

Supplementary file 1 – ⁴⁰Ar/³⁹Ar analytical data

1.1.

| Relative Abundances | ³⁹ Ar [fA] | %1σ | ³⁷ Ar [fA] | %1σ | ³⁹ Ar [fA] | %1σ | ³⁹ Ar [fA] | %1σ | ⁴⁰ Ar [fA] | %1σ | ⁴⁰ Ar/ ³⁹ Ar _K ± 1σ | J-value ± 1σ (Flux Monitor) | ⁴⁰ Ar (%) | K/Ca ± 1σ | |
|---------------------|-----------------------|-----------|-----------------------|-----------|-----------------------|-----------|-----------------------|-----------|-----------------------|-----------|--|-----------------------------|-----------------------|-----------|--------------|
| 89_VU101-F8 | 4 | 0.002325 | 7.783 | 0.176093 | 13.824 | 0.281934 | 0.313 | 24.52996 | 0.111 | 123.6373 | 0.032 | 5.01160 ± 0.00619 | 0.0030962 ± 0.0000038 | 99.43 | 59.7 ± 8.3 |
| 90_VU101-F8 | 4 | 0.0053753 | 4.566 | 0.5152000 | 5.297 | 0.658957 | 0.285 | 56.69098 | 0.105 | 286.3546 | 0.030 | 5.02273 ± 0.00564 | 0.0030983 ± 0.0000035 | 99.44 | 47.3 ± 2.5 |
| 91_VU101-F8 | 4 | 0.0034662 | 6.178 | 0.4003544 | 5.455 | 0.624089 | 0.284 | 53.85904 | 0.104 | 271.2900 | 0.033 | 5.01757 ± 0.00561 | 0.0030925 ± 0.0000035 | 99.61 | 57.8 ± 3.2 |
| 93_VU101-F8 | 4 | 0.0057754 | 4.479 | 0.5377946 | 5.473 | 0.747863 | 0.271 | 64.62421 | 0.105 | 326.5690 | 0.030 | 5.02650 ± 0.00562 | 0.0030970 ± 0.0000035 | 99.47 | 51.7 ± 2.8 |
| 94_VU101-F8 | 4 | 0.0040615 | 6.849 | 0.3097059 | 7.437 | 0.493975 | 0.299 | 42.44287 | 0.110 | 214.2608 | 0.048 | 5.01938 ± 0.00632 | 0.0030914 ± 0.0000039 | 99.43 | 58.9 ± 4.4 |
| 95_VU101-F8 | 4 | 0.0033149 | 7.601 | 0.3712244 | 8.414 | 0.380756 | 0.307 | 32.79453 | 0.109 | 165.3203 | 0.038 | 5.01098 ± 0.00621 | 0.0030966 ± 0.0000038 | 99.40 | 38.0 ± 3.2 |
| 97_VU101-F8 | 4 | 0.0054989 | 4.487 | 0.3703941 | 7.504 | 0.573981 | 0.263 | 49.86371 | 0.107 | 252.1712 | 0.031 | 5.02405 ± 0.00579 | 0.0030985 ± 0.0000036 | 99.34 | 57.9 ± 4.3 |
| 98_VU101-F9 | 4 | 0.0029331 | 6.617 | 0.218775 | 9.277 | 0.327386 | 0.389 | 28.36214 | 0.107 | 142.9799 | 0.030 | 5.01012 ± 0.00593 | 0.0030971 ± 0.0000037 | 99.38 | 55.7 ± 5.2 |
| 99_VU101-F9 | 4 | 0.0060354 | 3.666 | 0.3069693 | 8.170 | 0.440956 | 0.323 | 38.51916 | 0.109 | 195.0851 | 0.035 | 5.01764 ± 0.00602 | 0.0030925 ± 0.0000037 | 99.07 | 54.0 ± 4.4 |
| 101_VU101-F9 | 4 | 0.0023126 | 7.755 | 0.1294320 | 13.935 | 0.225340 | 0.289 | 19.51844 | 0.113 | 98.4678 | 0.029 | 5.00917 ± 0.00646 | 0.0030977 ± 0.0000040 | 99.29 | 64.8 ± 9.0 |
| 102_VU101-F9 | 4 | 0.0026934 | 10.987 | 0.4345913 | 6.486 | 0.672145 | 0.286 | 58.17969 | 0.108 | 292.7887 | 0.038 | 5.01842 ± 0.00594 | 0.0030920 ± 0.0000037 | 99.72 | 57.6 ± 3.7 |
| 103_VU101-F9 | 4 | 0.0027532 | 8.723 | 0.1584047 | 12.672 | 0.426519 | 0.290 | 36.86053 | 0.109 | 185.8884 | 0.031 | 5.02021 ± 0.00602 | 0.0030908 ± 0.0000037 | 99.55 | 100.1 ± 12.7 |
| 108_VU101-F9 | 4 | 0.0020503 | 11.920 | 1.8253917 | 1.955 | 0.547128 | 0.287 | 47.56467 | 0.106 | 239.6392 | 0.028 | 5.02760 ± 0.00572 | 0.0030963 ± 0.0000035 | 99.79 | 11.2 ± 0.2 |
| 110_VU101-F9 | 4 | 0.0107944 | 2.332 | 0.4022729 | 5.441 | 0.459329 | 0.308 | 39.66995 | 0.106 | 202.3024 | 0.031 | 5.01837 ± 0.00584 | 0.0030920 ± 0.0000036 | 98.41 | 42.4 ± 2.3 |
| 111_VU101-F10 | 4 | 0.0097333 | 2.791 | 0.3894811 | 8.058 | 0.495213 | 0.285 | 42.78403 | 0.106 | 218.3414 | 0.030 | 5.03531 ± 0.00588 | 0.0030916 ± 0.0000036 | 98.67 | 47.2 ± 3.8 |
| 113_VU101-F10 | 4 | 0.0076113 | 3.590 | 0.3724742 | 6.321 | 0.658973 | 0.300 | 57.08002 | 0.111 | 289.1362 | 0.036 | 5.02532 ± 0.00606 | 0.0030977 ± 0.0000037 | 99.21 | 65.9 ± 4.2 |
| 114_VU101-F10 | 4 | 0.0039631 | 6.766 | 0.2509541 | 11.090 | 0.392589 | 0.273 | 33.62278 | 0.117 | 170.4084 | 0.033 | 5.03281 ± 0.00657 | 0.0030832 ± 0.0000040 | 99.30 | 57.6 ± 6.4 |
| 115_VU101-F10 | 4 | 0.0148724 | 2.192 | 0.4442233 | 8.374 | 0.783011 | 0.276 | 67.26024 | 0.104 | 343.4622 | 0.031 | 5.03968 ± 0.00568 | 0.0030789 ± 0.0000035 | 98.70 | 65.1 ± 5.5 |
| 117_VU101-F10 | 4 | 0.0030722 | 8.855 | 0.2273613 | 11.222 | 0.274930 | 0.352 | 23.86694 | 0.113 | 120.6397 | 0.036 | 5.01617 ± 0.00686 | 0.0030934 ± 0.0000042 | 99.24 | 45.1 ± 5.1 |
| 118_VU101-F10 | 4 | 0.0057066 | 4.744 | 0.4722994 | 5.943 | 0.493993 | 0.252 | 42.71198 | 0.107 | 216.3405 | 0.028 | 5.02525 ± 0.00587 | 0.0030978 ± 0.0000036 | 99.21 | 38.9 ± 2.3 |
| 119_VU101-F10 | 4 | 0.0018802 | 16.263 | 0.2044352 | 13.869 | 0.398795 | 0.285 | 34.32335 | 0.105 | 172.9431 | 0.028 | 5.02192 ± 0.00608 | 0.0030898 ± 0.0000037 | 99.67 | 72.2 ± 10.0 |
| Σ | | 0.1062364 | 1.099 | 8.5183497 | 1.448 | 10.357841 | 0.066 | 895.13519 | 0.024 | 4528.0260 | 0.008 | | | | |

