

$^{40}\text{Ar}/^{39}\text{Ar}$  geochronology results for the Hurricane, Kolob  
Reservoir, and The Guardian Angels quadrangles, Utah

by

New Mexico Geochronology Research Laboratory and Utah  
Geological Survey

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paginated.

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.**

<b>Sample #</b>	<b>7.5' quadrangle</b>	<b>Latitude</b>	<b>Longitude</b>	<b>Reference</b>
ZP-0405	The Guardian Angels	37.374	113.060	Willis and Hylland (2002)
ZP-0502	The Guardian Angels	37.338	113.117	Willis and Hylland (2002)
ZP-0503	The Guardian Angels	37.350	113.107	Willis and Hylland (2002)
ZP-0601	Kolob Reservoir	37° 23' 14"	113° 02' 19"	Biek (2006)
ZP-0602	Kolob Reservoir	37° 23' 08"	113° 02' 20"	Biek (2006)
ZP-0605	The Guardian Angels	37.298	113.099	Willis and Hylland (2002)
ZP-0606	The Guardian Angels	37.279	113.096	Willis and Hylland (2002)
ZP-0607	The Guardian Angels	37.280	113.096	Willis and Hylland (2002)
VR123-11	Hurricane	37.126	113.364	Biek (2003)
VR113-4	Hurricane	37.243	113.297	Biek (2003)

## **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., 2003, Geologic map of the Hurricane quadrangle, Washington County, Utah: Utah Geological Survey Map 187, 61 p., 2 plates, scale 1:24,000.

—2006, Geologic map of the Kolob Reservoir quadrangle, Washington and Iron Counties, Utah: Utah Geological Survey Map 220, 2 plates, scale 1:24,000.

Willis, G.C., and Hylland, M.D., 2002, Interim geologic map of The Guardian Angels quadrangle, Washington County, Utah: Utah Geological Survey Open-File Report 395, 27 p., scale 1:24,000.

**Report On  $^{40}\text{Ar}/^{39}\text{Ar}$  Analysis for:**

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## Introduction

Two basalts from the Hurricane Quadrangle and eight basalts from Zion National Park, Utah were provided by Robert Biek for  $^{40}\text{Ar}/^{39}\text{Ar}$  age determinations.

## $^{40}\text{Ar}/^{39}\text{Ar}$ 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. Details of the overall operation of the New Mexico Geochronology Research Laboratory are provided in the Appendix.

All eight of the ZP and both of the VR groundmass samples yield age spectra resulting in plateau conforming to the criteria of Fleck et al (1977)(figures 1-5). With the exception of ZP-0605, all of the plateaus achieved at least 85% of the total cumulative  $^{39}\text{Ar}_K$  released; the plateau for ZP-0605 achieved only 65% of the total cumulative  $^{39}\text{Ar}_K$  released (figure 3b). Despite the comparatively low percent  $^{39}\text{Ar}$  plateau for ZP-0605, the plateau age is not significantly different than the total gas age, indicating that argon loss is not a considerable affect. Radiogenic yield varies among the samples. In half of the samples, the radiogenic yield is less than 10% (table 2). The K/Ca and Cl/K ratios (plotted on each age spectrum) do vary within each sample but are not consistently correlative to any increases or decreases in apparent age.

Isochron analyses were conducted on both groundmass separates (figures 6-10). Isochron ages for all of the groundmass samples are concordant with the plateau ages and yield trapped initial  $^{40}\text{Ar}/^{36}\text{Ar}$  compositions within error of the atmospheric value (295.5).

## **Discussion**

Plateau ages range from  $1.14 \pm 0.14$  to  $0.22 \pm 0.03$  Ma, as summarized in table 3 and figure 11. All of the plateau ages produced in this study are interpreted to be the age of eruption. Precision on the individual plateau ages varies from 2% to 14%. These values are not atypical for basalts in this age range. The lower precision ages are from samples with lower radiogenic yields, possibly reflecting alteration or hydration of groundmass glass or other phases. Alteration increases the quantity of atmospheric argon associated with a sample, partially or completely overwhelming the radiogenic argon. Additional sample preparation and/or analytical manipulation are generally not effective for improving the quality of data from low radiogenic yield basalts.

## References Cited

- Deino, A., and Potts, R., 1990. Single-Crystal  $^{40}\text{Ar}/^{39}\text{Ar}$  dating of the Olorgesailie Formation, Southern Kenya Rift, *J. Geophys. Res.*, 95, 8453-8470.
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Table 1. Analytical methods.

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**Sample preparation and irradiation:**

Basalt samples provided by James Luhr.

Samples packaged and irradiated in machined Al discs for 0.5 hours in L67 position, Ford Research Reactor, University of Michigan.

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.

Samples step-heated in Mo double-vacuum resistance furnace. Heating duration 10 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 filament operated at ~2000°C.

**Analytical parameters:**

Electron multiplier sensitivity averaged  $1 \times 10^{16}$  moles/pA.

Total system blank and background for the furnace averaged 4200, 10, 9, 5,  $15 \times 10^{18}$  moles at masses 40, 39, 38, 37, and 36, respectively for temperatures <1300°C.

J-factors determined to a precision of  $\pm 0.1\%$  by CO<sub>2</sub> laser-fusion of 4 single crystals from each of 3 radial positions around the irradiation tray.

Correction factors for interfering nuclear reactions were determined using K-glass and CaF<sub>2</sub> and are as follows:

$$({}^{40}\text{Ar}/{}^{39}\text{Ar})_k = 0.025 \pm 0.005; ({}^{36}\text{Ar}/{}^{37}\text{Ar})_c = 0.00026 \pm 0.00002; \text{ and } ({}^{39}\text{Ar}/{}^{37}\text{Ar})_c = 0.00070 \pm 0.00005.$$

**Age calculations:**

Total gas ages and errors calculated by weighting individual steps by the fraction of <sup>39</sup>Ar released.

Plateau definition: 3 or more analytically indistinguishable contiguous steps comprising at least 50% of the total <sup>39</sup>Ar (Fleck et al., 1977).

Preferred age calculated for indicated steps when the sample does not meet plateau criteria.

Plateau or preferred ages calculated by weighting each step by the inverse of the variance.

Plateau and preferred age errors calculated using the method of (Taylor, 1982).

MSWD values are calculated for n-1 degrees of freedom for plateau and preferred ages.

Isochron ages, <sup>40</sup>Ar/<sup>36</sup>Ar, 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  $\pm 2\sigma$ , unless otherwise noted.

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Table 2. <sup>40</sup>Ar/<sup>39</sup>Ar analytical data for ZP- and VR- groundmass concentrates.

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	Cl/K (x 10 <sup>-3</sup> )	<sup>40</sup> Ar* (%)	<sup>39</sup> Ar (%)	Age (Ma)	±2σ (Ma)
<b>ZP-0405</b> , E4:90, 142.76mg groundmass concentrate, J=0.000079363±0.11%, D=1.00513±0.00109, NM-90, Lab#=9235-01											
A	400	4228.4	0.2515	14129.5	0.826	2.0	72.3	1.3	2.6	8	59
B	625	745.1	0.6446	2487.5	2.77	0.79	46.2	1.4	11.3	1.4	2.2
C	700	397.1	1.271	1309.0	2.99	0.40	23.9	2.6	20.7	1.48	0.76
D	800	192.2	1.179	620.1	5.58	0.43	7.0	4.7	38.3	1.30	0.24
E	875	126.1	0.8025	403.2	5.60	0.64	3.7	5.6	55.9	1.00	0.14
F	975	123.6	0.9191	389.3	5.99	0.56	4.2	7.0	74.8	1.24	0.13
G	1075	229.0	1.041	751.2	3.07	0.49	9.3	3.1	84.5	1.01	0.31
H	1250	389.0	4.787	1272.6	3.38	0.11	13.3	3.4	95.1	1.92	0.66
I	1650	387.4	7.601	1255.0	1.55	0.067	10.7	4.4	100.0	2.47	0.67
<b>total gas age</b>			n=9		31.8	0.51				1.5	2.0
<b>plateau</b>			n=5		steps C-G	23.2	0.52		73.2	1.14	0.14
<b>ZP-0502</b> , F2:90, 152.38mg groundmass concentrate, J=0.000078508±0.11%, D=1.00513±0.00109, NM-90, Lab#=9237-01											
A	400	695.8	0.2939	2312.0	0.716	1.7	74.8	1.8	2.0	1.8	1.3
B	625	57.96	0.3298	192.1	5.67	1.5	45.9	2.1	18.1	0.17	0.08
C	700	31.86	0.5645	102.4	6.09	0.90	32.3	5.1	35.3	0.23	0.04
D	800	29.76	0.9078	95.90	8.23	0.56	19.3	4.9	58.6	0.21	0.04
E	875	33.74	1.225	108.9	5.47	0.42	14.3	4.8	74.0	0.23	0.06
F	975	44.30	1.547	145.0	3.28	0.33	16.9	3.5	83.3	0.22	0.08
G	1075	54.84	1.594	178.3	0.751	0.32	21.6	4.1	85.4	0.32	0.21
H	1250	73.24	5.437	242.4	2.79	0.094	18.9	2.8	93.3	0.29	0.13
I	1650	79.23	7.256	259.7	2.37	0.070	18.7	3.8	100.0	0.43	0.13
<b>total gas age</b>			n=9		35.4	0.68				0.27	0.10
<b>plateau</b>			n=7		steps B-H	32.3	0.71		91.3	0.22	0.03
<b>ZP-0503</b> , E2:90, 176.78mg groundmass concentrate, J=0.00007857±0.11%, D=1.00513±0.00109, NM-90, Lab#=9234-01											
A	400	695.2	0.5289	2332.2	0.799	0.96	76.0	0.9	2.3	0.9	2.9
B	625	30.23	0.6955	95.54	7.90	0.73	56.5	6.7	25.4	0.29	0.04
C	700	18.80	0.8219	56.80	6.62	0.62	36.1	10.9	44.7	0.29	0.03
D	800	26.56	1.033	83.29	6.27	0.49	25.6	7.5	63.0	0.28	0.03
E	875	30.16	1.398	95.73	2.78	0.36	16.1	6.5	71.1	0.28	0.06
F	975	39.67	1.771	127.5	1.34	0.29	16.7	5.3	75.0	0.30	0.10
G	1075	34.87	1.963	113.1	0.641	0.26	18.6	4.5	76.9	0.22	0.19
H	1250	58.49	6.748	191.1	7.48	0.076	19.0	4.3	98.8	0.36	0.07
I	1650	72.49	6.170	230.3	0.427	0.083	17.1	6.8	100.0	0.70	0.31
<b>total gas age</b>			n=9		34.3	0.47				0.32	0.12
<b>plateau</b>			n=7		steps B-H	33.0	0.46		96.4	0.29	0.02
<b>ZP-0601</b> , E6:90, 159.90mg groundmass concentrate, J=0.000079097±0.11%, D=1.00513±0.00109, NM-90, Lab#=9236-01											
A	400	1774.1	0.5335	5890.8	0.230	0.96	125.9	1.9	0.8	4.8	7.6
B	625	121.0	1.408	397.2	1.35	0.36	33.6	3.1	5.2	0.53	0.23
C	700	36.08	1.977	97.59	2.25	0.26	15.9	20.4	12.5	1.05	0.08
D	800	17.69	1.389	34.83	5.41	0.37	7.4	42.3	30.3	1.07	0.03
E	875	13.86	0.9948	22.60	5.52	0.51	4.0	52.2	48.3	1.03	0.03
F	975	15.50	0.8726	29.12	6.63	0.58	4.7	44.7	70.0	0.99	0.03
G	1075	36.32	0.8414	99.18	3.94	0.61	13.5	19.4	82.9	1.01	0.06
H	1250	72.34	2.134	221.3	2.97	0.24	16.4	9.8	92.7	1.01	0.11
I	1650	87.33	7.705	274.2	2.24	0.066	19.3	7.9	100.0	0.98	0.14
<b>total gas age</b>			n=9		30.6	0.43				1.03	0.12
<b>plateau</b>			n=7		steps C-I	29.0	0.43		94.8	1.02	0.03

