

CA-ID-TIMS zircon dating method

CA-ID-TIMS procedures described here are modified from Mundil et al., 2004, Mattinson, 2005 and Scoates and Friedman, 2008. After rock samples have undergone standard mineral separation procedures zircons are handpicked in alcohol. The clearest, crack- and inclusion-free grains are selected, photographed and then annealed in quartz glass crucibles at 900°C for 60 hours. Annealed grains are transferred into 3.5 mL PFA screwtop beakers, ultrapure HF (up to 50% strength, 500 mL) and HNO₃ (up to 14 N, 50 mL) are added and caps are closed finger tight. The beakers are placed in 125 mL PTFE liners (up to four per liner) and about 2 mL HF and 0.2 mL HNO₃ of the same strength as acid within beakers containing samples are added to the liners. The liners are then slid into stainless steel Parr™ high pressure dissolution devices, which are sealed and brought up to a maximum of 200°C for 8-16 hours (typically 175°C for 12 hours). Beakers are removed from liners and zircon is separated from leachate. Zircons are rinsed with >18 MΩ.cm water and subboiled acetone. Then 2 mL of subboiled 6N HCl is added and beakers are set on a hotplate at 80°-130°C for 30 minutes and again rinsed with water and acetone. Masses are estimated from the dimensions (volumes) of grains. Single grains are transferred into clean 300 mL PFA microcapsules (crucibles), and 50 mL 50% HF and 5 mL 14 N HNO₃ are added. Each is spiked with a ²³³⁻²³⁵U-²⁰⁵Pb tracer solution (EARTHTIME ET535), capped and again placed in a Parr liner (8-15 microcapsules per liner). HF and nitric acids in a 10:1 ratio, respectively, are added to the liner, which is then placed in Parr high pressure device and dissolution is achieved at 220°C for 40 hours. The resulting solutions are dried on a hotplate at 130°C, 50 mL 6N HCl is added to microcapsules and fluorides are dissolved in high pressure Parr devices for 12 hours at 180°C. HCl solutions are transferred into clean 7 mL PFA beakers and dried with 2 mL of 0.5 N H₃PO₄. Samples are loaded onto degassed, zone-refined Re filaments in 2 mL of silicic acid emitter (Gerstenberger and Haase, 1997).

Isotopic ratios are measured with a single collector VG 354S thermal ionization mass spectrometer equipped with Sector 54 electronics and analogue Daly photomultiplier. Analytical blanks are 0.1 pg for U and up to 0.6 pg for Pb. U fractionation was determined directly on individual runs using the EARTHTIME ET535 mixed ²³³⁻²³⁵U-²⁰⁵Pb isotopic tracer and Pb isotopic ratios were corrected for fractionation of 0.25 ± 0.04%/amu, based on replicate analyses of NBS-982 reference material and the values recommended by Thirlwall (2000). Data reduction employed the excel-based program of Schmitz and Schoene (2007). Standard concordia diagrams were constructed and regression intercepts, weighted averages calculated with Isoplot (Ludwig, 2003). Unless otherwise noted all errors are quoted at the 2 sigma or 95% level of confidence. Isotopic dates are calculated with the decay constants $\lambda_{238}=1.55125\text{E-}10$ and $\lambda_{235}=9.8485\text{E-}10$ (Jaffe et al, 1971) and a 238U/235U ratio of 137.88. EARTHTIME U-Pb synthetic solutions are analyzed on an on-going basis to monitor the accuracy of results. H. Lin did the mineral separation. T. Ockerman conducted activities in the clean lab and H. Lin and assisted with mass spectrometry.

