

# $^{40}\text{Ar}/^{39}\text{Ar}$ Geochronology Results from the Antelope Peak, Central East, Goldstrike, Page Ranch, and Saddle Mountain Quadrangles, Utah

by

Utah Geological Survey and  
New Mexico Geochronology Research Laboratory

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**OPEN-FILE REPORT 508**  
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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 funded or partially supported by the Utah Geological Survey (UGS). The references listed in table 1 report the age of the samples and provide additional information such as the sample location, geologic setting, and significance or interpretation of the samples in the context of the area in 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.

<b>Table 1. Sample numbers and locations.</b>				
<b>Sample #</b>	<b>7.5' quadrangle</b>	<b>Latitude (N)</b>	<b>Longitude (W)</b>	<b>Reference</b>
SMQ-4	Saddle Mountain	37° 19.191'	113° 33.264'	Biek and others (2007)
CEQ-18	Central East	37° 24.217'	113° 36.791'	Biek and others (2007)
CEQ-14	Central East	37° 24.483'	113° 31.750'	Biek and others (2007)
SMQ-1	Saddle Mountain	37° 16.450'	113° 36.355'	Biek and others (2007)
CEQ-8	Central East	37° 24.194'	113° 31.069'	Biek and others (2007)
EMD046	Central East	37° 26.361'	113° 35.382'	Biek and others (2007)
MM-1	Goldstrike	37° 24.802'	113° 59.169'	Biek and others (2007)
CEQ-22	Central East	37° 29.795'	113° 32.915'	Biek and others (2007)
IM-1	Page Ranch	37° 37.124'	113° 22.636'	Rowley and others (2006)
PP-2	Page Ranch	37° 31.444'	113° 28.380'	Rowley and others (2006)
TB-1	Antelope Peak	37° 51.032'	113° 29.008'	Rowley and others (2006)
Latitude and longitude in NAD83.				

## **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**

Biek, R.F., Rowley, P.D., Hacker, D.B., Hayden, J.M., Willis, G.C., Hintze, L.F., Anderson, R.E., and Brown, K.D., 2007, Interim geologic map of the St. George 30' x 60' quadrangle, Washington and Iron Counties, Utah: UGS Open-File Report 478, 70 p., 2 plates., scale 1:100,000.

Rowley, P.D., Williams, V.S., Vice, G.S., Maxwell, D.J., Hacker, D.B., Snee, L.W., and Mackin, J.H., 2006, Interim geologic map of the Cedar City 30' x 60' quadrangle, Iron and Washington Counties, Utah: Utah Geological Survey Open-File Report 476DM, scale 1:100,000.

# $^{40}\text{Ar}/^{39}\text{Ar}$ Geochronology Results From the Pine Valley Mountains

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## Introduction

Eleven samples from the Pine Valley Mountains in Utah were submitted for dating by the Utah Geological Survey. These samples were collected by David Hacker of Kent State University. The sample numbers, minerals dated and age assignments are briefly summarized in Table 1.

**Table 1. Brief summary of results.**

Sample	Phase	Age $\pm 2\sigma$ (Ma)	Comments
SMQ-4	Groundmass concentrate	0.45 $\pm$ 0.86	integrated age
CEQ-18	Groundmass concentrate	0.61 $\pm$ 0.04	plateau age
CEQ-14	Groundmass Concentrate	0.67 $\pm$ 0.07	plateau age
SMQ-1	Groundmass concentrate	1.02 $\pm$ 0.36	integrated age
CEQ-8	Groundmass concentrate	1.08 $\pm$ 0.13	plateau age
EMD046	Biotite	2.03 $\pm$ 0.76	isochron age
MM-1	Plagioclase concentrate	12.1 $\pm$ 1.9	integrated age
CEQ-22	Groundmass concentrate	17.39 $\pm$ 0.12	plateau age
IM-1	Plagioclase concentrate	21.85 $\pm$ 0.31	isochron age
PP-2	Biotite	21.96 $\pm$ 0.11	plateau age
TB-1	Biotite	22.51 $\pm$ 0.09	plateau age

## <sup>40</sup>Ar/<sup>39</sup>Ar Analytical Methods and Results

Samples being prepared as groundmass concentrates were crushed and cleaned with dilute hydrochloric acid while those being prepared as biotite and plagioclase concentrates were crushed and cleaned with only water. Biotite and plagioclase were separated from the crushed rock with standard heavy liquid, magnetic separator and handpicking techniques. The mineral separates were loaded into aluminum discs and irradiated for 7 hours at the Nuclear Science Center in College Station, Texas.

The prepared separates were analyzed with the furnace incremental heating age spectrum method. Abbreviated analytical methods for the dated samples are given in Table 2, 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 Tables 1 and 2 and listed in Tables 3-6.

**CEQ-8**      Weighted Mean Age=1.08±0.13 Ma      n/n<sub>total</sub>=9/9      MSWD=2.0

Groundmass concentrate from sample CEQ-8 yielded a well-behaved age spectrum and a weighted mean age of 1.08±0.13 Ma is calculated from all of the heating steps (Figure 1a). The radiogenic yields rise consistently over 81% of the age spectrum to a high of 7.1% and then decline over the remainder of the age spectrum. The K/Ca values reveal an overall decline from 0.97 to 0.03. The data was evaluated with the inverse isochron technique and was found to have a <sup>40</sup>Ar/<sup>36</sup>Ar ratio of 294.9±3.6, within error of the atmospheric ratio of 295.5 (Figure 1b). The isochron age (1.12±0.27 Ma) agrees within error to the age calculated from the age spectrum.

**CEQ-18**      Weighted Mean Age=0.61±0.04 Ma      n/n<sub>total</sub>=9/9      MSWD=1.0

Groundmass concentrate from sample CEQ-18 yielded a well-behaved age spectrum and a weighted mean age of 0.61±0.04 Ma is calculated from all of the heating steps. The radiogenic yields rise to a high of 28.6% radiogenic with ~58% of the <sup>39</sup>Ar released. The K/Ca values reveal an overall decline from 0.47 to 0.01. Inverse isochron analysis of the data revealed a <sup>40</sup>Ar/<sup>36</sup>Ar intercept of 293.0±2.6 with an isochron age of 0.64±0.04 Ma (Figure 2b).

**TB-1**                      Weighted Mean Age=22.51±0.09 Ma                      n/n<sub>total</sub>=10/10                      MSWD=6.4

TB-1 biotite yielded a slightly disturbed age spectrum (Figure 3a). A weighted mean age of 22.51±0.09 Ma was calculated from 100% of the <sup>39</sup>Ar released. The radiogenic yields rise to a high of 97.1% with 52.7% of the <sup>39</sup>Ar released and then remain fairly constant. K/Ca values rise over one third of the age spectrum to a high value 16.6 and oscillate between this value and 1.4 over the remainder of the age spectrum. Inverse isochron analysis of steps A-J reveals a <sup>40</sup>Ar/<sup>36</sup>Ar intercept of 292.5±6.5, slightly lower than the atmospheric value (Figure 3b). The calculated isochron age of 22.53±0.10 Ma agrees within error to the weighted mean from the age spectrum.

**PP-2**                      Weighted Mean Age=21.96±0.11 Ma                      n/n<sub>total</sub>=6/10                      MSWD=4.6

PP-2 biotite yielded a somewhat disturbed age spectrum with increasing apparent ages over the initial 26.2% of <sup>39</sup>Ar released during heating (Figure 4a). The remaining 73.8% of the <sup>39</sup>Ar released is used to calculate a weighted mean age of 21.96±0.11 Ma. The radiogenic yields and K/Ca values increase over 52.7% of the <sup>39</sup>Ar released, to 83% and a value of 38.0, respectively. The radiogenic yields are somewhat oscillatory for the remainder of the heating steps, while the K/Ca values reveal an overall decrease to a value of ~4. Inverse isochron analysis of steps D-J reveals a <sup>40</sup>Ar/<sup>36</sup>Ar intercept of 287±20 and an isochron age of 22.13±0.35 Ma (Figure 4b).

**CEQ-22**                      Weighted Mean Age=17.39±0.12 Ma                      n/n<sub>total</sub>=5/9                      MSWD=0.73

CEQ-22 groundmass concentrate yielded a somewhat disturbed age spectrum that reveals an overall increase in apparent ages from about 13 to 18 Ma (Figure 5a). A weighted mean age of 17.39±0.12 Ma is calculated from the mid 70.7% of the <sup>39</sup>Ar released. The radiogenic yields rise to 51.1% radiogenic over the initial 52.1% of the <sup>39</sup>Ar released and then drop to 23.6% radiogenic over the remainder of the age spectrum. The K/Ca values are fairly consistent at ~0.74-0.88 until the final 21.6% of the <sup>39</sup>Ar released where they drop to 0.27. Inverse isochron analysis of steps D-H reveals a

$^{40}\text{Ar}/^{36}\text{Ar}$  intercept of  $294.6\pm 5.1$  with an isochron age of  $17.46\pm 0.36$  Ma that agrees within error to the age calculated from the age spectrum (Figure 5b).

**CEQ-14**      Weighted Mean Age= $0.67\pm 0.08$  Ma       $n/n_{\text{total}}=8/9$       MSWD=3.1

CEQ-14 groundmass concentrate yielded a slightly disturbed age spectrum with an anomalously young apparent age ( $-4.11\pm 1.51$ ) in the initial heating step (Figure 6a). A weighted mean age of  $0.67\pm 0.08$  Ma is calculated from the remaining steps. The radiogenic yields increase to a high of 31.5% over the initial 66.3% and then decrease to 0.9% over the remainder of the age spectrum. The K/Ca values reveal a slight increase over the majority of the  $^{39}\text{Ar}$  released ( $\sim 0.2$  to  $0.3$ ) and then decrease to 0.007 over the final  $\sim 10\%$  of the  $^{39}\text{Ar}$  released. Inverse isochron analysis of steps B-I reveals a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept of  $295.6\pm 5.7$  and an isochron age of  $0.67\pm 0.08$  Ma (Figure 6b).

**EMD046**      Isochron Age= $2.03\pm 0.76$  Ma       $n/n_{\text{total}}=8/11$       MSWD=4.9

EMD046 biotite yielded a slightly disturbed, saddle-shaped age spectrum (Figure 7a). A weighted mean age of  $2.09\pm 0.22$  Ma is calculated for steps B-G that represent the overall youngest steps of the saddle. The radiogenic yields are consistently low ( $<9\%$ ). The K/Ca values are fairly consistent at  $\sim 11$  until the final  $\sim 6\%$  of the  $^{39}\text{Ar}$  released where the K/Ca values drop to  $<1$ . When evaluated with the inverse isochron technique, the data points cluster near the  $^{36}\text{Ar}/^{40}\text{Ar}$  axis, due to the consistent, low radiogenic yields (Figure 7b). Steps A-H yield an isochron age of  $2.03\pm 0.76$  Ma with a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept of  $296.8\pm 5.4$ .

**IM-1**      Isochron Age= $21.85\pm 0.31$  Ma       $n/n_{\text{total}}=7/12$       MSWD=6.5

IM-1 plagioclase concentrate yielded a disturbed saddle-shaped age spectrum (Figure 8a). Apparent ages decrease from 26.73 Ma to 21.73 Ma over the initial 41.7% of the  $^{39}\text{Ar}$  released and then reveal an overall increase to 24.38 Ma over the remainder of the heating steps. The radiogenic yields and K/Ca values reveal a rough inverse correlation to the apparent ages with the radiogenic yields varying from 5.9% to 76.5%

and the K/Ca values varying between 0.59 and 3.5. Steps A-G yielded an isochron age of  $21.85 \pm 0.31$  Ma with a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept of  $298.8 \pm 5.1$  (Figure 8b).

**SMQ-1**      Integrated Age= $1.02 \pm 0.36$  Ma

SMQ-1 groundmass concentrate yielded a slightly hump-shaped age spectrum (Figure 9a). The apparent ages increase slightly from -0.81 Ma to 1.28 Ma over the initial 59.8% of the  $^{39}\text{Ar}$  released and then decrease to 0.71 Ma until the final 3.3% of the age spectrum where the apparent age increases to 2.15 Ma. A weighted mean age of  $1.20 \pm 0.08$  Ma is calculated from the initial 70.2% of the  $^{39}\text{Ar}$  released. The radiogenic yields rise from -0.1% to 22.5% over the initial 59.8% of the age spectrum and then decrease to 1.4% radiogenic over the remainder of the gas released. The K/Ca values are fairly consistent at  $\sim 0.5$ -0.6 until 59.8% of the  $^{39}\text{Ar}$  was released after which the K/Ca values decrease to 0.054. Inverse isochron analysis of steps A-I (Figure 9b) yields an isochron age,  $1.20 \pm 0.17$  Ma, that agrees with both the weighted mean age calculated for the age spectrum and the integrated age ( $1.02 \pm 0.36$  Ma).

