$^{40}Ar/^{39}Ar$ geochronology results from Zion National Park (Cogswell Point, Kolob Arch, and Kolob Reservoir quadrangles), Utah

by

New Mexico Geochronology Research Laboratory and Utah Geological Survey

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This Open-File Report makes available raw analytical data from laboratory procedures completed to determine the age of rock samples collected during geologic mapping partially supported by the Utah Geological Survey (UGS). The references listed in table 1 report the age of the samples and generally provide additional information such as the sample location, geologic setting, and significance or interpretation of the samples in the context of the area from which they were collected. This report was prepared by the New Mexico Geochronology Research Laboratory under contract to the UGS. These data are highly technical in nature and proper interpretation requires considerable training in the applicable geochronologic techniques.

Table 1. Sample numbers and locations.

Sample #	7.5' quadrangle	Latitude	Longitude	Reference
CP71900-1	Cogswell Point	37° 24' 47"	112° 58' 32"	Biek and Hylland (2006)
CP71900-6	Cogswell Point	37° 25' 18"	112° 55' 45"	Biek and Hylland (2006)
CP83100-3	Cogswell Point	37° 27' 05"	112° 59' 58"	Biek and Hylland (2006)
KA92600-1	Kolob Arch	37° 28' 43"	113° 09' 32"	Biek (in preparation)
KR7200-6	Kolob Reservoir	37° 26' 27"	113° 02' 40"	Biek (2006)
KR81200-1	Kolob Reservoir	37° 25' 21"	113° 04' 02"	Biek (2006)

Disclaimer

This Open-File release is intended as a data repository for technical analytical information gathered in support of various geologic mapping projects. The data are presented as received from the New Mexico Geochronology Research Laboratory and do not necessarily conform to UGS technical or editorial standards. Therefore, it may be premature for an individual or group to take actions based on the contents of this report.

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References to geologic reports that cite or explain samples analyzed in this report

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$^{40}Ar/^{39}Ar$ Geochronology Results from Zion National Park

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Introduction

Six basalts from Zion National Park were submitted by Robert Biek for $^{40}Ar/^{39}Ar$ age determinations. Groundmass concentrate was prepared from the basalts (sample preparation in Table 1).

$^{40}Ar/^{39}Ar$ Analytical Methods and Results

The groundmass separates were analyzed by the furnace incremental heating age spectrum method. Abbreviated analytical methods for the dated samples are given in Table 1, and details of the overall operation of the New Mexico Geochronology Research Laboratory are provided in the Appendix. The argon isotopic results are summarized in Table 1 and listed in Table 2.

Four of the Zion National Park basalts yield somewhat discordant age spectra (Figures 1a-5a). Weighted mean ages (KA92600-1, 1.03±0.06 Ma; KR81200-1, 1.05±0.05 Ma; CP71900-6, 0.34±0.03 Ma and CP71900-1, 0.37±0.02 Ma) were assigned to 100% of the ^{39}Ar released during heating. The MSWD values for these weighted mean ages are outside the 95% confidence interval (Mahon, 1996) so the errors have been increased accordingly. Sample KA92600-1 was analyzed twice. A weighted mean age was also assigned to 100% of the ^{39}Ar released during the heating of the duplicate sample (Figure 5a), this age (1.03±0.10 Ma) is within error of the age assigned to the first analysis (1.03±0.06 Ma). These samples were also evaluated with the inverse isochron technique (Figures 1b-5b) and found to have isochron ages concordant with the age spectrum ages and trapped initial $^{40}Ar/^{36}Ar$ compositions within error of the atmospheric value (295.5).

CP83100-3 groundmass concentrate yielded an age spectrum concordant across the first ~80% of the ^{39}Ar released during heating (Figure 6a). A weighted mean age of (0.76±0.02 Ma) is calculated from this portion of the age spectrum. This is followed by an increase in apparent age that is correlated with a decrease in radiogenic yield and K/Ca. Excess Ar in late degassing, high Ca phenocrysts, such as olivine and pyroxene, are a possible explanation for this increase in apparent age. Inverse isochron analysis (Figure 6b) of this sample reveals an isochron age (0.74±0.05 Ma) that agrees within error to the age spectrum (0.76±0.02 Ma) but a trapped initial $^{40}Ar/^{36}Ar$ composition (302.5±6.2) slightly higher than the atmospheric value (295.5).

KR7200-6 yielded a disturbed age spectrum (Figure 7a). The first heating step yields the anomalously old age of 24.06±2.7 Ma. The remainder of the age spectrum displays apparent ages that increase from 0.97±0.18 Ma to 81±11 Ma. This rise in ages is correlated to dropping radiogenic yields and K/Ca ratios. Inverse isochron analysis reveals that the analysis of this basalt is non-isochronous (probably indicating a non-uniform trapped $^{40}Ar/^{36}Ar$ component, Figure 7b). We suggest that the disturbed nature of the age spectrum is due to melt inclusions that have been degassed to varying degrees resulting in multi-trapped $^{40}Ar/^{36}Ar$ components.

Discussion

The weighted mean ages assigned to four of the six Zion National Park basalts are interpreted as the eruption ages of these samples (KA92600-1, 1.03±0.06 Ma; KR81200-1, 1.05±0.05 Ma; CP71900-1, 0.37±0.02 Ma; CP71900-6, 0.034±0.03 Ma). Although the isochron age (0.74±0.05 Ma) and the weighted mean age (0.076±0.02 Ma) of CP83100-3 agree within error, the $^{40}Ar/^{36}Ar$ composition (302.5±6.2) suggests that there is slight excess Ar in this sample. For this reason, we have chosen the isochron age as the preferred eruption age of CP83100-3. We were unable to assign an eruption age to KR7200-6 but suggest that the apparent age of the second heating step (0.97±0.18 Ma) represents a maximum age for the eruption of this basalt. We note that three episodes of volcanism are defined by the assigned eruption ages (Figure 8). The earliest episode occurred ~1 Ma and included flows from which KR81200-1 and KA92600-1 were collected. This was followed by an event at ~0.75 Ma (CP83100-3) and one ~0.35 Ma (CP71900-1 and CP71900-6). We cannot say to which if any of these events sample KR7200-6 belongs.

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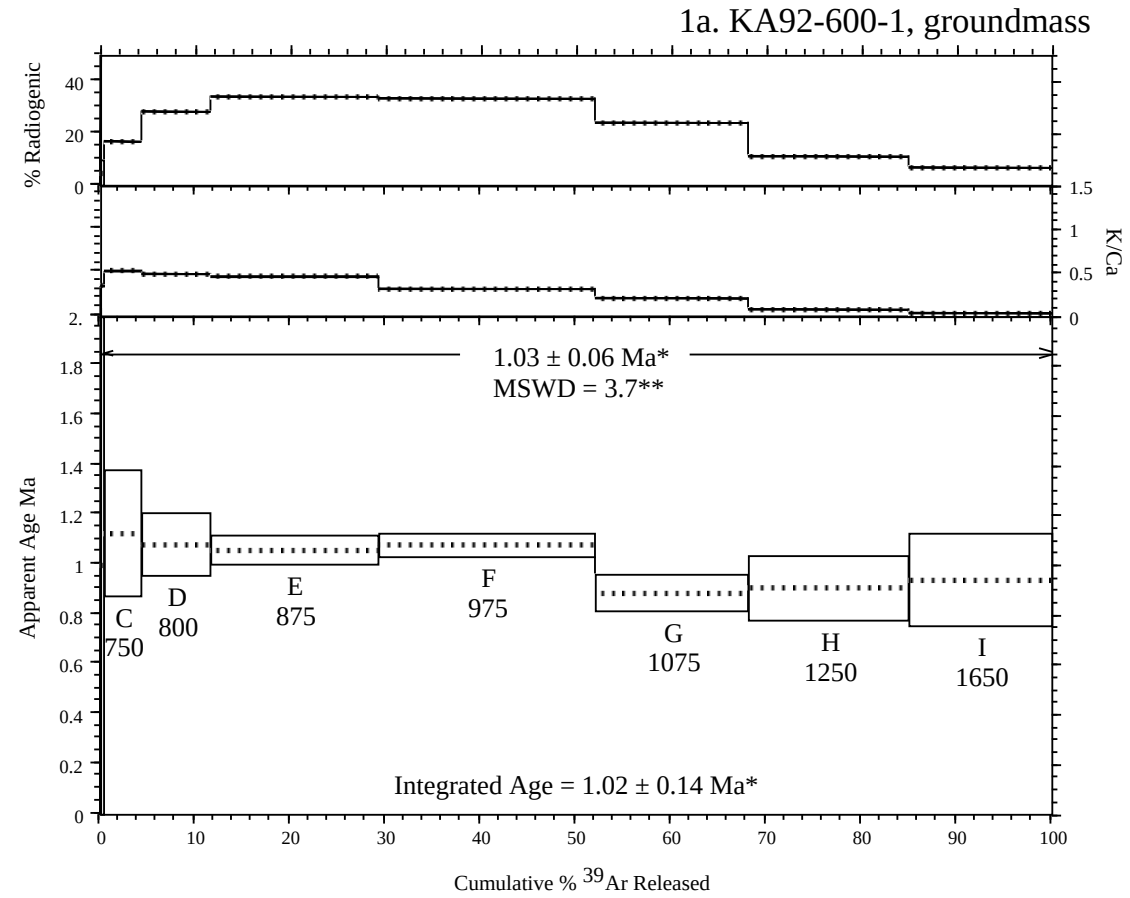