Table 1A. CA-ID-TIMS ages data

Sample	Wt.	U	Pb	Compositional Parameters	Radiogenic Isotope Ratios	Isotopic Ages
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	mg	ppm	ppm	Th/U	$^{206}\text{Pb}^*$ x10 ⁻¹³ mol	mol % $^{206}\text{Pb}^*$	Pb* Pb _c	Pb _c (pg)	$\frac{^{206}\text{Pb}}{^{204}\text{Pb}}$	$\frac{^{208}\text{Pb}}{^{206}\text{Pb}}$	$\frac{^{207}\text{Pb}}{^{206}\text{Pb}}$	% err	$\frac{^{207}\text{Pb}}{^{235}\text{U}}$	% err	$\frac{^{206}\text{Pb}}{^{238}\text{U}}$	% err	corr. coef.	$\frac{^{207}\text{Pb}}{^{206}\text{Pb}}$	±	$\frac{^{207}\text{Pb}}{^{235}\text{U}}$	±	$\frac{^{206}\text{Pb}}{^{238}\text{U}}$	±
2404 -a																							
A	0.005	148	3.1	0.702	0.5425	99.20%	40	0.36	2312	0.224	0.04860	2.633	0.12562	2.758	0.0187	0.352	0.411	129	62	120.2	3.1	119.73	0.42
C	0.012	156	3.5	0.575	1.6093	99.77%	136	0.30	8098	0.183	0.06155	0.347	0.17812	0.403	0.0210	0.161	0.522	658.50	7.44	166.44	0.62	133.90	0.21
D	0.009	98	2.0	0.544	0.6600	99.36%	47	0.35	2874	0.174	0.04865	0.511	0.12597	0.564	0.0188	0.119	0.529	130.8	12.0	120.47	0.64	119.95	0.14
2404 - b																							
B	0.003	218	4.6	0.616	0.5151	98.70%	24	0.56	1426	0.197	0.04775	2.793	0.12348	2.941	0.0188	0.330	0.495	87	66	118.2	3.3	119.78	0.39
C	0.005	98	2.2	0.788	0.3950	98.41%	20	0.53	1161	0.252	0.04758	2.496	0.12289	2.639	0.0187	0.263	0.579	78	59	117.7	2.9	119.65	0.31
D	0.009	102	2.1	0.682	0.6923	99.08%	34	0.53	2015	0.218	0.04830	1.119	0.12415	1.195	0.0186	0.192	0.463	114	26	118.8	1.3	119.06	0.23

LA-ICP-MS dating method

The zircon is glued to the glass slide with double-sided tape, covered with a PVC ring, and then the epoxy resin and the curing agent are thoroughly mixed and injected into the PVC ring. After the resin is fully cured, the sample target is peeled off from the slide glass, and It is ground and polished, and then the sample on the target is subjected to reflected light and transmitted light photography under the microscope and cathode fluorescent photography. According to zircon cathode luminescence, reflected light and transmitted light photograph, the appropriate (interesting) dating domain of zircon is selected. The zircon is etched by 193 nm excimer laser, and the laser ablation material carries sample with He as carrier gas. The pool, mixed with argon gas, is sent to Neptune, and the dynamic zoom can be used to expand the dispersion to simultaneously receive U and Pb isotopes with large differences in mass for simultaneous in-situ simultaneous determination of zircon U-Pb isotopes. GJ-1 and 91500 were used as the external zircon age standard. Data processing was performed using the ICPMS DataCal program developed by Dr. Liu Yongsheng of China University of Geosciences (Liu et al., 2010) and the Isoplot (Ludwig, 2003) program by Kenneth RLudwig. Common lead ^{204}Pb calibration method are based on (Andersen, 2004). The Pb, U, and Th contents of the zircon samples were calculated using the SRM610 glass standard as an external standard.

During the experiment, the four ion counters (IC2, IC3, IC4, IC5) of the mass spectrometer receive ^{202}Hg , $^{204}\text{Pb}+^{204}\text{Hg}$, ^{206}Pb , ^{207}Pb signals respectively, and the three Faraday cups (L4, H2, H4) receive ^{208}Pb , ^{232}Th , ^{238}U signals respectively. The RF power is 1200 W, the integration time is 0.066 s, and the signal acquisition time is 30 s (the first 10 s is blank). Laser energy density 9~10 J/cm², ablation beam spot diameter: single point ablation mode 10 μm, curve scan ablation mode 5 μm; laser frequency: single point ablation mode 5~6 Hz, curve scan mode 12~15 Hz, scan Rate 3~4 μm/s