**MM-1**      Integrated Age= $12.1 \pm 1.9$  Ma

MM-1 plagioclase concentrate yielded a disturbed saddle-shaped age spectrum (Figure 10a). The apparent ages decrease from 19.96 Ma to 9.89 Ma over the initial 55.0% of the  $^{39}\text{Ar}$  released and then increase throughout the remainder of the age spectrum to 30.34 Ma. A weighted mean age has not been calculated. The integrated age for this sample is  $12.1 \pm 1.9$  Ma. Both the radiogenic yields and K/Ca values are oscillatory. The radiogenic yields vary from 0.1% to 14.2% and while the K/Ca values vary from 0.44 to 14.2. An isochron age of  $12.22 \pm 1.87$  Ma has been calculated from steps A-J (Figure 10b). This isochron age has a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept of  $295.5 \pm 3.7$ .

**SMQ-4**      Integrated Age= $0.45 \pm 0.86$  Ma

SMQ-4 groundmass concentrate yielded a somewhat disturbed saddle-shaped age spectrum (Figure 11a). Five of the nine heating steps yield negative apparent ages. A weighted mean age has not been calculated. The integrated age is  $0.46 \pm 0.89$  Ma.

Radiogenic yields are extremely low (<3%) with 5 of the heating steps yielding values below zero. The K/Ca values decrease consistently throughout the heating schedule (0.98-0.20) until the final heating step reveals a slight increase (0.23). Inverse isochron analysis of points A-I reveal a negative apparent age of  $-0.75 \pm 0.55$  Ma with a  $^{40}\text{Ar}/^{36}\text{Ar}$  value of  $298.4 \pm 5.4$  (Figure 11b).

## Discussion

The weighted mean ages assigned to the flattest portions of the well-behaved age spectra (CEQ-8,  $1.08 \pm 0.13$  Ma; CEQ-18,  $0.61 \pm 0.04$  Ma; TB-1,  $22.51 \pm 0.09$  Ma) provide reliable eruption or emplacement ages. The increasing apparent ages correlated with increasing radiogenic yields revealed by the early heating steps of the PP-2, CEQ-22 and CEQ-14 age spectra are suggestive of alteration and accompanying Ar loss. However, the weighted mean ages assigned to these samples contain well over 50% of the  $^{39}\text{Ar}$  released: 73.8%, PP-2; 70.7%, CEQ-22; and 91.4%, CEQ-14. We therefore feel fairly comfortable in assigning the calculated weighted mean ages as the eruption ages. Saddle-shaped age spectra such as those revealed by EMD046 biotite and IM-1 plagioclase concentrate can be caused by excess Ar (Harrison and McDougal, 1981). Inverse isochron analysis of steps A-H for EMD046 biotite and A-G for IM-1 plagioclase reveals a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept slightly over the atmospheric intercept. We assign the isochron ages of  $2.03 \pm 0.76$  Ma (EMD046) and  $21.85 \pm 0.31$  Ma (IM-1) as the emplacement ages for these samples. The integrated ages have been assigned as our best estimate of the eruption or emplacement ages for the disturbed SMQ-1, MM-1, and SMQ-4 age spectra ( $1.02 \pm 0.36$  Ma,  $12.1 \pm 1.9$  Ma and  $0.46 \pm 0.89$  Ma, respectively). The humped or saddle shapes revealed by these spectra are most likely the result of recoil redistributing the  $^{39}\text{Ar}$  created at the reactor from high K sites to low K sites. As mentioned above, excess Ar can also result in a saddle-shaped spectrum but neither of these three spectra yields a  $^{40}\text{Ar}/^{36}\text{Ar}$  intercept above the atmospheric intercept. Recoil has the affect of increasing the apparent ages of the sites that have lost  $^{39}\text{Ar}$  and decreasing the apparent ages of those

sites that have gained  $^{39}\text{Ar}$  (Figure 12). In situations where recoil is suspected, the integrated age is usually assigned as the best estimate of the age of the sample. The preferred ages for the Pine Valley Mountains are shown plotted on Figures 13 and 14.

## References Cited

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- Renne, P.R., Owens, T.L., DePaolo, D.J., Swisher, C.C., Deino, A.L., and Darner, D.B., 1998. Intercalibration of standards, absolute ages and uncertainties in  $^{40}\text{Ar}/^{39}\text{Ar}$  dating, 145, 117-152.
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Table 2. Summary of  $^{40}\text{Ar}/^{39}\text{Ar}$  results and analytical methods

Sample	Lab #	Irradiation	mineral/phase	age analysis	steps	Age	$\pm 2\sigma$	MSWD	40Ar/36Ar intercept	comments
SMQ-4	55428	NM-186	groundmass concentrate	furnace step-heat	9	0.46	0.89	-	-	integrated age
CEQ-18	55431	NM-186	groundmass concentrate	furnace step-heat	9	0.61	0.04	1.0	-	
CEQ-14	55430	NM-186	groundmass concentrate	furnace step-heat	8	0.67	0.08	3.1	-	
SMQ-1	55427	NM-186	groundmass concentrate	furnace step-heat	9	1.02	0.36	-	-	integrated age
CEQ-8	55429	NM-186	groundmass concentrate	furnace step-heat	9	1.08	0.13	2.0	-	
EMD046	55413	NM-186	biotite	furnace step-heat	8	2.03	0.76	4.90	296.8 $\pm$ 5.4	isochron age
MM-1	55434	NM-186	plagioclase	furnace step-heat	11	12.1	1.9	-	-	integrated age
CEQ-22	55432	NM-186	groundmass concentrate	furnace step-heat	5	17.39	0.12	0.73	-	
IM-1	55433	NM-186	plagioclase	furnace step-heat	7	21.85	0.31	6.5	298.8 $\pm$ 5.1	isochron age
PP-2	55412	NM-186	biotite	furnace step-heat	6	21.96	0.11	4.6	-	
TB-1	55410	NM-186	biotite	furnace step-heat	10	22.51	0.09	6.4	-	

**Sample preparation and irradiation:**

Minerals separated with standard heavy liquid, Franz Magnetic and hand-picking techniques.

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-2). Assigned age = 28.02 Ma (Reine et al., 1998).

**Instrumentation:**

Mass Analyzer Products 215-50 mass spectrometer on line with automated all-metal extraction system.

Mineral separates step-heated for 8-10 minutes using a Mo double-vacuum resistance furnace.

Reactive gases removed during furnace analysis by reaction with 3 SAES GP-50 getters, 2 operated at ~450°C and

1 at 20°C. Gas also exposed to a W filament operated at ~2000°C.

**Analytical parameters:**

Electron multiplier sensitivity averaged  $2.24 \times 10^{16}$  moles/pA.

Total system blank and background averaged 2330, 8.72, 2.50, 1.56,  $9.13 \times 10^{18}$  moles at masses 40, 39, 38, 37 and 36, respectively.

J-factors determined to a precision of  $\pm 0.1\%$  by  $\text{CO}_2$  laser-fusion of 6 single crystals from each of 6 or 10 radial positions around the irradiation tray.

Correction factors for interfering nuclear reactions were determined using K-glass and  $\text{CaF}_2$  and are as follows:

$(^{40}\text{Ar}/^{39}\text{Ar})_c = 0.0000 \pm 0.0004$ ;  $(^{36}\text{Ar}/^{37}\text{Ar})_c = 0.00028 \pm 0.00001$ ; and  $(^{39}\text{Ar}/^{37}\text{Ar})_c = 0.0007 \pm 0.00005$ .

**Table 3.  $^{40}\text{Ar}/^{39}\text{Ar}$  analytical data.**

ID	Temp (°C)	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{37}\text{Ar}/^{39}\text{Ar}$	$^{36}\text{Ar}/^{39}\text{Ar}$ ( $\times 10^{-3}$ )	$^{39}\text{Ar}_K$ ( $\times 10^{-15}$ mol)	K/Ca	$^{40}\text{Ar}^*$ (%)	$^{39}\text{Ar}$ (%)	Age (Ma)	$\pm 1\sigma$ (Ma)
<b>CEQ-8</b> , Groundmass Concentrate, J=0.0007367±0.10%, D=1.0055±0.001, NM-186H, Lab#=55429-01										
A	625	213.9	0.5260	726.0	4.2	0.97	-0.3	7.1	-0.79	1.28
B	700	18.48	0.8523	60.76	15.2	0.60	3.2	33.0	0.79	0.12
C	750	15.00	1.216	48.15	8.6	0.42	5.9	47.7	1.17	0.10
D	800	12.30	1.147	39.39	10.2	0.44	6.2	65.2	1.01	0.10
E	875	12.15	1.334	38.57	9.3	0.38	7.1	81.0	1.15	0.09
F	975	15.13	1.827	49.02	5.0	0.28	5.3	89.5	1.07	0.15
G	1075	21.80	2.179	70.71	2.2	0.23	5.0	93.3	1.45	0.25
H	1250	33.15	14.20	112.7	1.7	0.036	3.2	96.2	1.41	0.38
I	1700	44.26	17.17	150.0	2.21	0.030	3.2	100.0	1.87	0.44
<b>Integrated age <math>\pm 2\sigma</math></b>			n=9		58.7	0.38 ±0.58		K2O=1.13%	0.94	0.34
<b>Plateau <math>\pm 2\sigma</math></b>	<b>steps A-I</b>		<b>n=9</b>	<b>MSWD=2.0</b>	<b>58.7</b>	<b>0.38 ±0.58</b>		<b>100.0</b>	<b>1.08</b>	<b>0.13</b>
<b>Isochron<math>\pm 2\sigma</math></b>	steps A-I		n=9	MSWD=2.2		$^{40}\text{Ar}/^{36}\text{Ar}=294.9\pm 3.6$			1.12	0.27
<b>CEQ-18</b> , Groundmass Concentrate, J=0.0007397±0.07%, D=1.0055±0.001, NM-186H, Lab#=55431-01										
A	625	87.03	1.197	295.0	2.2	0.43	0.0	6.4	-0.05	0.57
B	700	2.231	1.093	6.276	8.0	0.47	21.1	30.2	0.63	0.03
C	750	1.767	1.380	4.873	4.72	0.37	25.2	44.3	0.59	0.05
D	800	1.715	1.395	4.546	4.5	0.37	28.6	57.7	0.66	0.07
E	875	1.827	1.592	5.116	4.1	0.32	24.7	70.0	0.60	0.05
F	975	2.541	2.440	7.613	3.0	0.21	19.7	79.1	0.67	0.09
G	1075	3.519	2.970	11.51	2.1	0.17	10.6	85.4	0.50	0.14
H	1250	9.850	18.38	37.37	4.0	0.028	3.8	97.3	0.51	0.15
I	1700	27.39	41.56	106.2	0.89	0.012	-1.6	100.0	-0.60	0.56
<b>Integrated age <math>\pm 2\sigma</math></b>			n=9		33.5	0.26 ±0.33		K2O=0.67%	0.53	0.12
<b>Plateau <math>\pm 2\sigma</math></b>	<b>steps A-I</b>		<b>n=9</b>	<b>MSWD=1.0</b>	<b>33.5</b>	<b>0.26 ±0.33</b>		<b>100.0</b>	<b>0.61</b>	<b>0.04</b>
<b>Isochron<math>\pm 2\sigma</math></b>	steps A-I		n=9	MSWD=0.67		$^{40}\text{Ar}/^{36}\text{Ar}=293.0\pm 2.6$			0.64	0.04
<b>TB-1</b> , biotite, J=0.00074±0.05%, D=1.0055±0.001, NM-186E, Lab#=55410-01										
A	650	177.6	0.2706	548.7	3.99	1.9	8.7	4.3	20.49	0.94
B	750	20.05	0.0980	10.18	6.25	5.2	85.0	11.0	22.62	0.07
C	850	19.07	0.0993	7.529	10.9	5.1	88.4	22.6	22.36	0.06
D	920	17.75	0.0307	2.076	9.9	16.6	96.6	33.2	22.73	0.05
E	1000	17.49	0.0353	1.744	17.9	14.5	97.1	52.3	22.53	0.04
F	1075	17.69	0.0438	2.355	15.2	11.7	96.1	68.6	22.55	0.04
G	1110	17.72	0.0494	3.065	8.6	10.3	94.9	77.7	22.31	0.06
H	1180	19.04	0.2414	6.983	6.23	2.1	89.3	84.4	22.55	0.07
I	1210	18.09	0.1476	4.235	8.3	3.5	93.2	93.2	22.35	0.06
J	1250	18.00	0.0529	3.479	5.75	9.6	94.3	99.4	22.53	0.07
<b>Integrated age <math>\pm 2\sigma</math></b>			n=12		93.5	7.0 ±10.6		K2O=4.66%	22.42	0.14
<b>Plateau <math>\pm 2\sigma</math></b>	<b>steps A-L</b>		<b>n=12</b>	<b>MSWD=5.4</b>	<b>93.5</b>	<b>7.0 ±10.6</b>		<b>100.0</b>	<b>22.51</b>	<b>0.09</b>
<b>Isochron<math>\pm 2\sigma</math></b>	steps A-I		n=12	MSWD=5.2		$^{40}\text{Ar}/^{36}\text{Ar}=292.3\pm 1.3$			22.53	0.02

