

$^{40}\text{Ar}/^{39}\text{Ar}$ Geochronology Results for the Tintic Mountain and Champlin Peak Quadrangles, Utah

by

Utah Geological Survey and
New Mexico Geochronology Research Laboratory

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Introduction

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 generally provide additional information such as 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 (NMGRL) 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 (N) | Longitude (W) | Reference |
|-----------------|------------------------|---------------------|----------------------|---|
| TM-5 | Tintic Mountain | 39° 45' 25" | 112° 03' 55" | Keith and others (in prep.); Moore and others (in press) |
| TM-4 | Tintic Mountain | 39° 47' 49" | 112° 01' 47" | Keith and others (in prep.) |
| TM-3 | Tintic Mountain | 39° 47' 53" | 112° 02' 16" | Keith and others (in prep.) |
| CP-3 | Champlin Peak | 39° 34' 59" | 112° 07' 30" | Hayden and others (in press) |
| TM-1 | Tintic Mountain | 39° 50' 27" | 112° 02' 18" | Keith and others (in prep.) |

Location data based on NAD27.

Disclaimer

This Open-File Report 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 NMGRL 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 Reports that Cite or Explain Samples Analyzed in this Report

Hayden, J.M., Lawton, T.F., and Clark, D.L., in press, Provisional geologic map of the Champlin Peak quadrangle, Juab and Millard Counties, Utah: Utah Geological Survey Miscellaneous Publication 07-6, scale 1:24,000.

Keith, J.D., Tingey, D.G., Hannah, J.L., Nelson, S.T., Moore, D.K., Cannan, T.M., Macbeth, A., and Pulsipher, T., in preparation, Provisional geologic map of the Tintic Mountain quadrangle, Juab County, Utah: Utah Geological Survey Miscellaneous Publication, scale 1:24,000.

Moore, D.K., Keith, J.D., Christiansen, E.H., Kim, C.S., Tingey, D.G., Nelson, S.T., and Flamm, D.S., 2007, Petrogenesis of the Oligocene East Tintic volcanic field, Utah, *in* Willis, G.C., Hylland, M.D., Clark, D.L., and Chidsey, T.C., Jr., editors, Central Utah – diverse geology of a dynamic landscape: Utah Geological Association Publication 36.

$^{40}\text{Ar}/^{39}\text{Ar}$ Geochronology Results

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Introduction

Five samples from various locations in Utah were submitted by the Utah Geological Survey. These samples were collected by Donald Clark, Project Geologist. This information is briefly summarized in Table 1.

Table 1. Brief summary of results.

| Sample | Phase | Location/Unit | Age $\pm 2\sigma$ (Ma) | Comments |
|--------|------------------------|---------------------------|------------------------|------------------------|
| TM-5 | Sanidine | Fernow Qtz Latite | 34.94 \pm 0.10 | |
| TM-4 | Biotite | Latite Ridge Latite | 34.64 \pm 0.17 | |
| TM-3 | Plagioclase | Rattlesnake Peak andesite | 35.05 \pm 0.63 | disturbed age spectrum |
| CP-3 | Groundmass concentrate | West Fork Reservoir Cgl | 37.86 \pm 0.30 | disturbed age spectrum |
| TM-1 | Groundmass concentrate | Rattlesnake Peak andesite | 38.08 \pm 0.41 | disturbed age spectrum |

⁴⁰Ar/³⁹Ar Analytical Methods and Results

Samples being prepared as groundmass concentrates were crushed and cleaned with dilute hydrochloric acid. Those being prepared as sanidine and plagioclase separates were crushed and cleaned with dilute hydrofluoric acid, while those being prepared as biotite separates were crushed and cleaned with only water. The analyzed phases were then separated 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 groundmass concentrates, biotite and plagioclase were analyzed with the furnace incremental heating age spectrum method. Sanidine was analyzed by the single-

crystal laser fusion 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 and 4.

TM-5 Weighted Mean Age=34.94±0.10 Ma n/n_{total}=13/15 MSWD=1.2

Thirteen of the fifteen analyzed TM-5 sanidine crystals yielded a near Gaussian population (Figure 1). The youngest and oldest crystals, which were among the smallest in ⁴⁰Ar signal size, were not included in the weighted mean age calculation. The radiogenic yields are all over 99% and the K/Ca values are quite consistent, with values ranging from 49.9 to 104.4.

TM-4 Weighted Mean Age=34.64±0.17 Ma n/n_{total}=9/12 MSWD=8.69

Biotite from TM-4 yielded a fairly well-behaved age spectrum (Figure 2a). The initial 3.4% of the ³⁹Ar released yields rapidly rising apparent ages, radiogenic yields and K/Ca values. The apparent ages are more consistent over the remainder of the age spectrum and all steps are used to calculate an apparent age of 34.65 ±0.17 Ma. The radiogenic yields are correlated with the apparent ages, increasing over the initial 17.9% of the ³⁹Ar released and then decreasing slightly before rising again to ~95% radiogenic and remaining constant for the rest of the age spectrum. The K/Ca values are correlated roughly to the radiogenic yields, rising over the initial 17.9%, decreasing and then rising again over the remaining 56.6% of the age spectrum. Inverse isochron analysis of steps D-L yields a ⁴⁰Ar/³⁶Ar intercept of 278±13, slightly below the atmospheric intercept of 295.5 (Figure 2b). The isochron age calculated from these points, 34.88±0.24 Ma, is within error of the weighted mean age calculated from the age spectrum.

CP-3 Weighted Mean Age=37.86±0.30 Ma n/n_{total}=5/9 MSWD=0.77

CP-3 groundmass concentrate yielded a fairly disturbed age spectrum with increasing apparent ages over the majority of the age spectrum (increasing from 28.3 Ma to 42.49 Ma, Figure 3a). A weighted mean age of 37.86±0.30 Ma is calculated from the

flattest portion of the age spectrum. The radiogenic yields rise over the initial 32.4% to a high of 44.1% radiogenic and then decline over all but the final step, which reveals a slight increase in radiogenic yield. The K/Ca values are fairly consistent varying from 0.1 to 0.88. Steps C-G were evaluated with the inverse isochron technique and were found to have a $^{40}\text{Ar}/^{36}\text{Ar}$ ratio (296 ± 6), within error of the atmospheric ratio of 295.5. The isochron age calculated from points C-G (37.8 ± 1.2 Ma) is within error of the weighted mean age calculated from the age spectrum (Figure 3b).