Table 2.  $^{40}\text{Ar}/^{39}\text{Ar}$  analytical data for ZP- and VR- groundmass concentrates.

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	Cl/K ( $\times 10^{-3}$ )	$^{40}\text{Ar}^*$ (%)	$^{39}\text{Ar}$ (%)	Age (Ma)	$\pm 2\sigma$ (Ma)
<b>ZP-0602</b> , G2:90, 143.32mg groundmass concentrate, $J=0.000077653\pm 0.12\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9240-01											
A	400	1268.5	0.8623	4224.8	0.069	0.59	189.2	1.6	0.3	3	11
B	625	136.2	1.479	449.4	0.425	0.34	26.4	2.6	2.0	0.50	0.49
C	700	34.52	2.198	88.98	0.777	0.23	4.9	24.3	5.3	1.17	0.18
D	800	15.98	1.674	27.08	3.12	0.30	2.3	50.6	18.2	1.13	0.04
E	875	11.28	1.332	12.50	4.22	0.38	1.5	67.9	35.7	1.07	0.03
F	975	10.53	1.081	9.612	6.40	0.47	2.0	73.6	62.2	1.09	0.02
G	1075	11.89	1.010	14.83	4.53	0.51	5.3	63.6	81.0	1.06	0.03
H	1250	24.32	1.265	57.01	2.68	0.40	13.6	31.0	92.1	1.06	0.06
I	1650	79.76	9.108	247.3	1.90	0.056	31.8	9.2	100.0	1.04	0.13
<b>total gas age</b>			n=9		24.1	0.39				1.08	0.09
<b>plateau</b>			n=7		steps C-I	23.6	0.39		98.0	1.08	0.02
<b>ZP-0605</b> , F6:90, 163.18mg groundmass concentrate, $J=0.000078227\pm 0.12\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9239-01											
A	400	372.1	0.5623	1266.3	0.329	0.91	91.4	-0.5	1.6	-0.3	1.1
B	625	138.4	1.004	468.7	3.21	0.51	58.1	-0.1	17.4	-0.01	0.18
C	700	78.95	1.619	264.4	3.54	0.32	47.2	1.2	34.7	0.13	0.12
D	800	41.95	2.161	134.2	5.11	0.24	28.7	5.8	59.8	0.34	0.07
E	875	17.71	2.042	53.37	3.37	0.25	8.2	11.7	76.4	0.29	0.05
F	975	15.86	1.981	47.61	2.33	0.26	5.3	12.1	87.8	0.27	0.06
G	1075	33.55	2.514	105.2	0.842	0.20	15.3	7.8	92.0	0.37	0.16
H	1250	57.90	13.72	191.1	0.710	0.037	21.7	4.3	95.4	0.35	0.24
I	1650	47.16	25.16	156.9	0.928	0.020	20.4	5.8	100.0	0.39	0.17
<b>total gas age</b>			n=9		20.4	0.29				0.23	0.12
<b>plateau</b>			n=6		steps D-I	13.3	0.22		65.3	0.31	0.04
<b>ZP-0606</b> , D6:90, 164.27mg groundmass concentrate, $J=0.000079722\pm 0.11\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9233-01											
A	400	1469.5	0.4525	4932.4	0.381	1.1	109.3	0.8	0.9	1.7	9.9
B	625	50.76	1.040	166.3	2.42	0.49	63.0	3.3	6.4	0.24	0.08
C	700	24.06	1.179	75.86	3.29	0.43	25.8	7.1	14.0	0.25	0.05
D	800	13.14	0.7135	38.43	8.37	0.72	7.2	13.8	33.1	0.26	0.02
E	875	8.530	0.4514	22.64	7.50	1.1	3.9	21.7	50.3	0.27	0.02
F	975	7.882	0.4396	20.56	9.03	1.2	4.4	23.0	71.0	0.26	0.02
G	1075	14.64	0.6222	43.73	3.96	0.82	7.4	11.9	80.1	0.25	0.04
H	1250	26.59	2.944	83.56	2.16	0.17	8.6	7.9	85.0	0.30	0.07
I	1650	27.07	3.772	85.51	6.54	0.14	9.4	7.6	100.0	0.30	0.04
<b>total gas age</b>			n=9		43.7	0.74				0.28	0.12
<b>plateau</b>			n=8		steps B-I	43.3	0.74		99.1	0.26	0.01
<b>ZP-0607</b> , F4:90, 156.47mg groundmass concentrate, $J=0.000078648\pm 0.11\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9238-01											
A	400	816.6	0.3078	2737.8	0.792	1.7	131.7	0.9	1.8	1.1	1.6
B	625	46.48	0.4191	151.0	5.95	1.2	71.2	4.0	15.0	0.27	0.07
C	700	25.17	0.7198	79.28	5.48	0.71	48.5	7.0	27.1	0.25	0.04
D	800	29.54	0.8074	93.34	9.48	0.63	18.8	6.8	48.2	0.28	0.04
E	875	26.71	0.6495	83.98	7.85	0.79	5.1	7.2	65.6	0.27	0.04
F	975	26.26	0.5929	83.82	6.50	0.86	4.2	5.8	80.1	0.21	0.04
G	1075	33.07	0.7970	105.7	2.01	0.64	7.2	5.7	84.5	0.27	0.09
H	1250	57.02	4.005	186.2	1.95	0.13	9.6	4.0	88.9	0.32	0.11
I	1650	77.67	4.431	254.8	5.01	0.12	9.7	3.5	100.0	0.38	0.12
<b>total gas age</b>			n=9		45.0	0.72				0.29	0.09
<b>plateau</b>			n=7		steps B-H	39.2	0.78		87.1	0.26	0.03

Table 2.  $^{40}\text{Ar}/^{39}\text{Ar}$  analytical data for ZP- and VR- groundmass concentrates.

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	Cl/K ( $\times 10^{-3}$ )	$^{40}\text{Ar}^*$ (%)	$^{39}\text{Ar}$ (%)	Age (Ma)	$\pm 2\sigma$ (Ma)
<b>VR-12311</b> , G4:90, 160.20mg groundmass concentrate, $J=0.000077877\pm 0.12\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9241-01											
A	450	951.8	1.607	3185.5	0.111	0.32	87.0	1.1	0.7	1.5	8.4
B	625	83.52	4.565	261.9	0.411	0.11	15.2	7.7	3.3	0.91	0.23
C	700	25.33	4.249	62.14	0.805	0.12	5.1	28.7	8.3	1.02	0.09
D	800	14.61	2.982	25.08	2.77	0.17	6.0	50.7	25.7	1.04	0.03
E	875	11.80	1.738	15.34	2.80	0.29	7.5	62.5	43.2	1.04	0.03
F	975	12.96	1.037	18.88	3.25	0.49	12.8	57.4	63.5	1.04	0.02
G	1075	18.91	0.6597	39.48	2.16	0.77	18.6	38.4	77.0	1.02	0.04
H	1250	28.33	3.858	73.89	1.68	0.13	13.2	23.9	87.6	0.95	0.05
I	1650	33.66	12.68	92.85	1.99	0.040	11.8	21.3	100.0	1.02	0.05
<b>total gas age</b>			n=9		16.0	0.32				1.02	0.10
<b>plateau</b>			n=8		steps B-I	15.9	0.32		99.3	1.03	0.02
<b>VR-113-4</b> , G6:90, 177.20mg groundmass concentrate, $J=0.000078164\pm 0.12\%$ , $D=1.00513\pm 0.00109$ , NM-90, Lab#=9242-01											
A	450	396.4	2.046	1350.6	0.101	0.25	85.8	-0.7	0.7	-0.4	2.8
B	625	53.86	5.691	165.2	0.355	0.090	13.5	10.1	3.3	0.77	0.22
C	700	20.02	5.124	48.26	0.972	0.100	3.8	30.6	10.2	0.87	0.07
D	800	13.28	3.079	24.03	3.09	0.17	2.3	48.1	32.3	0.90	0.02
E	875	11.24	2.061	17.14	3.03	0.25	2.0	56.2	53.9	0.89	0.02
F	975	13.08	2.174	23.97	3.34	0.23	2.9	47.0	77.8	0.87	0.02
G	1075	24.10	2.512	60.40	1.46	0.20	6.1	26.6	88.2	0.91	0.06
H	1250	80.67	6.681	254.1	0.908	0.076	8.4	7.5	94.7	0.86	0.13
I	1650	132.3	27.86	435.8	0.740	0.018	7.9	4.3	100.0	0.82	0.34
<b>total gas age</b>			n=9		14.0	0.18				0.87	0.08
<b>plateau</b>			n=8		steps B-I	13.9	0.18		99.3	0.89	0.02

Isotopic ratios corrected for blank, radioactive decay, and mass discrimination, not corrected for interfering reactions. Individual analyses show analytical error only; mean age errors also include error in J and irradiation parameters. Analyses in italics are excluded from mean age calculations.