ID	Temp (°C)	<sup>40</sup> Ar/ <sup>39</sup> Ar	<sup>37</sup> Ar/ <sup>39</sup> Ar	<sup>36</sup> Ar/ <sup>39</sup> Ar (x 10 <sup>-3</sup> )	<sup>39</sup> Ar <sub>K</sub> (x 10 <sup>-15</sup> mol)	K/Ca	<sup>40</sup> Ar* (%)	<sup>39</sup> Ar (%)	Age (Ma)	±1σ (Ma)
<b>PP-2</b> , biotite, J=0.0007412±0.06%, D=1.0055±0.001, NM-186E, Lab#=55412-01										
# A	650	358.2	0.0535	1177.7	2.48	9.5	2.8	1.9	13.59	2.06
# B	750	46.02	0.0542	109.5	2.51	9.4	29.7	3.8	18.18	0.29
# C	850	24.35	0.0225	28.04	11.1	22.6	66.0	12.2	21.36	0.09
D	920	20.60	0.0144	13.05	18.3	35.4	81.3	26.1	22.25	0.06
E	1000	19.95	0.0134	11.47	34.8	38.0	83.0	52.6	22.00	0.05
F	1075	20.47	0.0260	13.50	20.7	19.6	80.5	68.4	21.91	0.06
G	1110	22.41	0.0912	20.78	7.9	5.6	72.6	74.3	21.64	0.09
H	1180	19.07	0.1600	8.440	20.6	3.2	87.0	90.0	22.05	0.05
I	1210	18.23	0.1514	6.106	4.90	3.4	90.2	93.7	21.85	0.09
J	1250	17.83	0.1310	4.195	8.0	3.9	93.1	99.8	22.07	0.06
K	1300	19.47	0.4263	12.81	0.120	1.2	80.8	99.9	20.91	2.05
L	1700	69.77	0.6855	172.7	0.144	0.74	26.9	100.0	24.95	2.17
<b>Integrated age ± 2σ</b>			n=12		131.4	12.3 ±29.9		K2O=6.54%	21.72	0.17
<b>Plateau ± 2σ</b>	<b>steps D-L</b>		<b>n=9</b>	<b>MSWD=5.7</b>	<b>115.4</b>	<b>12.3 ±29.9</b>		<b>87.8</b>	<b>22.01</b>	<b>0.11</b>
<b>Isochron±2σ</b>	steps D-K		n=8	MSWD=6.2		<sup>40</sup> Ar/ <sup>36</sup> Ar=287±20			22.13	0.35
<b>CEQ-22</b> , Groundmass Concentrate, J=0.0007399±0.09%, D=1.0055±0.001, NM-186H, Lab#=55432-01										
# A	625	294.3	0.8373	962.2	5.1	0.61	3.4	3.8	13.39	1.61
# B	700	31.40	0.4707	64.69	10.3	1.1	39.2	11.6	16.38	0.14
# C	750	25.71	0.6861	44.56	8.5	0.74	49.0	18.0	16.76	0.12
D	800	26.12	0.6882	44.37	10.6	0.74	50.0	26.0	17.37	0.11
E	875	26.29	0.5800	44.56	13.9	0.88	50.1	36.5	17.50	0.11
F	975	25.60	0.5877	42.49	20.6	0.87	51.1	52.1	17.40	0.11
G	1075	32.25	0.5823	65.49	34.7	0.88	40.1	78.4	17.21	0.14
H	1250	47.31	1.836	116.2	13.6	0.28	27.8	88.7	17.47	0.24
# I	1700	58.95	1.883	152.9	14.9	0.27	23.6	100.0	18.50	0.28
<b>Integrated age ± 2σ</b>			n=9		132.0	0.73 ±0.52		K2O=2.76%	17.21	0.37
<b>Plateau ± 2σ</b>	<b>steps D-H</b>		<b>n=5</b>	<b>MSWD=0.73</b>	<b>93.3</b>	<b>0.73 ±0.52</b>		<b>70.7</b>	<b>17.39</b>	<b>0.12</b>
<b>Isochron±2σ</b>	steps A-I		n=9	MSWD=12		<sup>40</sup> Ar/ <sup>36</sup> Ar=295.5±6.9			17.21	0.62
<b>CEQ-14</b> , Groundmass Concentrate, J=0.0007382±0.08%, D=1.0055±0.001, NM-186H, Lab#=55430-01										
# A	625	245.3	2.475	841.4	3.1	0.21	-1.3	8.6	-4.11	1.51
B	700	7.697	2.013	24.92	7.6	0.25	6.6	29.8	0.67	0.07
C	750	2.521	1.899	7.039	6.5	0.27	23.9	47.8	0.80	0.05
D	800	1.589	1.592	4.144	6.7	0.32	31.5	66.3	0.67	0.04
E	875	1.477	1.570	4.039	6.2	0.33	28.3	83.4	0.56	0.04
F	975	2.254	2.771	6.539	3.2	0.18	24.8	92.2	0.74	0.08
G	1075	7.104	5.480	24.81	0.9	0.093	3.4	94.8	0.32	0.25
H	1250	36.07	44.48	135.3	1.4	0.011	-0.3	98.7	-0.14	0.51
I	1700	192.3	72.65	666.0	0.477	0.007	0.9	100.0	2.37	2.07
<b>Integrated age ± 2σ</b>			n=9		36.0	0.18 ±0.26		K2O=0.72%	0.26	0.33
<b>Plateau ± 2σ</b>	<b>steps B-I</b>		<b>n=8</b>	<b>MSWD=3.1</b>	<b>32.9</b>	<b>0.18 ±0.26</b>		<b>91.4</b>	<b>0.67</b>	<b>0.08</b>
<b>Isochron±2σ</b>	steps A-I		n=9	MSWD=3.8		<sup>40</sup> Ar/ <sup>36</sup> Ar=293.2±3.9			0.69	0.08

ID	Temp (°C)	<sup>40</sup> Ar/ <sup>39</sup> Ar	<sup>37</sup> Ar/ <sup>39</sup> Ar	<sup>36</sup> Ar/ <sup>39</sup> Ar (x 10 <sup>-3</sup> )	<sup>39</sup> Ar <sub>K</sub> (x 10 <sup>-15</sup> mol)	K/Ca	<sup>40</sup> Ar* (%)	<sup>39</sup> Ar (%)	Age (Ma)	±1σ (Ma)
<b>EMD046</b> , biotite, J=0.0007424±0.07%, D=1.0055±0.001, NM-186E, Lab#=55413-01										
# A	650	465.5	0.0460	1555.0	19.9	11.1	1.3	17.5	8.06	2.66
B	750	62.32	0.0434	206.1	26.7	11.8	2.3	41.0	1.90	0.34
C	850	33.35	0.0456	106.8	17.4	11.2	5.4	56.4	2.42	0.19
D	920	30.64	0.0477	99.09	11.7	10.7	4.5	66.7	1.83	0.19
E	1000	33.37	0.0596	108.2	11.8	8.6	4.2	77.1	1.90	0.20
F	1075	31.72	0.0516	102.2	19.6	9.9	4.8	94.3	2.05	0.18
G	1110	29.58	0.1447	94.02	4.08	3.5	6.1	97.9	2.42	0.24
# H	1180	27.08	0.8490	83.66	1.93	0.60	9.0	99.6	3.26	0.26
# I	1210	28.27	2.069	87.18	0.193	0.25	9.5	99.8	3.59	1.13
# J	1250	32.33	4.140	96.01	0.139	0.12	13.3	99.9	5.79	1.65
# L	1700	222.5	0.9481	679.8	0.093	0.54	9.8	100.0	28.89	4.37
Integrated age ± 2σ			n=11		113.4	9.3 ±6.1		K2O=4.97%	3.1	1.3
<b>Plateau ± 2σ</b>			steps B-G	n=6	MSWD=1.6	91.2	9.3 ±6.1	80.4	2.09	0.22
<b>Isochron±2σ</b>			<b>steps A-H</b>	<b>n=8</b>	<b>MSWD=4.9</b>	<b><sup>40</sup>Ar/<sup>36</sup>Ar=296.8±5.4</b>			<b>2.03</b>	<b>0.76</b>
<b>IM-1</b> , plagioclase, J=0.0007344±0.10%, D=1.0055±0.001, NM-186J, Lab#=55433-01										
# A	650	343.8	0.4167	1094.8	7.64	1.2	5.9	2.5	26.73	1.87
B	775	46.38	0.8711	99.17	51.0	0.59	37.0	19.1	22.59	0.19
C	850	23.93	0.2325	24.44	41.5	2.2	69.9	32.7	22.04	0.07
D	925	21.55	0.2242	17.17	24.6	2.3	76.5	40.7	21.73	0.06
E	1000	22.95	0.2580	20.75	17.0	2.0	73.4	46.3	22.18	0.08
F	1100	30.24	0.1466	45.51	43.1	3.5	55.6	60.3	22.13	0.10
G	1175	45.41	0.1771	98.02	32.0	2.9	36.2	70.8	21.68	0.19
# H	1250	58.51	0.6715	135.5	82.2	0.76	31.6	97.6	24.38	0.26
# I	1350	72.56	1.510	166.0	2.79	0.34	32.6	98.5	31.07	0.45
# J	1450	88.06	2.689	191.2	2.8	0.19	36.1	99.4	41.73	0.44
# K	1675	106.0	4.462	165.1	1.7	0.11	54.3	100.0	74.92	0.52
Integrated age ± 2σ			n=11		306.2	2.2 ±2.0		K2O=5.78%	23.39	0.39
<b>Plateau ± 2σ</b>			steps B-G	n=6	MSWD=7.8	209.0	2.2 ±2.0	68.3	21.98	0.21
<b>Isochron±2σ</b>			<b>steps A-G</b>	<b>n=7</b>	<b>MSWD=6.5</b>	<b><sup>40</sup>Ar/<sup>36</sup>Ar=298.8</b>			<b>21.85</b>	<b>0.31</b>
<b>SMQ-1</b> , Groundmass Concentrate, J=0.0007384±0.07%, D=1.0055±0.001, NM-186H, Lab#=55427-01										
A	625	614.1	0.9655	2080.5	1.7	0.53	-0.1	3.0	-0.81	3.61
B	700	13.81	0.8186	44.38	8.7	0.62	5.5	18.3	1.02	0.10
C	750	7.334	0.8189	22.31	5.7	0.62	11.1	28.4	1.08	0.09
D	800	5.507	0.8024	15.71	9.7	0.64	17.0	45.4	1.24	0.05
E	875	4.285	0.9007	11.50	8.2	0.57	22.5	59.8	1.28	0.06
F	975	5.379	1.354	15.61	5.9	0.38	16.4	70.2	1.18	0.07
# G	1075	8.696	1.587	28.04	4.4	0.32	6.3	77.9	0.73	0.10
# H	1250	28.51	8.674	97.21	10.7	0.059	1.9	96.7	0.71	0.21
# I	1700	114.5	9.445	384.6	1.85	0.054	1.4	100.0	2.15	0.83
Integrated age ± 2σ			<b>n=9</b>		<b>56.8</b>	<b>0.56 ±0.20</b>		<b>K2O=1.11%</b>	<b>1.02</b>	<b>0.36</b>
<b>Plateau ± 2σ</b>			steps A-F	n=6	MSWD=1.6	39.9	0.56 ±0.20	70.2	1.20	0.08
<b>Isochron±2σ</b>			steps A-I	n=9	MSWD=4.6	<b><sup>40</sup>Ar/<sup>36</sup>Ar=293.8±3.9</b>			1.20	0.17