Table 2A. LA-ICP-MS ages data

2405-598

	Pb (ppm)	U (ppm)	Radiogenic Isotope Ratios															Isotopic Ages					
			²⁰⁶ Pb/ ²³⁸ U	1σ	err%	²⁰⁷ Pb/ ²³⁵ U	1σ	err%	²⁰⁷ Pb/ ²⁰⁶ Pb	1σ	err%	²⁰⁸ Pb/ ²³² Th	1σ	err%	²³² Th/ ²³⁸ U	1σ	err%	²⁰⁶ Pb/ ²³⁸ U	1σ	²⁰⁷ Pb/ ²³⁵ U	1σ	²⁰⁷ Pb/ ²⁰⁶ Pb	1σ
1	7	367	0.0184	0.0001	0.57	0.1230	0.0049	4.02	0.0485	0.0019	4.00	0.0062	0.0001	0.81	0.5217	0.0023	0.44	118	1	118	5	122	94
2	7	384	0.0183	0.0001	0.56	0.1218	0.0055	4.49	0.0483	0.0021	4.40	0.0063	0.0001	2.01	0.4114	0.0021	0.51	117	1	117	5	114	104
3	11	539	0.0182	0.0001	0.60	0.1221	0.0034	2.78	0.0485	0.0014	2.78	0.0061	0.0000	0.74	0.5546	0.0022	0.40	117	1	117	3	126	66
4	8	410	0.0184	0.0001	0.54	0.1208	0.0038	3.17	0.0477	0.0015	3.13	0.0059	0.0001	1.13	0.5057	0.0023	0.46	117	1	116	4	86	74
5	8	430	0.0183	0.0001	0.56	0.1222	0.0042	3.45	0.0483	0.0016	3.40	0.0054	0.0000	0.87	0.5183	0.0020	0.38	117	1	117	4	115	80
6	7	353	0.0184	0.0001	0.58	0.1226	0.0057	4.63	0.0483	0.0022	4.55	0.0056	0.0001	1.79	0.4496	0.0024	0.54	118	1	117	5	112	107
7	9	443	0.0184	0.0001	0.59	0.1224	0.0032	2.59	0.0482	0.0013	2.65	0.0048	0.0000	0.81	0.6578	0.0012	0.18	118	1	117	3	111	62
8	8	403	0.0185	0.0001	0.59	0.1222	0.0049	4.03	0.0480	0.0019	3.94	0.0047	0.0000	0.94	0.7486	0.0015	0.20	118	1	117	5	97	93
9	3	142	0.0186	0.0002	1.30	0.1236	0.0185	14.99	0.0482	0.0072	14.87	0.0042	0.0003	6.63	0.4759	0.0006	0.12	119	2	118	18	108	351
10	7	368	0.0185	0.0004	1.93	0.1225	0.0142	11.62	0.0481	0.0041	8.52	0.0047	0.0003	6.21	0.6020	0.0018	0.29	118	2	117	14	106	201
11	7	354	0.0184	0.0001	0.59	0.1236	0.0058	4.72	0.0486	0.0023	4.65	0.0038	0.0000	0.90	0.8266	0.0005	0.06	118	1	118	6	130	109
12	13	654	0.0180	0.0001	0.68	0.1217	0.0100	8.19	0.0491	0.0040	8.06	0.0035	0.0000	1.14	1.2596	0.0006	0.05	115	1	117	10	150	189
13	9	480	0.0177	0.0001	0.63	0.1215	0.0073	5.97	0.0498	0.0030	5.95	0.0037	0.0001	1.75	0.7832	0.0017	0.21	113	1	116	7	184	139
14	9	465	0.0183	0.0002	0.87	0.1237	0.0068	5.50	0.0492	0.0021	4.31	0.0038	0.0001	3.15	0.7154	0.0026	0.36	117	1	118	7	156	101
15	7	356	0.0181	0.0001	0.61	0.1206	0.0066	5.44	0.0484	0.0026	5.41	0.0033	0.0000	1.37	0.7833	0.0031	0.40	115	1	116	6	120	127
16	8	412	0.0181	0.0001	0.73	0.1211	0.0088	7.26	0.0486	0.0034	7.07	0.0034	0.0001	1.94	0.6297	0.0004	0.06	115	1	116	8	129	166
17	6	320	0.0180	0.0001	0.58	0.1213	0.0067	5.49	0.0489	0.0026	5.41	0.0048	0.0001	1.45	0.5036	0.0007	0.13	115	1	116	6	145	127
18	8	453	0.0180	0.0001	0.66	0.1205	0.0109	9.04	0.0487	0.0043	8.90	0.0041	0.0001	2.55	0.6017	0.0011	0.19	115	1	116	10	132	209
19	5	295	0.0180	0.0001	0.81	0.1226	0.0093	7.61	0.0494	0.0037	7.57	0.0046	0.0001	3.12	0.4753	0.0012	0.26	115	1	117	9	166	177
20	6	343	0.0183	0.0001	0.57	0.1216	0.0055	4.53	0.0483	0.0022	4.50	0.0056	0.0001	1.48	0.4816	0.0044	0.91	117	1	117	5	114	106
21	7	276	0.0223	0.0001	0.59	0.2035	0.0085	4.19	0.0661	0.0028	4.19	0.0080	0.0001	1.35	0.5416	0.0013	0.25	142	1	188	8	808	88