TM-1 Weighted Mean Age= 38.08 ± 0.41 Ma $n/n_{\text{total}}=6/9$ MSWD=3.0

TM-1 groundmass concentrate yielded an age spectrum very similar to that of CP-3 groundmass concentrate, with ages increasing from 26.00 Ma to 43.77 Ma (Figure 4a). As with CP-3, a weighted mean age of 38.08 ± 0.41 Ma, is calculated from the flattest mid-portion of the age spectrum which contains 58.9% of the ^{39}Ar released. The radiogenic yields also display a similar pattern to those from CP-3, rising from 2.7% to 52.7% radiogenic over the initial 38.4% of the ^{39}Ar released and then falling to 30.7% radiogenic. The K/Ca values are fairly consistent, varying from 0.11 to 0.83. Inverse isochron analysis of steps B-G yields an isochron age of 38.9 ± 1.7 Ma with an $^{40}\text{Ar}/^{36}\text{Ar}$ intercept of 290 ± 12 (Figure 4b).

TM-3 Weighted Mean Age= 35.05 ± 0.63 Ma $n/n_{\text{total}}=6/11$ MSWD=1.5

TM-3 plagioclase yields an age spectrum more disturbed than the previous samples with apparent ages increasing over the majority of the age spectrum (from 24.8 to 62.7 Ma, Figure 5a). A weighted mean age of 35.05 ± 0.63 Ma is calculated from the flattest mid-portion of the age spectrum. This portion of the age spectrum contains 53.7% of the ^{39}Ar released. The radiogenic yields are oscillatory but not correlated to changes in the apparent ages. The K/Ca values are fairly constant varying from 0.023 to 0.051. Points B-H were evaluated with the inverse isochron technique and revealed an isochron age of 34.8 ± 2.4 Ma. The $^{40}\text{Ar}/^{36}\text{Ar}$ intercept for these points is 294 ± 24 (Figure 5b).

Discussion

The TM-5 data are straightforward and provide a reliable eruption age for the Fernow Quartz latite. The age calculated from thirteen of the sanidine crystals analyzed for sample TM-5 (34.94 ± 0.10 Ma) is assigned as the eruption age of the Fernow Quartz Latite.

The increasing apparent ages correlated with increasing radiogenic yields revealed by the initial heating steps of the TM-4 biotite age spectrum are suggestive of alteration and accompanying Ar loss. Because the majority of the age spectrum is fairly well-behaved we feel comfortable in assigning this age (34.65 ± 0.17 Ma) as the eruption age of the Latite Ridge Latite.

The remaining age spectra reveal not only increasing apparent ages in the early heating steps but also anomalously old apparent ages in the final 20-30% of the spectra. We, therefore have less confidence in the weighted mean ages calculated from the flat mid-portions of these spectra than of that from TM-4. In addition to alteration and accompanying Ar loss, these samples have most likely been affected by one or more of the following: xenocrystic contaminants, ^{39}Ar recoil (redistribution of the ^{39}Ar created at the reactor from high K sites to low K sites), and excess Ar ($^{40}\text{Ar}/^{36}\text{Ar}$ intercept greater than the atmospheric intercept of 295.5). In cases where alteration is suspected, the oldest age is usually assigned as a minimum age. If ^{39}Ar recoil is suspected, the integrated age is usually assigned as our best estimate of the sample's age. Unfortunately, Ar loss would result in the integrated age being anomalously young. Although we can not definitively say that the mid-portions of these age spectra are unaffected by the above mentioned phenomenon, we feel that the weighted mean ages calculated from the flattest portions of the age spectra are our best estimates of the eruption ages for samples CP-3, TM-1 and TM-3 (37.86 ± 0.30 Ma, 38.03 ± 0.41 Ma, and 35.05 ± 0.63 , respectively). We do caution that our confidence in these ages is not as high as the other samples in this project.

References Cited

- Harrison, T.M., and McDougall, I. 1981. Excess ^{40}Ar in metamorphic rocks from Broken Hill, New South Wales: Implications for $^{40}\text{Ar}/^{39}\text{Ar}$ age spectra and the thermal history of the region. *Earth Planet. Sci. Lett.*, 55, 123-149.
- 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.
- Samson, S.D., and Alexander, E.C., Jr., 1987. Calibration of the interlaboratory $^{40}\text{Ar}/^{39}\text{Ar}$ dating standard, Mmhb-1, *Chem. Geol.*, 66, 27-34.
- Steiger, R.H., and Jäger, E., 1977. Subcommittee on geochronology: Convention on the use of decay constants in geo- and cosmochronology. *Earth and Planet. Sci. Lett.*, 36, 359-362.
- Taylor, J.R., 1982. *An Introduction to Error Analysis: The Study of Uncertainties in Physical Measurements.* Univ. Sci. Books, Mill Valley, Calif., 270 p.

Table 2. Summary of $^{40}\text{Ar}/^{39}\text{Ar}$ results and analytical methods

| Sample | Lab # | Irradiation | mineral/phase | age analysis | steps/crystals | Age | $\pm 2\sigma$ | MSWD | comments |
|--------|-------|-------------|------------------------|--------------------|----------------|-------|---------------|------|---------------------|
| TM-5 | 55446 | NM-186 | sanidine | laser total fusion | 13 | 34.94 | 0.1 | 1.20 | well-behaved |
| TM-4 | 55445 | NM-186 | biotite | furnace step-heat | 9 | 34.64 | 0.17 | 8.69 | fairly well-behaved |
| CP-3 | 55435 | NM-186 | groundmass concentrate | furnace step-heat | 5 | 37.86 | 0.30 | 0.77 | disturbed |
| TM-1 | 55436 | NM-186 | groundmass concentrate | furnace step-heat | 6 | 38.08 | 0.41 | 3.0 | disturbed |
| TM-3 | 55437 | NM-186 | plagioclase | furnace step-heat | 6 | 35.05 | 0.63 | 1.5 | disturbed |

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 (Renne et al., 1998).

Instrumentation:

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

Plagioclase, biotite and groundmass concentrate step-heated for 6-10 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 $\sim 450^\circ\text{C}$ and

1 at 20°C . Gas also exposed to a W filament operated at $\sim 2000^\circ\text{C}$.

Sample and flux monitor fused by a 50 watt Synrad CO₂ laser.

Reactive gases removed during a 2 minute reaction with 2 SAES GP-50 getters, 1 operated at $\sim 450^\circ\text{C}$ and

1 at 20°C . Gas also exposed to a W filament operated at $\sim 2000^\circ\text{C}$ and a cold finger operated at -140°C .

Analytical parameters:

Electron multiplier sensitivity averaged 2.24×10^{-16} moles/pA for furnace analyses.