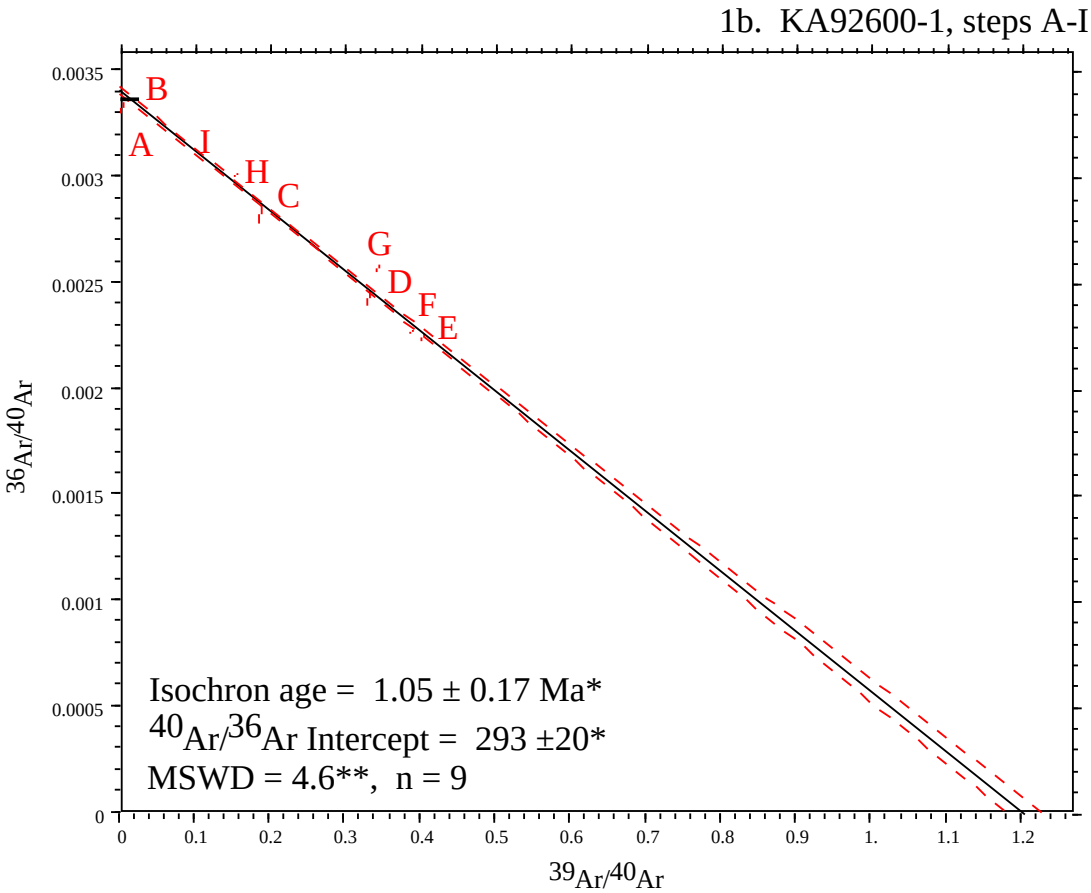
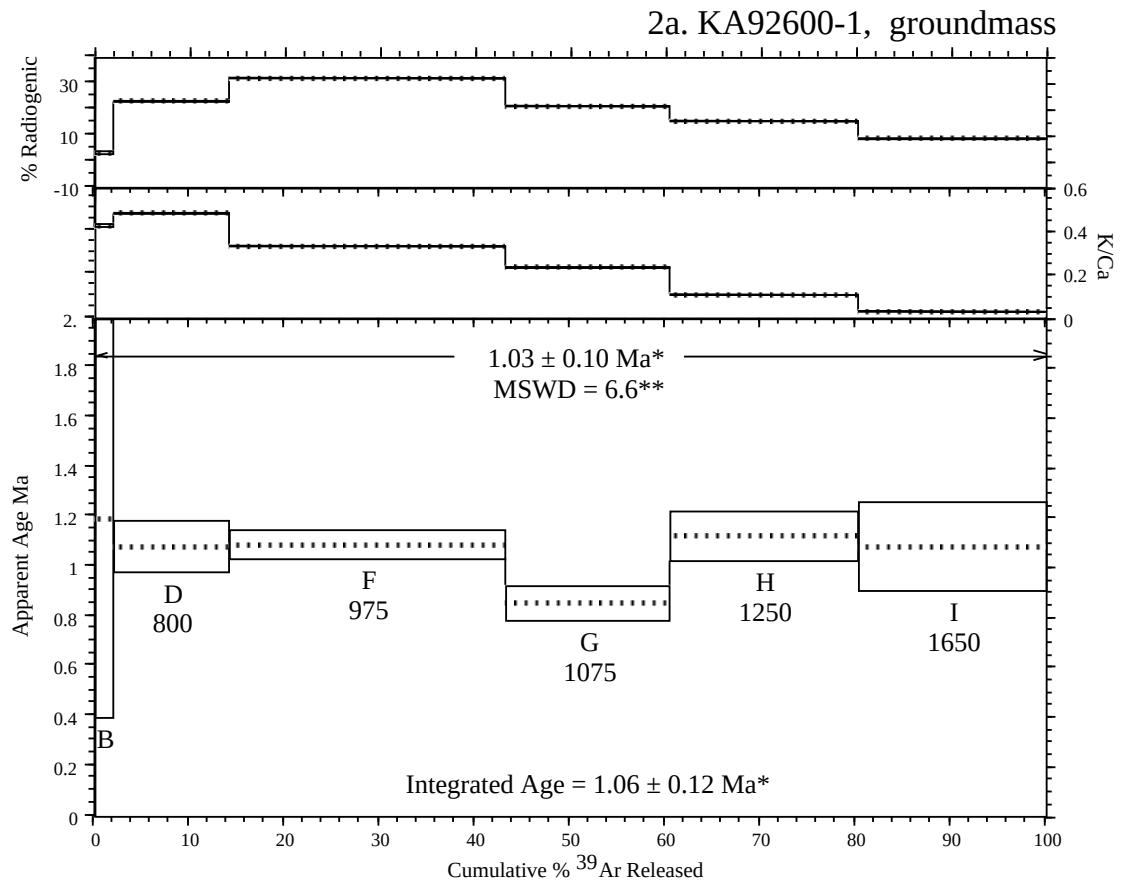


Figure 1. Age spectrum (1a) and isochron (1b) for groundmass concentrate from basalt sample KA92600-1. * 2σ **MSWD outside 95% confidence interval



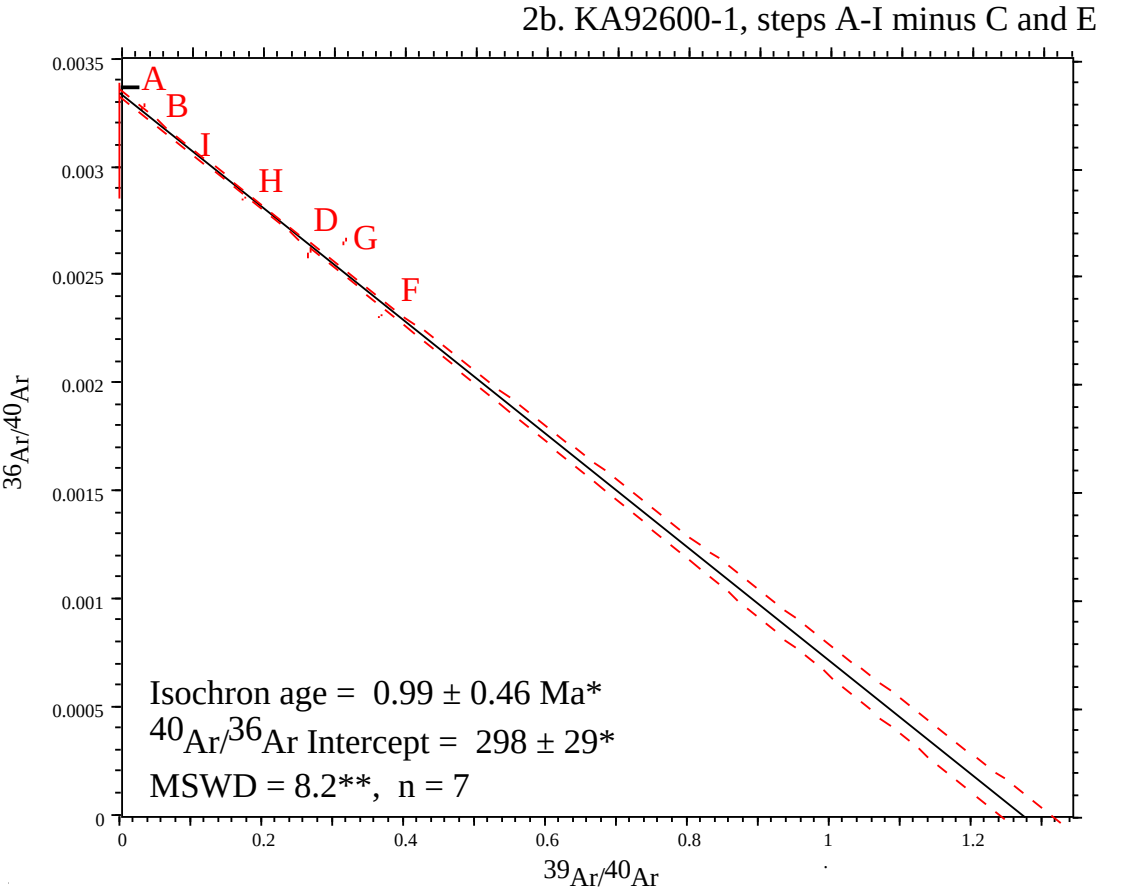
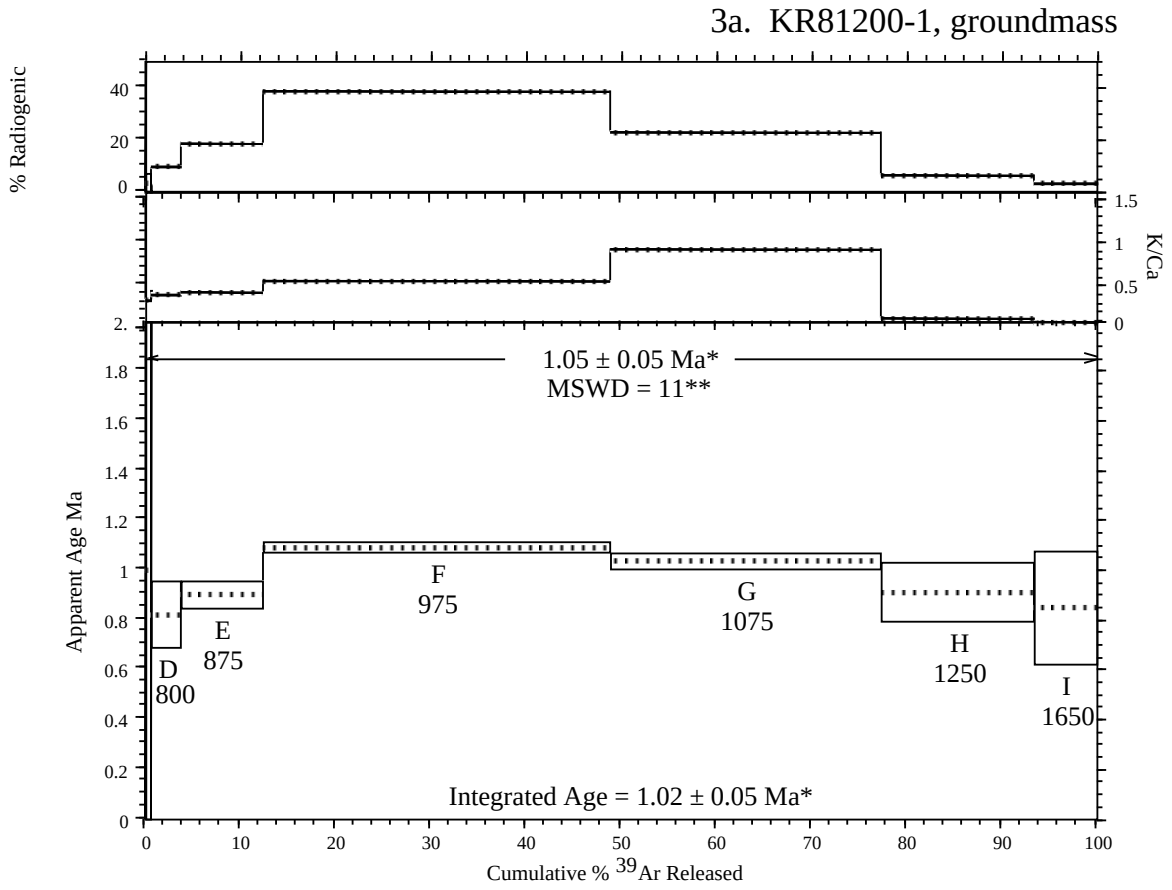