22	4	203	0.0187	0.0002	0.83	0.1214	0.0104	8.59	0.0471	0.0040	8.55	0.0082	0.0003	3.51	0.3841	0.0005	0.12	119	1	116	10	55	204
23	3	132	0.0222	0.0002	0.85	0.1999	0.0180	8.99	0.0653	0.0059	9.08	0.0105	0.0003	2.76	0.4637	0.0024	0.51	142	1	185	17	783	191
24	4	120	0.0258	0.0002	0.79	0.2297	0.0181	7.90	0.0647	0.0051	7.92	0.0087	0.0002	1.81	0.8374	0.0077	0.92	164	1	210	17	763	167
25	5	126	0.0307	0.0002	0.80	0.5991	0.0279	4.65	0.1417	0.0062	4.35	0.0235	0.0005	1.96	0.5346	0.0010	0.19	195	2	477	22	2249	75

2404-450

	Pb (ppm)	U (ppm)	Radiogenic Isotope Ratios															Isotopic Ages					
			²⁰⁶ Pb/ ²³⁸ U	1σ	err%	²⁰⁷ Pb/ ²³⁵ U	1σ	err%	²⁰⁷ Pb/ ²⁰⁶ Pb	1σ	err%	²⁰⁸ Pb/ ²³² Th	1σ	err%	²³² Th/ ²³⁸ U	1σ	err%	²⁰⁶ Pb/ ²³⁸ U	1σ	²⁰⁷ Pb/ ²³⁵ U	1σ	²⁰⁷ Pb/ ²⁰⁶ Pb	1σ
1	10	503	0.0181	0.0002	0.92	0.1163	0.0057	4.89	0.0467	0.0023	4.86	0.0050	0.0002	3.34	0.6355	0.0024	0.37	115	1	112	5	33	117
2	13	686	0.0180	0.0002	0.88	0.1363	0.0048	3.49	0.0549	0.0019	3.45	0.0048	0.0001	2.93	0.6283	0.0018	0.28	115	1	130	5	408	77
3	4	219	0.0183	0.0002	1.29	0.1264	0.0156	12.36	0.0501	0.0062	12.46	0.0046	0.0002	4.88	0.4092	0.0005	0.11	117	1	121	15	200	289
4	10	500	0.0184	0.0002	0.97	0.1404	0.0087	6.19	0.0554	0.0034	6.17	0.0060	0.0002	2.80	0.6222	0.0013	0.21	117	1	133	8	429	138
5	10	546	0.0185	0.0002	0.98	0.1408	0.0063	4.51	0.0553	0.0025	4.44	0.0051	0.0002	3.39	0.4431	0.0008	0.18	118	1	134	6	423	99
6	6	337	0.0179	0.0002	1.23	0.1444	0.0166	11.51	0.0585	0.0068	11.62	0.0047	0.0002	4.12	0.4594	0.0016	0.35	114	1	92	11	-469	307
7	8	428	0.0184	0.0002	1.00	0.1305	0.0075	5.77	0.0514	0.0030	5.75	0.0054	0.0002	3.31	0.5044	0.0011	0.22	118	1	125	7	257	132
8	9	450	0.0186	0.0002	1.04	0.1211	0.0150	12.41	0.0472	0.0058	12.37	0.0060	0.0003	4.38	0.4298	0.0009	0.22	119	1	116	14	58	295
9	13	652	0.0183	0.0002	0.95	0.1355	0.0058	4.26	0.0537	0.0021	3.96	0.0062	0.0002	3.97	0.5814	0.0022	0.38	117	1	173	7	1030	80
10	11	589	0.0182	0.0002	0.98	0.1281	0.0055	4.31	0.0511	0.0022	4.23	0.0046	0.0002	3.41	0.4875	0.0038	0.77	116	1	122	5	246	97
11	6	359	0.0178	0.0002	1.15	0.1136	0.0166	14.63	0.0462	0.0067	14.58	0.0042	0.0002	4.57	0.4722	0.0010	0.21	114	1	109	16	6	351
12	11	572	0.0182	0.0002	1.13	0.1327	0.0116	8.72	0.0530	0.0046	8.75	0.0042	0.0001	3.42	0.5056	0.0007	0.14	116	1	127	11	327	199
13	10	572	0.0183	0.0002	0.99	0.1283	0.0064	5.01	0.0510	0.0025	4.94	0.0037	0.0001	3.06	0.4506	0.0017	0.38	117	1	123	6	240	114
14	14	771	0.0182	0.0002	1.12	0.1208	0.0052	4.27	0.0482	0.0020	4.16	0.0036	0.0001	2.94	0.6377	0.0014	0.21	116	1	116	5	110	98
15	19	1044	0.0181	0.0002	0.90	0.1280	0.0043	3.32	0.0513	0.0016	3.09	0.0033	0.0001	3.05	0.7612	0.0013	0.17	116	1	122	4	255	71
16	7	411	0.0181	0.0002	1.17	0.1338	0.0083	6.21	0.0537	0.0034	6.25	0.0038	0.0002	4.26	0.4760	0.0020	0.41	116	1	128	8	358	141
17	27	1434	0.0182	0.0002	1.09	0.1524	0.0055	3.63	0.0608	0.0020	3.21	0.0066	0.0003	4.61	0.3595	0.0011	0.29	116	1	229	8	1637	60