Electron multiplier sensitivity averaged 1.28×10^{-16} moles/pA for laser analyses.

Total system blank and background for furnace analyses averaged 2642, 9.63, 2.01, 10.5, 2.24×10^{-18} moles at masses 40, 39, 38, 37 and 36, respectively.

Total system blank and background for laser analyses averaged 137, 3.69, 0.94, 1.07, 1.81×10^{-18} moles at masses 40, 39, 38, 37 and 36, respectively.

J-factors determined to a precision of $\pm 0.1\%$ by CO₂ 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 CaF, and are as follows:

$$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.0 \pm 0.0004; (^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.000289 \pm 0.000005; \text{ and } (^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 0.00068 \pm 0.00002.$$

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

| ID | $^{40}\text{Ar}/^{39}\text{Ar}$ | $^{37}\text{Ar}/^{39}\text{Ar}$ | $^{36}\text{Ar}/^{39}\text{Ar}$ ($\times 10^{-3}$) | $^{38}\text{Ar}_k$ ($\times 10^{-15}$ mol) | K/Ca | $^{40}\text{Ar}^*$ (%) | Age (Ma) | $\pm 1\sigma$ (Ma) |
|--|---------------------------------|---------------------------------|---|--|----------------------------------|---------------------------|--------------|-----------------------|
| TM-5 , Sanidine, J=0.0007347 \pm 0.14%, D=1.0055 \pm 0.001, NM-186K, Lab#=55446 | | | | | | | | |
| # 12 | 26.52 | 0.0059 | 0.5391 | 2.543 | 86.0 | 99.4 | 34.60 | 0.09 |
| 03 | 26.76 | 0.0102 | 0.7749 | 2.995 | 49.9 | 99.1 | 34.82 | 0.08 |
| 04 | 26.62 | 0.0054 | 0.2373 | 3.604 | 93.9 | 99.7 | 34.86 | 0.07 |
| 02 | 26.62 | 0.0079 | 0.1995 | 6.935 | 64.7 | 99.8 | 34.87 | 0.05 |
| 14 | 26.65 | 0.0057 | 0.2448 | 6.442 | 89.6 | 99.7 | 34.89 | 0.06 |
| 05 | 26.63 | 0.0050 | 0.1397 | 6.582 | 101.4 | 99.8 | 34.91 | 0.07 |
| 10 | 26.62 | 0.0057 | 0.0971 | 7.397 | 89.3 | 99.9 | 34.91 | 0.05 |
| 08 | 26.76 | 0.0056 | 0.5295 | 8.250 | 91.5 | 99.4 | 34.92 | 0.05 |
| 09 | 26.66 | 0.0056 | 0.1720 | 5.474 | 91.2 | 99.8 | 34.93 | 0.06 |
| 11 | 26.64 | 0.0060 | 0.0839 | 6.322 | 85.0 | 99.9 | 34.94 | 0.06 |
| 01 | 26.69 | 0.0053 | 0.0882 | 7.355 | 97.0 | 99.9 | 35.00 | 0.06 |
| 13 | 26.74 | 0.0057 | 0.2217 | 6.103 | 90.1 | 99.8 | 35.01 | 0.06 |
| 15 | 26.71 | 0.0052 | 0.1040 | 11.564 | 98.7 | 99.9 | 35.02 | 0.05 |
| 07 | 26.71 | 0.0049 | 0.0876 | 8.098 | 104.4 | 99.9 | 35.03 | 0.06 |
| # 06 | 26.79 | 0.0051 | -0.0247 | 3.825 | 99.2 | 100.0 | 35.18 | 0.07 |
| Mean age $\pm 2\sigma$ | | n=13 | MSWD=1.2 | | 88.2 \pm30.1 | | 34.94 | 0.10 |

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.

Mean age is weighted mean age of Taylor (1982). Mean age error is weighted error

of the mean (Taylor, 1982), multiplied by the root of the MSWD where MSWD>1, and also incorporates uncertainty in J factors and irradiation correction uncertainties.

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

symbol preceding sample ID denotes analyses excluded from mean age calculations.

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

Decay Constant (LambdaK (total)) = 5.543e-10

Discrimination = 1.0055 \pm 0.001

Correction factors:

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

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

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

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

Table 4. $^{40}\text{Ar}/^{39}\text{Ar}$ analytical data.