Figure 2. Age spectrum (2a) and isochron (2b) for groundmass concentrate from basalt sample KA92600-1. * 2σ **MSWD outside 95% confidence interval



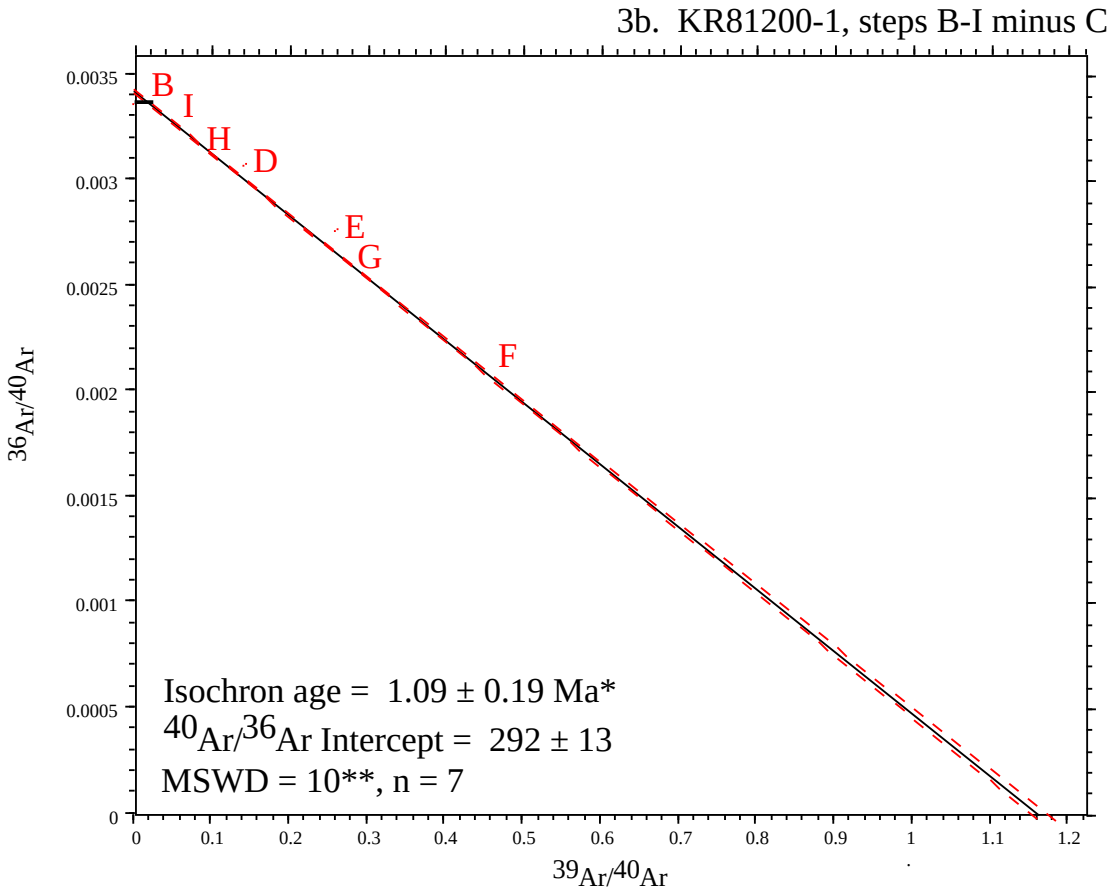


Figure 3. Age spectrum (3a) and isochron (3b) for groundmass concentrate from basalt sample KR81200-1. *2σ ** outside 95% confidence interval