Correction factors:

$$(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.00070\pm 0.00005$$

$$(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.00026\pm 0.00002$$

$$(^{38}\text{Ar}/^{39}\text{Ar})_K = 0.0119$$

$$(^{40}\text{Ar}/^{39}\text{Ar})_K = 0.0250\pm 0.0050$$

Table 3. Summary of <sup>40</sup>Ar/<sup>39</sup>Ar results

Sample	Unit	Location	L.#	min	age analysis	n=	K/Ca	±2σ	Age	±2σ	Comments
ZP-0405		Zion National Park	9235-01	142.76mg	groundmass	5	0.5		1.14 ±	0.16	
ZP-0502		Zion National Park	9237-01	152.38mg	groundmass	7	0.7		0.22 ±	0.03	
ZP-0503		Zion National Park	9234-01	176.78mg	groundmass	7	0.5		0.29 ±	0.02	
ZP-0601		Zion National Park	9236-01	159.90mg	groundmass	7	0.4		1.02 ±	0.03	
ZP-0602		Zion National Park	9240-01	143.32mg	groundmass	7	0.4		1.08 ±	0.02	
ZP-0605		Zion National Park	9239-01	163.18mg	groundmass	6	0.2		0.31 ±	0.04	
ZP-0606		Zion National Park	9233-01	164.27mg	groundmass	8	0.7		0.26 ±	0.01	
ZP-0607		Zion National Park	9238-01	156.47mg	groundmass	7	0.8		0.26 ±	0.03	
VR123-11		Hurricane Quadrangle	9241-01	160.20mg	groundmass	8	0.3		1.03 ±	0.02	
VR113-4		Hurricane Quadrangle	9242-01	177.20mg	groundmass	8	0.2		0.89 ±	0.02	

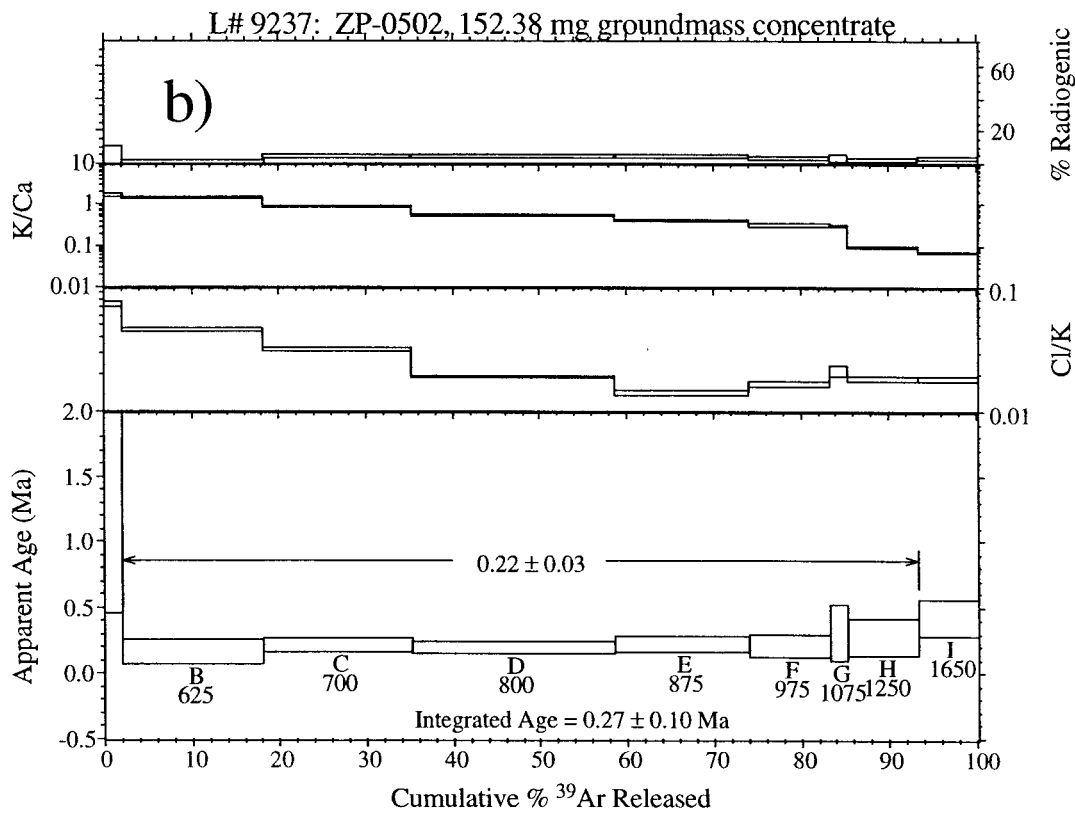
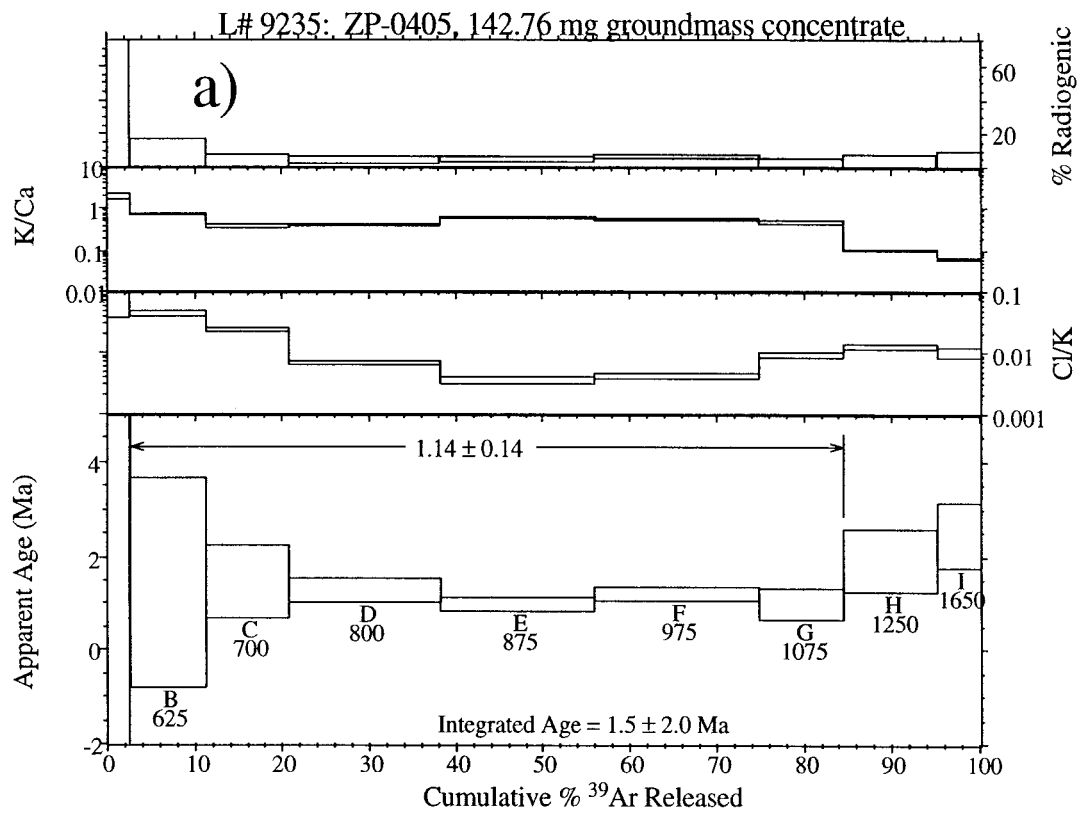


Figure 1

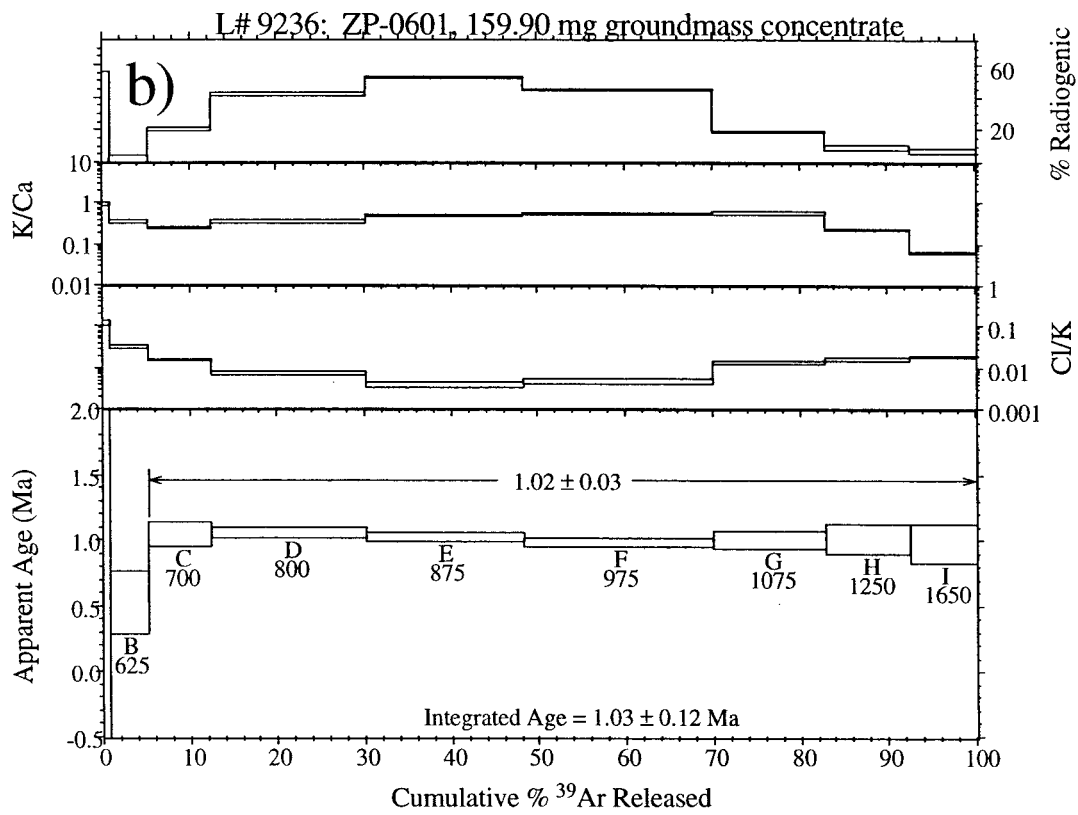
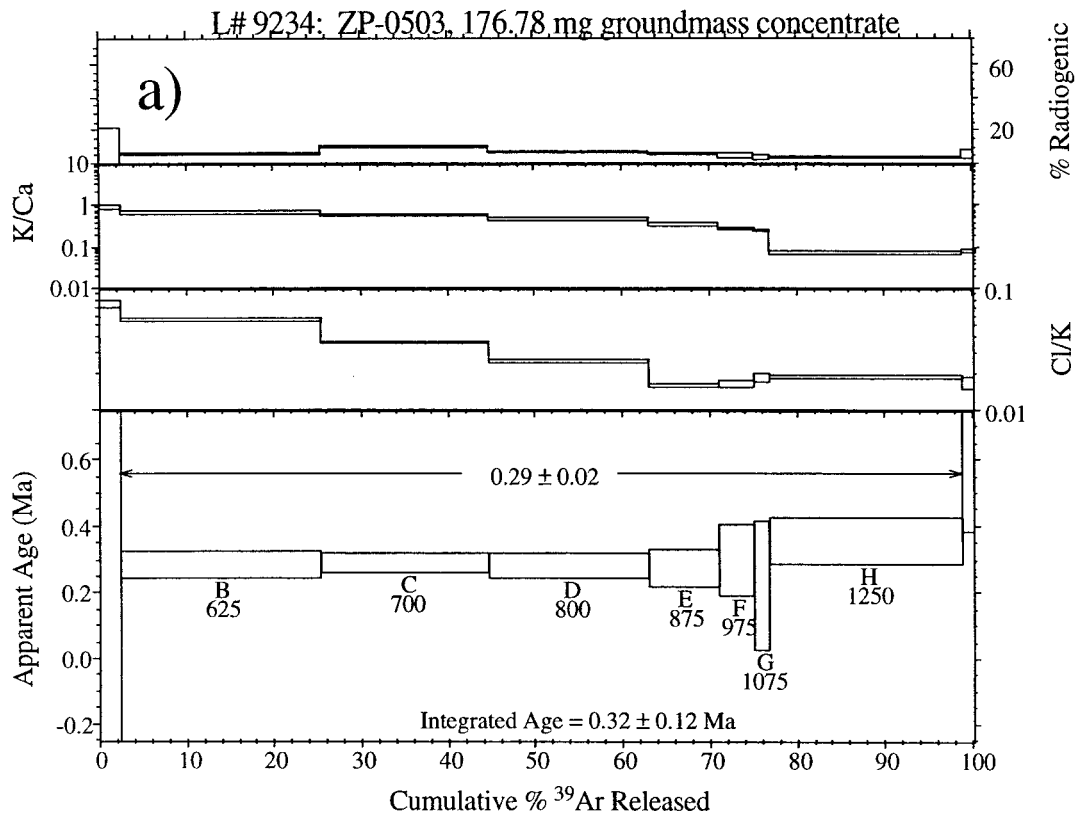