1.2.

| Information on Analysis and Constants Used in Calculations | ⁴⁰ Ar/ ³⁹ Ar _K ± 1σ | J-value ± 1σ (Flux Monitor) | MSWD | N | K/Ca ± 1σ |
|--|--|-------------------------------|--------------|----------------------|------------|
| Sample = VU101-F8 - F9 - F10 | | | | | |
| Material = sandine | | | | | |
| Location = FishCanyon | | | | | |
| Analyst = K. Kuiper / F. Markovic | | | | | |
| Project = VU101 | | | | | |
| Mass Discrimination Law = LIN | | | | | |
| Irradiation = VU101 | | | | | |
| Fish Canyon = 28.201 ± 0.023 Ma | | | | | |
| IGSN = Undefined | | | | | |
| Experiment Type = Undefined | | | | | |
| Extraction Method = Undefined | | | | | |
| Heating = 45 sec | | | | | |
| Isolation = 5.00 min | | | | | |
| Age Equations = Min et al. (2000) | | | | | |
| Negative Intensities = Allowed | | | | | |
| Decay Constant 40K = 5.460 ± 0.053 E-10 1/a | | | | | |
| Decay Constant 39Ar = 2.940 ± 0.016 E-07 1/h | | | | | |
| Decay Constant 37Ar = 8.230 ± 0.012 E-04 1/h | | | | | |
| Decay Constant 36Cl = 2.257 ± 0.015 E-06 1/a | | | | | |
| Decay Activity 40K(β ⁺) = 3.310 ± 0.030 1/g | | | | | |
| Decay Activity 40K(β ⁻) = 27.890 ± 0.150 1/g | | | | | |
| Atmospheric Ratio 40Ar/36Ar = 298.56 ± 0.31 | | | | | |
| Atmospheric Ratio 38Ar/36Ar = 0.1885 ± 0.0003 | | | | | |
| Production Ratio 39Ar/37Ar = 0.000673 ± 0.000004 | | | | | |
| Production Ratio 38Ar/36Ar = 0.000284 ± 0.000002 | | | | | |
| Production Ratio 40Ar/39K = 0.000860 ± 0.000070 | | | | | |
| Production Ratio 38Ar/36K = 0.012110 ± 0.000030 | | | | | |
| Production Ratio 38Ar/38Cl = 262.80 ± 1.71 | | | | | |
| Scaling Ratio K/Ca = 0.430 | | | | | |
| Abundance Ratio 40K/K = 1.1700 ± 0.0100 E-04 | | | | | |
| Atomic Weight K = 39.0983 ± 0.0001 g | | | | | |
| Weighted mean J | 5.02075 ± 0.00157 ± 0.03% | 0.0030906 ± 0.0000010 ± 0.03% | 1.37 | 20 | 13.6 ± 2.2 |
| Total Fusion J | 5.02297 ± 0.00135 ± 0.03% | 0.0030892 ± 0.0000008 ± 0.03% | 1.32 | 21 | 45.2 ± 0.7 |
| Normal Isochron | 5.02484 ± 0.00384 ± 0.08% | 0.0030880 ± 0.0000024 ± 0.08% | 1.73 | 20 | |
| | 270.54 ± 31.25 ± 11.55% | | 3% | 20 | |
| Inverse Isochron | 5.01803 ± 0.00350 ± 0.07% | 0.0030922 ± 0.0000022 ± 0.07% | 1.33 | 20 | |
| | 323.60 ± 26.98 ± 8.34% | | 1.32 | 20 | |
| | | | 1.1719 | 21 | |
| | | | 0.0000010278 | Convergence | |
| | | | 1 | Number of Iterations | |
| | | | 1.3146 | Error Magnification | |
| | | | 1.33 | 1σ Confidence Limit | |
| | | | 3 | Number of Iterations | |
| | | | 1.1790 | Error Magnification | |
| | | | 0.000155719 | Convergence | |
| | | | 1% | Spreading Factor | |

2.1.