| ID | Power (°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}Ar (%) | Age (Ma) | $\pm 1\sigma$ (Ma) |
|---|---------------|---------------------------------|---------------------------------|---|--|---|---------------------------|-------------------------|--------------|-----------------------|
| CP-3 , Groundmass Concentrate, J=0.000731 \pm 0.10%, D=1.0055 \pm 0.001, NM-186J, Lab#=55435-01 | | | | | | | | | | |
| # A | 625 | 588.9 | 0.6890 | 1919.9 | 2.1 | 0.74 | 3.7 | 2.9 | 28.3 | 3.2 |
| # B | 700 | 103.6 | 0.7992 | 259.6 | 4.2 | 0.64 | 26.0 | 8.7 | 35.20 | 0.51 |
| C | 750 | 105.4 | 0.8908 | 258.5 | 5.5 | 0.57 | 27.6 | 16.4 | 37.96 | 0.49 |
| D | 800 | 71.38 | 0.6571 | 142.7 | 4.1 | 0.78 | 41.0 | 22.1 | 38.21 | 0.33 |
| E | 875 | 66.11 | 0.5818 | 125.3 | 7.5 | 0.88 | 44.1 | 32.4 | 38.04 | 0.28 |
| F | 975 | 66.43 | 0.5881 | 127.6 | 10.5 | 0.87 | 43.3 | 46.9 | 37.57 | 0.28 |
| G | 1075 | 81.33 | 0.6053 | 177.9 | 8.5 | 0.84 | 35.4 | 58.6 | 37.63 | 0.36 |
| # H | 1250 | 107.4 | 0.6246 | 259.1 | 25.3 | 0.82 | 28.7 | 93.6 | 40.27 | 0.48 |
| # I | 1700 | 103.9 | 4.985 | 243.2 | 4.6 | 0.10 | 31.3 | 100.0 | 42.49 | 0.52 |
| Integrated age $\pm 2\sigma$ | | | n=9 | | 72.4 | 0.79 \pm 0.25 | | | 38.54 | 0.88 |
| Plateau $\pm 2\sigma$ steps C-G | | | n=5 | MSWD=0.77 | 36.1 | 0.79 \pm0.25 | | 49.9 | 37.86 | 0.30 |
| Isochron$\pm 2\sigma$ steps C-G | | | n=5 | MSWD=0.96 | | $^{40}\text{Ar}/^{36}\text{Ar}=296\pm 6$ | | | 37.80 | 1.20 |
| TM-1 , Groundmass Concentrate, J=0.0007317 \pm 0.10%, D=1.0055 \pm 0.001, NM-186J, Lab#=55436-01 | | | | | | | | | | |
| A | 625 | 731.9 | 0.7631 | 2410.1 | 1.6 | 0.67 | 2.7 | 2.9 | 26.0 | 4.1 |
| B | 700 | 92.14 | 1.217 | 215.8 | 4.5 | 0.42 | 30.9 | 11.0 | 37.22 | 0.45 |
| C | 750 | 83.61 | 1.021 | 184.7 | 5.6 | 0.50 | 34.8 | 21.0 | 38.07 | 0.40 |
| D | 800 | 58.02 | 0.6150 | 96.28 | 5.4 | 0.83 | 51.1 | 30.7 | 38.70 | 0.22 |
| E | 875 | 55.22 | 0.6651 | 88.59 | 4.3 | 0.77 | 52.7 | 38.4 | 38.02 | 0.25 |
| F | 975 | 60.70 | 0.6739 | 107.3 | 7.4 | 0.76 | 47.9 | 51.7 | 37.97 | 0.25 |
| G | 1075 | 72.20 | 0.6765 | 147.5 | 5.6 | 0.75 | 39.7 | 61.7 | 37.47 | 0.33 |
| # H | 1250 | 97.58 | 0.6778 | 229.0 | 18.7 | 0.75 | 30.7 | 95.4 | 39.17 | 0.44 |
| # I | 1700 | 103.5 | 4.621 | 238.2 | 2.6 | 0.11 | 32.3 | 100.0 | 43.77 | 0.53 |
| Integrated age $\pm 2\sigma$ | | | n=9 | | 55.6 | 0.67 \pm 0.34 | | | 38.27 | 0.83 |
| Plateau $\pm 2\sigma$ steps B-G | | | n=6 | MSWD=3.0 | 32.8 | 0.67 \pm0.34 | | 58.9 | 38.08 | 0.41 |
| Isochron$\pm 2\sigma$ steps B-G | | | n=6 | MSWD=2.1 | | $^{40}\text{Ar}/^{36}\text{Ar}=290\pm 12$ | | | 38.9 | 1.7 |
| TM-3 , Plagioclase, J=0.0007338 \pm 0.08%, D=1.0055 \pm 0.001, NM-186J, Lab#=55437-01 | | | | | | | | | | |
| # A | 650 | 349.6 | 9.926 | 1122.8 | 0.5 | 0.051 | 5.4 | 9.5 | 24.8 | 2.3 |
| # B | 775 | 40.13 | 16.42 | 54.68 | 0.9 | 0.031 | 63.2 | 26.4 | 33.66 | 0.41 |
| C | 850 | 38.66 | 17.82 | 46.15 | 0.4 | 0.029 | 68.7 | 34.3 | 35.23 | 0.64 |
| D | 925 | 38.48 | 19.20 | 46.63 | 0.5 | 0.027 | 68.5 | 43.0 | 34.99 | 0.59 |
| E | 1000 | 53.37 | 20.70 | 99.35 | 0.473 | 0.025 | 48.3 | 51.6 | 34.29 | 0.64 |
| F | 1100 | 46.06 | 17.12 | 70.11 | 0.9 | 0.030 | 58.2 | 67.5 | 35.55 | 0.45 |
| G | 1175 | 71.58 | 15.82 | 156.1 | 0.5 | 0.032 | 37.5 | 76.4 | 35.52 | 0.77 |
| H | 1250 | 92.81 | 15.58 | 235.6 | 0.203 | 0.033 | 26.4 | 80.1 | 32.5 | 1.2 |
| # I | 1350 | 120.5 | 14.78 | 292.6 | 0.2 | 0.035 | 29.3 | 83.4 | 46.7 | 1.5 |
| # J | 1450 | 91.59 | 16.14 | 153.4 | 0.3 | 0.032 | 52.0 | 88.2 | 62.7 | 1.2 |
| # K | 1675 | 67.08 | 22.43 | 111.2 | 0.6 | 0.023 | 53.9 | 100.0 | 47.94 | 0.65 |
| Integrated age $\pm 2\sigma$ | | | n=11 | | 5.5 | 0.029 \pm 0.006 | | | 37.06 | 0.81 |
| Plateau $\pm 2\sigma$ steps C-H | | | n=6 | MSWD=1.5 | 3.0 | 0.029\pm0.006 | | 53.7 | 35.05 | 0.63 |
| Isochron$\pm 2\sigma$ steps B-H | | | n=7 | MSWD=3.0 | | $^{40}\text{Ar}/^{36}\text{Ar}=294\pm 24$ | | | 34.8 | 2.4 |

| ID | Power (°C) | ⁴⁰ Ar/ ³⁹ Ar | ³⁷ Ar/ ³⁹ Ar | ³⁶ Ar/ ³⁹ Ar (x 10 ⁻³) | ³⁹ Ar _k (x 10 ⁻¹⁵ mol) | K/Ca | ⁴⁰ Ar* (%) | ³⁹ Ar (%) | Age (Ma) | ±1σ (Ma) |
|--|---------------|------------------------------------|------------------------------------|---|--|-------------------------------------|--------------------------|-------------------------|--------------|-------------|
| TM-4 , Biotite, 12.74 mg, J=0.0007395±0.04%, D=1.0055±0.001, NM-186K, Lab#=55445-01 | | | | | | | | | | |
| # A | 650 | 766.0 | 0.0916 | 2562.0 | 1.47 | 5.6 | 1.2 | 0.6 | 11.84 | 4.38 |
| # B | 750 | 81.88 | 0.0829 | 209.1 | 2.08 | 6.2 | 24.6 | 1.6 | 26.63 | 0.49 |
| # C | 850 | 41.68 | 0.0577 | 58.97 | 4.09 | 8.8 | 58.2 | 3.4 | 32.07 | 0.20 |
| D | 920 | 32.13 | 0.0217 | 20.83 | 13.0 | 23.5 | 80.8 | 9.1 | 34.33 | 0.09 |
| E | 1000 | 28.73 | 0.0135 | 8.698 | 19.8 | 37.9 | 91.1 | 17.9 | 34.57 | 0.08 |
| F | 1075 | 29.11 | 0.0234 | 10.20 | 18.5 | 21.8 | 89.7 | 26.0 | 34.48 | 0.08 |
| G | 1110 | 30.84 | 0.0636 | 16.92 | 10.5 | 8.0 | 83.8 | 30.7 | 34.16 | 0.09 |
| H | 1180 | 32.96 | 0.1275 | 22.96 | 28.6 | 4.0 | 79.4 | 43.4 | 34.61 | 0.08 |
| I | 1210 | 28.21 | 0.0388 | 6.260 | 66.2 | 13.2 | 93.5 | 72.6 | 34.83 | 0.06 |
| J | 1250 | 27.56 | 0.0076 | 3.908 | 56.2 | 67.1 | 95.8 | 97.5 | 34.88 | 0.06 |
| K | 1300 | 27.61 | 0.0167 | 4.436 | 4.78 | 30.6 | 95.3 | 99.6 | 34.75 | 0.11 |
| L | 1700 | 41.64 | 0.0896 | 53.10 | 0.854 | 5.7 | 62.3 | 100.0 | 34.30 | 0.54 |
| Integrated age ± 2σ | | | n=12 | | 226.0 | 12.8 | | | 34.43 | 0.17 |
| Plateau ± 2σ steps D-L | | | n=9 | MSWD=8.69 | 218.4 | 29.5 ±40.0 | | 96.6 | 34.64 | 0.17 |
| Isochron±2σ steps D-L | | | n=9 | MSWD=4.78 | | ⁴⁰ Ar/ ³⁶ Ar= | 278.4±5.9 | | 34.88 | 0.10 |