Figure 2

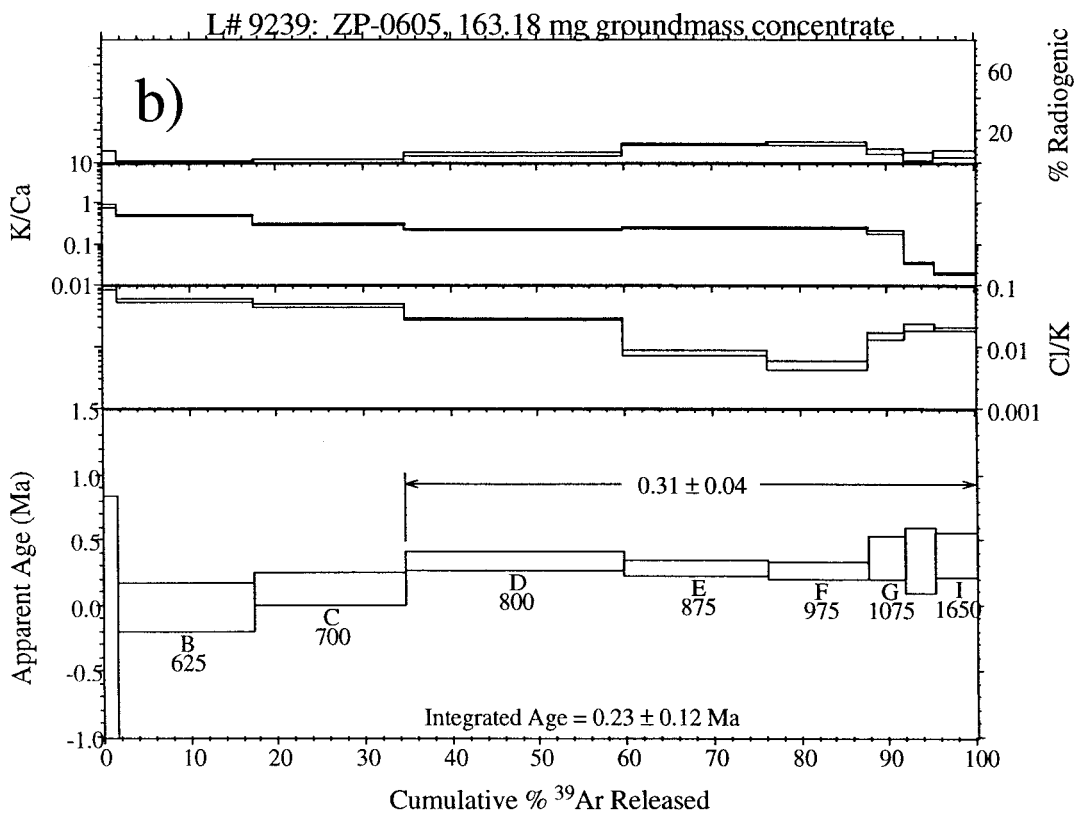
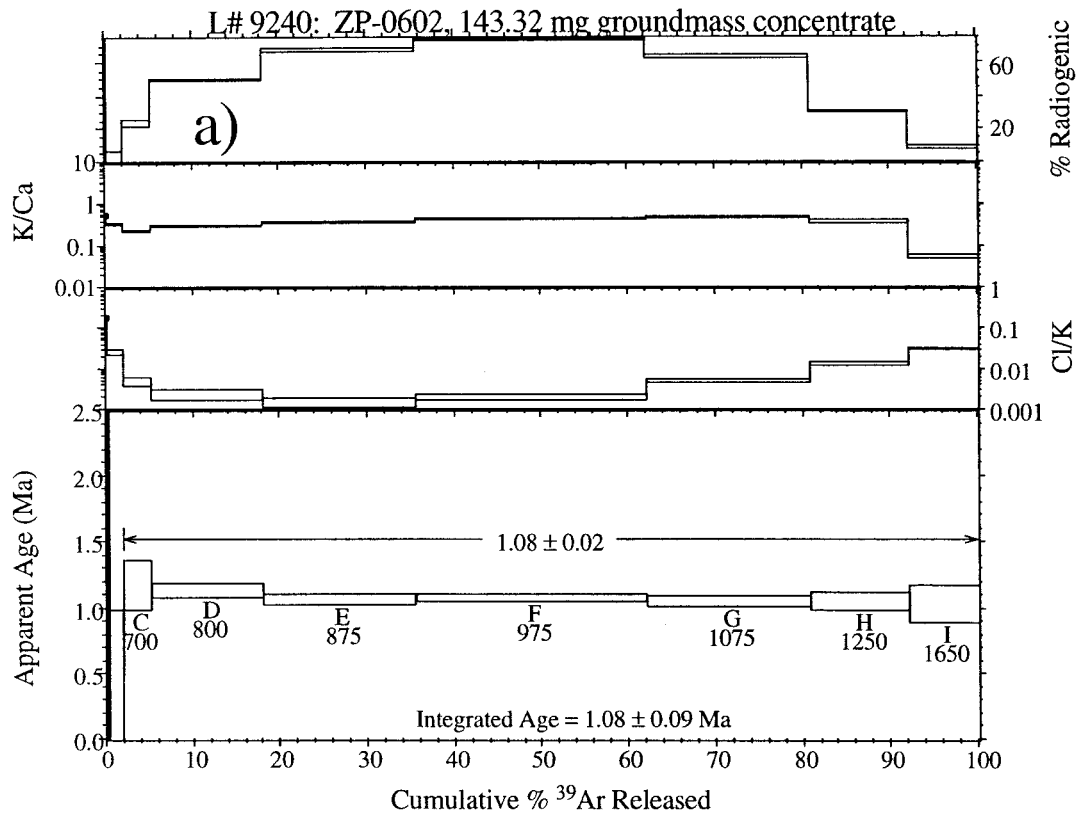