| Relative Abundances | | ^{36}Ar [FA] | %1 σ | ^{37}Ar [FA] | %1 σ | ^{39}Ar [FA] | %1 σ | ^{40}Ar [FA] | %1 σ | $^{40}\text{Ar}/^{39}\text{Ar}/\text{Ar}_K \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | ^{40}Ar (%) | K/Ca $\pm 1\sigma$ |
|---------------------|------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|---|------------------------|----------------------|--------------------|
| 129_VU101-R2 | 8 °C | 0.0135454 | 1.517 | 37.80022 | 0.833 | 0.0414716 | 0.801 | 10.44119 | 0.228 | 2.65858 \pm 0.02069 | 14.99 \pm 0.12 | 89.77 | 0.040 \pm 0.000 |
| 131_VU101-R2 | 8 °C | 0.0143895 | 2.523 | 39.97288 | 0.818 | 0.0516754 | 0.622 | 12.81004 | 0.195 | 2.65945 \pm 0.02648 | 14.99 \pm 0.15 | 91.03 | 0.047 \pm 0.000 |
| 122_VU101-R2 | 8 °C | 0.0132076 | 2.244 | 36.75849 | 0.816 | 0.0538798 | 0.799 | 12.94729 | 0.217 | 2.68718 \pm 0.02207 | 15.15 \pm 0.12 | 91.89 | 0.052 \pm 0.000 |
| 121_VU101-R2 | 8 °C | 0.0167614 | 1.580 | 39.63710 | 0.840 | 0.0521478 | 0.696 | 13.35176 | 0.189 | 2.70234 \pm 0.02101 | 15.23 \pm 0.12 | 85.89 | 0.046 \pm 0.000 |
| 123_VU101-R2 | 8 °C | 0.0238272 | 1.442 | 52.05310 | 0.818 | 0.1127186 | 0.356 | 27.80559 | 0.130 | 2.72623 \pm 0.01288 | 15.37 \pm 0.07 | 89.14 | 0.075 \pm 0.001 |
| 130_VU101-R2 | 8 °C | 0.0128609 | 1.914 | 33.36867 | 0.849 | 0.0681448 | 0.872 | 17.10004 | 0.166 | 2.72964 \pm 0.01436 | 15.39 \pm 0.08 | 92.90 | 0.075 \pm 0.001 |
| 126_VU101-R2 | 8 °C | 0.0158322 | 1.924 | 43.89354 | 0.856 | 0.0647320 | 0.595 | 16.10785 | 0.159 | 2.74660 \pm 0.01878 | 15.48 \pm 0.11 | 92.10 | 0.063 \pm 0.000 |
| 125_VU101-R2 | 8 °C | 0.0161414 | 1.967 | 44.09337 | 0.823 | 0.0664039 | 0.623 | 16.72728 | 0.205 | 2.76571 \pm 0.01929 | 15.59 \pm 0.11 | 91.94 | 0.064 \pm 0.000 |
| 127_VU101-R2 | 8 °C | 0.0134158 | 2.588 | 42.58682 | 0.860 | 0.0751477 | 0.598 | 18.09525 | 0.157 | 2.77538 \pm 0.01808 | 15.64 \pm 0.10 | 96.38 | 0.063 \pm 0.001 |
| 133_VU101-R2 | 8 °C | 0.0280806 | 1.382 | 29.00053 | 0.872 | 0.2912686 | 0.341 | 75.00564 | 0.106 | 2.79733 \pm 0.00574 | 15.77 \pm 0.03 | 91.84 | 0.365 \pm 0.003 |
| Σ | | 0.1680621 | 0.588 | 399.16473 | 0.267 | 0.8779902 | 0.182 | 220.39193 | 0.053 | 73.66000 | 0.020 | | |

2.2.

| Information on Analysis and Constants Used in Calculations | | $^{40}\text{Ar}/^{36}\text{Ar}_K \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | MSWD | N | K/Ca $\pm 1\sigma$ |
|--|--|---|--------------------------------|--------------|-----------------------------|----------------------|
| Sample = VU101-R2 | Age Equations = Min et al. (2000) | | | | | |
| Material = volcanic glass | Negative Intensities = Allowed | | | | | |
| Location = Jovac | Decay Constant $^{40}\text{K} = 5.460 \pm 0.053 \text{ E-10 } 1/\text{a}$ | 2.67826 \pm 0.0112 | 15.10 \pm 0.06 | 0.96 | | 0.046 \pm 0.002 |
| Analyst = K. Kuiper / F. Markovic | Decay Constant $^{39}\text{Ar} = 2.940 \pm 0.016 \text{ E-07 } 1/\text{h}$ | \pm 0.42% | \pm 0.43% | 41% | 4 | |
| Project = VU101 | Decay Constant $^{37}\text{Ar} = 8.230 \pm 0.012 \text{ E-04 } 1/\text{h}$ | Full External Error \pm 0.17 | Full External Error \pm 0.17 | 1.82 | 1 σ Confidence Limit | |
| Mass Discrimination Law = LIN | Decay Constant $^{36}\text{Cl} = 2.257 \pm 0.015 \text{ E-06 } 1/\text{a}$ | Analytical Error \pm 0.06 | Analytical Error \pm 0.06 | 1.0000 | Error Magnification | |
| Irradiation = VU101 | Decay Activity $^{40}\text{K}(\text{EC},\beta^-) = 3.310 \pm 0.030 \text{ } 1/\text{gs}$ | | | | | |
| J = 0.00309060 \pm 0.00000309 | Decay Activity $^{40}\text{K}(\beta^-) = 27.890 \pm 0.150 \text{ } 1/\text{gs}$ | 2.74801 \pm 0.00455 | 15.49 \pm 0.03 | | 10 | 0.079 \pm 0.000 |
| Fish Canyon = 28.201 \pm 0.023 Ma | Atmospheric Ratio $^{40}\text{Ar}/^{36}\text{Ar} = 298.56 \pm 0.31$ | \pm 0.17% | \pm 0.19% | | | |
| IGSN = Undefined | Atmospheric Ratio $^{39}\text{Ar}/^{36}\text{Ar} = 0.1885 \pm 0.0003$ | Full External Error \pm 0.16 | Full External Error \pm 0.16 | | | |
| Preferred Age = Undefined | Production Ratio $^{39}\text{Ar}/^{36}\text{Ar} = 0.000673 \pm 0.000004$ | Analytical Error \pm 0.03 | Analytical Error \pm 0.03 | | | |
| Classification = Undefined | Production Ratio $^{37}\text{Ar}/^{36}\text{Ar} = 0.000264 \pm 0.000002$ | | | | | |
| Experiment Type = Undefined | Production Ratio $^{40}\text{Ar}/^{39}\text{Ar} = 0.000860 \pm 0.000070$ | 346.66 \pm 47.56 | 14.80 \pm 0.29 | 0.67 | 4 | |
| Extraction Method = Undefined | Production Ratio $^{38}\text{Ar}/^{36}\text{Ar} = 0.012110 \pm 0.000030$ | \pm 13.72% | Full External Error \pm 0.33 | 51% | 4 | |
| Heating = 45 sec | Production Ratio $^{36}\text{Ar}/^{36}\text{Ar} = 262.80 \pm 1.71$ | | Analytical Error \pm 0.29 | 2.00 | 1 σ Confidence Limit | |
| Isolation = 5.00 min | Scaling Ratio K/Ca = 0.430 | | | 1.0000 | Error Magnification | |
| Instrument = HELIX | Abundance Ratio $^{40}\text{K}/\text{K} = 1.1700 \pm 0.0100 \text{ E-04}$ | | | 100 | Number of Iterations | |
| Lithology = Undefined | Atomic Weight K = 39.0983 \pm 0.0001 g | 348.33 \pm 47.23 | 14.80 \pm 0.29 | 0.0001212391 | Convergence | |
| Lat-Lon = Undefined - Undefined | | \pm 13.56% | Full External Error \pm 0.33 | | | |
| Feature = Undefined | | | Analytical Error \pm 0.29 | | | |
| | | | | | 2 | Number of Iterations |
| | | | | | 6% | Spreading Factor |
| | | | | | 0.0012211843 | Convergence |