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 (LambdaK (total)) = 5.543e-10

Discrimination = 1.0055 ± 0.001

Correction factors:

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

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

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

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

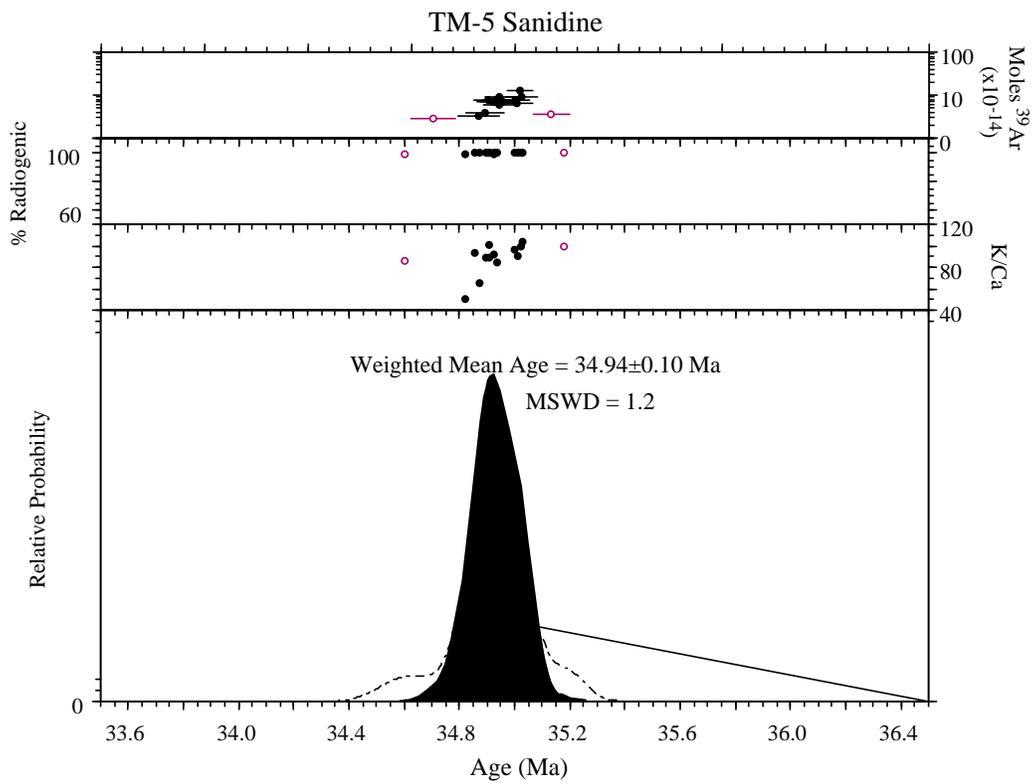


Figure 1 . Age probability distribution diagram of TM-5 sanidine. All errors quoted at 2 sigma.