18	9	518	0.0180	0.0002	0.96	0.1226	0.0054	4.44	0.0493	0.0022	4.40	0.0034	0.0001	3.38	0.5182	0.0016	0.31	115	1	117	5	163	103
19	7	377	0.0181	0.0002	1.12	0.1364	0.0093	6.84	0.0546	0.0037	6.75	0.0033	0.0001	4.13	0.4983	0.0025	0.51	116	1	130	9	397	151
20	14	757	0.0179	0.0002	1.15	0.1201	0.0069	5.76	0.0488	0.0028	5.72	0.0033	0.0001	3.07	0.6729	0.0017	0.26	114	1	115	7	136	134
21	11	610	0.0183	0.0002	1.02	0.1311	0.0053	4.02	0.0519	0.0020	3.94	0.0033	0.0001	3.39	0.5283	0.0012	0.23	117	1	125	5	280	90
22	7	378	0.0183	0.0002	1.08	0.1652	0.0069	4.19	0.0653	0.0027	4.08	0.0036	0.0001	3.72	0.5220	0.0021	0.41	117	1	155	6	784	86
23	10	550	0.0181	0.0002	0.98	0.1493	0.0083	5.57	0.0599	0.0033	5.53	0.0035	0.0001	3.75	0.6937	0.0008	0.12	115	1	141	8	600	120
24	9	461	0.0182	0.0002	1.07	0.1433	0.0085	5.95	0.0570	0.0034	6.02	0.0045	0.0002	4.39	0.5668	0.0019	0.34	117	1	179	11	1118	120
25	7	368	0.0178	0.0002	1.09	0.1570	0.0122	7.77	0.0638	0.0049	7.74	0.0048	0.0002	4.75	0.5654	0.0003	0.05	114	1	148	12	735	164
26	14	811	0.0181	0.0002	0.89	0.1238	0.0036	2.92	0.0496	0.0014	2.86	0.0059	0.0003	4.60	0.0927	0.0003	0.33	116	1	119	3	174	67
27	13	799	0.0180	0.0002	0.90	0.1156	0.0038	3.28	0.0467	0.0015	3.18	0.0045	0.0003	5.99	0.0871	0.0002	0.22	115	1	111	4	32	76
28	13	803	0.0179	0.0002	0.93	0.1224	0.0038	3.14	0.0496	0.0015	2.97	0.0044	0.0003	7.20	0.1117	0.0003	0.31	114	1	117	4	175	69
29	14	831	0.0182	0.0002	1.03	0.1225	0.0044	3.59	0.0488	0.0015	3.05	0.0045	0.0004	9.82	0.0999	0.0007	0.70	116	1	117	4	138	72
30	39	2306	0.0181	0.0002	0.96	0.1164	0.0025	2.18	0.0466	0.0008	1.71	0.0036	0.0004	11.02	0.1179	0.0009	0.79	116	1	112	2	27	41

⁴⁰Ar-³⁹Ar dating method

The samples are crushed in a ring mill, washed in distilled water and ethanol, and sieved when dry to -40+60mesh. Appropriate mineral grains were picked out of the bulk fraction. The samples were wrapped in aluminum foil and stacked in an irradiation capsule with similar-aged samples and neutron flux monitors (Fish Canyon Tuff sanidine (FCs), 28.201±0.046 Ma (Kuiper et al., 2008)).

The samples were irradiated in July 2017 at the McMaster Nuclear Reactor in Hamilton, Ontario, for 134 MWH in the medium flux site 8E. Analyses (n=54) of 16 neutron flux monitor positions produced errors of <0.5% in the J value.