ID	Temp (°C)	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{37}\text{Ar}/^{39}\text{Ar}$	$^{36}\text{Ar}/^{39}\text{Ar}$ ( $\times 10^{-3}$ )	$^{39}\text{Ar}_K$ ( $\times 10^{-15}$ mol)	K/Ca	$^{40}\text{Ar}^*$ (%)	$^{39}\text{Ar}$ (%)	Age (Ma)	$\pm 1\sigma$ (Ma)
<b>MM-1</b> , plagioclase, J=0.0007324±0.08%, D=1.0055±0.001, NM-186J, Lab#=55434-01										
A	650	12963.2	0.4848	43817.5	0.2	1.1	0.1	0.4	19.96	74.60
B	775	168.6	0.4097	535.9	10.0	1.2	6.1	18.2	13.58	0.89
C	850	73.38	0.1032	214.9	2.54	4.9	13.5	22.8	13.03	0.52
D	925	80.81	0.1549	242.8	3.3	3.3	11.2	28.6	11.95	0.53
E	1000	113.0	0.0684	353.8	2.4	7.5	7.5	32.9	11.12	0.76
F	1100	132.8	0.0437	424.0	12.4	11.7	5.7	55.0	9.89	0.72
G	1175	146.9	0.0359	468.7	12.6	14.2	5.7	77.5	11.12	0.78
H	1250	120.2	0.0735	373.7	10.8	6.9	8.1	96.8	12.90	0.63
I	1350	130.9	0.1779	406.5	0.8	2.9	8.2	98.2	14.18	1.14
J	1450	183.4	0.5615	580.5	0.5	0.91	6.5	99.1	15.68	1.62
K	1675	162.9	1.153	473.4	0.5	0.44	14.2	100.0	30.34	1.42
<b>Integrated age <math>\pm 2\sigma</math></b>			<b>n=11</b>		<b>56.2</b>	<b>5.5 <math>\pm 9.2</math></b>			<b>12.1</b>	<b>1.9</b>
<b>Isochron <math>\pm 2\sigma</math></b>		steps A-J	n=10	MSWD=3.5		$^{40}\text{Ar}/^{36}\text{Ar}=295.5\pm 3.7$			12.2	1.9
<b>SMQ-4</b> , Groundmass Concentrate, J=0.0007368±0.08%, D=1.0055±0.001, NM-186H, Lab#=55428-01										
A	625	195.1	0.5194	661.7	8.2	0.98	-0.2	10.4	-0.45	1.11
B	700	32.59	0.7392	110.4	15.7	0.69	0.1	30.3	0.05	0.21
C	750	41.11	0.9165	140.9	10.1	0.56	-1.1	43.2	-0.58	0.24
D	800	45.48	1.011	156.6	11.3	0.50	-1.6	57.4	-0.96	0.27
E	875	60.25	1.098	205.4	9.2	0.46	-0.6	69.0	-0.48	0.38
F	975	94.79	1.197	319.9	5.54	0.43	0.4	76.0	0.49	0.60
G	1075	202.4	1.431	688.4	2.2	0.36	-0.4	78.8	-1.13	1.25
# H	1250	220.1	2.520	729.7	5.6	0.20	2.1	85.8	6.25	1.30
# I	1700	66.67	2.262	220.6	11.2	0.23	2.5	100.0	2.24	0.40
<b>Integrated age <math>\pm 2\sigma</math></b>			<b>n=9</b>		<b>79.0</b>	<b>0.57 <math>\pm 0.42</math></b>	<b>K2O=1.53%</b>		<b>0.46</b>	<b>0.89</b>
<b>Plateau <math>\pm 2\sigma</math></b>		steps A-G	n=7	MSWD=2.0	62.2	0.57 $\pm 0.42$		78.8	-0.39	0.35
<b>Isochron <math>\pm 2\sigma</math></b>		steps A-I	n=9	MSWD=9.2		$^{40}\text{Ar}/^{36}\text{Ar}=298.4\pm 5.4$			-0.75	0.55

**Notes:**

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

Errors quoted for individual analyses include analytical error only, without interfering reaction or J uncertainties.

Integrated age is volume-weighted mean of all steps.

Integrated age calculated by recombining isotopic measurements of all steps.

Integrated age error calculated by recombining errors of isotopic measurements of all steps.

Plateau age is inverse-variance-weighted mean of selected steps.

Plateau age error is inverse-variance-weighted mean error (Taylor, 1982) times root MSWD where MSWD>1.

Decay constants and isotopic abundances after Steiger and Jaeger (1977).

# symbol preceding sample ID denotes analyses excluded from plateau age calculations.

Ages calculated relative to FC-2 Fish Canyon Tuff sanidine interlaboratory standard at 28.02 Ma

Decay Constant ( $\lambda_{\text{total}}$ ) =  $5.543\text{e-}10$

Discrimination =  $1.0055 \pm 0.001$

Correction factors:

$$(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.00068 \pm 2\text{e-}05$$

$$(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.000289 \pm 5\text{e-}06$$

$$(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.0132$$

$$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0 \pm 0.0004$$

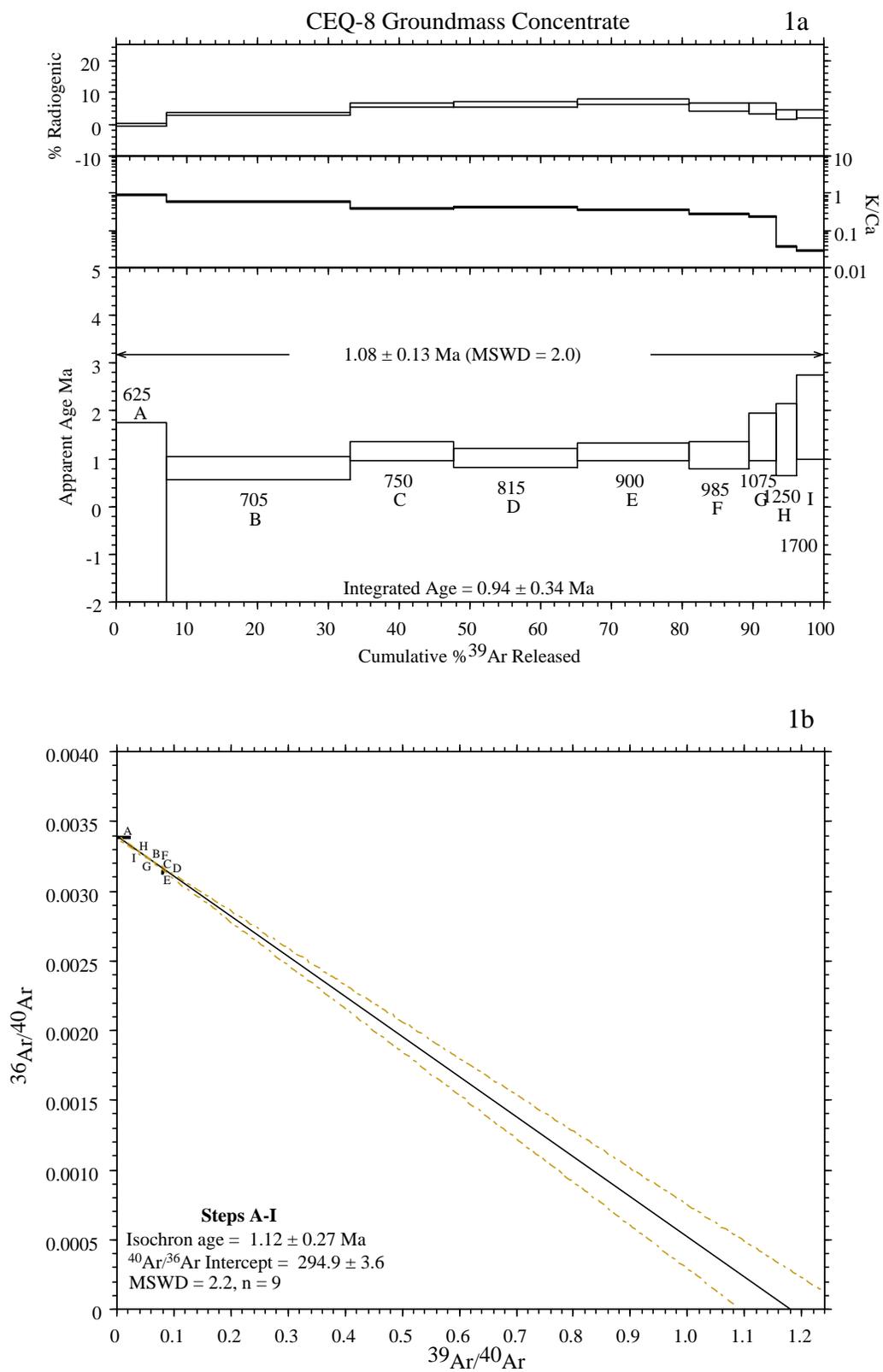


Figure 1. Age spectrum (1a) and isochron (1b) for CEQ-8 groundmass concentrate. All errors quoted at two sigma.

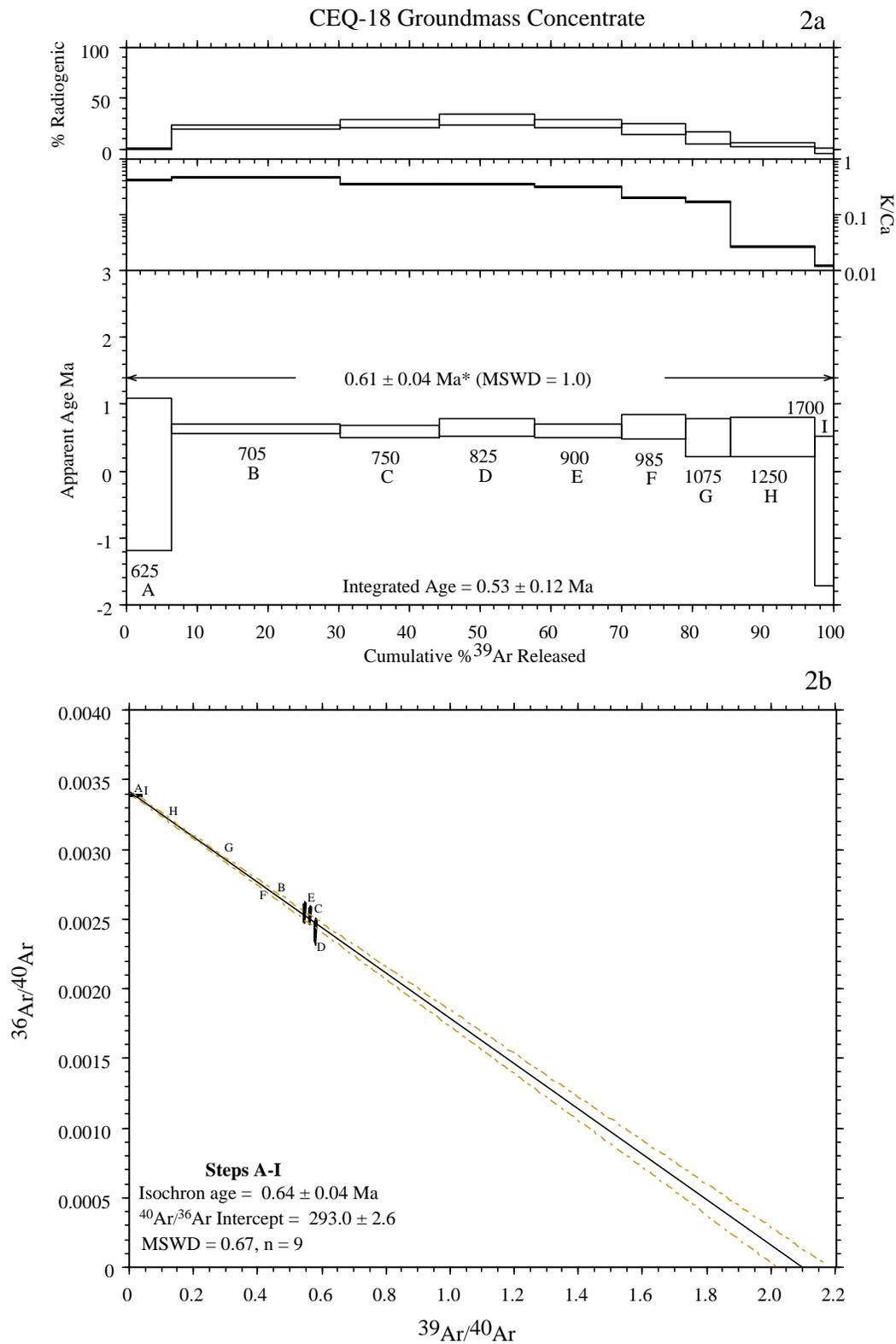


Figure 2. Age spectrum (2a) and isochron (2b) for CEQ-18 groundmass concentrate. All errors quoted at two sigma.