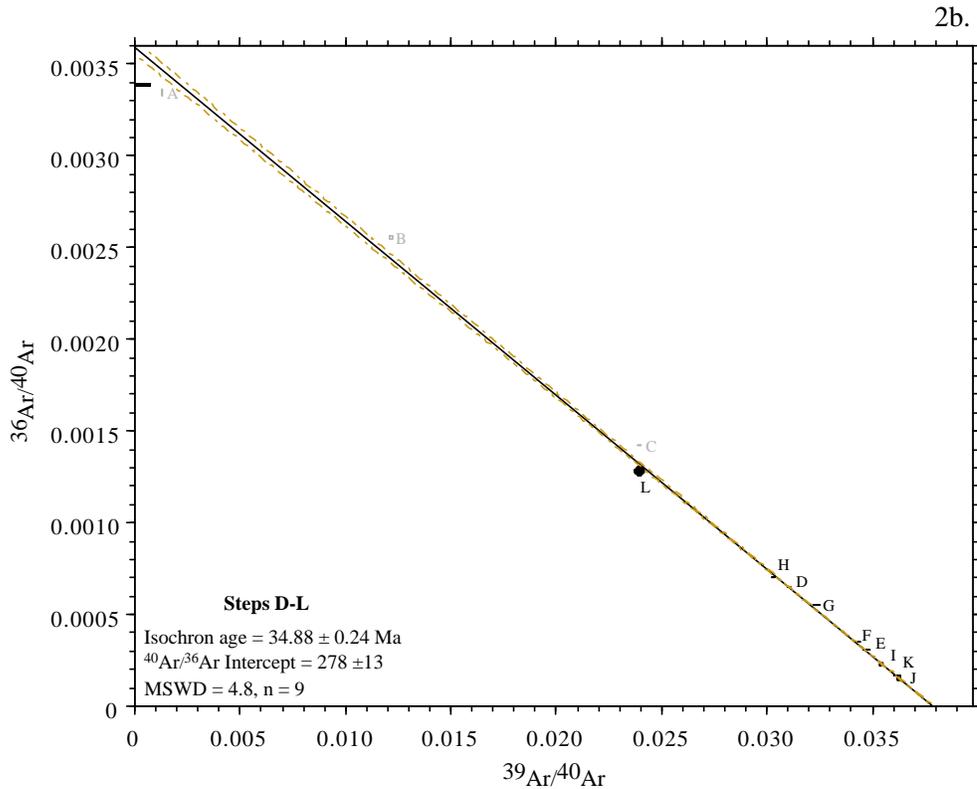
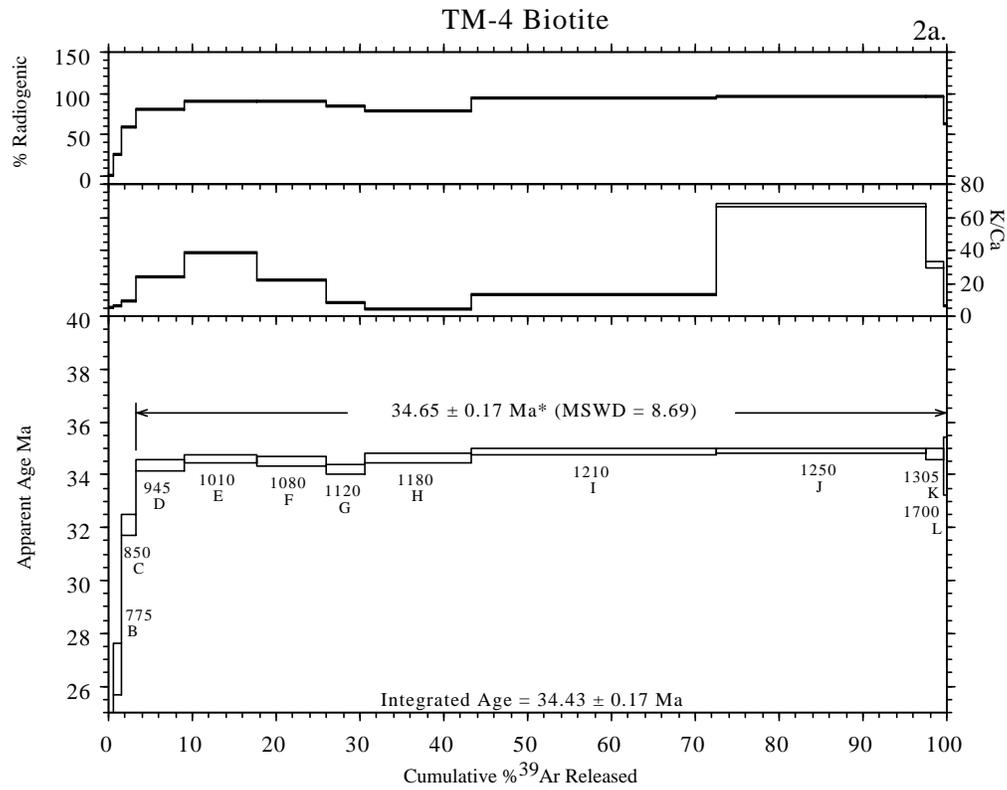


Figure 2. Age spectrum (2a) and isochron (2b) for TM-4 biotite. Points shown in purple not included in isochron. All errors quoted at two sigma.

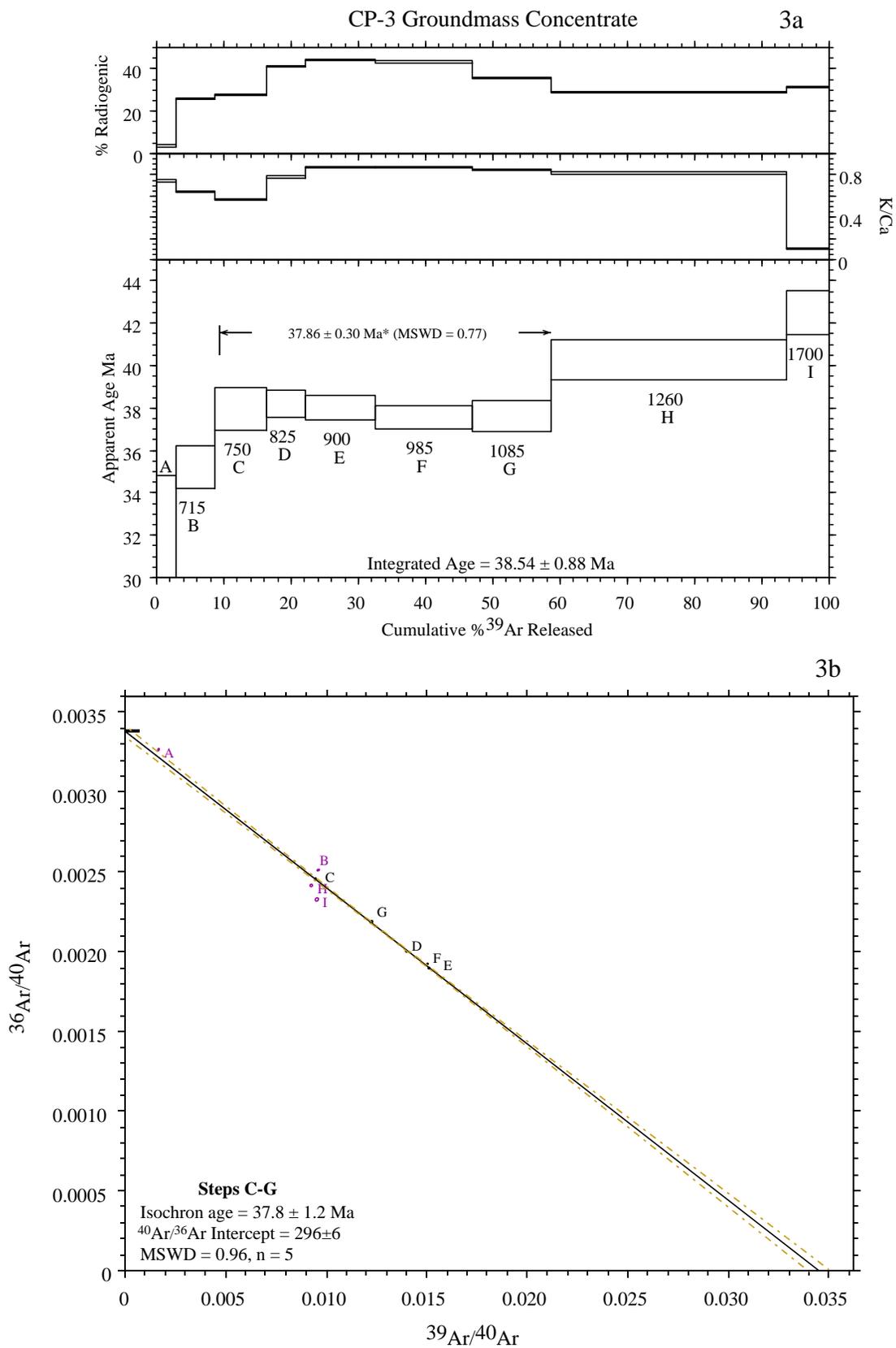


Figure 3. Age spectrum (3a) and isochron (3b) for CP-3 groundmass concentrate. Points shown in purple not included in isochron. All errors quoted at two sigma.