Figure 3

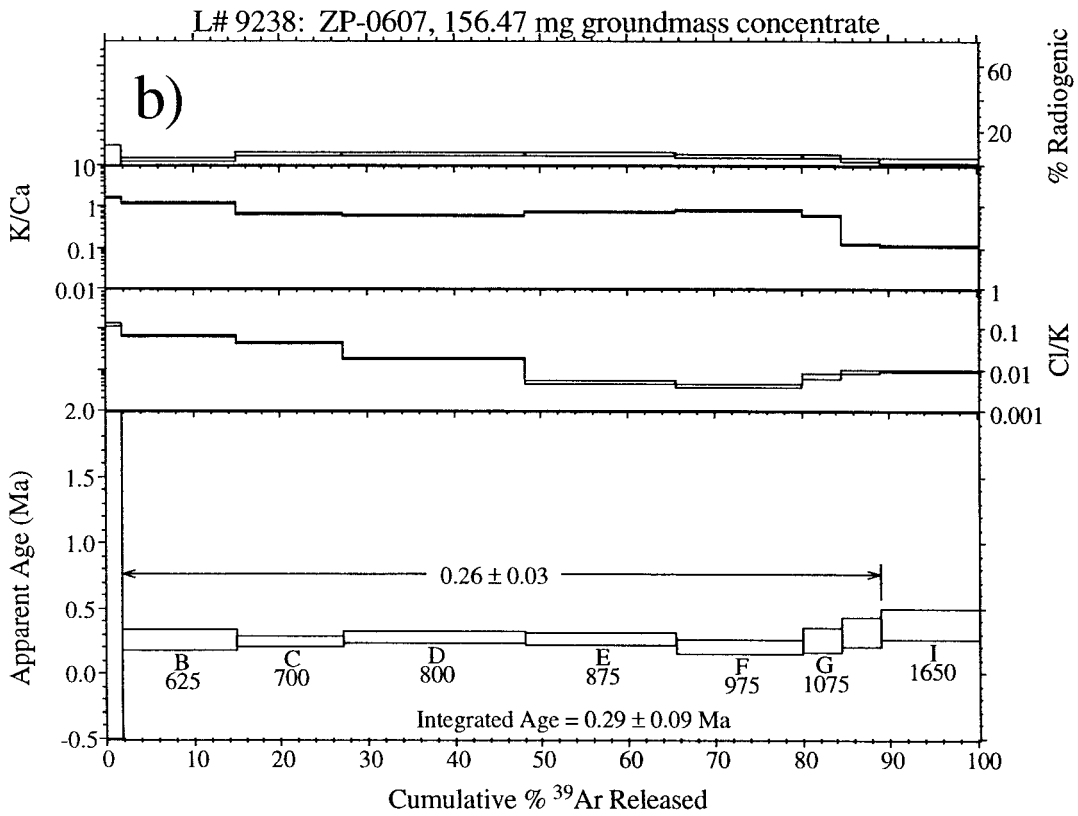
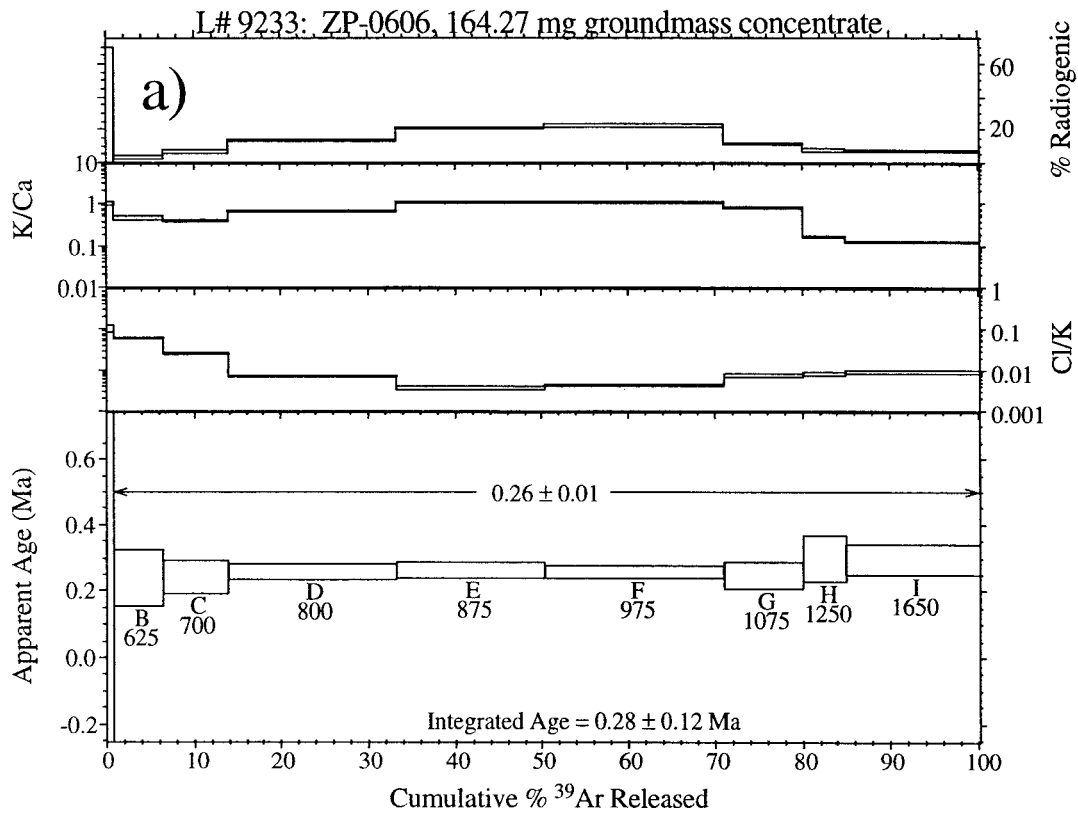


Figure 4



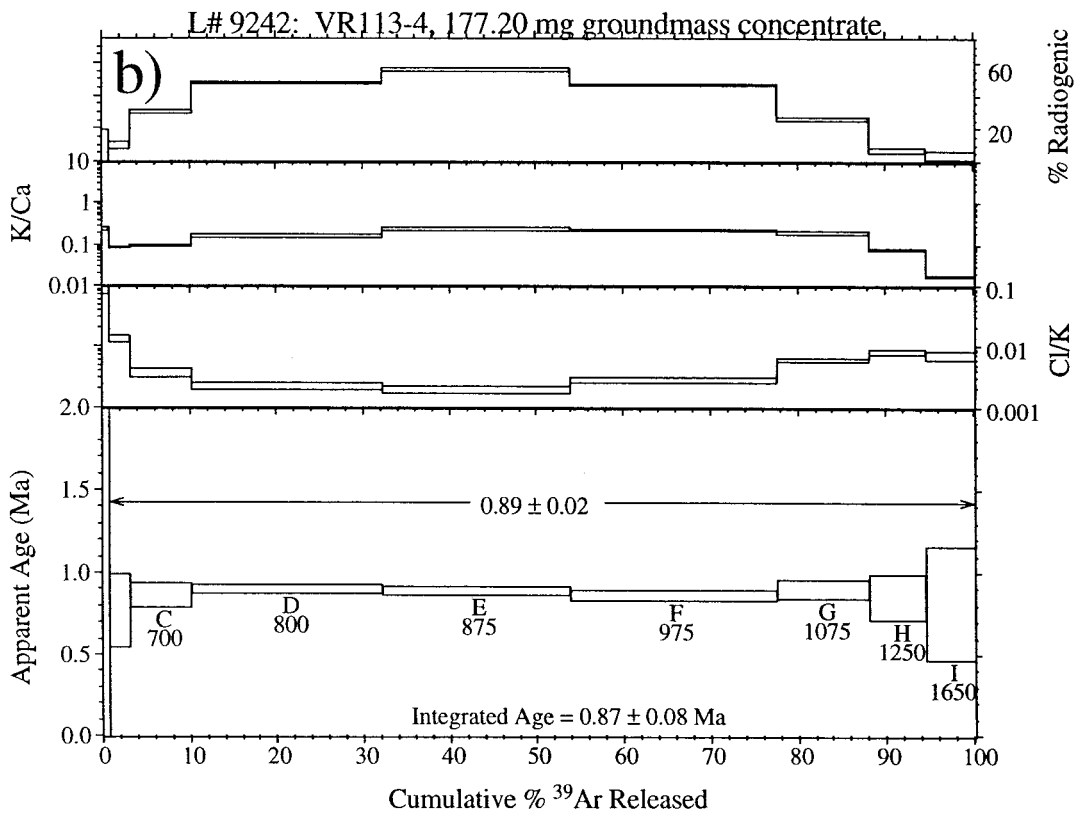
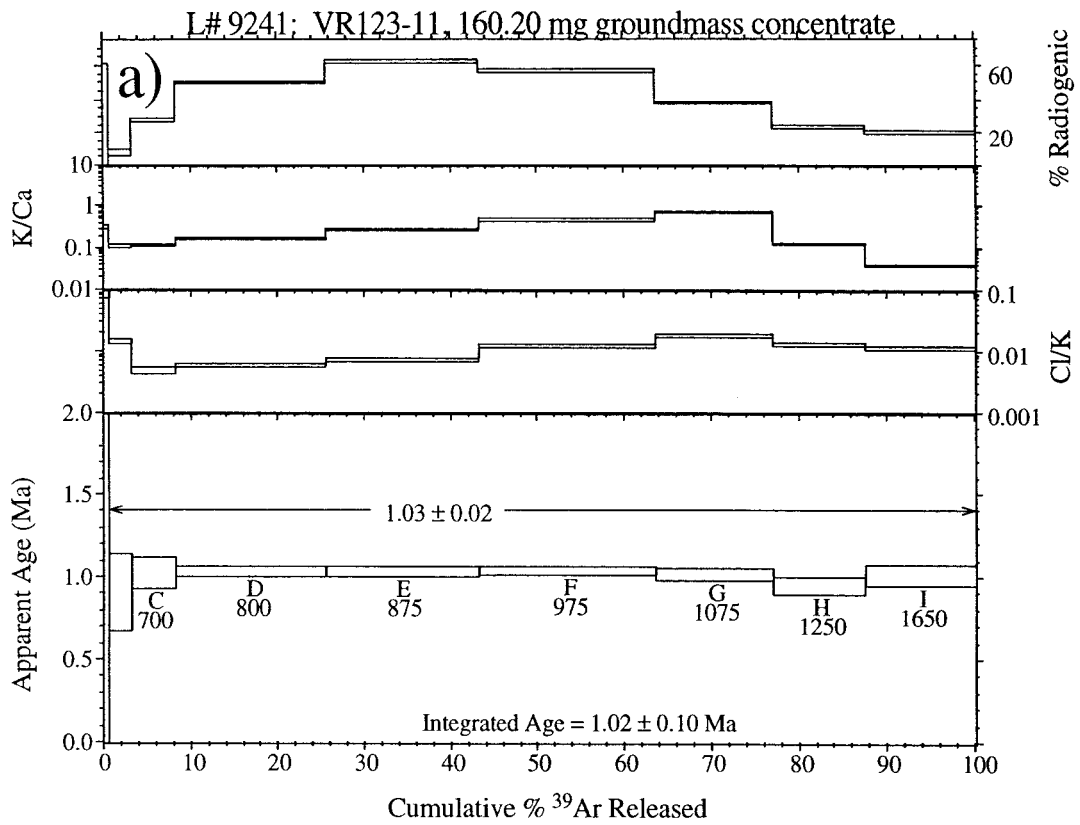