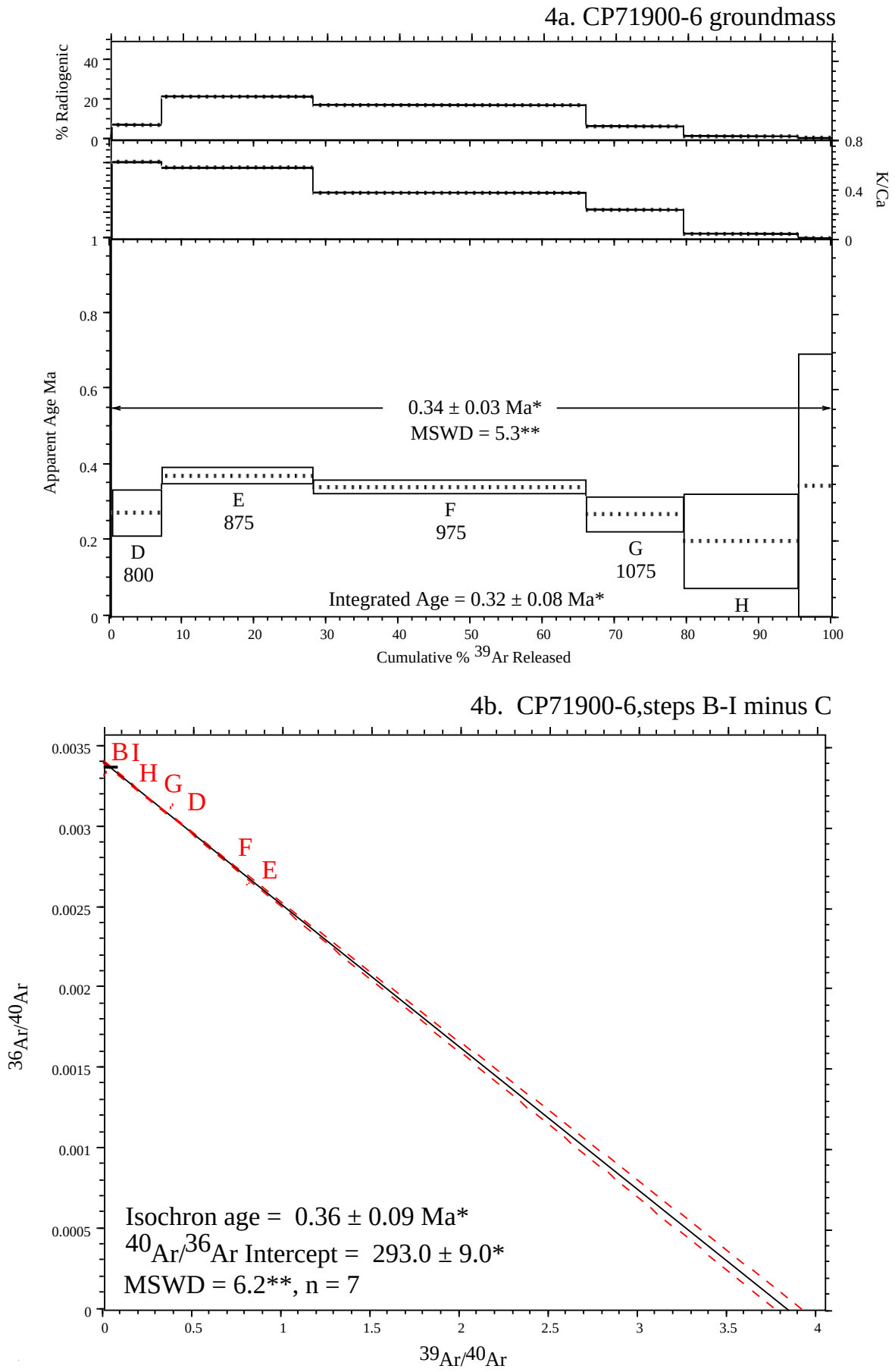
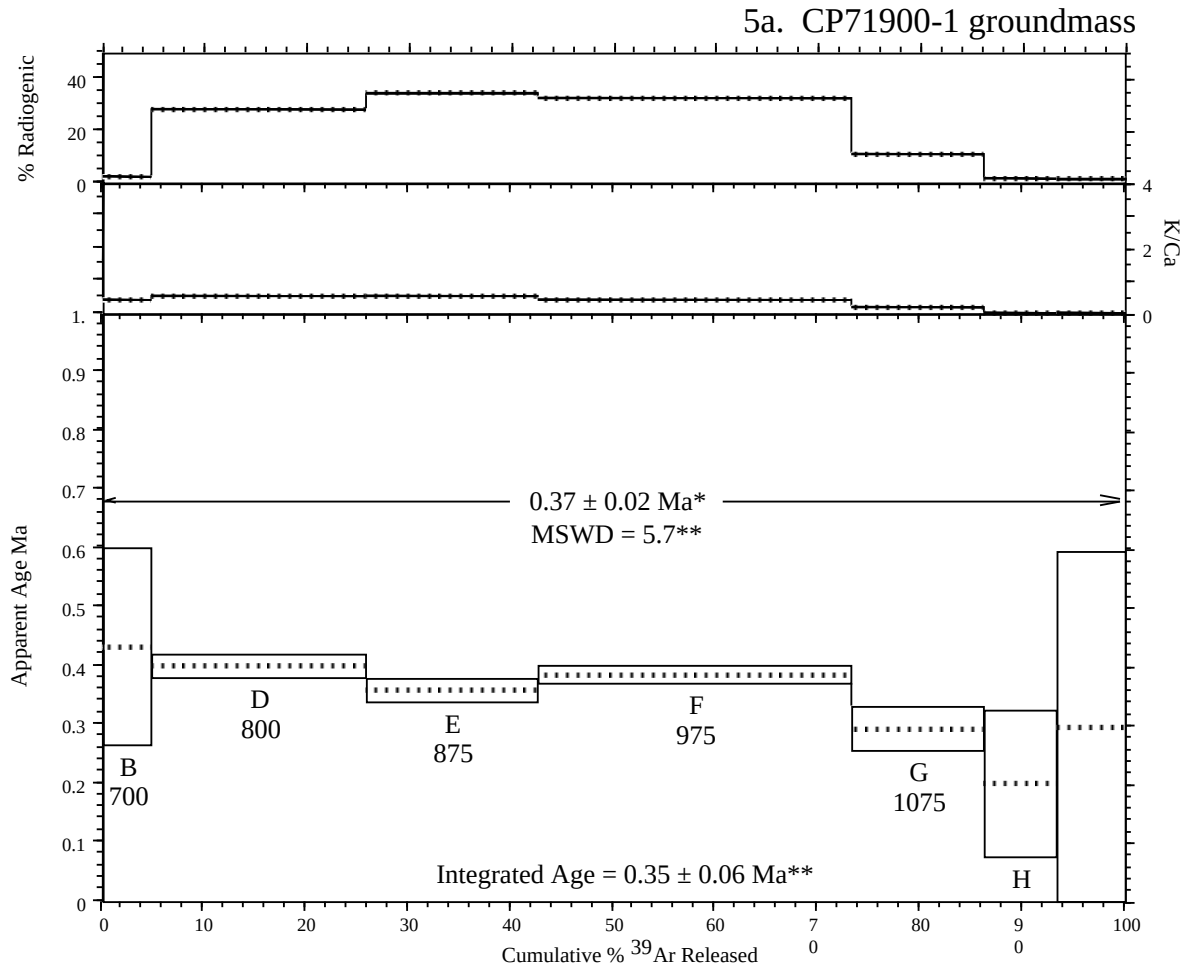


Figure 4. Age spectrum (4a) and isochron (4b) for groundmass concentrate from basalt sample CP71900-6. *2σ ** outside 95% confidence interval



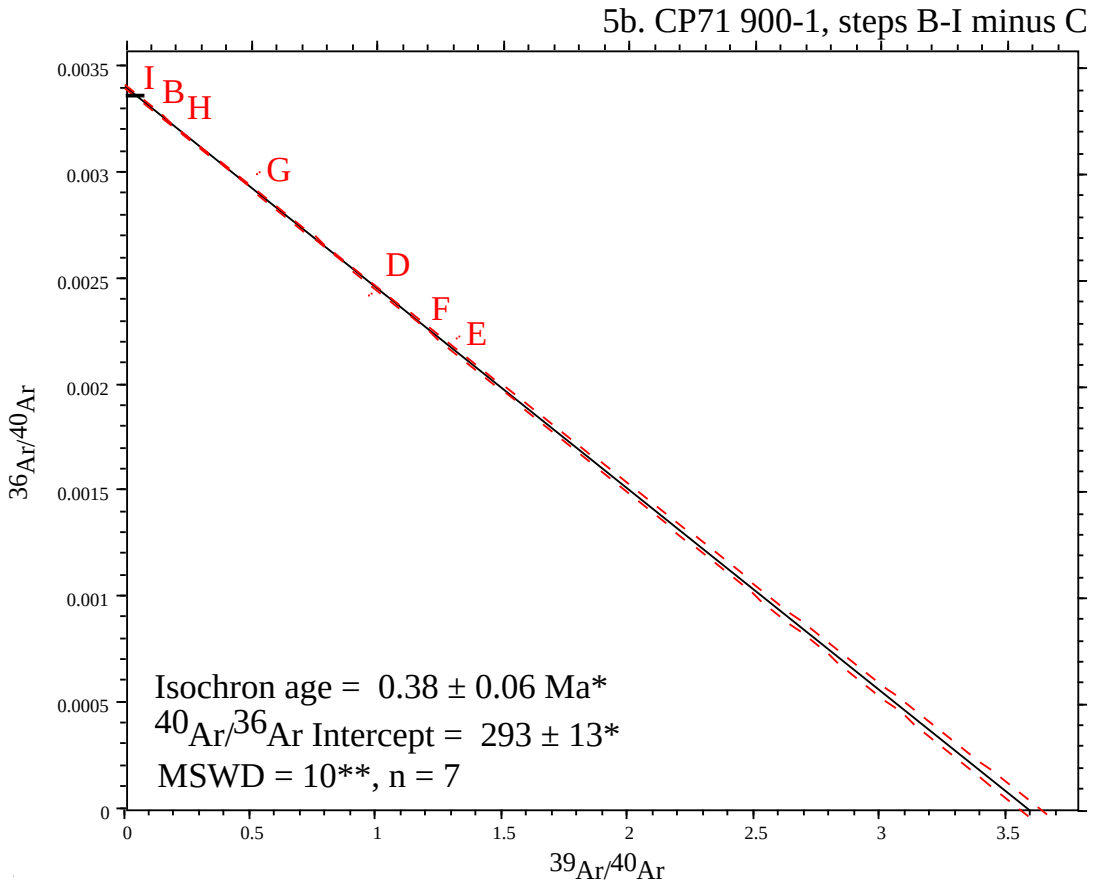
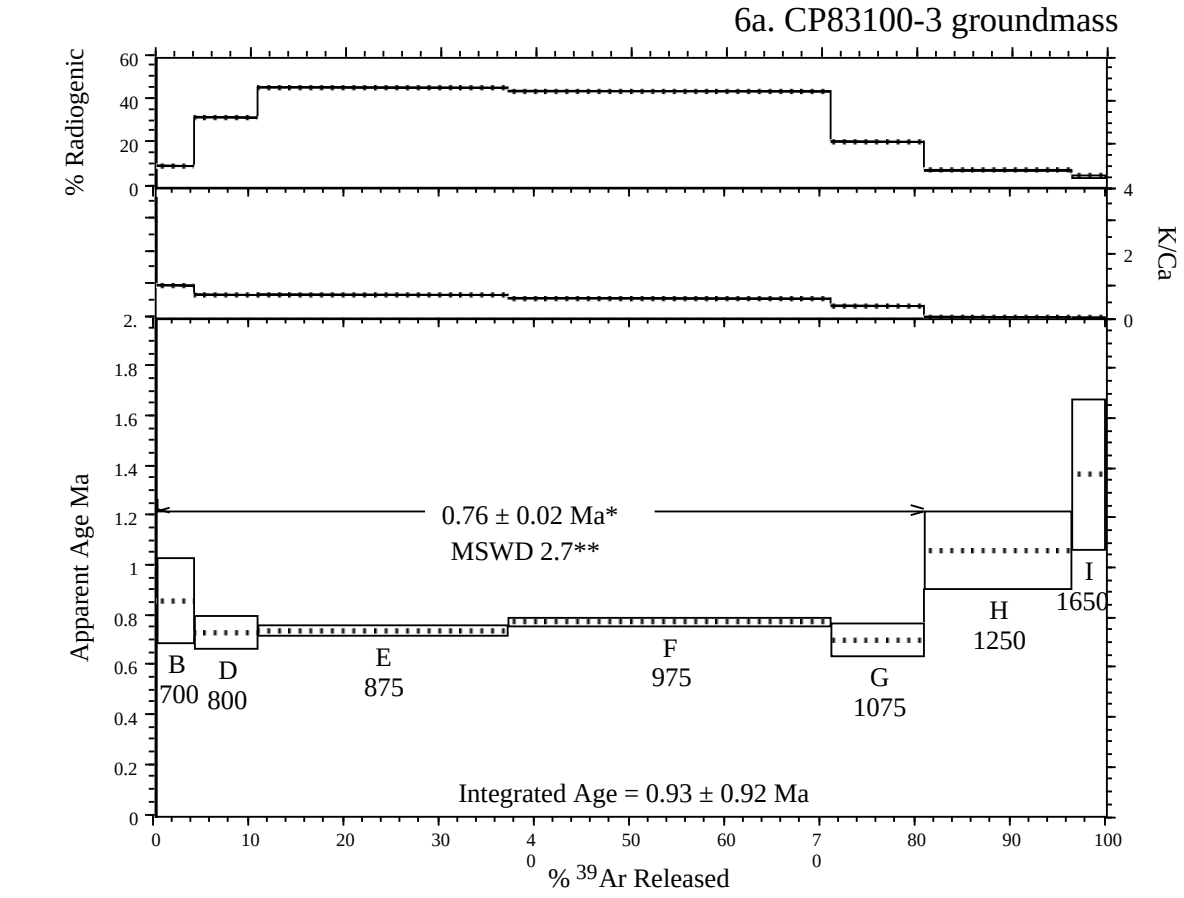