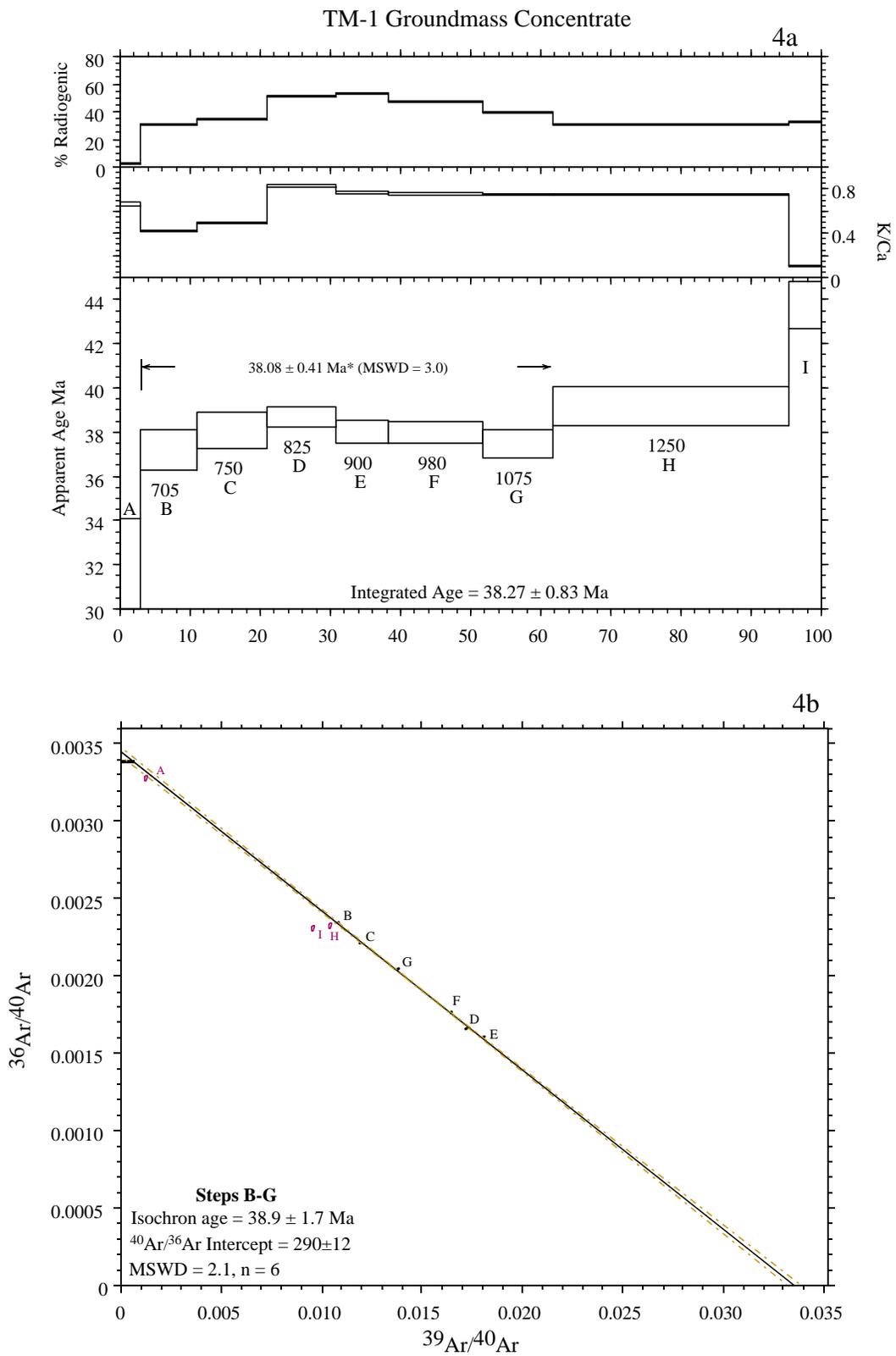


Figure 4. Age spectrum (4a) and isochron (4b) for TM-1 groundmass concentrate. Points in purple not included in isochron. All errors quoted at two sigma.