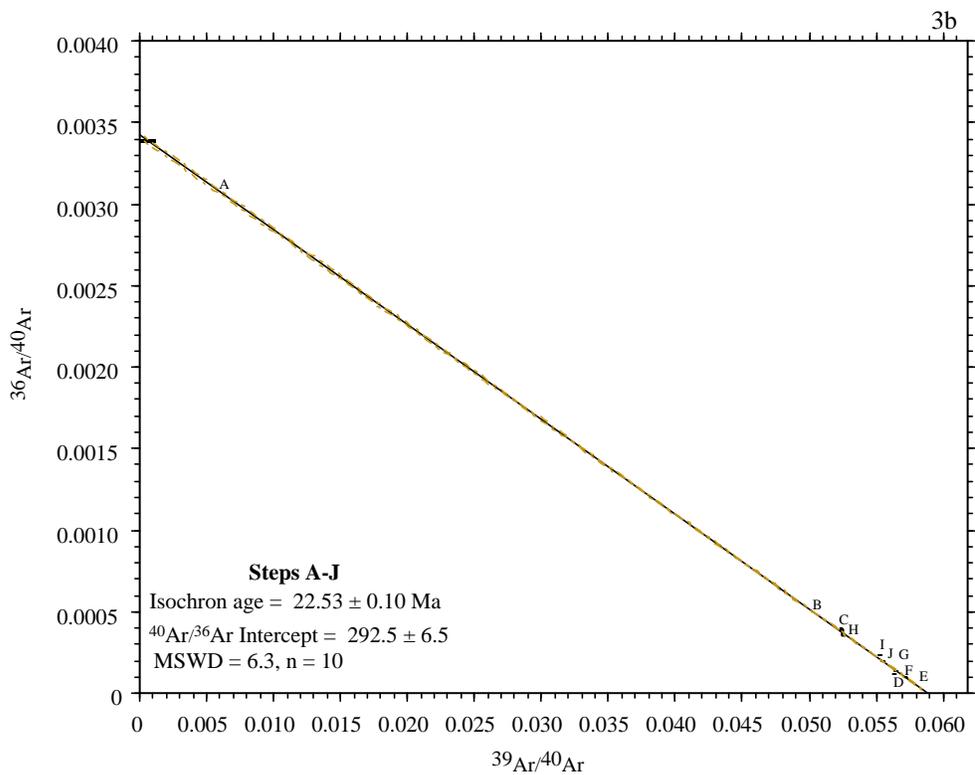
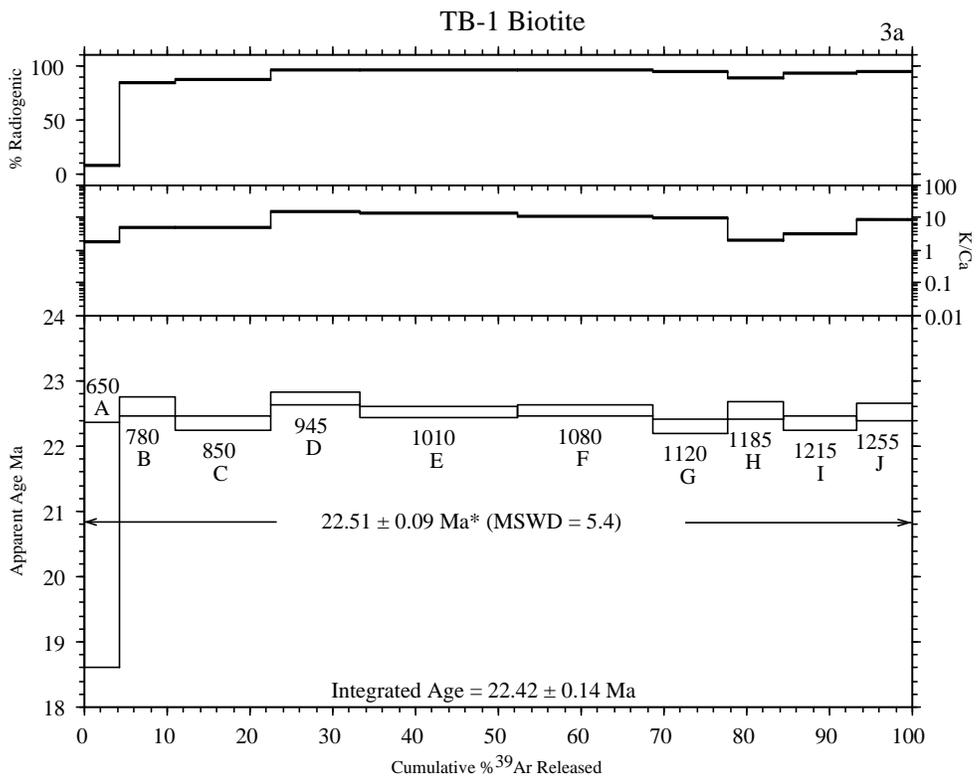


Figure 3. Age spectrum (3a) and isochron (3b) for TB-1 groundmass concentrate. All errors quoted at 2 sigma.

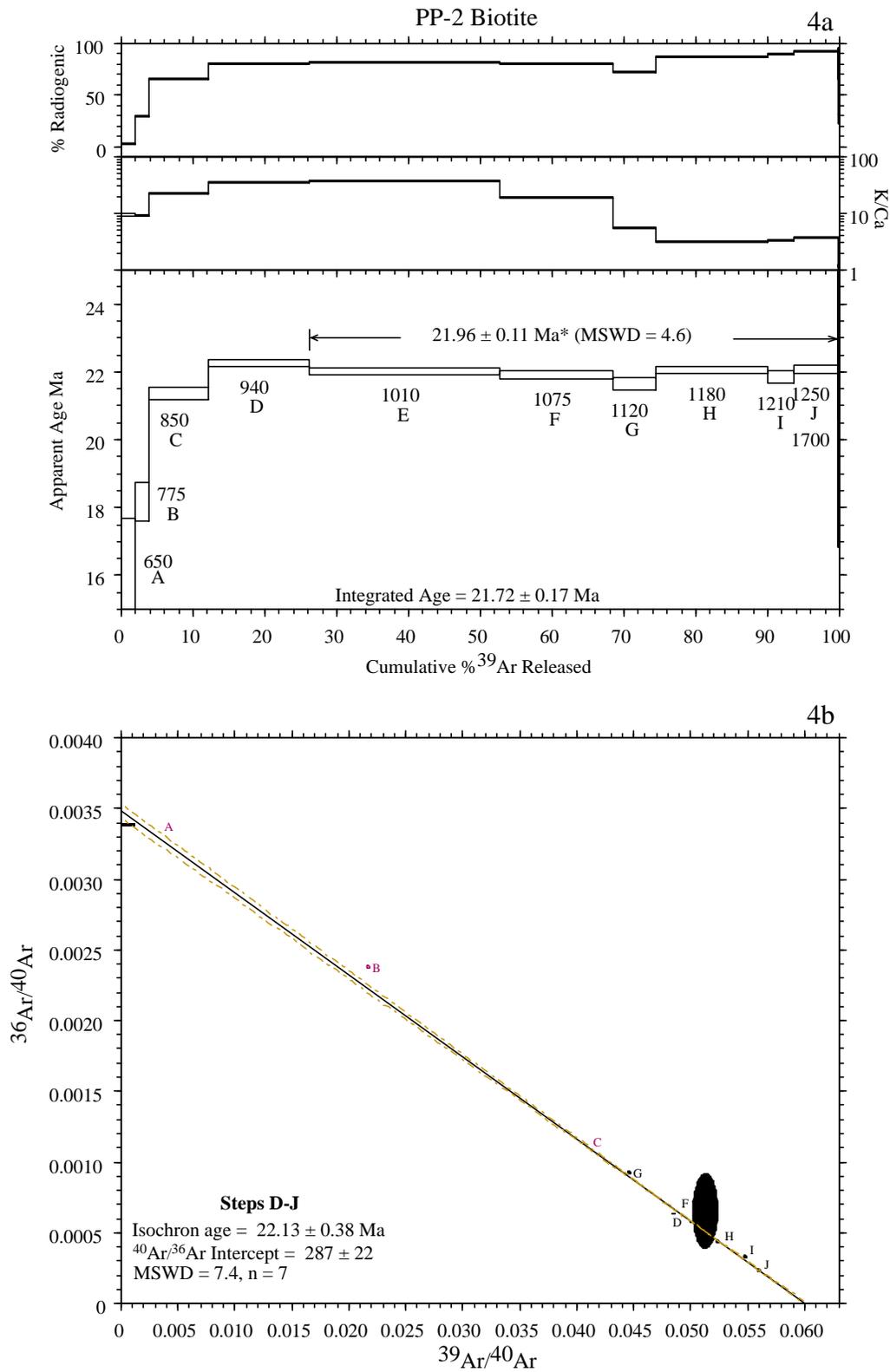


Figure 4. Age spectrum (4a) and isochron (4b) for PP-2 biotite. Points in purple not included in isochron. All errors quoted at two sigma.