2.3.

| Results | | $^{40}\text{Ar}/^{36}\text{Ar}_K \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | MSWD | N | K/Ca $\pm 1\sigma$ |
|--------------------------|--|---|--------------------------------|--------------|-----------------------------|--------------------|
| Weighted mean age | | | | | | |
| | | 2.67826 \pm 0.0112 | 15.10 \pm 0.06 | 0.96 | | 0.046 \pm 0.002 |
| | | \pm 0.42% | \pm 0.43% | 41% | 4 | |
| | | Full External Error \pm 0.17 | Full External Error \pm 0.17 | 1.82 | 1 σ Confidence Limit | |
| | | Analytical Error \pm 0.06 | Analytical Error \pm 0.06 | 1.0000 | Error Magnification | |
| Total fusion age | | | | | | |
| | | 2.74801 \pm 0.00455 | 15.49 \pm 0.03 | | 10 | 0.079 \pm 0.000 |
| | | \pm 0.17% | \pm 0.19% | | | |
| | | Full External Error \pm 0.16 | Full External Error \pm 0.16 | | | |
| | | Analytical Error \pm 0.03 | Analytical Error \pm 0.03 | | | |
| Normal isochron | | | | | | |
| | | 346.66 \pm 47.56 | 14.80 \pm 0.29 | 0.67 | | |
| | | \pm 13.72% | \pm 1.99% | 51% | 4 | |
| | | Full External Error \pm 0.33 | Full External Error \pm 0.33 | 2.00 | 1 σ Confidence Limit | |
| | | Analytical Error \pm 0.29 | Analytical Error \pm 0.29 | 1.0000 | Error Magnification | |
| | | | | 100 | Number of Iterations | |
| | | | | 0.0001212391 | Convergence | |
| Inverse isochron | | | | | | |
| | | 348.33 \pm 47.23 | 14.80 \pm 0.29 | 0.70 | | |
| | | \pm 13.56% | \pm 1.94% | 49% | 4 | |
| | | Full External Error \pm 0.33 | Full External Error \pm 0.33 | 2.00 | 1 σ Confidence Limit | |
| | | Analytical Error \pm 0.29 | Analytical Error \pm 0.29 | 1.0000 | Error Magnification | |
| | | | | 2 | Number of Iterations | |
| | | | | 0.0012211843 | Convergence | |
| | | | | 6% | Spreading Factor | |

3.1.

| Relative Abundances | ³⁶ Ar [fA] | %Iσ | ³⁷ Ar [fA] | %Iσ | ³⁸ Ar [fA] | %Iσ | ³⁹ Ar [fA] | %Iσ | ⁴⁰ Ar [fA] | %Iσ | ⁴⁰ Ar/ ³⁹ Ar _K ± Iσ | Age ± Iσ (Ma) | ⁴⁰ Ar (%) | K/Ca ± Iσ |
|---------------------|-----------------------|-----------|-----------------------|-----------|-----------------------|-----------|-----------------------|----------|-----------------------|-----------|--|---------------|----------------------|---------------|
| 163_VU101-R6 | 4 | 0.0046989 | 3.7917 | 1.409 | 0.0090790 | 1.846 | 0.740826 | 0.912 | 3.01411 | 0.342 | 2.58021 ± 0.09118 | 14.55 ± 0.51 | 63.20 | 0.085 ± 0.001 |
| 153_VU101-R6 | 4 | 0.0104225 | 2.375 | 5.95677 | 1.163 | 0.0145071 | 1.806 | 1.088176 | 0.757 | 5.49671 | 2.63202 ± 0.07184 | 14.84 ± 0.40 | 51.91 | 0.078 ± 0.001 |
| 179_VU101-R6 | 4 | 0.0051711 | 4.350 | 6.07914 | 1.170 | 0.0140682 | 1.627 | 1.113099 | 0.518 | 4.00384 | 2.64936 ± 0.06294 | 14.94 ± 0.35 | 73.38 | 0.078 ± 0.001 |
| 175_VU101-R6 | 4 | 0.0071964 | 4.475 | 5.29734 | 1.116 | 0.0133086 | 1.419 | 1.059390 | 0.496 | 4.53339 | 2.65333 ± 0.09240 | 14.96 ± 0.52 | 61.80 | 0.086 ± 0.001 |
| 161_VU101-R6 | 4 | 0.0044480 | 4.291 | 5.67944 | 1.061 | 0.0124666 | 2.006 | 1.055330 | 0.613 | 3.72341 | 2.70293 ± 0.05739 | 15.24 ± 0.32 | 76.33 | 0.080 ± 0.001 |
| 155_VU101-R6 | 4 | 0.0049349 | 5.250 | 5.59972 | 1.244 | 0.0125439 | 2.401 | 1.060322 | 0.674 | 3.89022 | 2.70437 ± 0.07618 | 15.24 ± 0.43 | 73.45 | 0.081 ± 0.001 |
| 154_VU101-R6 | 4 | 0.0047215 | 4.988 | 5.78538 | 1.026 | 0.0141135 | 1.530 | 1.151661 | 0.630 | 4.06595 | 2.71077 ± 0.06421 | 15.28 ± 0.36 | 76.52 | 0.085 ± 0.001 |
| 166_VU101-R6 | 4 | 0.0116881 | 2.348 | 5.34338 | 1.103 | 0.0139116 | 1.681 | 1.050145 | 0.796 | 5.95670 | 2.75896 ± 0.08293 | 15.55 ± 0.47 | 48.47 | 0.084 ± 0.001 |
| 173_VU101-R6 | 4 | 0.0037302 | 6.745 | 5.53174 | 1.210 | 0.0126864 | 2.299 | 1.038421 | 0.822 | 3.55593 | 2.78087 ± 0.07675 | 15.67 ± 0.43 | 80.92 | 0.080 ± 0.001 |
| 178_VU101-R6 | 4 | 0.0036334 | 6.457 | 5.76773 | 1.221 | 0.0112837 | 1.257 | 0.955174 | 0.782 | 3.29326 | 2.79859 ± 0.07749 | 15.77 ± 0.43 | 80.84 | 0.071 ± 0.001 |
| 177_VU101-R6 | 4 | 0.0046286 | 5.305 | 4.52306 | 1.353 | 0.0106405 | 1.871 | 0.857754 | 0.793 | 3.46825 | 2.85847 ± 0.08957 | 16.11 ± 0.50 | 70.42 | 0.081 ± 0.001 |
| 157_VU101-R6 | 4 | 0.0095439 | 2.556 | 8.79593 | 1.057 | 0.0201950 | 1.261 | 1.625679 | 0.438 | 6.78790 | 2.85869 ± 0.04736 | 16.11 ± 0.27 | 68.22 | 0.079 ± 0.001 |
| 167_VU101-R6 | 8 °C | 0.0031101 | 7.419 | 4.93554 | 1.270 | 0.0110378 | 1.786 | 0.958169 | 0.835 | 3.30015 | 2.89030 ± 0.07664 | 16.29 ± 0.43 | 83.63 | 0.083 ± 0.001 |
| 159_VU101-R6 | 8 °C | 0.0124989 | 2.512 | 6.69431 | 1.168 | 0.0160745 | 1.862 | 1.173621 | 0.498 | 6.16682 | 2.91827 ± 0.08233 | 16.44 ± 0.46 | 51.56 | 0.075 ± 0.001 |
| 158_VU101-R6 | 8 °C | 0.0061468 | 5.540 | 5.20975 | 1.431 | 0.0124439 | 1.658 | 0.961452 | 0.735 | 4.24271 | 2.94101 ± 0.10888 | 16.57 ± 0.61 | 66.40 | 0.079 ± 0.001 |
| 151_VU101-R6 | 8 °C | 0.0048688 | 5.901 | 7.56159 | 1.105 | 0.0140790 | 1.579 | 1.193920 | 0.351 | 4.39505 | 2.94506 ± 0.07523 | 16.59 ± 0.42 | 79.66 | 0.068 ± 0.001 |
| 165_VU101-R6 | 8 °C | 0.0027495 | 11.022 | 3.46378 | 1.307 | 0.0078722 | 2.215 | 0.639388 | 1.009 | 2.47996 | 3.03196 ± 0.14613 | 17.08 ± 0.82 | 77.89 | 0.079 ± 0.001 |
| 174_VU101-R6 | 8 °C | 0.0213795 | 1.762 | 4.83505 | 1.392 | 0.0141003 | 1.400 | 0.866298 | 0.769 | 6.69274 | 3.11687 ± 0.13339 | 17.56 ± 0.75 | 30.95 | 0.077 ± 0.001 |
| 181_VU101-R6 | 8 °C | 0.0040638 | 6.156 | 9.04791 | 1.168 | 0.0117346 | 1.801 | 0.959508 | 0.671 | 4.01609 | 3.68534 ± 0.08335 | 20.74 ± 0.47 | 87.53 | 0.045 ± 0.001 |
| 162_VU101-R6 | 8 °C | 0.0086448 | 2.581 | 5.58881 | 1.243 | 0.0252203 | 1.082 | 2.100699 | 0.288 | 27.10545 | 11.90462 ± 0.04738 | 66.17 ± 0.26 | 92.10 | 0.161 ± 0.002 |
| Σ | | 0.1383987 | 0.865 | 115.43656 | 0.272 | 0.2713688 | 0.377 | 21.64942 | 0.142 | 112.63964 | 0.038 | | | |