Figure 5. Age spectrum (5a) and isochron (5b) for groundmass concentrate from basalt sample CP71900-1. * 2σ ** outside 95% confidence interval



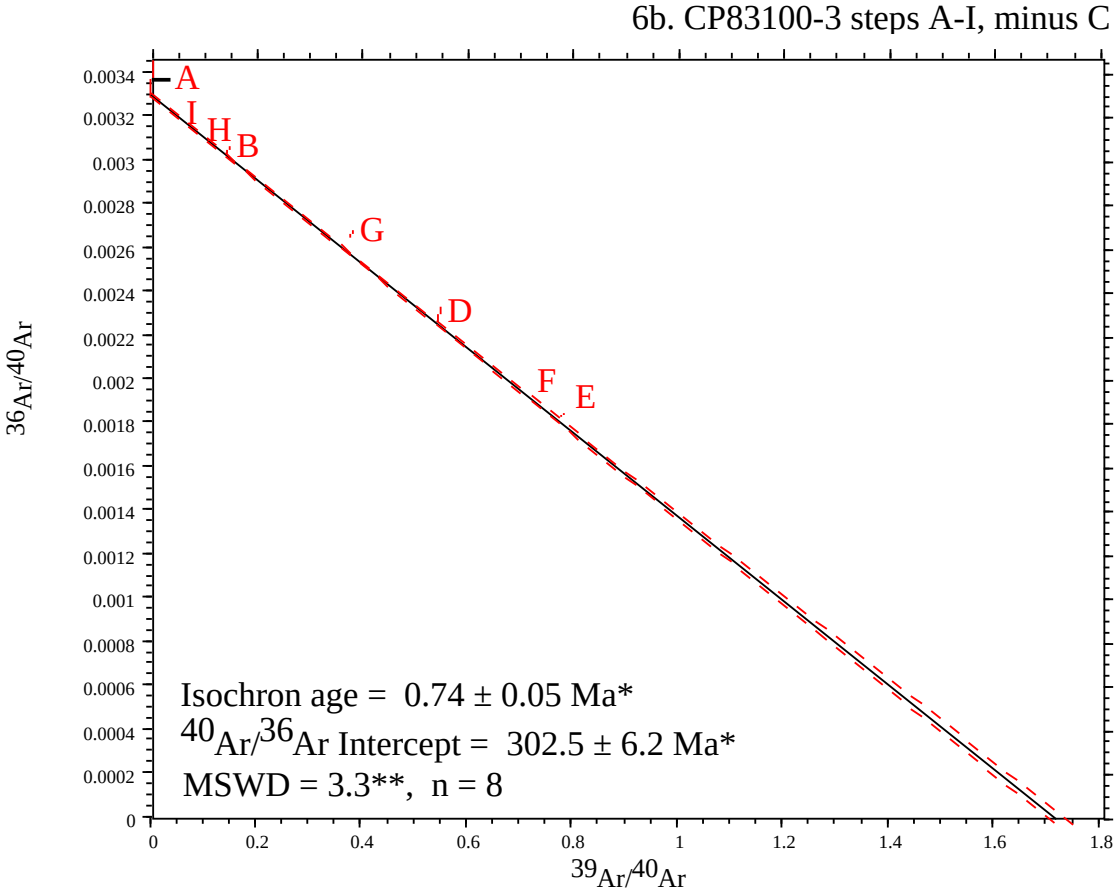
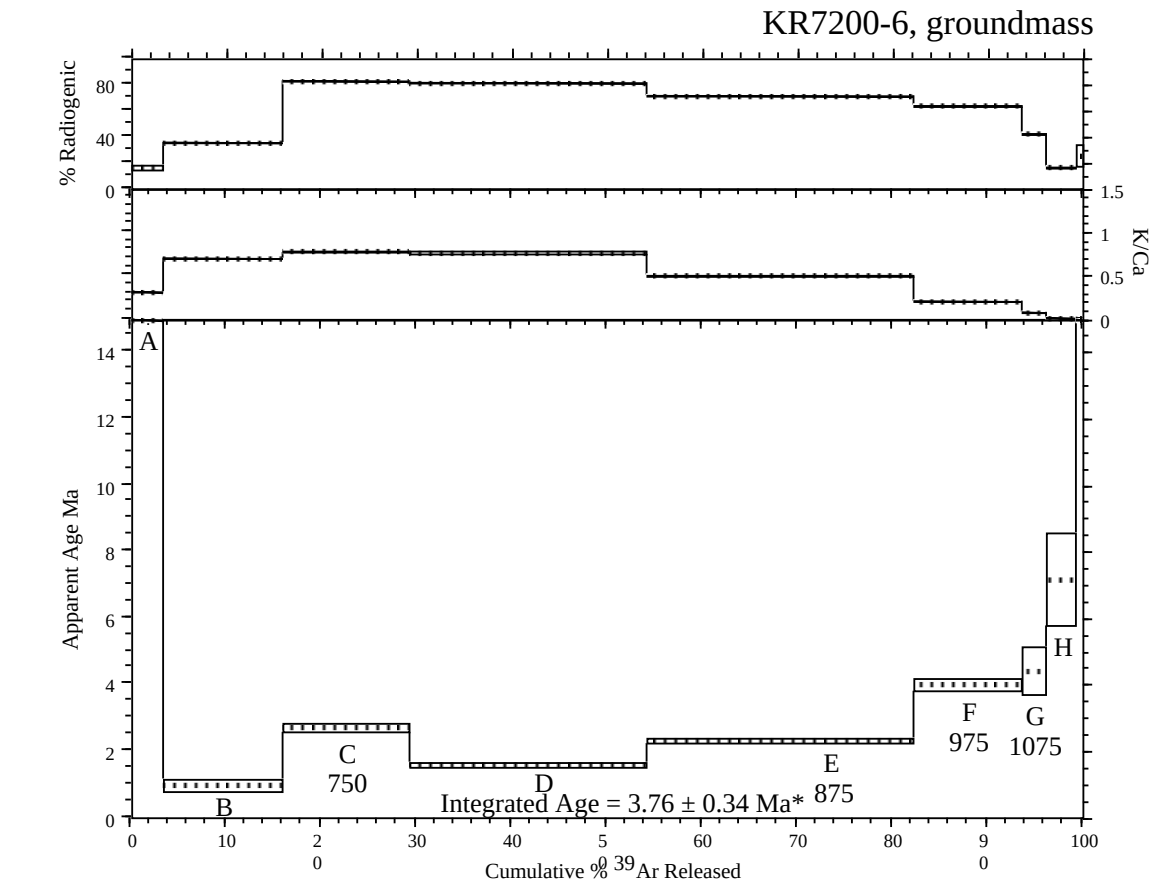


Figure 6. Age spectrum (6a) and isochron (6b) for groundmass concentrate from basalt sample CP83100-3 groundmass. * 2σ ** outside 95% confidence interval