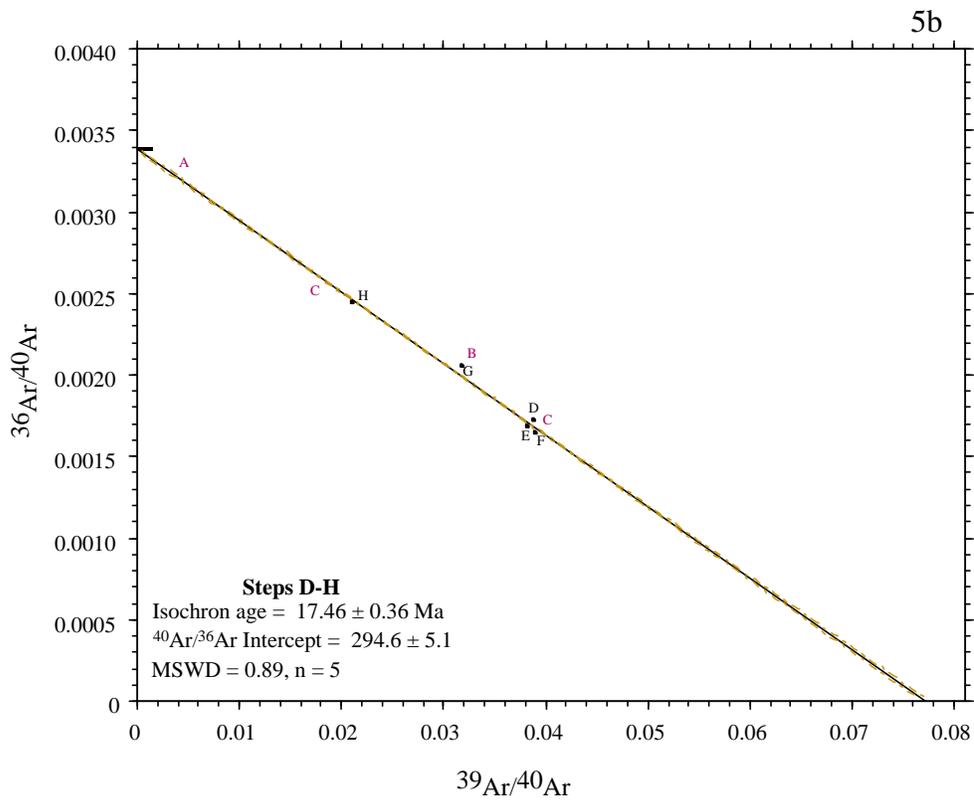
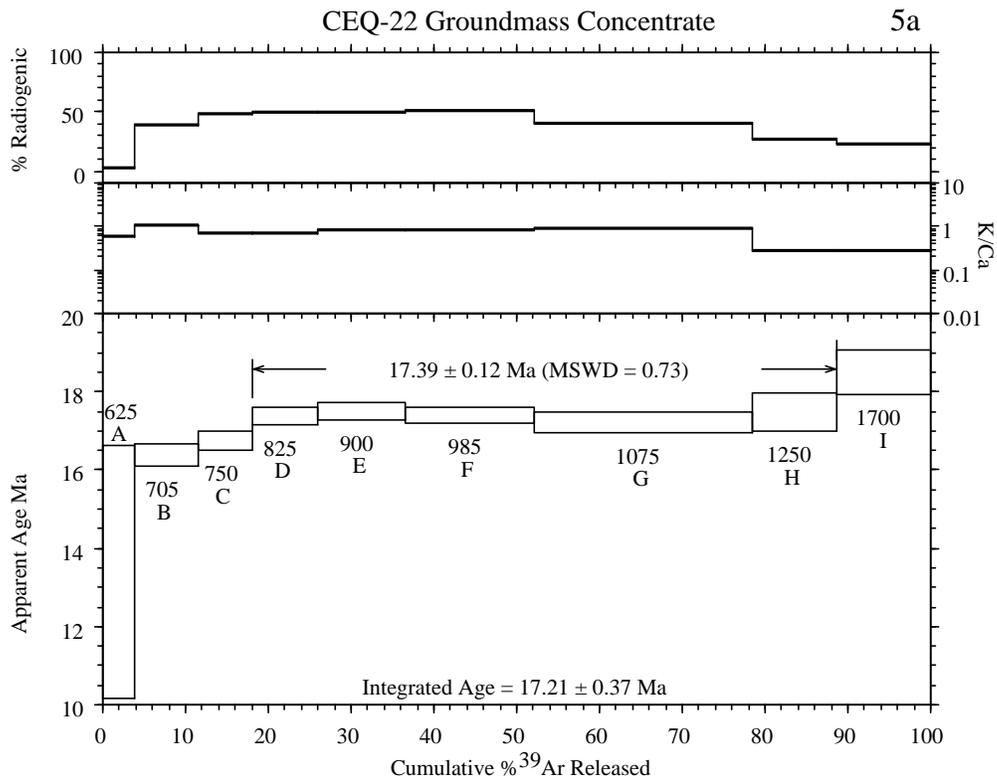


Figure 5. Age spectrum (5a) and isochron (5b) for CEQ-22 groundmass concentrate. Points in purple not included in isochron. All errors quoted at 2 sigma.

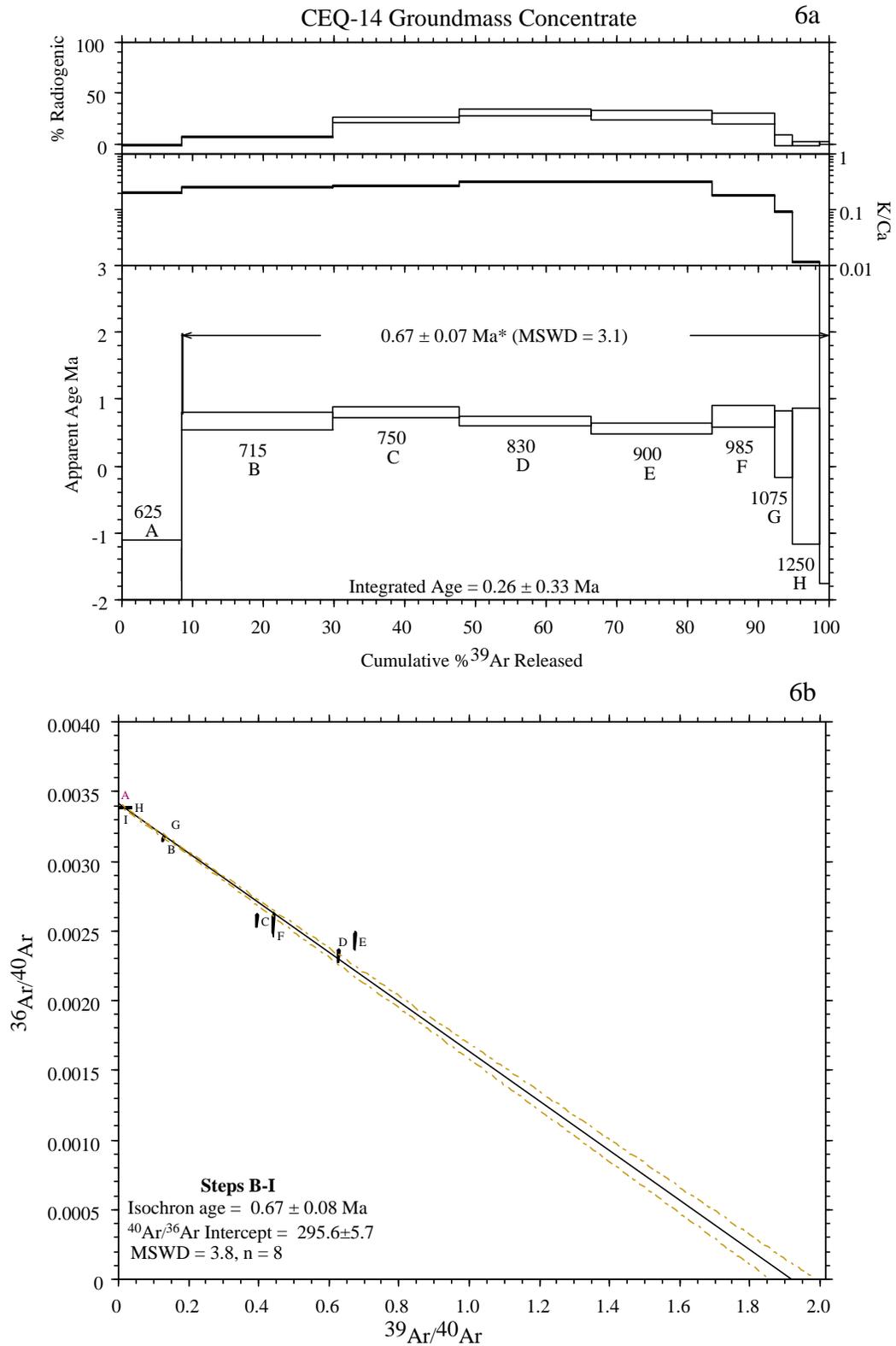


Figure 6. Age spectrum (6a) and isochron (6b) for CEQ-14 groundmass concentrate. Points shown in purple not included in isochron. All errors quoted at two sigma.

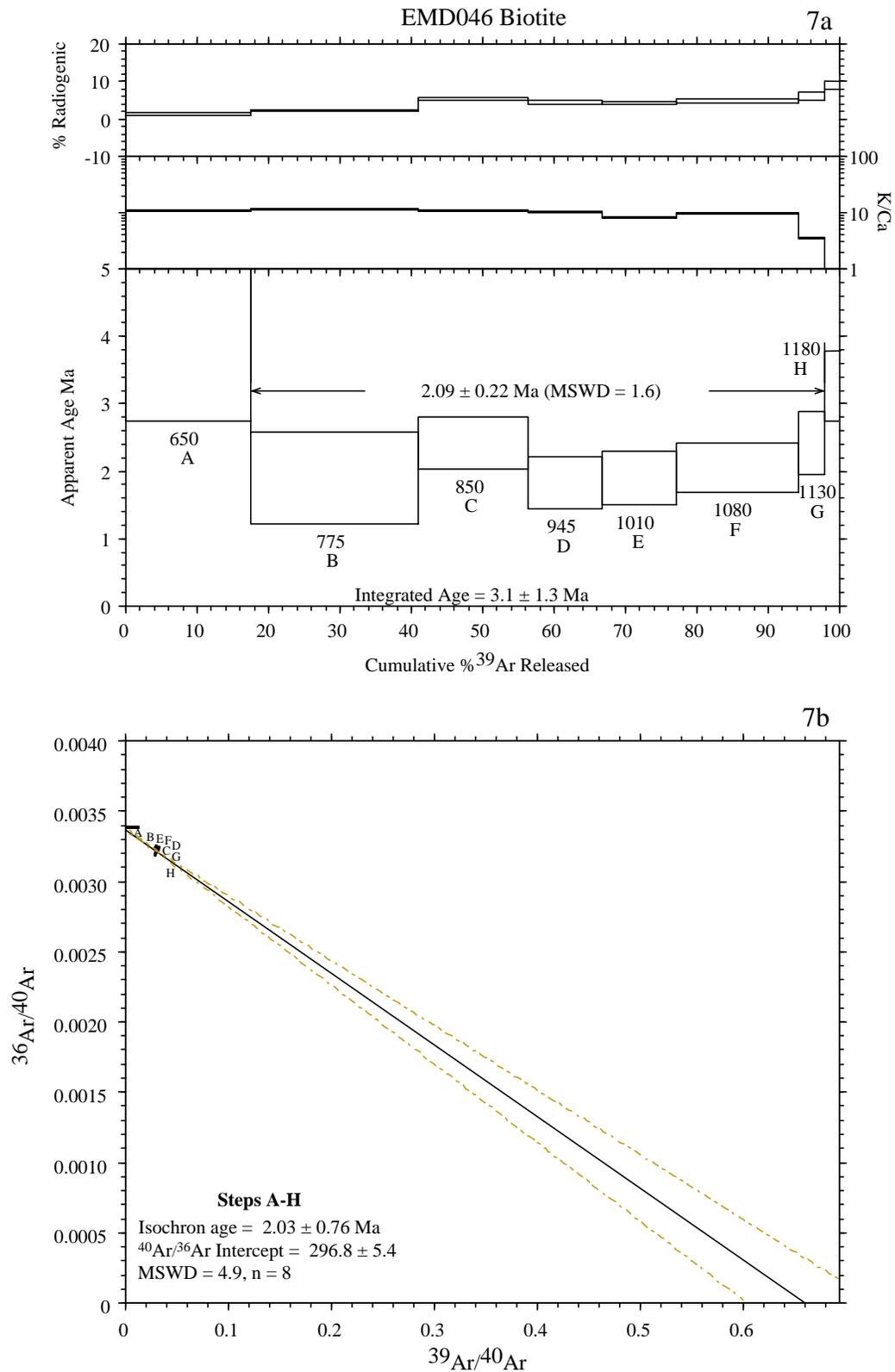


Figure 7. Age spectrum (7a) and isochron (7b) for EMD046 biotite. All errors quoted at 2 sigma.