Figure 5

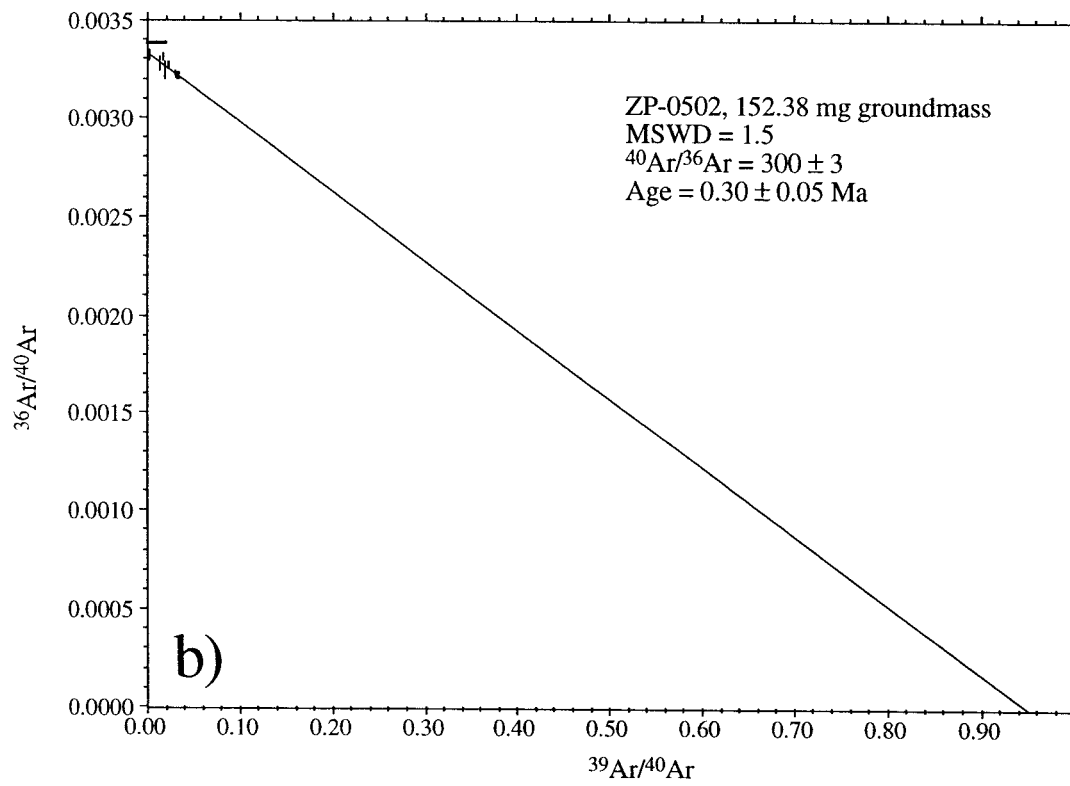
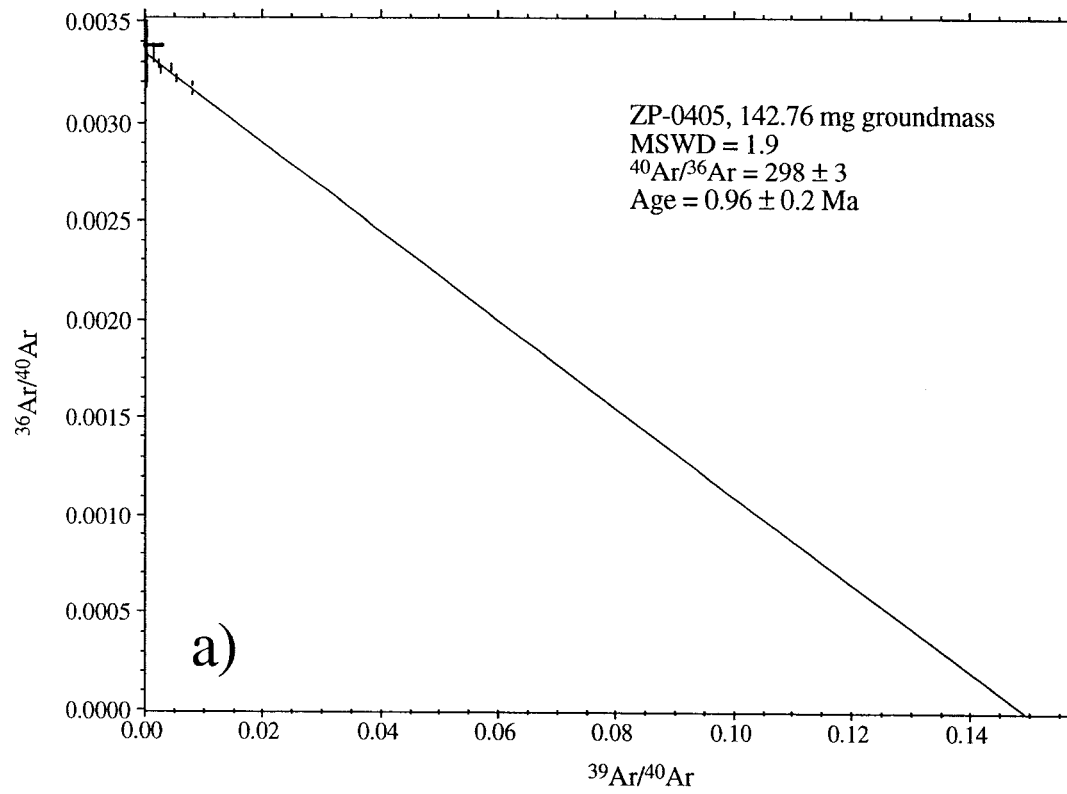


Figure 6

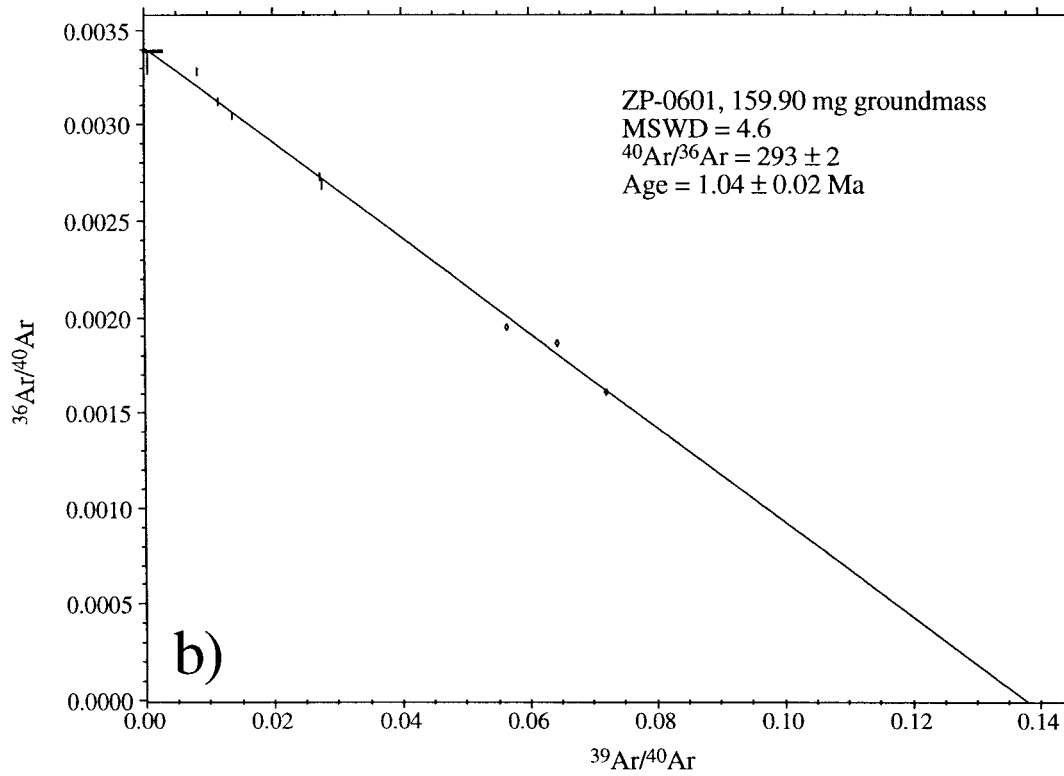
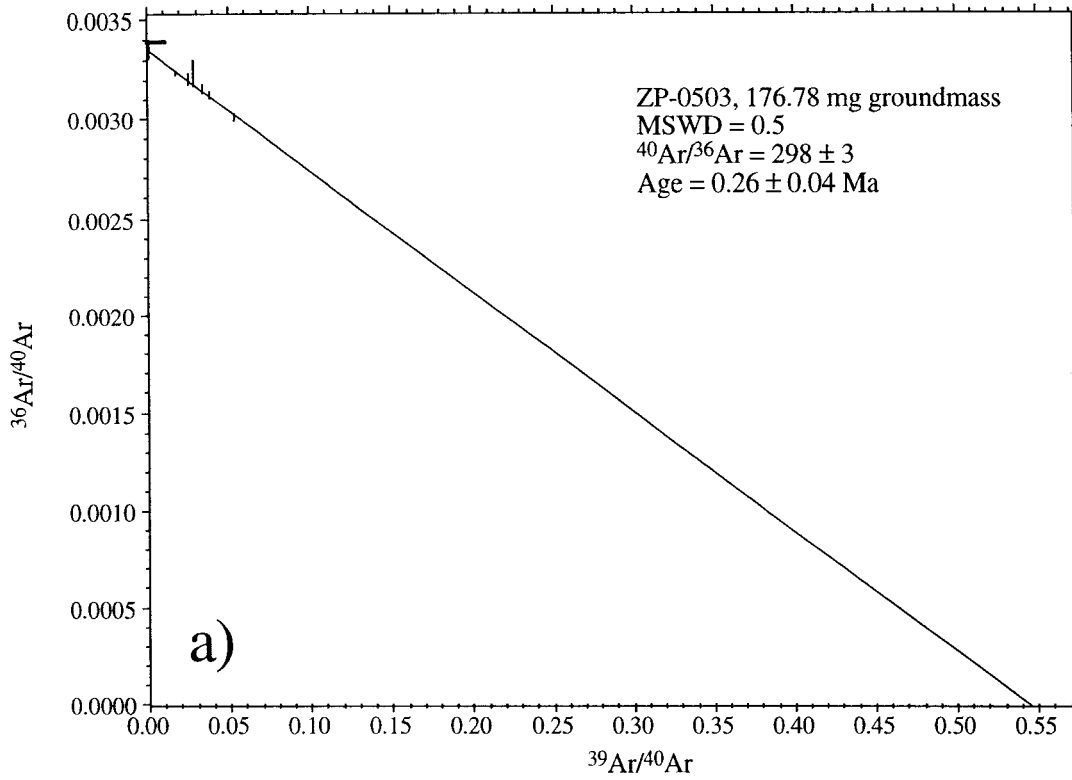


