G, Ludwig, RL, Mundil, R, Min, K, . (2011). Response to the comment by W.H. Schwarz et al. on "Joint determination of $(40)\text{K}$ decay constants and $(40)\text{Ar}^*/(40)\text{K}$ for the Fish Canyon sanidine standard, and improved accuracy for $(40)\text{Ar}/(39)\text{Ar}$ geochronology" by PR Renne et al. (2010). *Geochimica Cosmochimica Acta*, 75, 5097-5100.
- [3] York, D, Personal Communication - McMaster reactor

Regression method:

York, D. (1969). Least-squares fitting of a straight line with correlated errors. *Earth Planet. Sci. Lett.* 5, 320-4.

Ages and errors of Hb3gr and TCs monitors refers to:

Rennes, PR, Balco, G, Ludwig, Mundil, R, Min, K, . (2011). Response to the comment by W.H. Schwarz et al. on "Joint determination of $(40)\text{K}$ decay constants and $(40)\text{Ar}^*/(40)\text{K}$ for the Fish Canyon sanidine standard, and improved accuracy for $(40)\text{Ar}/(39)\text{Ar}$ geochronology" by PR Renne et al. (2010). *Geochimica Cosmochimica Acta*, 75, 5097-5100.