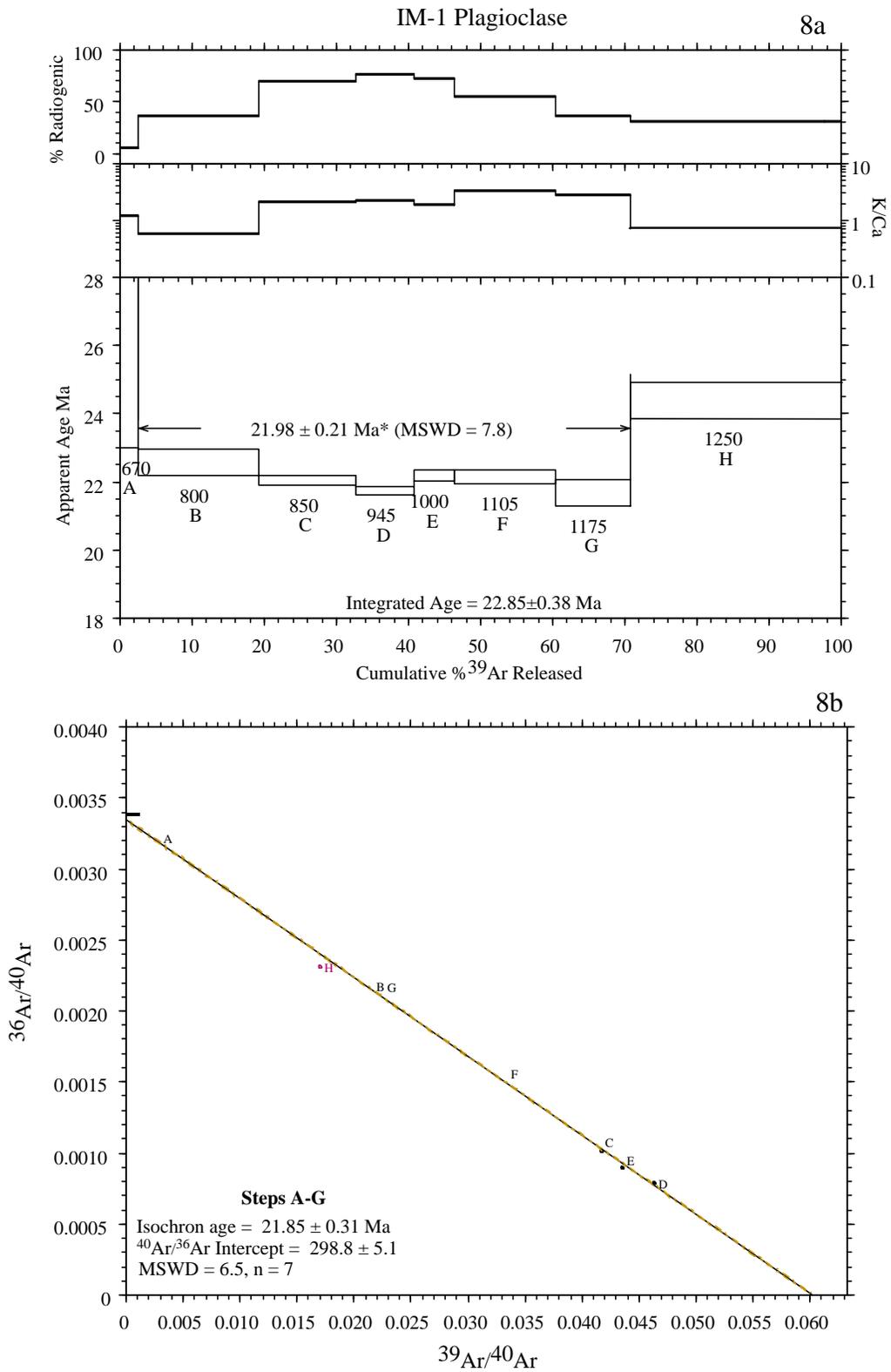


Figure 8. Age spectrum (8a) and isochron (8b) for IM-1 plagioclase. Point shown in purple not included in isochron. All errors quoted at 2 sigma.

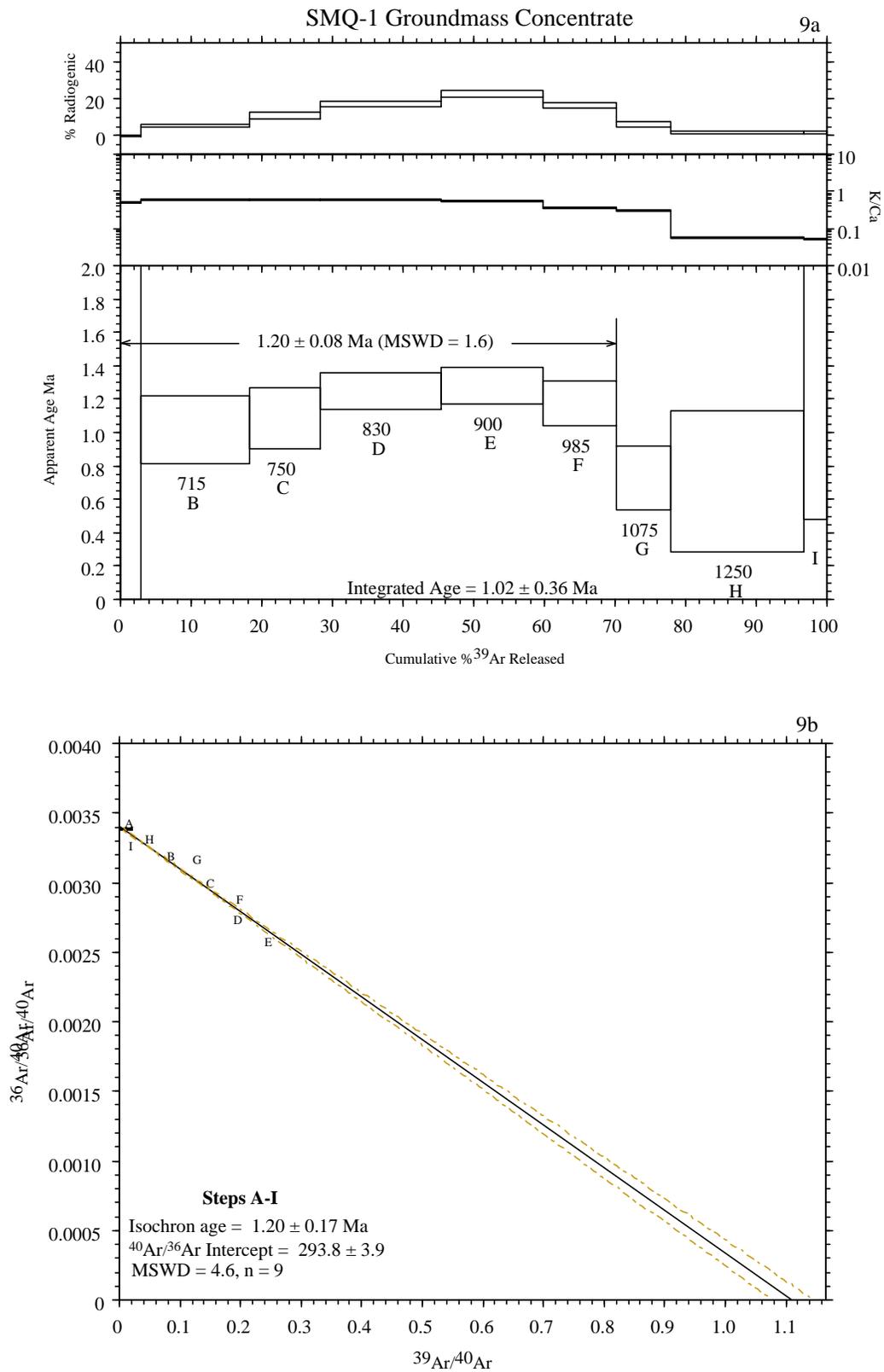


Figure 9. Age spectrum (9a) and isochron (9b) for SMQ-1 groundmass concentrate. All errors quoted at 2 sigma.

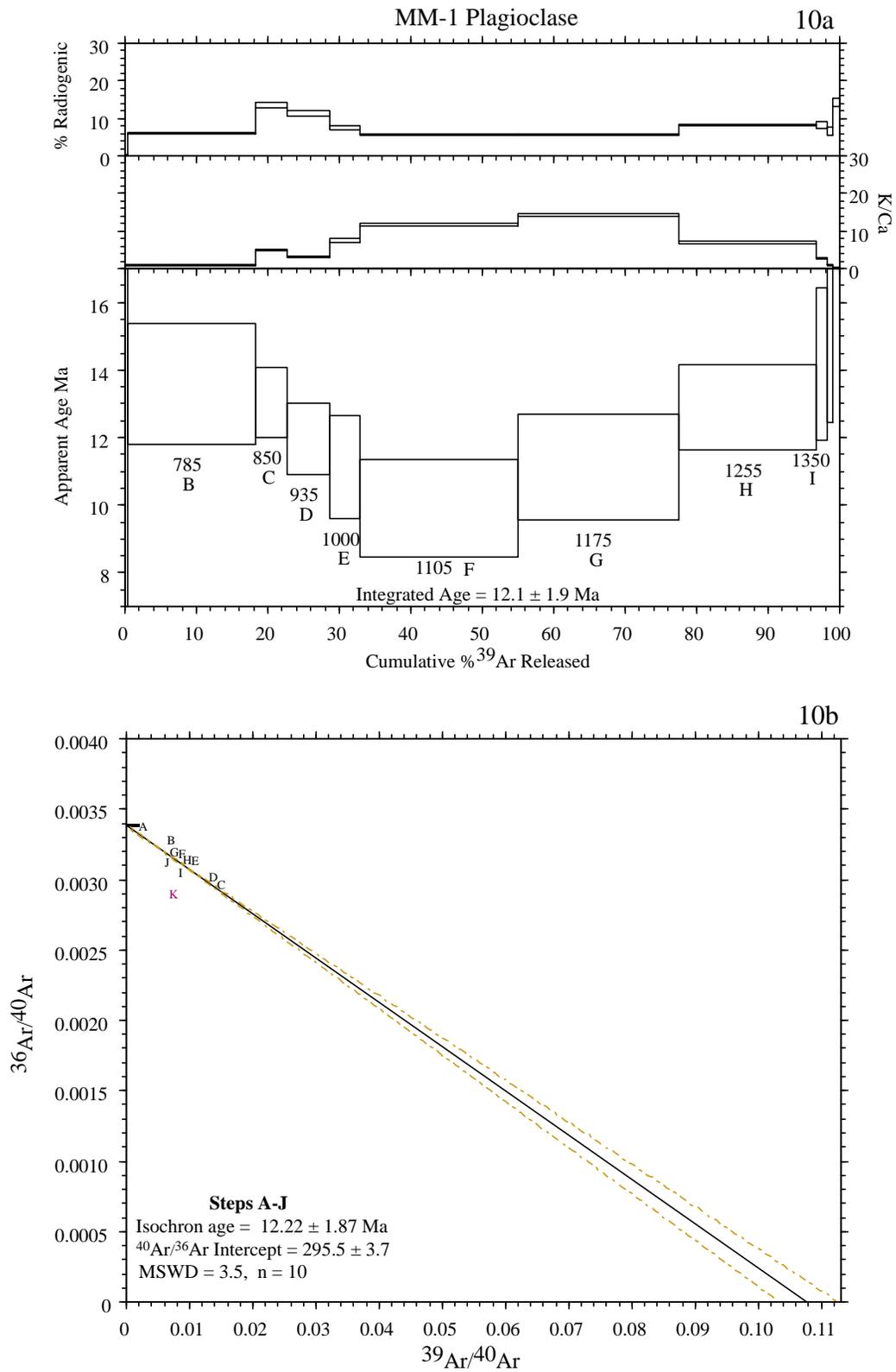


Figure 10. Age spectrum (10a) and isochron (10b) for MM-1 plagioclase. Points shown in purple not included in isochron. All errors quoted at 2 sigma.