3.2.

| Information on Analysis and Constants Used in Calculations | ⁴⁰ Ar/ ³⁹ Ar _K ± Iσ | Age ± Iσ (Ma) | MSWD | N | K/Ca ± Iσ |
|--|--|----------------------------|--------|----|---------------------|
| Sample = VU101-R6 | 2.73571 | 15.42 ± 0.15 | 1.64 | 12 | 0.080 ± 0.001 |
| Material = volcanic glass | ± 0.02584 | ± 0.95% | 8% | 12 | |
| Location = Laz | ± 0.94% | | | | |
| Analyst = K. Kujper / F. Markovic | Full External Error ± 0.22 | Full External Error ± 0.22 | 1.43 | 10 | 1σ Confidence Limit |
| Project = VU101 | Analytical Error ± 0.15 | Analytical Error ± 0.15 | 1.2799 | | Error Magnification |
| Mass Discrimination Law = LIN | | | | | |
| Irradiation = VU101 | 3.72708 ± 0.01757 | 20.98 ± 0.10 | | 20 | 0.080 ± 0.000 |
| J = 0.00309060 ± 0.00000309 | ± 0.47% | ± 0.48% | | | |
| Fish Canyon = 28.201 ± 0.023 Ma | Full External Error ± 0.24 | Full External Error ± 0.24 | | | |
| IGSN = Undefined | Analytical Error ± 0.10 | Analytical Error ± 0.10 | | | |
| Preferred Age = Undefined | 2.72643 ± 0.06017 | 15.37 ± 0.34 | 1.73 | 12 | |
| Classification = Undefined | ± 2.21% | ± 2.20% | 7% | 12 | |
| Experiment Type = Undefined | Full External Error ± 0.37 | Full External Error ± 0.37 | 1.45 | 10 | 1σ Confidence Limit |
| Extraction Method = Undefined | Analytical Error ± 0.34 | Analytical Error ± 0.34 | 1.3141 | | Error Magnification |
| Heating = 45 sec | 58 | Number of Iterations | | | |
| Isolation = 5.00 min | 0.0000249838 | Convergence | | | |
| Instrument = HELIX | | | | | |
| Lithology = Undefined | 1.80 | 15.45 ± 0.34 | | | |
| Lat-Lon = Undefined | 5% | ± 2.20% | | 12 | |
| Feature = Undefined | 1.45 | 1σ Confidence Limit | | | |
| | 1.3427 | Full External Error ± 0.38 | | | |
| | 3 | Analytical Error ± 0.34 | | | |
| | 3 | Number of Iterations | | | |
| | 0.0000695995 | Convergence | | | |
| | 32% | Spreading Factor | | | |

4.1.