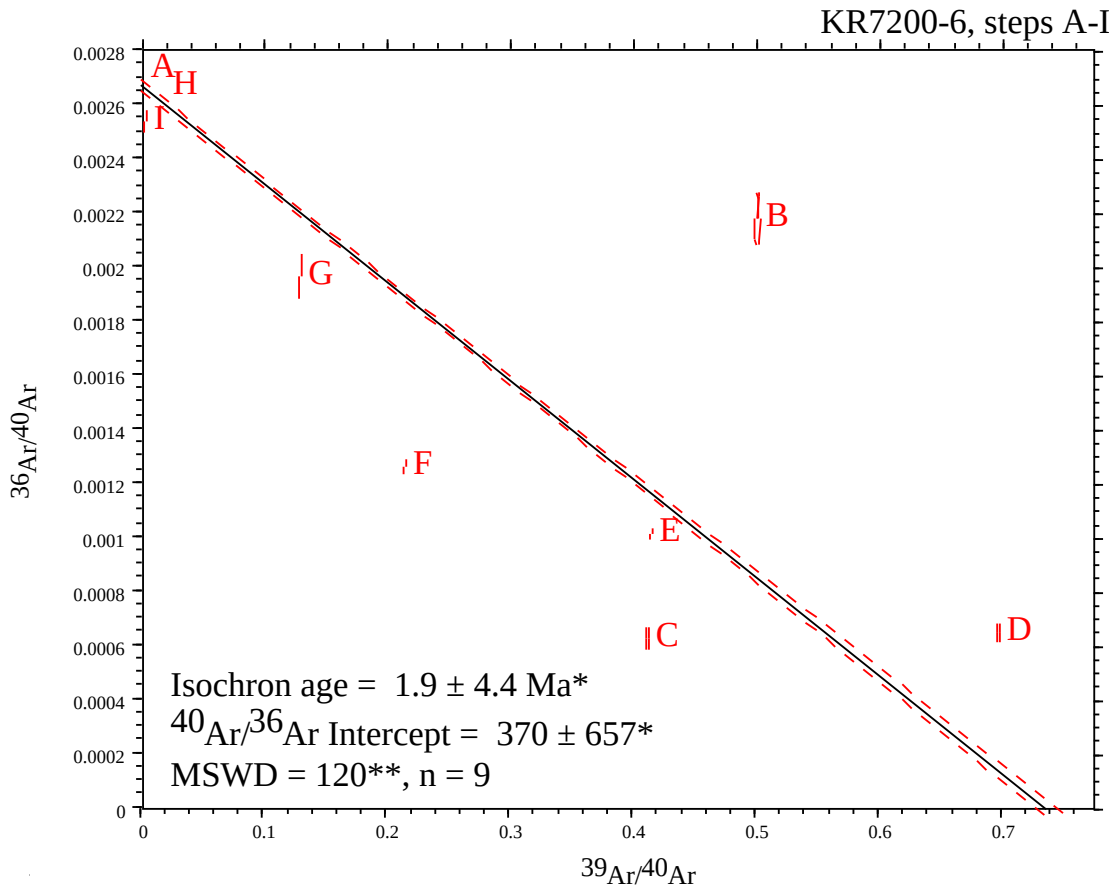


Figure 7. Age spectrum (7a) and isochron (7b) for groundmass concentrate from basalt sample KR7200-6 groundmass. * 2σ ** outside 95% confidence interval

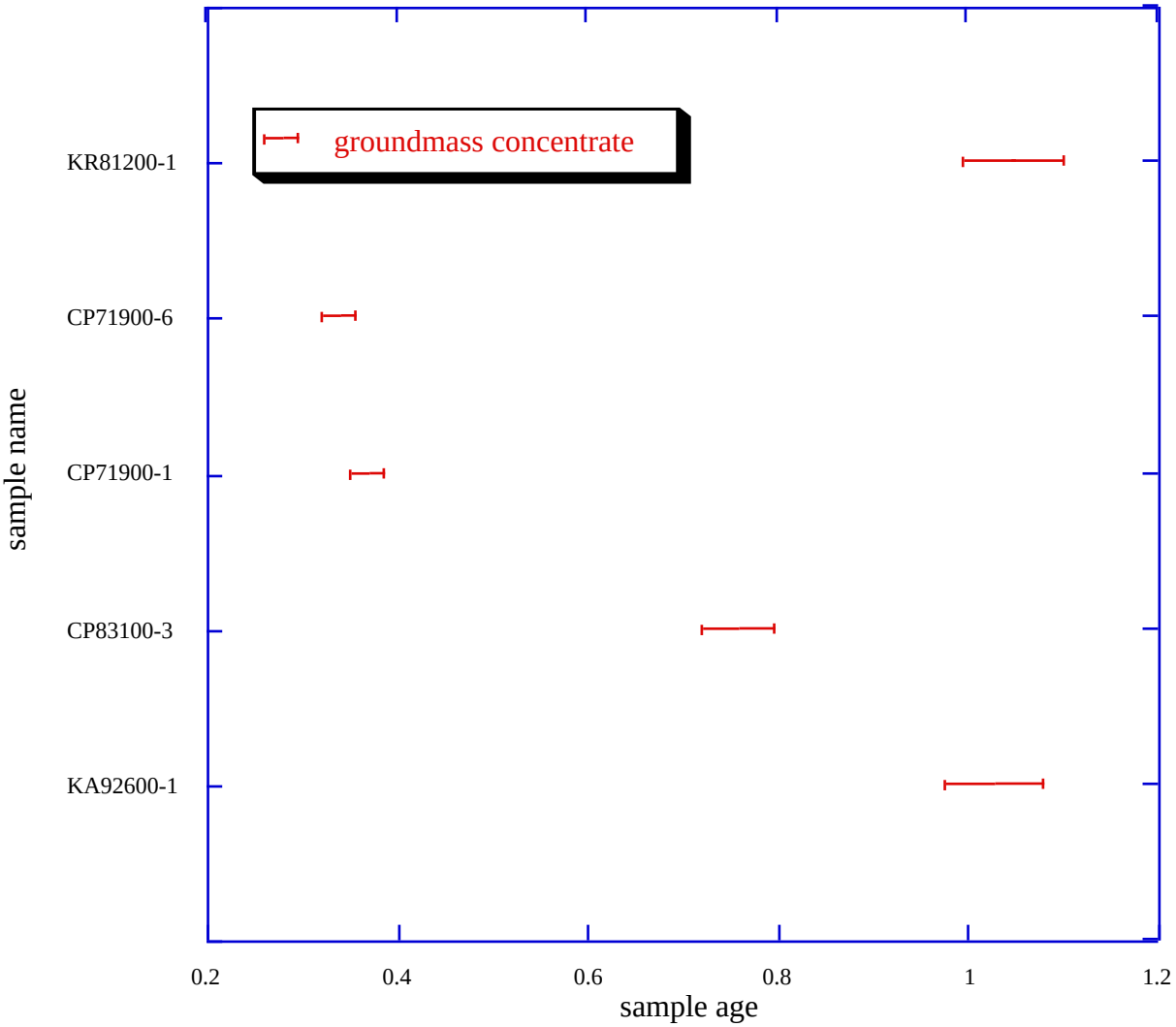


Figure 8. Eruption ages of Zion National Park basalts.

Table 1. Summary of $^{40}Ar/^{39}Ar$ results and analytical methods

Sample	Location	Lab #	Irradiation	mineral	Age	±2σ	comments
KA92600-1	Zion National Park	5226	NM-137	groundmass concentrate	1.03	0.06	plateau age
KR81200-1	Zion National Park	5229	NM-137	groundmass concentrate	1.05	0.05	plateau age
CP71900-6	Zion National Park	52263	NM-137	groundmass concentrate	0.34	0.03	plateau age
CP71900-1	Zion National Park	52263	NM-137	groundmass concentrate	0.37	0.02	plateau age
CP83100-3	Zion National Park	5227	NM-137	groundmass concentrate	0.74	0.05	isochron age
KR7200-6	Zion National Park	52317	NM-127	groundmass concentrate	0.97	0.18	maximum age

Sample preparation and irradiation:

The groundmass concentrates were prepared using standard hand-picking techniques.
The samples were loaded into a machined Al disc and irradiated for 7 hours in D-3 position, Nuclear Science Center, College Station, TX.
Neutron flux monitor Fish Canyon Tuff sanidine (FC-1). Assigned age = 27.84 Ma (Deino and Potts, 1990)
relative to Mmhb-1 at 520.4 Ma (Samson and Alexander, 1987).

Instrumentation:

Mass Analyzer Products 215-50 mass spectrometer on line with automated all-metal extraction system.
Groundmass concentrates were step-heated in Mo double-vacuum resistance furnace. Heating duration 7 minutes.
Reactive gases removed by reaction with 3 SAES GP-50 getters, 2 operated at ~450°C and
1 at 20°C. Gas also exposed to a W filiment operated at ~2000°C.

Analytical parameters:

Electron multiplier sensitivity averaged 1.33 $x10^{-16}$ moles/pA.
Total system blank and background for the laser averaged 512, 10.7, 0.05, 7.3, 2.4 x 10^{-18} moles
at masses 40, 39, 38, 37, and 36, respectively.
J-factors determined to a precision of ± 0.1% by CO_2 laser-fusion of 4 single crystals from each of 6 radial positions around the irradiation tray.
Correction factors for interfering nuclear reactions were determined using K-glass and CaF_2 and are as follows:
$(^{40}Ar/^{39}Ar)_K = 0.00020\pm0.0003$; $(^{36}Ar/^{37}Ar)_{Ca} = 0.00028\pm0.000006$; and $(^{39}Ar/^{37}Ar)_{Ca} = 0.0007\pm0.00002$.

Age calculations:

Total gas age and error calculated by weighting individual steps by the fraction of ^{39}Ar released.
Plateau age or preferred age calculated for the indicated steps by weighting each step by the inverse of the variance.
Plateau age error calculated using the method of (Taylor, 1982).
MSWD values are calculated for n-1 degrees of freedom for plateau age.
Isochron ages, $^{40}Ar/^{36}Ar_i$ and MSWD values calculated from regression results obtained by the methods of York (1969).
Decay constants and isotopic abundances after Steiger and Jäger (1977).
All final errors reported at ±2σ, unless otherwise noted.

Table 2. Argon isotopic results for furnace step-heating analyses.