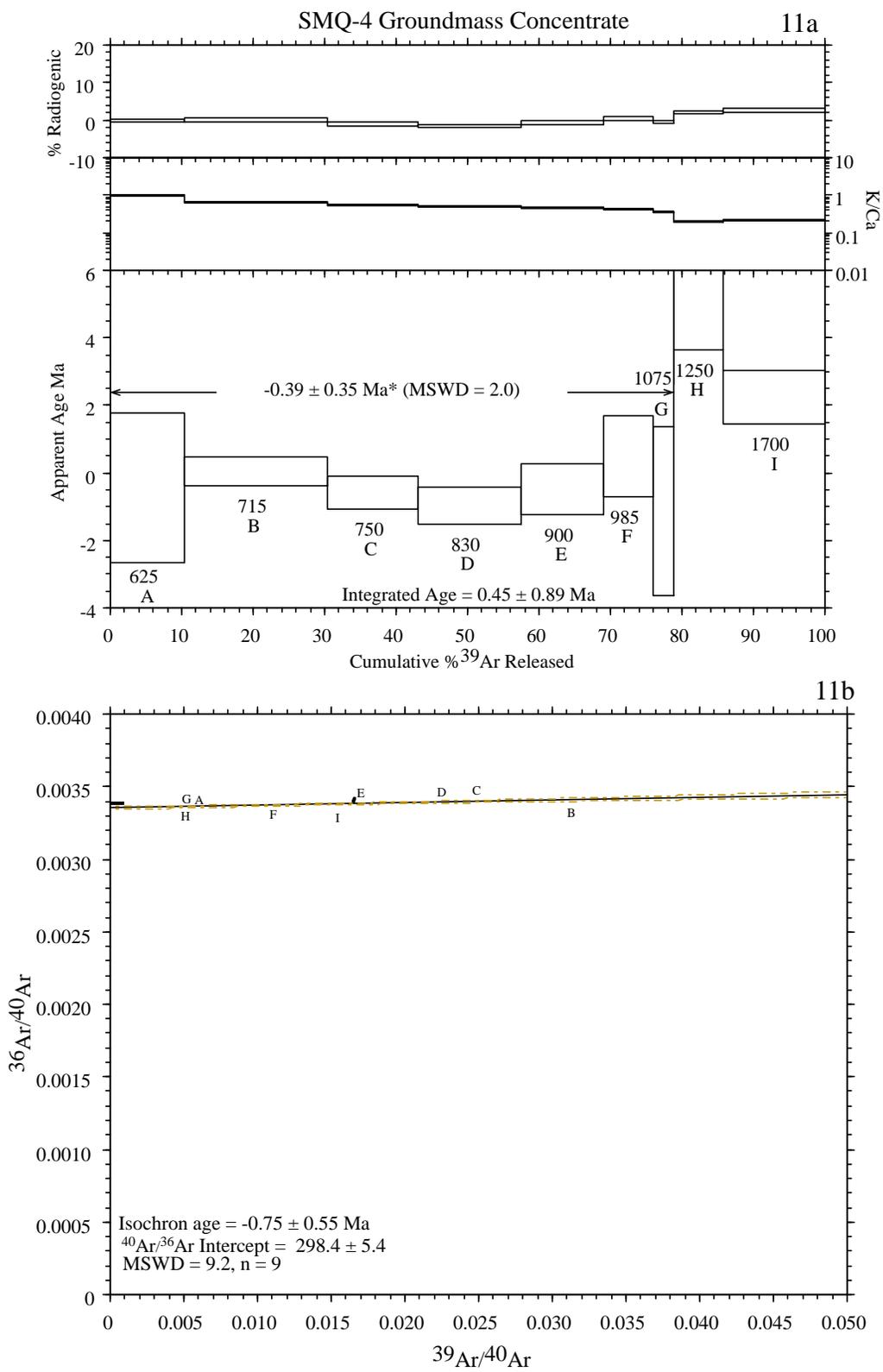


Figure 11. Age spectrum (11a) and isochron (11b) for SMQ-4 groundmass concentrate. All errors quoted at 2 sigma.

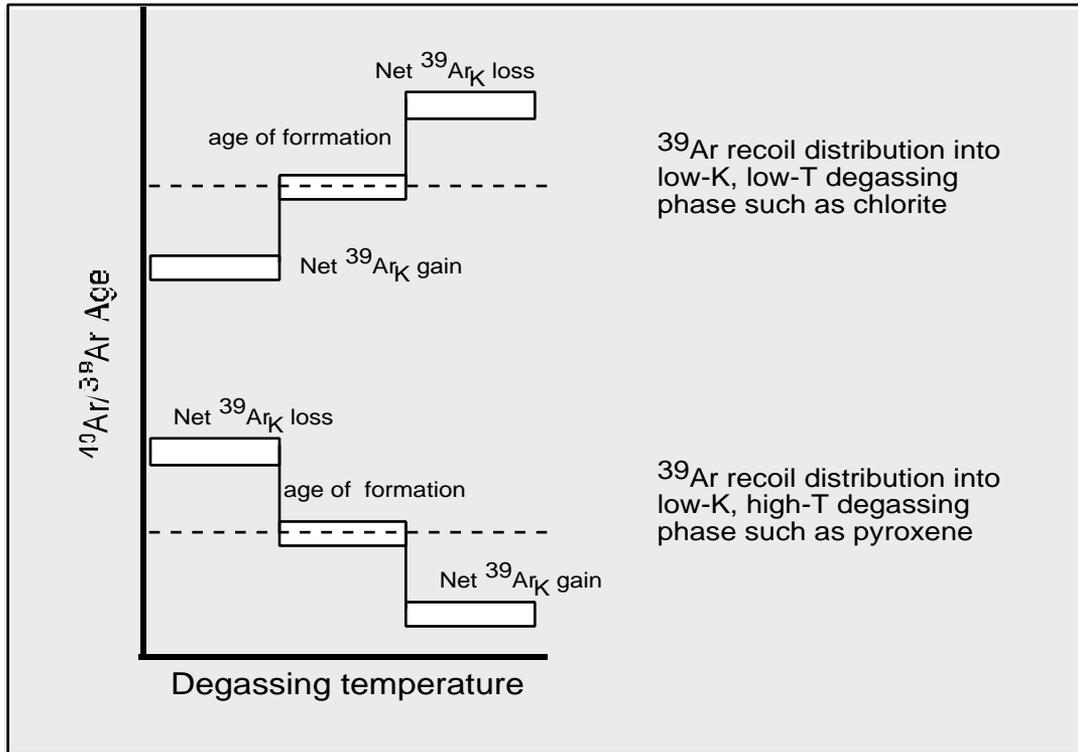


Figure 12. Cartoon showing potential effects of  $^{39}\text{Ar}$  recoil on an otherwise flat age spectrum.

### Summary of Young Pine Valley Mountain Samples

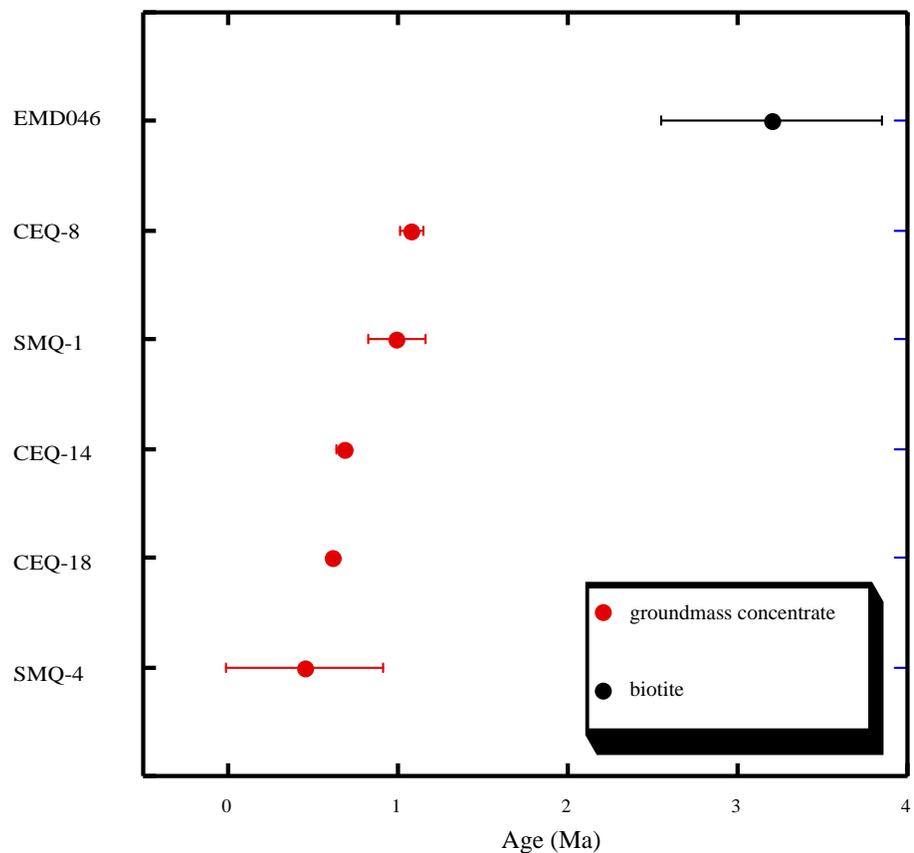


Figure 13. Summary of the Pine Valley Mountain samples with apparent ages less than 4 Ma.

### Summary of 12-23 Ma Pine Valley Mountain Samples

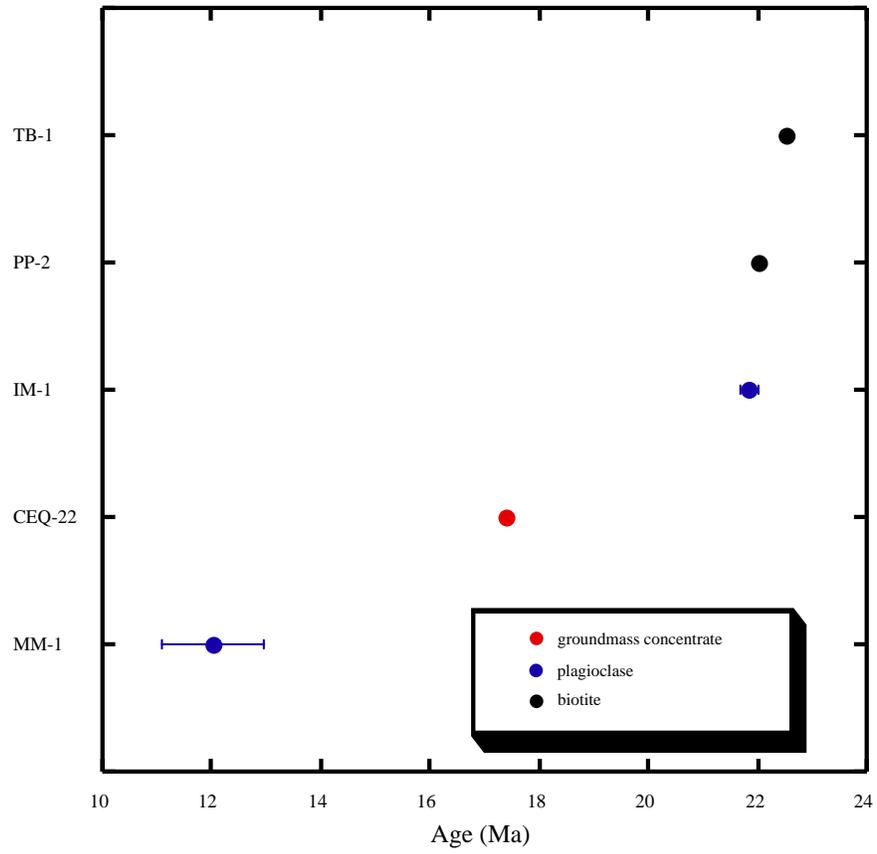


Figure 14. Summary of the Pine Valley Mountain samples with apparent ages between 12 and 23 Ma.

**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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## **$^{40}\text{Ar}/^{39}\text{Ar}$ 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  $^{40}\text{Ar}^*$  and  $^{40}\text{K}$ ) limits the precision of the K-Ar method to approximately 1% and also, the technique provides limited potential to evaluate underlying assumptions. In the  $^{40}\text{Ar}/^{39}\text{Ar}$  variant of the K-Ar technique, a sample is irradiated with fast neutrons thereby converting  $^{39}\text{K}$  to  $^{39}\text{Ar}$  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  $^{40}\text{Ar}/^{39}\text{Ar}$  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  $^{40}\text{Ar}/^{39}\text{Ar}$  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  $^{40}\text{Ar}/^{39}\text{Ar}$  method requires comparison of the measured  $^{40}\text{Ar}/^{39}\text{Ar}$  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  $^{40}\text{Ar}/^{39}\text{Ar}$  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<sub>2</sub> 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<sub>2</sub> 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 getting 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<sub>2</sub>, respectively. Typically, 3-5 individual pieces of the salt or glass are fused with the CO<sub>2</sub> 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 % <sup>39</sup>Ar<sub>K</sub> 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 <sup>40</sup>K) has the modern day atmospheric <sup>40</sup>Ar/<sup>36</sup>Ar value of 295.5. Additional parameters for each heating step are often plotted versus the cumulative % <sup>39</sup>Ar<sub>K</sub> released. These auxiliary parameters can aid age spectra interpretation and may include radiogenic yield (percent of <sup>40</sup>Ar which is not atmospheric), K/Ca (determined from measured Ca-derived <sup>37</sup>Ar and K-derived <sup>39</sup>Ar) and/or K/Cl (determined from measured Cl-derived <sup>38</sup>Ar and K-derived <sup>39</sup>Ar). Incremental heating analysis is often effective at revealing complex argon systematics related to excess argon, alteration, contamination, <sup>39</sup>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  $^{40}\text{Ar}$  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  $^{36}\text{Ar}/^{40}\text{Ar}$  ratio is plotted versus the  $^{39}\text{Ar}/^{40}\text{Ar}$  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  $^{40}\text{Ar}^*/^{39}\text{Ar}_K$  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 performed 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  $^{39}\text{Ar}$  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 ( $1\sigma$ ) for each age analysis is generally shown by the horizontal lines in the moles of  $^{39}\text{Ar}$  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,  $^{40}\text{Ar}/^{36}\text{Ar}_i$  values and MSWD values are calculated from the regression results obtained by the York (1969) method.

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