| Relative Abundances | | ^{36}Ar [fA] | %1 σ | ^{37}Ar [fA] | %1 σ | ^{38}Ar [fA] | %1 σ | ^{39}Ar [fA] | %1 σ | ^{40}Ar [fA] | %1 σ | $^{40}\text{Ar}/^{39}\text{Ar}_K \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | ^{40}Ar (%) | MSWD | N | K/Ca $\pm 1\sigma$ | |
|---------------------|------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|---|------------------------|----------------------|------|---|--------------------|-------|
| 193_VU101-R7 | 8 °C | 4 | 0.0159890 | 1.895 | 39.41153 | 0.837 | 0.0495800 | 0.568 | 4.139366 | 12.37153 | 0.083 | 2.60179 \pm 0.02402 | 14.67 \pm 0.13 | 86.49 | | | 0.0449 \pm 0.00C | |
| 194_VU101-R7 | 8 °C | 4 | 0.0171836 | 1.505 | 48.75740 | 0.814 | 0.0583399 | 0.715 | 4.865156 | 13.92638 | 0.070 | 2.61466 \pm 0.01864 | 14.74 \pm 0.10 | 90.73 | | | 0.0426 \pm 0.00C | |
| 195_VU101-R7 | 8 °C | 4 | 0.0110715 | 2.474 | 30.87482 | 0.825 | 0.0398130 | 0.719 | 3.155407 | 9.21809 | 0.118 | 2.66175 \pm 0.02878 | 15.01 \pm 0.16 | 90.51 | | | 0.0437 \pm 0.00C | |
| 199_VU101-R7 | 8 °C | 4 | 0.0101852 | 2.926 | 26.06134 | 0.837 | 0.0340507 | 0.880 | 2.740711 | 8.25218 | 0.104 | 2.66716 \pm 0.03452 | 15.04 \pm 0.19 | 88.01 | | | 0.0449 \pm 0.00C | |
| 190_VU101-R7 | 8 °C | | 0.0139201 | 2.403 | 38.79632 | 0.864 | 0.0577881 | 0.642 | 4.352513 | 12.74455 | 0.085 | 2.69112 \pm 0.02519 | 15.17 \pm 0.14 | 91.36 | | | 0.0480 \pm 0.00C | |
| 187_VU101-R7 | 8 °C | | 0.0120688 | 2.793 | 38.11509 | 0.850 | 0.0366099 | 0.782 | 3.100359 | 8.94644 | 0.116 | 2.71400 \pm 0.03520 | 15.30 \pm 0.20 | 93.27 | | | 0.0347 \pm 0.00C | |
| 183_VU101-R7 | 8 °C | | 0.0106672 | 3.297 | 24.83340 | 0.796 | 0.0356623 | 0.862 | 2.868956 | 9.01734 | 0.102 | 2.73030 \pm 0.03826 | 15.39 \pm 0.21 | 86.36 | | | 0.0494 \pm 0.00C | |
| 182_VU101-R7 | 8 °C | | 0.0268724 | 1.398 | 37.22994 | 0.847 | 0.0462461 | 0.614 | 3.640101 | 15.04373 | 0.068 | 2.75295 \pm 0.03320 | 15.52 \pm 0.19 | 66.15 | | | 0.0418 \pm 0.00C | |
| 186_VU101-R7 | 8 °C | | 0.0131945 | 2.146 | 33.24870 | 0.876 | 0.0377463 | 0.710 | 3.168763 | 10.00735 | 0.124 | 2.76061 \pm 0.02926 | 15.56 \pm 0.16 | 86.80 | | | 0.0407 \pm 0.00C | |
| 191_VU101-R7 | 8 °C | | 0.0153418 | 1.711 | 40.34068 | 0.842 | 0.0485828 | 0.890 | 4.029500 | 13.87028 | 0.072 | 3.11468 \pm 0.02241 | 17.55 \pm 0.13 | 89.88 | | | 0.0427 \pm 0.00C | |
| Σ | | | 0.1464941 | 0.670 | 357.66922 | 0.270 | 0.4404190 | 0.234 | 36.060832 | 113.39787 | 0.073 | | | | | | | 0.029 |

4.2.

Information on Analysis and Constants Used in Calculations

| | |
|-------------------------------------|--|
| Sample = VU101-R7 | Age Equations = Min et al. (2000) |
| Material = volcanic glass | Negative Intensities = Allowed |
| Location = Cuceje | Decay Constant $40K = 5.460 \pm 0.053 E-10$ 1/a |
| Analyst = K. Kuiper / F. Markovic | Decay Constant $39Ar = 2.940 \pm 0.016 E-07$ 1/h |
| Project = VU101 | Decay Constant $37Ar = 8.230 \pm 0.012 E-04$ 1/h |
| Mass Discrimination Law = LIN | Decay Constant $36Cl = 2.257 \pm 0.015 E-06$ 1/a |
| Irradiation = VU101 | Decay Activity $40K(\beta^-) = 3.310 \pm 0.030$ 1/gS |
| J = 0.00309060 \pm 0.00000309 | Decay Activity $40K(\beta^-) = 27.890 \pm 0.150$ 1/gS |
| Fish Canyon = 28.201 \pm 0.023 Ma | Atmospheric Ratio $40Ar/36Ar(a) = 298.56 \pm 0.31$ |
| IGSN = Undefined | Atmospheric Ratio $38Ar/36Ar(a) = 0.1885 \pm 0.0003$ |
| Preferred Age = Undefined | Production Ratio $36Ar/37Ar(ca) = 0.000673 \pm 0.000004$ |
| Classification = Undefined | Production Ratio $36Ar/39Ar(k) = 0.000264 \pm 0.000002$ |
| Experiment Type = Undefined | Production Ratio $40Ar/39Ar(k) = 0.000860 \pm 0.000070$ |
| Extraction Method = Undefined | Production Ratio $38Ar/39Ar(k) = 0.012110 \pm 0.000030$ |
| Heating = 45 sec | Production Ratio $36Ar/38Ar(cl) = 262.80 \pm 1.71$ |
| Isolation = 5.00 min | Scaling Ratio K/Ca = 0.430 |
| Instrument = HELIX | Abundance Ratio $40K/K = 1.1700 \pm 0.0100 E-04$ |
| Lithology = Undefined | Atomic Weight K = 39.0983 \pm 0.0001 g |
| Lat-Lon = Undefined - Undefined | |
| Feature = Undefined | |

4.3.