ID	Temp (°C)	$^{40}Ar/^{39}Ar$	$^{37}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$ (x 10^{-3})	$^{39}Ar_K$ (x 10^{-16} mol)	K/Ca	$^{40}Ar^*$ (%)	^{39}Ar (%)	Age (Ma)	±1σ (Ma)
KA92600-1, 96.77 mg groundmass, J=0.0007047, NM-137, Lab#=52219-01										
B	700	321.0	1.478	1067.9	4.10	0.35	1.7	0.4	7.03	5.12
C	750	5.320	0.9934	15.29	39.5	0.51	16.6	4.4	1.13	0.13
D	800	3.009	1.053	7.595	71.9	0.48	28.3	11.6	1.08	0.06
E	875	2.470	1.121	5.850	177.1	0.46	33.8	29.4	1.06	0.03
F	975	2.567	1.660	6.285	225.5	0.31	33.0	52.1	1.08	0.03
G	1075	2.904	2.429	8.148	159.7	0.21	24.0	68.1	0.89	0.04
H	1250	6.375	7.690	21.32	167.3	0.066	11.1	84.9	0.91	0.06
I	1650	10.44	17.25	37.68	149.7	0.030	7.0	100.0	0.94	0.09
total gas age			n=8		994.8	0.26			1.02	0.07
plateau		MSWD = 3.7**	n=8	steps B-I	994.8	0.26		100.0	1.03	0.06 *
KA92600-1, 96.00 mg groundmass, J=0.0007033, NM-137, Lab#=5226-01										
B	700	30.99	1.197	102.0	17.9	0.43	3.1	2.0	1.20	0.41
D	800	3.731	1.067	10.04	110.3	0.48	22.8	14.2	1.08	0.05
F	975	2.716	1.554	6.719	261.1	0.33	31.6	43.2	1.09	0.03
G	1075	3.148	2.214	9.002	155.4	0.23	21.3	60.5	0.85	0.04
H	1250	5.659	4.728	17.49	178.6	0.11	15.6	80.3	1.12	0.05
I	1650	9.709	18.02	35.05	177.1	0.028	8.7	100.0	1.08	0.08
total gas age			n=6		900.4	0.23			1.06	0.06
plateau		MSWD = 6.6**	n=6	steps B-I	900.4	0.23		100.0	1.03	0.10 *
KR 81200-1, 99.60 mg groundmass, J=0.0007038, NM-137, Lab#=5229-01										
B	700	528.0	1.575	1778.8	10.6	0.32	0.5	0.6	3.23	3.89
D	800	6.970	1.320	21.77	61.1	0.39	9.3	3.8	0.82	0.07
E	875	3.851	1.236	10.98	161.8	0.41	18.4	12.4	0.90	0.03
F	975	2.226	0.9434	4.894	690.4	0.54	38.5	49.0	1.09	0.01
G	1075	3.608	0.5600	9.608	537.4	0.91	22.6	77.4	1.04	0.02
H	1250	12.03	4.035	39.43	301.8	0.13	6.0	93.4	0.91	0.06
I	1650	22.36	7.366	75.50	124.1	0.069	3.0	100.0	0.85	0.11
total gas age			n=7		1887.3	0.53			1.02	0.05
plateau		MSWD = 11**	n=7	steps B-I	1887.3	0.53		100.0	1.05	0.05*
CP71900-6, groundmass 160 mg, J=0.0007659, NM-137, Lab#=52264-01										
B	700	113.0	0.0000	376.1	5.15	-	1.6	0.3	2.55	1.40
D	800	2.592	0.8228	8.334	106.3	0.62	7.6	7.1	0.27	0.03
E	875	1.234	0.8913	3.515	330.1	0.57	21.8	28.2	0.37	0.01
F	975	1.416	1.379	4.342	592.9	0.37	17.5	66.0	0.34	0.01
G	1075	2.776	2.158	9.335	210.5	0.24	7.1	79.4	0.27	0.02
H	1250	8.821	14.32	33.38	251.5	0.036	1.6	95.5	0.20	0.06
I	1650	19.98	67.99	86.01	70.9	0.008	0.9	100.0	0.27	0.15
total gas age			n=7		1567.2	0.34			0.32	0.04
plateau		MSWD = 5.3**	n=7	steps B-I	1567.2	0.34		100.0	0.34	0.03*
CP71900-1, groundmass 156.4 mg, J=0.0007659, NM-137, Lab#=52263-01										
B	700	12.69	1.255	42.24	79.9	0.41	2.5	4.8	0.43	0.08
D	800	1.026	0.9045	2.745	353.7	0.56	28.2	25.8	0.40	0.01
E	875	0.7560	0.9161	1.937	281.5	0.56	34.3	42.6	0.36	0.01
F	975	0.8502	1.144	2.255	514.5	0.45	32.8	73.2	0.39	0.01
G	1075	1.878	2.390	6.304	218.3	0.21	11.3	86.2	0.29	0.02
H	1250	8.030	12.83	30.28	119.5	0.040	1.8	93.3	0.20	0.06
I	1650	12.35	64.64	59.32	112.0	0.008	1.4	100.0	0.25	0.10
total gas age			n=7		1679.4	0.40			0.35	0.03
plateau		MSWD = 5.7**	n=7	steps B-I	1679.4	0.40		100.0	0.37	0.02*

Table 2. Argon isotopic results for furnace step-heating analyses.

ID	Temp (°C)	$^{40}Ar/^{39}Ar$	$^{37}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$ (x 10^{-3})	$^{39}Ar_K$ (x 10^{-16} mol)	K/Ca	$^{40}Ar^*$ (%)	^{39}Ar (%)	Age (Ma)	±1σ (Ma)
CP83100-3, 92.2 mg groundmass concentrate, J=0.000704, NM-137, Lab#=5227-02										
A	*625*	*8239.3*	*0.1540*	*27797.9*	*3.05*	*3.3*	*0.3*	*0.3*	*31.57*	*298.10*
B	700	6.803	0.5113	20.86	39.3	1.00	10.0	4.1	0.86	0.09
C	750	-0.2101	0.2794	-9.3817	1.53	1.8	-1229.1	4.3	3.28	1.25
D	800	1.822	0.7290	4.400	69.4	0.70	32.0	11.0	0.74	0.03
E	875	1.282	0.6950	2.548	269.6	0.73	45.8	37.2	0.75	0.01
F	975	1.395	0.8684	2.885	349.6	0.59	44.0	71.2	0.78	0.01
G	1075	2.617	1.476	7.392	100.5	0.35	21.2	81.0	0.71	0.03
H	*1250*	*10.87*	*20.67*	*39.78*	*157.8*	*0.025*	*7.6*	*96.3*	*1.07*	*0.07*
I	*1650*	*19.74*	*23.14*	*69.68*	*37.7*	*0.022*	*5.4*	*100.0*	*1.37*	*0.14*
total gas age			n=9		1028.5	0.53			0.93	0.92
plateau		MSWD = 2.7**	n=6	steps B-G	829.9	0.64		80.7	0.76	0.02*
KR7200-6 wr, 20.90 mg groundmass, J=0.000758, NM-138, Lab#=52317-01										
A	*625*	*115.3*	*1.616*	*330.8*	*12.0*	*0.32*	*15.3*	*3.5*	*24.06*	*1.35*
B	700	1.991	0.7334	4.549	43.5	0.70	35.5	15.9	0.97	0.09
C	750	2.427	0.6678	1.697	46.8	0.76	81.6	29.4	2.71	0.06
D	800	1.434	0.6718	1.112	86.4	0.76	81.0	54.2	1.59	0.04
E	875	2.405	1.019	2.715	97.8	0.50	70.2	82.2	2.31	0.04
F	*975*	*4.657*	*2.546*	*6.575*	*40.0*	*0.20*	*62.8*	*93.7*	*4.00*	*0.09*
G	*1075*	*7.703*	*6.718*	*17.03*	*8.55*	*0.076*	*41.9*	*96.2*	*4.43*	*0.36*
H	*1250*	*32.72*	*48.64*	*107.2*	*10.8*	*0.010*	*15.5*	*99.2*	*7.16*	*0.68*
I	*1650*	*237.5*	*48.63*	*617.3*	*2.62*	*0.010*	*24.9*	*100.0*	*81.78*	*5.94*
total gas age			n=9		348.4	0.55			3.76	0.17

Notes:

Isotopic ratios corrected for blank, radioactive decay, and mass discrimination, not corrected for interferring reactions.

Individual analyses show analytical error only; plateau and total gas age errors include error in J and irradiation parameters.

n= number of heating steps

K/Ca = molar ratio calculated from reactor produced $^{39}Ar_K$ and $^{37}Ar_{Ca}$.

* 2σ error

** MSWD outside 95% confidence interval

New Mexico Bureau of Mines and Mineral Resources

Procedures of the New Mexico Geochronology Research Laboratory

For the Period June 1998 – present

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$^{40}Ar/^{39}Ar$ and K-Ar dating

Often, large bulk samples (either minerals or whole rocks) are required for K-Ar dating and even small amounts of xenocrystic, authigenic, or other non-ideal behavior can lead to inaccuracy. The K-Ar technique is susceptible to sample inhomogeneity as separate aliquots are required for the potassium and argon determinations. The need to determine absolute quantities (i.e. moles of $^{40}Ar^*$ and ^{40}K) limits the precision of the K-Ar method to approximately 1% and also, the technique provides limited potential to evaluate underlying assumptions. In the $^{40}Ar/^{39}Ar$ variant of the K-Ar technique, a sample is irradiated with fast neutrons thereby converting ^{39}K to ^{39}Ar through a (n,p) reaction. Following irradiation, the sample is either fused or incrementally heated and the gas analyzed in the same manner as in the conventional K-Ar procedure, with one exception, no argon spike need be added.

Some of the advantages of the $^{40}Ar/^{39}Ar$ method over the conventional K-Ar technique are:

1. A single analysis is conducted on one aliquot of sample thereby reducing the sample size and eliminating sample inhomogeneity.
2. Analytical error incurred in determining absolute abundances is reduced by measuring only isotopic ratios. This also eliminates the need to know the exact weight of the sample.
3. The addition of an argon spike is not necessary.
4. The sample does not need to be completely fused, but rather can be incrementally heated. The $^{40}Ar/^{39}Ar$ ratio (age) can be measured for each fraction of argon released and this allows for the generation of an age spectrum.

The age of a sample as determined with the $^{40}Ar/^{39}Ar$ method requires comparison of the measured $^{40}Ar/^{39}Ar$ ratio with that of a standard of known age. Also, several isotopes of other elements (Ca, K, Cl, Ar) produce argon during the irradiation procedure and must be corrected for. Far more in-depth details of the determination of an apparent age via the $^{40}Ar/^{39}Ar$ method are given in Dalrymple et al. (1981) and McDougall and Harrison (1988).