The sample is analyzed at the Noble Gas Laboratory, Pacific Centre for Isotopic and Geochemical Research, University of British Columbia, Vancouver, BC, Canada. The mineral separates are step-heated at incrementally higher powers in the defocused beam of a 10W CO₂ laser (New Wave Research MIR10) until fused. The gas evolved from each step is analyzed by a VG5400 mass spectrometer equipped with an ion-counting electron multiplier. All measurements are corrected for total system blank, mass spectrometer sensitivity, mass discrimination, radioactive decay during and subsequent to irradiation, as well as interfering Ar from atmospheric contamination and the irradiation of Ca, Cl and K (Isotope production ratios: (⁴⁰Ar/³⁹Ar)_K=0.0302±0.00006, (³⁷Ar/³⁹Ar)_{Ca}=1416.4±0.5, (³⁶Ar/³⁹Ar)_{Ca}=0.3952±0.0004, Ca/K=1.83±0.01(³⁷Ar_{Ca}/³⁹Ar_K)).

Details of the analyses, including plateau (spectrum) and inverse correlation plots, are presented in Excel spreadsheets. Initial data entry and calculations were carried out using the software ArArCalc (Koppers, 2002). The plateau and correlation ages were calculated using Isoplot ver.3.09 (Ludwig, 2003). Errors are quoted at the 2-sigma (95% confidence) level and are propagated from all sources except mass spectrometer sensitivity and age of the flux monitor. The best statistically-justified plateau and plateau age were picked based on the following criteria:

1. Three or more contiguous steps comprising more than 60% of the ^{39}Ar ;
2. Probability of fit of the weighted mean age greater than 5%;
3. Slope of the error-weighted line through the plateau ages equals zero at 5% confidence;
4. Ages of the two outermost steps on a plateau are not significantly different from the weighted-mean plateau age (at 1.8 σ six or more steps only);
5. Outermost two steps on either side of a plateau must not have nonzero slopes with the same sign (at 1.8 σ nine or more steps only)

The “picks” for the correlation and plateau ages are arbitrary and should be considered illustrative only since they are made outside of a geological framework and may not correspond to the researcher’s criteria for plateau and correlation ages.

The Excel spreadsheet contains the data needed for recalculation using spreadsheet add-ins such as Isoplot.

Table 3A. ^{40}Ar - ^{39}Ar ages data

2404-737 muscovite

T steps (°C)	$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$(^{36}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$(^{37}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$^{40}\text{Ar}(\%)$	$F(^{40}\text{Ar}^*/^{39}\text{Ar})$	$^{39}\text{Ar}(\times 10^{-14}\text{mol})$	$^{39}\text{Ar}(\text{Cum.})(\%)$	Age(Ma)	$\pm 1\sigma(\text{Ma})$
630	31.0859	0.0079	0.0015	92.50	28.7558	2.07	5.88	118.48	0.61
710	30.7559	0.0020	0.0022	98.11	30.1755	5.17	14.69	124.13	0.60
760	30.0274	0.0007	0.0016	99.26	29.8060	4.26	12.08	122.66	0.60
810	29.7514	0.0006	0.0013	99.39	29.5704	4.74	13.47	121.72	0.60
860	29.5938	0.0008	0.0001	99.21	29.3599	4.33	12.29	120.89	0.59
910	29.8799	0.0019	0.0005	98.12	29.3189	3.88	11.01	120.72	0.59
960	30.1259	0.0033	0.0008	96.80	29.1623	2.84	8.06	120.10	0.60
1010	30.4537	0.0047	0.0014	95.46	29.0707	2.02	5.75	119.73	0.60
1100	31.2957	0.0070	0.0015	93.39	29.2273	4.37	12.41	120.36	0.61

1200	32.8318	0.0128	0.0031	88.52	29.0632	1.47	4.18	119.70	0.67
1400	65.3866	0.1312	0.1605	40.75	26.6493	0.06	0.18	110.05	3.89

1604-429 Alunite

T steps (°C)	$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$(^{36}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$(^{37}\text{Ar}/^{39}\text{Ar})_{\text{m}}$	$^{40}\text{Ar}(\%)$	$F(^{40}\text{Ar}^*/^{39}\text{Ar})$	$^{39}\text{Ar}(\times 10^{-14}\text{mol})$	$^{39}\text{Ar}(\text{Cum.})(\%)$	Age(Ma)	$\pm 1\sigma(\text{Ma})$
550	71.7868	0.1784	0.0976	26.57	19.0771	0.03	0.09	76.36	9.87
630	28.7112	0.0067	0.0108	93.06	26.7185	4.27	14.72	106.07	0.55
670	29.7342	0.0023	0.0063	97.68	29.0435	19.08	65.78	115.01	0.59
710	30.4608	0.0038	0.1124	96.33	29.3449	3.40	11.74	116.17	0.60
760	31.7078	0.0078	0.0380	92.78	29.4202	1.12	3.88	116.46	0.63
860	30.9139	0.0040	0.1583	96.20	29.7419	1.08	3.73	117.69	0.62
960	95.1441	0.2084	8.2089	35.98	34.4730	0.02	0.07	135.73	20.08