| Results | $^{40}\text{Ar}/^{39}\text{Ar}_A \pm 1\sigma$ | %1 σ | $^{40}\text{Ar}/^{39}\text{Ar}_K \pm 1\sigma$ | %1 σ | Age $\pm 1\sigma$ (Ma) | MSWD | N | K/Ca $\pm 1\sigma$ |
|--------------------------|---|--------------------------------|---|---------------|--------------------------------|--------------|-----------------------------|--------------------|
| Weighted mean age | | | | | | | | |
| | 2.62647 | ± 0.01476 | 2.62647 | ± 0.01476 | 14.81 | 1.45 | 4 | 0.0439 \pm 0.00C |
| | | $\pm 0.566\%$ | | | $\pm 0.57\%$ | 23% | | |
| | | Full External Error ± 0.18 | | | 1.82 | 1.82 | 1 σ Confidence Limit | |
| | | Analytical Error ± 0.08 | | | 1.2042 | 1.2042 | Error Magnification | |
| Total fusion age | | | | | | | | |
| | 2.73091 | ± 0.00888 | 2.73091 | ± 0.00888 | 15.39 | | 10 | 0.0431 \pm 0.00C |
| | | $\pm 0.33\%$ | | | $\pm 0.34\%$ | | | |
| | | Full External Error ± 0.17 | | | 15.39 | | | |
| | | Analytical Error ± 0.05 | | | 14.79 | 1.91 | 4 | |
| Normal Isochron | | | | | | | | |
| No Convergence | 297.26 | ± 81.42 | 2.62324 | ± 0.08844 | 14.79 | 15% | 4 | |
| | | $\pm 27.39\%$ | | | $\pm 3.36\%$ | 2.00 | 1 σ Confidence Limit | |
| | | | | | Full External Error ± 0.52 | 1.3818 | Error Magnification | |
| | | | | | Analytical Error ± 0.50 | 100 | Number of Iterations | |
| | | | | | 0.0001484535 | 0.0001484535 | Convergence | |
| Inverse Isochron | | | | | | | | |
| Error Chron | 323.66 | ± 102.41 | 2.59993 | ± 0.09980 | 14.66 | 2.11 | 4 | |
| | | $\pm 31.64\%$ | | | $\pm 3.82\%$ | 12% | | |
| | | | | | Full External Error ± 0.58 | 2.00 | 1 σ Confidence Limit | |
| | | | | | Analytical Error ± 0.56 | 1.4510 | Error Magnification | |
| | | | | | 0.0000737669 | 4 | Number of Iterations | |
| | | | | | 4% | | Spreading Factor | |

5.1.

| Relative Abundances | ^{36}Ar [fA] | %1 σ | ^{37}Ar [fA] | %1 σ | ^{38}Ar [fA] | %1 σ | ^{39}Ar [fA] | %1 σ | ^{40}Ar [fA] | %1 σ | $^{40}\text{Ar}^*/^{39}\text{Ar}_K \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ (%) | K/Ca $\pm 1\sigma$ |
|---------------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|-----------------------|-------------|---|------------------------|---|--------------------|
| 207_VU101-R9 | 1.7044141 | 0.462 | 8.424777 | 0.976 | 0.4536470 | 0.325 | 12.40920 | 0.121 | 531.35779 | 0.033 | 1.86570 \pm 0.19468 | 10.53 \pm 1.10 | 4.36 | 0.63 \pm 0.01 |
| 197_VU101-R9 | 0.0111327 | 3.034 | 5.583872 | 1.089 | 0.2247165 | 0.375 | 19.33322 | 0.108 | 52.05184 | 0.041 | 2.54283 \pm 0.00601 | 14.34 \pm 0.03 | 94.43 | 1.49 \pm 0.02 |
| 211_VU101-R9 | 0.0052452 | 4.995 | 11.781819 | 1.051 | 0.4791445 | 0.261 | 41.75628 | 0.116 | 107.32202 | 0.047 | 2.55456 \pm 0.00372 | 14.40 \pm 0.02 | 99.37 | 1.52 \pm 0.02 |
| 195_VU101-R9 | 0.0093560 | 2.803 | 9.121925 | 1.017 | 0.2296483 | 0.380 | 19.80711 | 0.130 | 52.75824 | 0.067 | 2.55881 \pm 0.00549 | 14.43 \pm 0.03 | 96.04 | 0.93 \pm 0.01 |
| 199_VU101-R9 | 0.0051009 | 4.939 | 8.701540 | 1.103 | 0.1133891 | 0.475 | 9.80567 | 0.123 | 26.24853 | 0.054 | 2.59219 \pm 0.00848 | 14.61 \pm 0.05 | 96.78 | 0.48 \pm 0.01 |
| 205_VU101-R9 | 0.0093705 | 3.289 | 16.345210 | 0.879 | 0.2131660 | 0.415 | 18.03734 | 0.116 | 48.57273 | 0.043 | 2.60995 \pm 0.00610 | 14.71 \pm 0.03 | 96.86 | 0.47 \pm 0.00 |
| 212_VU101-R9 | 0.0021877 | 12.848 | 7.124404 | 1.150 | 0.0636189 | 0.664 | 4.72142 | 0.195 | 12.69932 | 0.097 | 2.67218 \pm 0.01879 | 15.06 \pm 0.11 | 99.25 | 0.28 \pm 0.00 |
| 198_VU101-R9 | 0.0087367 | 3.497 | 5.223656 | 1.511 | 0.2407301 | 0.465 | 20.82423 | 0.114 | 58.33110 | 0.062 | 2.69522 \pm 0.00563 | 15.19 \pm 0.03 | 96.20 | 1.71 \pm 0.03 |
| 210_VU101-R9 | 0.0035512 | 7.522 | 6.169784 | 1.177 | 0.0246348 | 1.042 | 2.22924 | 0.318 | 6.81907 | 0.142 | 2.80582 \pm 0.03731 | 15.81 \pm 0.21 | 91.56 | 0.16 \pm 0.00 |
| 206_VU101-R9 | 0.0080037 | 3.767 | 10.373247 | 1.100 | 0.0306545 | 1.171 | 2.46794 | 0.253 | 8.54691 | 0.072 | 2.83338 \pm 0.03760 | 15.97 \pm 0.21 | 81.58 | 0.10 \pm 0.00 |
| Σ | 1.7670986 | 0.448 | 88.850233 | 0.348 | 2.0633497 | 0.136 | 151.39164 | 0.047 | 904.70755 | 0.021 | | | | |

5.2.