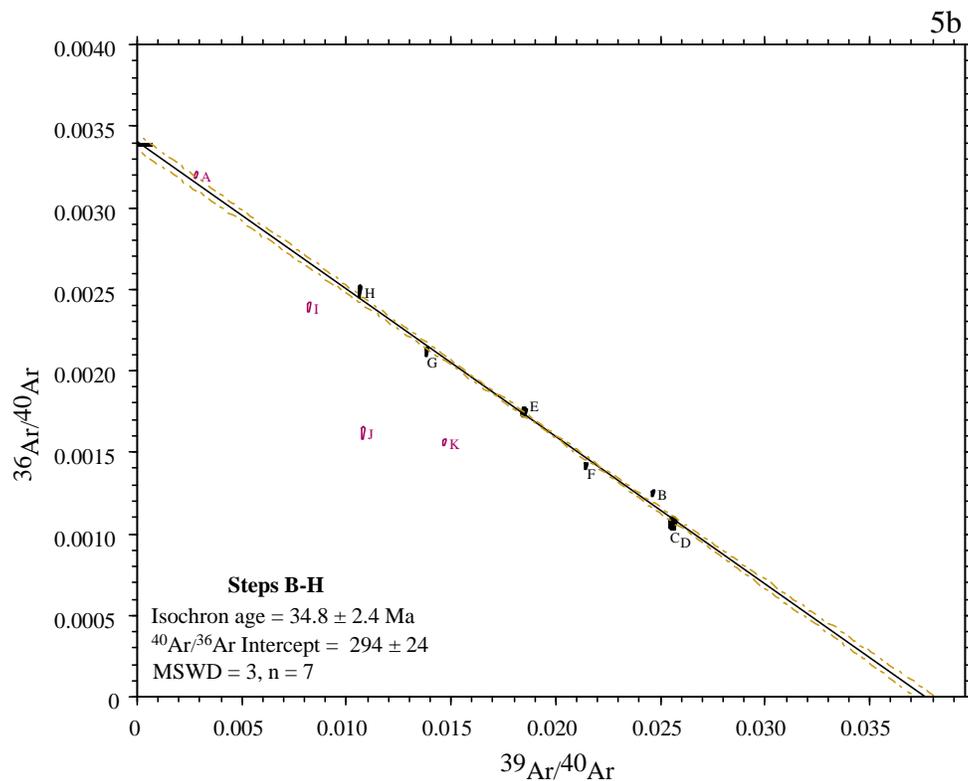
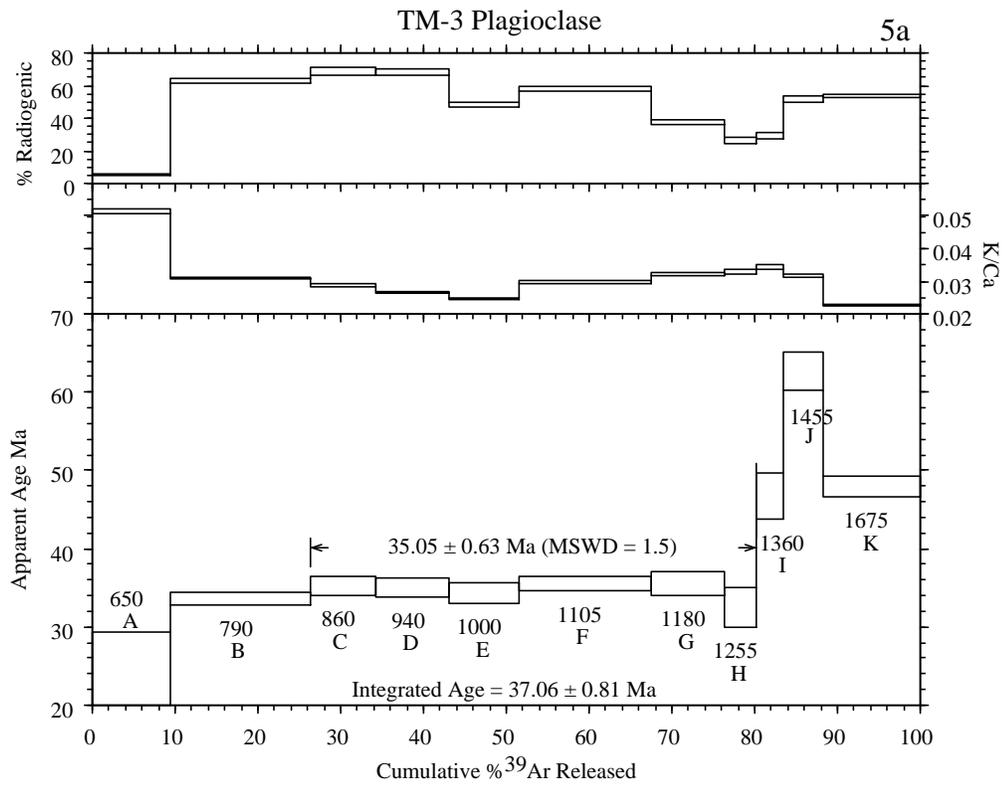


Figure 5. Age spectrum (5a) and isochron (5b) for TM-3 plagioclase. Points shown in purple not included in isochron. All errors quoted at two sigma.

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}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}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 $^{40}\text{Ar}/^{39}\text{Ar}$ 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 CO₂ 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 CO₂ 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 CaF₂, respectively. Typically, 3-5 individual pieces of the salt or glass are fused with the CO₂ 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 % ³⁹Ar_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 ⁴⁰K) has the modern day atmospheric ⁴⁰Ar/³⁶Ar value of 295.5. Additional parameters for each heating step are often plotted versus the cumulative % ³⁹Ar_K released. These auxiliary parameters can aid age spectra interpretation and may include radiogenic yield (percent of ⁴⁰Ar which is not atmospheric), K/Ca (determined from measured Ca-derived ³⁷Ar and K-derived ³⁹Ar) and/or K/Cl (determined from measured Cl-derived ³⁸Ar and K-derived ³⁹Ar). Incremental heating analysis is often effective at revealing complex argon systematics related to excess argon, alteration, contamination, ³⁹Ar 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 $^{36}\text{Ar}/^{40}\text{Ar}$ 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}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 σ) 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, $^{40}\text{Ar}/^{36}\text{Ar}_i$ values and MSWD values are calculated from the regression results obtained by the York (1969) method.

References cited

- Dalrymple, G.B., Alexander, E.C., Jr., Lanphere, M.A., and Kraker, G.P., 1981. Irradiation of samples for $^{40}\text{Ar}/^{39}\text{Ar}$ dating using the Geological Survey TRIGA reactor. U.S.G.S., Prof. Paper, 1176.
- 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.
- Deino, A., and Potts, R., 1992. Age-probability spectra from examination of single-crystal $^{40}\text{Ar}/^{39}\text{Ar}$ dating results: Examples from Olorgesailie, Southern Kenya Rift, *Quat. International*, 13/14, 47-53.
- Fleck, R.J., Sutter, J.F., and Elliot, D.H., 1977. Interpretation of discordant $^{40}\text{Ar}/^{39}\text{Ar}$ age-spectra of Mesozoic tholeiites from Antarctica, *Geochim. Cosmochim. Acta*, 41, 15-32.
- Heizler, M. T., and Harrison, T. M., 1988. Multiple trapped argon components revealed by $^{40}\text{Ar}/^{39}\text{Ar}$ analysis, *Geochim. Cosmochim. Acta*, 52, 295-1303.
- Mahon, K.I., 1996. The New "York" regression: Application of an improved statistical method to geochemistry, *International Geology Review*, 38, 293-303.
- McDougall, I., and Harrison, T.M., 1988. *Geochronology and thermochronology by the $^{40}\text{Ar}/^{39}\text{Ar}$ method*. Oxford University Press.
- Samson, S.D., and Alexander, E.C., Jr., 1987. Calibration of the interlaboratory $^{40}\text{Ar}/^{39}\text{Ar}$ dating standard, Mmhb-1, *Chem. Geol.*, 66, 27-34.
- Steiger, R.H., and Jäger, E., 1977. Subcommittee on geochronology: Convention on the use of decay constants in geo- and cosmochronology. *Earth and Planet. Sci. Lett.*, 36, 359-362.
- Taylor, J.R., 1982. *An Introduction to Error Analysis: The Study of Uncertainties in Physical Measurements*, Univ. Sci. Books, Mill Valley, Calif., 270 p.
- York, D., 1969. Least squares fitting of a straight line with correlated errors, *Earth and Planet. Sci. Lett.*, 5, 320-324.