2404-313 muscovite

Power (%)	40Ar/39Ar	22σ	36Ar/39Ar	2σ	39Ar/40Ar	2σ	36Ar/40Ar	2σ	Rho	K/Ca	%40Ar rad	f 39Ar	40Ar*/39ArK	Age	2σ
2.10	73.84	1.30	0.21	0.0108	0.014	0.000	0.00287	0.00014	0.010	120.83	14.28	0.34	10.543	78.07	± 22.09
2.30	42.31	0.66	0.10	0.0049	0.024	0.000	0.00241	0.00011	0.039	72.61	28.19	1.74	11.928	88.09	± 10.27
2.70	19.05	0.49	0.014	0.0007	0.053	0.001	0.00072	0.00003	0.017	29.90	78.54	10.17	14.959	109.81	± 3.12
2.90	15.78	0.38	0.004	0.0002	0.063	0.002	0.00023	0.00001	0.346	104.48	93.11	23.97	14.690	107.90	± 2.72
3.00	15.66	0.25	0.003	0.0002	0.064	0.001	0.00019	0.00001	0.071	126.05	94.40	13.80	14.779	108.53	± 1.72
3.10	15.60	0.22	0.003	0.0001	0.064	0.001	0.00018	0.00001	0.045	129.49	94.63	17.97	14.765	108.42	± 1.53
3.30	16.39	0.24	0.005	0.0003	0.061	0.001	0.00032	0.00002	0.083	110.37	90.43	10.16	14.824	108.85	± 1.71
3.50	16.21	0.26	0.004	0.0003	0.062	0.001	0.00026	0.00002	0.078	127.42	92.33	8.13	14.967	109.86	± 1.84
3.80	16.29	0.21	0.005	0.0003	0.061	0.001	0.00032	0.00002	0.021	128.52	90.52	7.99	14.750	108.32	± 1.51

4.30	15.96	0.38	0.005	0.0003	0.063	0.001	0.00033	0.00002	0.263	92.31	90.21	5.74	14.398	105.81	± 2.67
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1604-171 Alunite

<i>Power (%)</i>	<i>40Ar/39Ar</i>	<i>2σ</i>	<i>36Ar/39Ar</i>	<i>2σ</i>	<i>39Ar/40Ar</i>	<i>2σ</i>	<i>36Ar/40Ar</i>	<i>2σ</i>	<i>Rho</i>	<i>K/Ca</i>	<i>%40Ar rad</i>	<i>f 39Ar</i>	<i>40Ar*/39ArK</i>	<i>Age</i>	<i>2σ</i>
2.10	2382.90	5232.04	7.28	16.01	0.000	0.0009	0.00305	0.00037	0.002	0.17	8.82	0.00	210.928	1140.96	± 2157.47
2.90	223.17	7.27	0.73	0.05	0.004	0.0001	0.00328	0.00020	0.011	19.11	1.95	0.22	4.342	32.31	± 96.74
3.10	52.69	0.99	0.13	0.01	0.019	0.0004	0.00250	0.00016	0.017	42.38	25.36	0.65	13.362	97.67	± 18.35
3.30	21.31	0.37	0.0195	0.0015	0.047	0.0008	0.00091	0.00007	0.034	170.11	72.71	1.57	15.497	112.80	± 3.78
3.50	17.00	0.50	0.0058	0.0003	0.059	0.0017	0.00034	0.00002	0.046	216.51	89.86	16.41	15.280	111.27	± 3.29
3.60	16.38	0.26	0.0041	0.0003	0.061	0.0010	0.00025	0.00002	0.050	236.64	92.48	16.79	15.147	110.33	± 1.83
3.70	17.60	0.27	0.0070	0.0004	0.057	0.0009	0.00040	0.00002	0.022	280.64	88.12	22.48	15.512	112.91	± 1.88
3.80	21.90	0.32	0.0210	0.0010	0.046	0.0007	0.00096	0.00005	0.016	227.33	71.34	24.19	15.625	113.70	± 2.67
3.90	18.31	0.27	0.0104	0.0006	0.055	0.0008	0.00056	0.00003	0.017	175.35	83.12	17.69	15.223	110.86	± 1.98