Figure 7

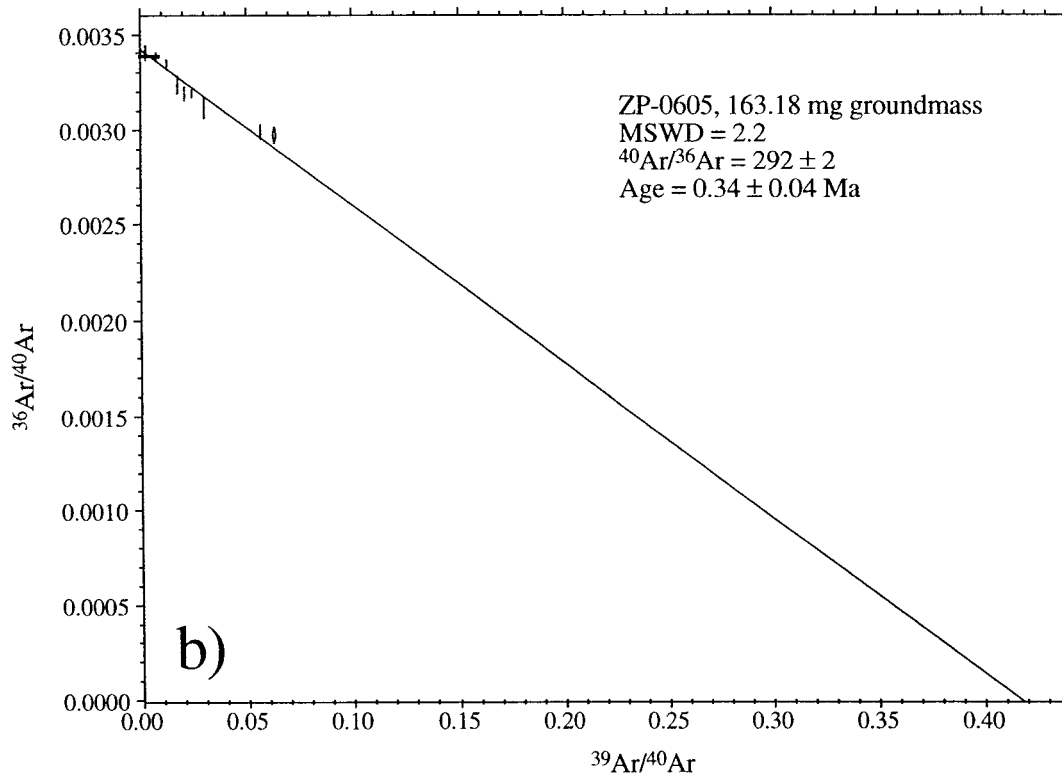
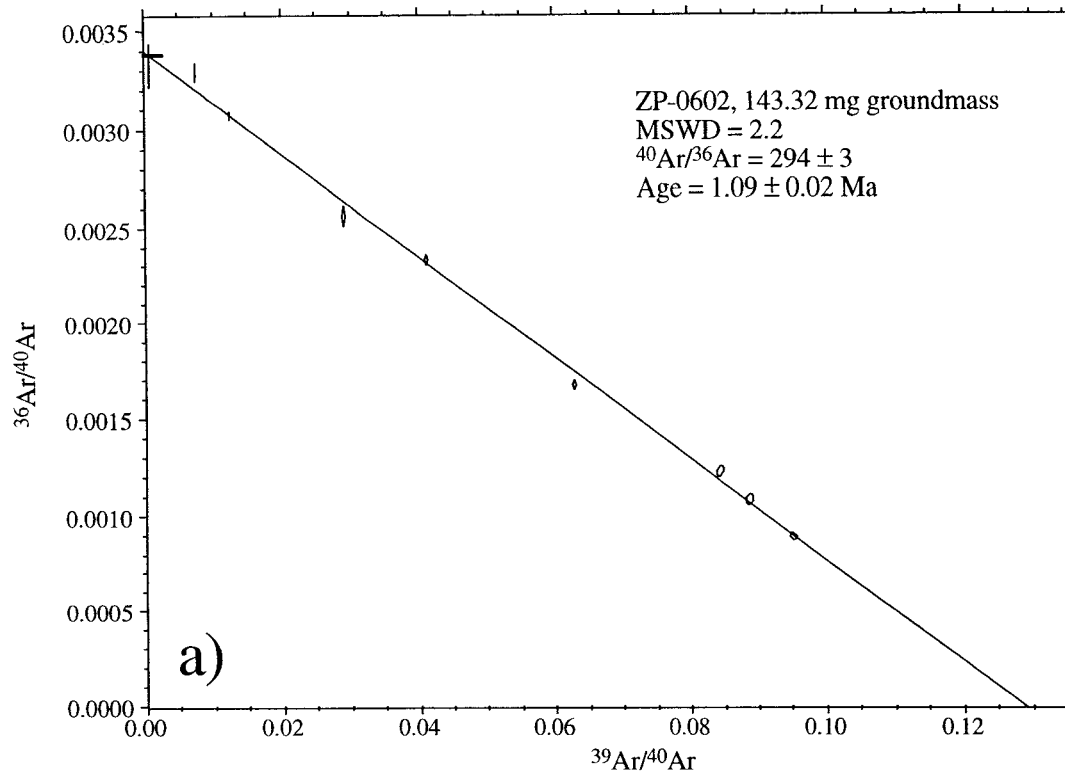


Figure 8

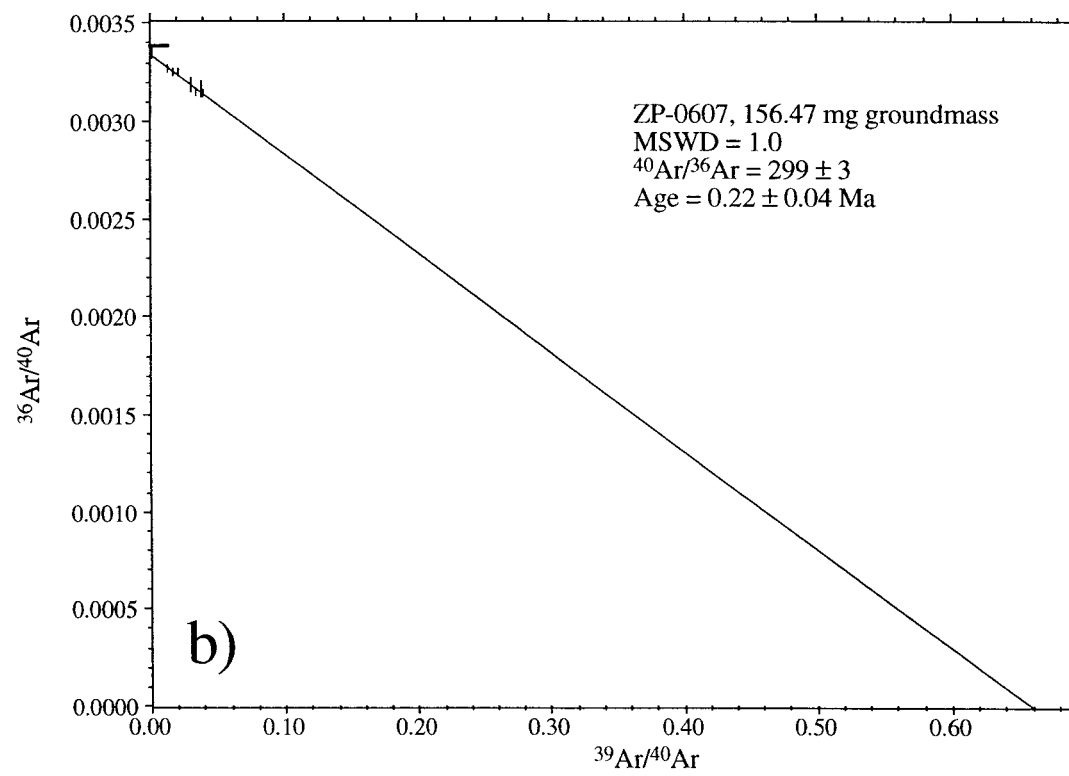
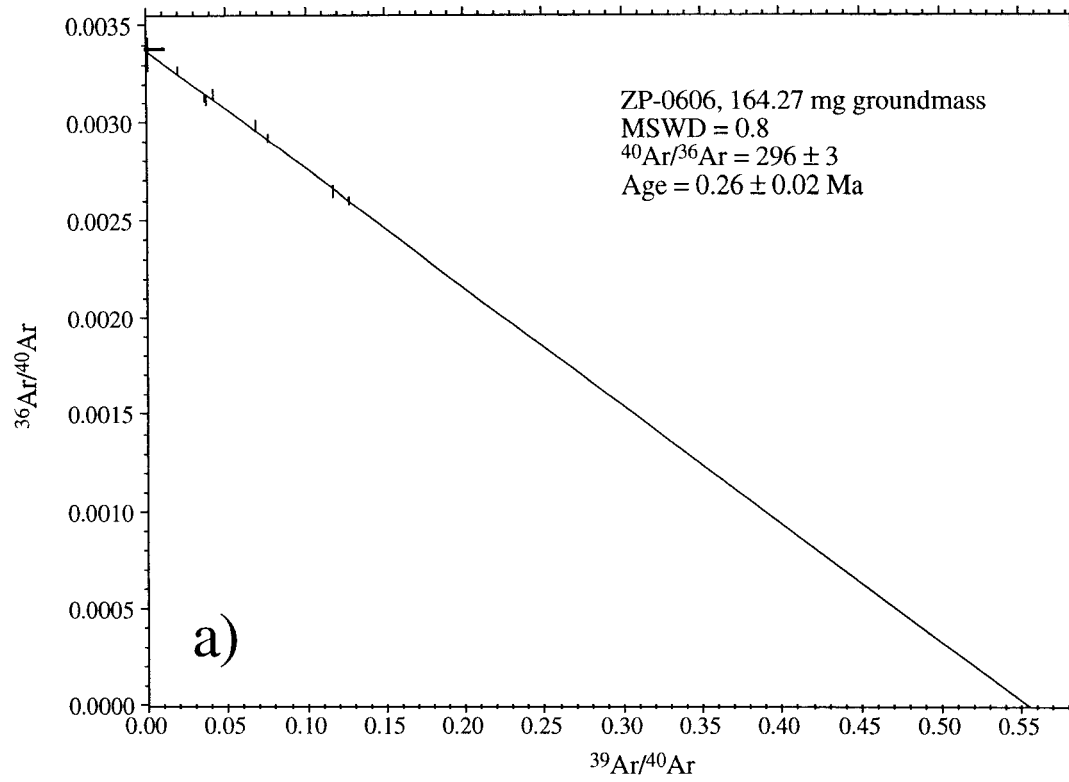