Analytical techniques

Sample Preparation and irradiation details

Mineral separates are obtained in various fashions depending upon the mineral of interest, rock type and grain size. In almost all cases the sample is crushed in a jaw crusher and ground in a disc grinder and then sized. The size fraction used generally corresponds to the largest size possible which will permit obtaining a pure mineral separate. Following sizing, the sample is washed and dried. For plutonic and metamorphic rocks and lavas, crystals are separated using standard heavy liquid, Franz magnetic and hand-picking techniques. For volcanic sanidine and plagioclase, the sized sample is reacted with 15% HF acid to remove glass and/or matrix and then thoroughly washed prior to heavy liquid and magnetic separation. For groundmass concentrates, rock fragments are selected which do not contain any visible phenocrysts.

The NMGRL uses either the Ford reactor at the University of Michigan or the Nuclear Science Center reactor at Texas A&M University. At the Ford reactor, the L67 position is used (unless otherwise noted) and the D-3 position is always used at the Texas A&M reactor. All of the Michigan irradiations are carried out underwater without any shielding for thermal neutrons, whereas the Texas irradiations are in a dry location which is shielded with B and Cd. Depending upon the reactor used, the mineral separates are loaded into either holes drilled into Al discs or into 6 mm I.D. quartz tubes. Various Al discs are used. For Michigan, either six hole or twelve hole, 1 cm diameter discs are used and all holes are of equal size. Samples are placed in the 0, 120 and 240° locations and standards in the 60, 180 and 300° locations for the six hole disc. For the twelve hole disc, samples are located at 30, 60, 120, 150, 210, 240, 300, and 330° and standards at 0, 90, 180 and 270 degrees. If samples are loaded into the quartz tubes, they are wrapped in Cu foil with standards interleaved at ~0.5 cm intervals. For Texas, 2.4 cm diameter discs contain either sixteen or six sample holes with smaller holes used to hold the standards. For the six hole disc, sample locations are 30, 90, 150, 210, 270 and 330° and standards are at 0, 60, 120, 180, 240 and 300°. Samples are located at 18, 36, 54, 72, 108, 126, 144, 162, 198, 216, 234, 252, 288, 306, 324, 342 degrees and standards at 0, 90, 180 and 270 degrees in the sixteen hole disc. Following sample loading into the discs, the discs are stacked, screwed together and sealed

in vacuo in either quartz (Michigan) or Pyrex (Texas) tubes.

Extraction Line and Mass Spectrometer details

The NMGRL argon extraction line has both a double vacuum Mo resistance furnace and a CO_2 laser to heat samples. The Mo furnace crucible is heated with a W heating element and the temperature is monitored with a W-Re thermocouple placed in a hole drilled into the bottom of the crucible. A one inch long Mo liner is placed in the bottom of the crucible to collect the melted samples. The furnace temperature is calibrated by either/or melting Cu foil or with an additional thermocouple inserted in the top of the furnace down to the liner. The CO_2 laser is a Synrad 10W laser equipped with a He-Ne pointing laser. The laser chamber is constructed from a 3 3/8" stainless steel conflat and the window material is ZnS. The extraction line is a two stage design. The first stage is equipped with a SAES GP-50 getter, whereas the second stage houses two SAES GP-50 getters and a tungsten filament. The first stage getter is operated at 450°C as is one of the second stage getters. The other second stage getter is operated at room temperature and the tungsten filament is operated at ~2000°C. Gases evolved from samples heated in the furnace are reacted with the first stage getter during heating. Following heating, the gas is expanded into the second stage for two minutes and then isolated from the first stage. During second stage cleaning, the first stage and furnace are pumped out. After gettering in the second stage, the gas is expanded into the mass spectrometer. Gases evolved from samples heated in the laser are expanded through a cold finger operated at -140°C and directly into the second stage. Following cleanup, the gas in the second stage and laser chamber is expanded into the mass spectrometer for analysis.

The NMGRL employs a MAP-215-50 mass spectrometer which is operated in static mode. The mass spectrometer is operated with a resolution ranging between 450 to 600 at mass 40 and isotopes are detected on a Johnston electron multiplier operated at ~2.1 kV with an overall gain of about 10,000 over the Faraday collector. Final isotopic intensities are determined by linear regression to time zero of the peak height versus time following gas introduction for each mass. Each mass intensity is corrected for mass spectrometer baseline and background and the extraction system blank.

Blanks for the furnace are generally determined at the beginning of a run while the furnace is cold and then between heating steps while the furnace is cooling. Typically, a blank is

run every three to six heating steps. Periodic furnace hot blank analysis reveals that the cold blank is equivalent to the hot blank for temperatures less than about 1300°C. Laser system blanks are generally determined between every four analyses. Mass discrimination is measured using atmospheric argon which has been dried using a Ti-sublimation pump. Typically, 10 to 15 replicate air analyses are measured to determine a mean mass discrimination value. Air pipette analyses are generally conducted 2-3 times per month, but more often when samples sensitive to the mass discrimination value are analyzed. Correction factors for interfering nuclear reactions on K and Ca are determined using K-glass and CaF_2, respectively. Typically, 3-5 individual pieces of the salt or glass are fused with the CO_2 laser and the correction factors are calculated from the weighted mean of the individual determinations.

Data acquisition, presentation and age calculation

Samples are either step-heated or fused in a single increment (total fusion). Bulk samples are often step-heated and the data are generally displayed on an age spectrum or isochron diagram. Single crystals are often analyzed by the total fusion method and the results are typically displayed on probability distribution diagrams or isochron diagrams.

The Age Spectrum Diagram

Age spectra plot apparent age of each incrementally heated gas fraction versus the cumulative % $^{39}Ar_K$ released, with steps increasing in temperature from left to right. Each apparent age is calculated assuming that the trapped argon (argon not produced by *in situ* decay of ^{40}K) has the modern day atmospheric $^{40}Ar/^{36}Ar$ value of 295.5. Additional parameters for each heating step are often plotted versus the cumulative $\%^{39}Ar_K$ released. These auxiliary parameters can aid age spectra interpretation and may include radiogenic yield (percent of ^{40}Ar which is not atmospheric), K/Ca (determined from measured Ca-derived ^{37}Ar and K-derived ^{39}Ar) and/or K/Cl (determined from measured Cl-derived ^{38}Ar and K-derived ^{39}Ar). Incremental heating analysis is often effective at revealing complex argon systematics related to excess argon, alteration, contamination, ^{39}Ar recoil, argon loss, etc. Often low-temperature heating steps have low radiogenic yields and apparent ages with relatively high errors due mainly to

loosely held, non-radiogenic argon residing on grain surfaces or along grain boundaries. An entirely or partially flat spectrum, in which apparent ages are the same within analytical error, may indicate that the sample is homogeneous with respect to K and Ar and has had a simple thermal and geological history. A drawback to the age spectrum technique is encountered when hydrous minerals such as micas and amphiboles are analyzed. These minerals are not stable in the ultra-high vacuum extraction system and thus step-heating can homogenize important details of the true ^{40}Ar distribution. In other words, a flat age spectrum may result even if a hydrous sample has a complex argon distribution.

The Isochron Diagram

Argon data can be plotted on isotope correlation diagrams to help assess the isotopic composition of Ar trapped at the time of argon closure, thereby testing the assumption that trapped argon isotopes have the composition of modern atmosphere which is implicit in age spectra. To construct an "inverse isochron" the $^{36}Ar/^{40}Ar$ ratio is plotted versus the $^{39}Ar/^{40}Ar$ ratio. A best fit line can be calculated for the data array which yields the value for the trapped argon (Y-axis intercept) and the $^{40}Ar^*/^{39}Ar_K$ value (age) from the X-axis intercept. Isochron analysis is most useful for step-heated or total fusion data which have a significant spread in radiogenic yield. For young or low K samples, the calculated apparent age can be very sensitive to the composition of the trapped argon and therefore isochron analysis should be preformed routinely on these samples (cf. Heizler and Harrison, 1988). For very old (>Mesozoic) samples or relatively old sanidines (>mid-Cenozoic) the data are often highly radiogenic and cluster near the X-axis thereby making isochron analysis of little value.

The Probability Distribution Diagram

The probability distribution diagram, which is sometimes referred to as an ideogram, is a plot of apparent age versus the summation of the normal distribution of each individual analysis (Deino and Potts, 1992). This diagram is most effective at displaying single crystal laser fusion data to assess the distribution of the population. The K/Ca, radiogenic yield, and the moles of ^{39}Ar for each analysis are also often displayed for each sample as this allows for visual ease in identifying apparent age correlations between, for instance, plagioclase contamination, signal size and/or radiogenic concentrations. The error (1σ) for each age analysis is generally shown by the horizontal lines in the moles of ^{39}Ar section. Solid symbols represent the analyses used for the weighted mean age calculation and the generation of the solid line on the ideogram, whereas open symbols represent data omitted from the age calculation. If shown, a dashed line represents the probability distribution of all of the displayed data. The diagram is most effective for displaying the form of the age distribution (i.e. gaussian, skewed, etc.) and for identifying xenocrystic or other grains which fall outside of the main population.

Error Calculations

For step-heated samples, a plateau for the age spectrum is defined by the steps indicated. The plateau age is calculated by weighting each step on the plateau by the inverse of the variance and the error is calculated by either the method of Samson and Alexander (1987) or Taylor (1982). A mean sum weighted deviates (MSWD) value is determined by dividing the Chi-squared value by n-1 degrees of freedom for the plateau ages. If the MSWD value is outside the 95% confidence window (cf. Mahon, 1996; Table 1), the plateau or preferred age error is multiplied by the square root of the MSWD.

For single crystal fusion data, a weighted mean is calculated using the inverse of the variance to weight each age determination (Taylor, 1982). Errors are calculated as described for the plateau ages above.

Isochron ages, $^{40}Ar/^{36}Ar_i$ values and MSWD values are calculated from the regression results obtained by the York (1969) method.

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