2412-511 Alunite

<i>Power (%)</i>	<i>40Ar/39Ar</i>	<i>2σ</i>	<i>36Ar/39Ar</i>	<i>2σ</i>	<i>39Ar/40Ar</i>	<i>2σ</i>	<i>36Ar/40Ar</i>	<i>2σ</i>	<i>Rho</i>	<i>K/Ca</i>	<i>%40Ar rad</i>	<i>f 39Ar</i>	<i>40Ar*/39ArK</i>	<i>Age</i>	<i>2σ</i>
2.20	218.94	11.73	0.53	0.05	0.0046	0.0002	0.00242	0.00017	0.010	57.21	27.75	0.05	60.750	410.82	± 71.66
2.50	20.80	0.33	0.0189	0.0012	0.048	0.001	0.00091	0.00006	0.012	187.35	72.90	0.87	15.167	111.52	± 3.09
2.60	15.99	0.39	0.0019	0.0002	0.063	0.002	0.00012	0.00001	0.023	259.61	96.37	6.73	15.406	113.22	± 2.74
2.70	15.91	0.36	0.0018	0.0001	0.063	0.001	0.00011	0.00001	0.002	285.37	96.66	11.01	15.376	113.01	± 2.49
2.80	15.75	0.23	0.0016	0.0001	0.064	0.001	0.00010	0.00001	0.012	341.48	97.04	13.10	15.281	112.33	± 1.61
2.90	15.81	0.23	0.0016	0.0001	0.063	0.001	0.00010	0.00001	0.008	252.62	97.02	12.93	15.337	112.73	± 1.61
3.00	15.82	0.24	0.0018	0.0001	0.063	0.001	0.00012	0.00001	0.006	148.76	96.53	10.89	15.269	112.25	± 1.69
3.00	15.71	0.23	0.0015	0.0001	0.064	0.001	0.00009	0.00001	0.008	429.85	97.17	12.79	15.264	112.21	± 1.59
3.10	15.76	0.44	0.0015	0.0001	0.063	0.002	0.00010	0.00001	0.017	271.56	97.07	8.00	15.302	112.48	± 3.04

3.20	15.82	0.28	0.0019	0.0001	0.063	0.001	0.00012	0.00001	0.101	171.72	96.44	15.31	15.258	112.17	± 1.95
3.20	15.92	0.25	0.0019	0.0001	0.063	0.001	0.00012	0.00001	0.046	164.63	96.47	8.32	15.358	112.89	± 1.78

3212-206 Alunite

<i>Power (%)</i>	<i>40Ar/39Ar</i>	<i>2σ</i>	<i>36Ar/39Ar</i>	<i>2σ</i>	<i>39Ar/40Ar</i>	<i>2σ</i>	<i>36Ar/40Ar</i>	<i>2σ</i>	<i>Rho</i>	<i>K/Ca</i>	<i>%40Ar rad</i>	<i>f.39Ar</i>	<i>40Ar*/39ArK</i>	<i>Age</i>	<i>2σ</i>
2.40	438.10	10.18	1.78	0.11	0.0023	0.0001	0.0041	0.0002	0.006	41.71	-14.28	1.61	62.582	-509.96	± 265.24
2.50	258.82	4.57	0.99	0.06	0.0039	0.0001	0.0038	0.0002	0.008	260.34	-7.53	1.91	19.491	-144.15	± 122.66
2.60	146.22	2.68	0.47	0.03	0.0068	0.0001	0.0032	0.0002	0.012	98.91	8.55	3.66	12.501	86.81	± 51.48
2.80	44.69	0.96	0.11	0.01	0.0224	0.0005	0.0024	0.0001	0.006	156.90	31.81	13.92	14.216	98.41	± 12.54
3.00	21.96	0.34	0.03	0.00	0.0455	0.0007	0.0012	0.0001	0.018	163.25	65.47	19.49	14.376	99.48	± 3.63
3.20	60.65	1.11	0.16	0.01	0.0165	0.0003	0.0027	0.0002	0.019	87.42	24.35	22.45	14.769	102.13	± 17.62
3.30	52.32	0.91	0.13	0.01	0.0191	0.0003	0.0025	0.0001	0.055	775.86	28.90	6.27	15.120	104.49	± 14.62
3.50	59.43	1.35	0.16	0.01	0.0168	0.0004	0.0026	0.0002	0.017	54.59	25.56	7.47	15.192	104.97	± 20.02
3.70	18.73	0.29	0.01	0.00	0.0534	0.0008	0.0008	0.0000	0.032	175.25	77.97	22.90	14.602	101.01	± 2.36
3.90	250.21	20.04	0.824	0.113	0.0040	0.0003	0.0033	0.0004	0.013	5.73	7.18	0.11	17.958	123.46	± 173.42
4.20	236.56	13.25	0.78	0.08	0.0042	0.0002	0.0033	0.0003	0.009	75.10	7.09	0.21	16.769	115.54	± 127.68

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