Figure 9

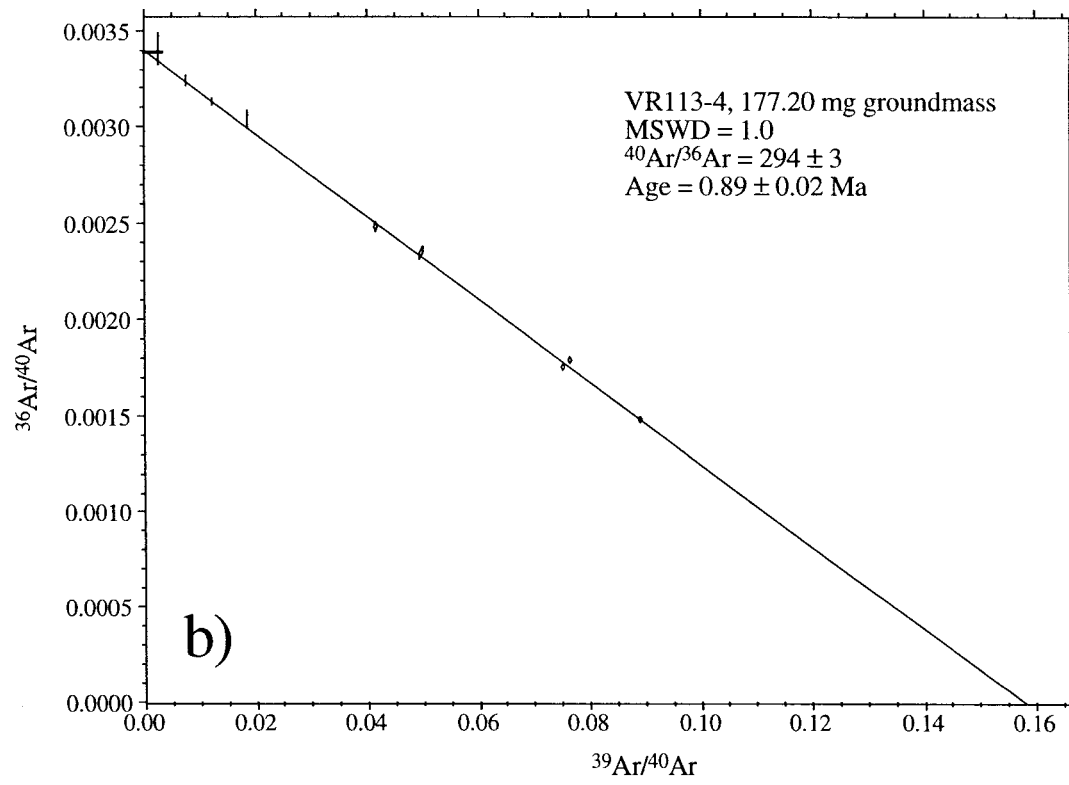
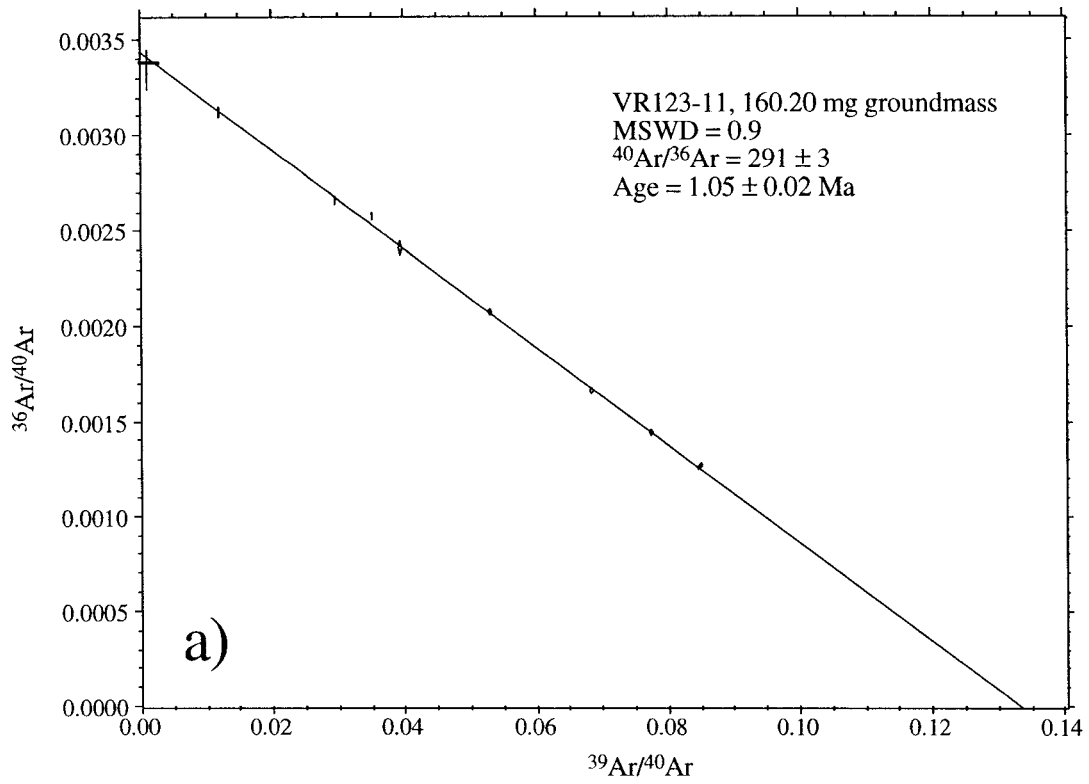


Figure 10

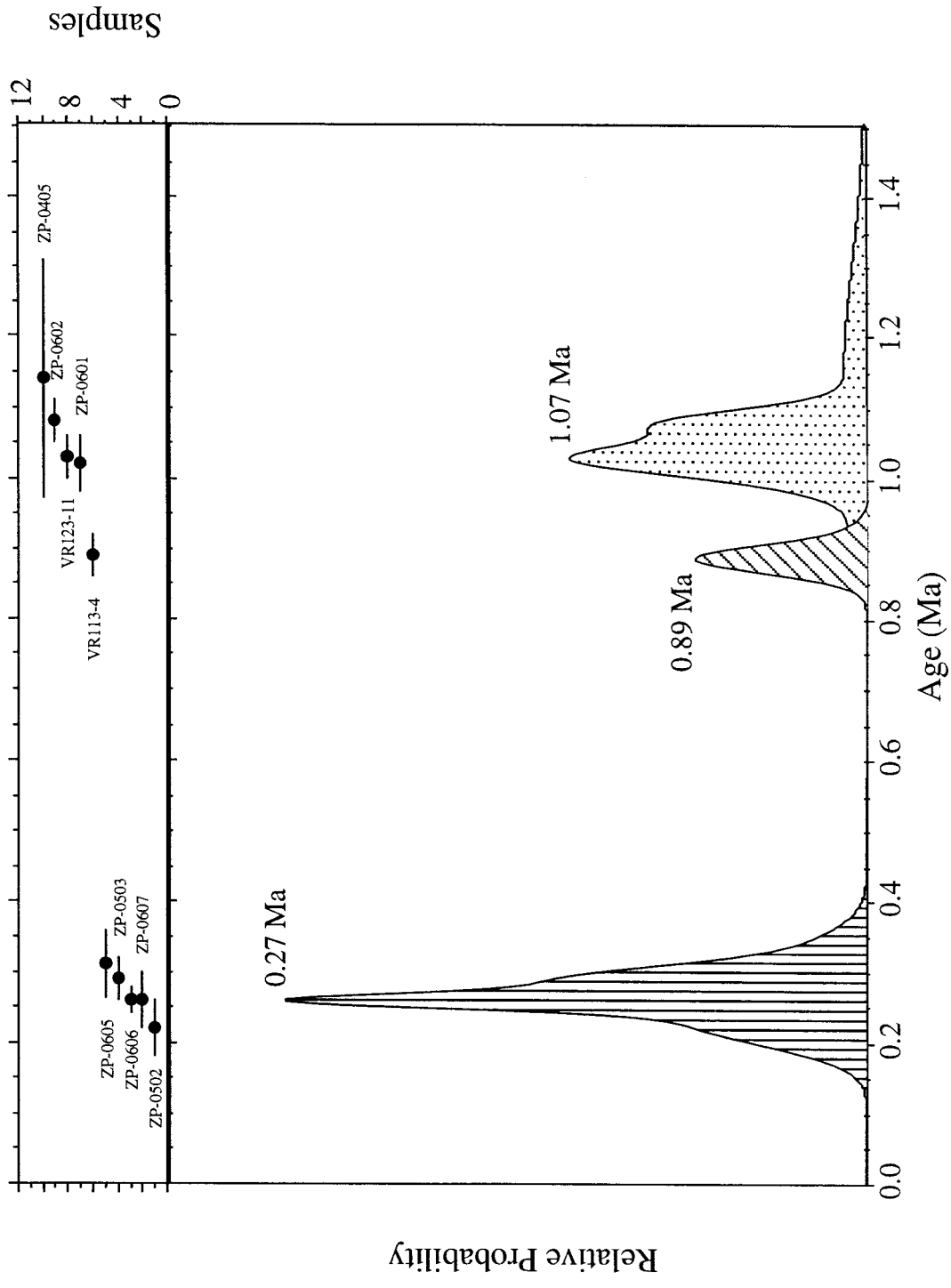


Figure 11

**New Mexico Bureau of Mines and Mineral Resources**

**Open file report #**

**Procedures of the New Mexico Geochronology Research  
Laboratory**



**For the Period June 1998 – present**

**Matthew Heizler  
William C. McIntosh  
Richard Esser  
Lisa Peters**



## **$^{40}\text{Ar}/^{39}\text{Ar}$ 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 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<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. Commonly, not all heating steps will yield identical ages, but a plateau is defined if 3 or more contiguous

heating steps, comprising at least 50% of the  $^{39}\text{Ar}$  agree within error (Fleck, et. al, 1977). In cases where a sample does not meet the rigorous plateau definition, a preferred age is calculated for the steps indicated. 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 if three or more contiguous heating steps, comprising at least 50% of the total  $^{39}\text{Ar}$  released, are analytically indistinguishable at  $2\sigma$  (Fleck et al., 1977). 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). Often a sample will not meet the defined plateau criteria. In these cases a preferred age is calculated for the steps indicated using the same equations used to determine a plateau age and error. A mean sum weighted deviates (MSWD) value is determined by dividing the Chi-squared value by  $n-1$  degrees of freedom for the plateau and preferred 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 MWSD values are calculated from the regression results obtained by the York (1969) method.

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