Information on Analysis and Constants Used in Calculations

Sample = VU101-R9
 Material = volcanic glass
 Location = Njzic
 Analyst = K. Kuiper / F. Markovic
 Project = VU101
 Mass Discrimination Law = LIN
 Irradiation = VU101
 $J = 0.00309060 \pm 0.00000309$
 Fish Canyon = 28.201 ± 0.023 Ma
 IGSN = **Undefined**
 Preferred Age = **Undefined**
 Classification = **Undefined**
 Experiment Type = **Undefined**
 Extraction Method = **Undefined**
 Heating = 45 sec
 Isolation = 5.00 min
 Instrument = HELIX
 Lithology = **Undefined**
 Lat-Lon = **Undefined - Undefined**
 Feature = **Undefined**

5.3.

| Results | $^{40}\text{Ar}_A/^{36}\text{Ar}_A \pm 1\sigma$ | $^{40}\text{Ar}^*/^{39}\text{Ar}_K \pm 1\sigma$ | MSWD | N | Age $\pm 1\sigma$ (Ma) | K/Ca $\pm 1\sigma$ |
|-------------------------------------|---|---|---------------|----|--------------------------------|-----------------------------|
| Weighted mean age | | | | | | |
| | | 2.55318 ± 0.00395 | 2.08 | | 14.40 ± 0.03 | 1.17 ± 0.20 |
| | | $\pm 0.15\%$ | 13% | 3 | $\pm 0.18\%$ | |
| | | | 2.00 | | Full External Error ± 0.15 | 1 σ Confidence Limit |
| | | | 1.4408 | | Analytical Error ± 0.02 | Error Magnification |
| Total fusion age | | | | | | |
| | | 2.53744 ± 0.01610 | | 10 | 14.31 ± 0.09 | 0.73 ± 0.00 |
| | | $\pm 0.63\%$ | | | $\pm 0.64\%$ | |
| | | | | | Full External Error ± 0.18 | |
| | | | | | Analytical Error ± 0.09 | |
| Normal Isochron Error Chron | | | | | | |
| | 286.15 ± 25.31 | 2.55497 ± 0.00749 | 3.26 | | 14.41 ± 0.04 | |
| | $\pm 8.84\%$ | $\pm 0.29\%$ | 7% | 3 | $\pm 0.31\%$ | |
| | | | 2.41 | | Full External Error ± 0.16 | 1 σ Confidence Limit |
| | | | 1.8044 | | Analytical Error ± 0.04 | Error Magnification |
| | | | 0.0000001872 | | 1 Number of Iterations | Convergence |
| Inverse Isochron Error Chron | | | | | | |
| | 284.48 ± 25.42 | 2.55626 ± 0.00745 | 3.24 | | 14.41 ± 0.04 | |
| | $\pm 8.94\%$ | $\pm 0.29\%$ | 7% | 3 | $\pm 0.31\%$ | |
| | | | 2.41 | | Full External Error ± 0.16 | 1 σ Confidence Limit |
| | | | 1.8012 | | Analytical Error ± 0.04 | Error Magnification |
| | | | 0.00000765828 | | 4 Number of Iterations | Convergence |
| | | | 5% | | Spreading Factor | |

5.4.

| Results | $^{40}\text{Ar}/^{39}\text{Ar}_k \pm 1\sigma$ | $^{40}\text{Ar}/^{39}\text{Ar}_k \pm 1\sigma$ | Age $\pm 1\sigma$ (Ma) | MSWD | N | K/Ca $\pm 1\sigma$ |
|--------------------------|---|---|---|-------------|----|---|
| Weighted mean age | 2.69834 ± 0.01476 $\pm 0.55\%$ | 2.69834 ± 0.01476 $\pm 0.55\%$ | 15.21 ± 0.08 Full External Error ± 0.18 Analytical Error ± 0.08 | 7.81 0% | 4 | 0.13 ± 0.04 1 σ Confidence Limit Error Magnification |
| Total fusion age | 2.53744 ± 0.01610 $\pm 0.63\%$ | 2.53744 ± 0.01610 $\pm 0.63\%$ | 14.31 ± 0.09 Full External Error ± 0.18 Analytical Error ± 0.09 | | 10 | 0.73 ± 0.00 |
| Normal Isochron | 384.97 ± 24.68 $\pm 6.41\%$ | 2.66497 ± 0.01118 $\pm 0.42\%$ | 15.02 ± 0.06 $\pm 0.43\%$ Full External Error ± 0.17 Analytical Error ± 0.06 | 0.75 47% | 4 | 1 σ Confidence Limit Error Magnification Number of Iterations Convergence |
| Inverse Isochron | 388.81 ± 25.31 $\pm 6.51\%$ | 2.66428 ± 0.01137 $\pm 0.43\%$ | 15.02 ± 0.07 $\pm 0.44\%$ Full External Error ± 0.17 Analytical Error ± 0.06 | 1.02 36% | 4 | 1 σ Confidence Limit Error Magnification Number of Iterations Convergence Spreading Factor |

6.1.

| Labbook* | Collector set-up (5 jumps from H2 to L2) | N | $[\text{CO}_2]_{\text{H}2}/[\text{CO}_2]_{\text{L}2}$ | $[\text{CO}_2]_{\text{H}1}/[\text{CO}_2]_{\text{L}2}$ | $[\text{CO}_2]_{\text{AX}}/[\text{CO}_2]_{\text{L}2}$ | $[\text{CO}_2]_{\text{L}1}/[\text{CO}_2]_{\text{L}2}$ |
|-------------------|--|----|---|---|---|---|
| 20141210_CO2_0026 | H2F – H1C – AXC – L1C – L2C | 10 | 0.9571 ± 0.0123 (1.29%; n=128) | 0.9980 ± 0.0100 (0.45%; n=132) | 1.0075 ± 0.0045 (0.45%; n=134) | 1.0159 ± 0.0043 (0.42%; n=133) |
| 20141215_CO2_0027 | H2F – H1C – AXC – L1C – L2C | 35 | N/A | 1.0006 ± 0.0022 (0.22%; n=490) | 1.0109 ± 0.0022 (0.22%; n=488) | N/A |
| 20150105_CO2_0001 | H2F – H1F – AXC – L1C – L2C | 30 | 0.9570 ± 0.0106 (1.11%; n=699) | 0.9775 ± 0.0100 (1.03%; n=701) | 1.0025 ± 0.0024 (0.24%; n=702) | 1.0124 ± 0.0023 (0.23%; n=709) |
| 20150119_C0002 | H2F – H1C – AXC – L1C – L2C | 22 | 0.9282 ± 0.0131 (1.41%; n=260) | 0.9957 ± 0.0026 (0.26%; n=265) | 1.0041 ± 0.0024 (0.24%; n=263) | 1.0140 ± 0.0023 (0.22%; n=261) |
| 20150201_C0003 | H2F – H1C – AXC – L1C – L2C | 21 | 0.9602 ± 0.0119 (1.23%; n=290) | 0.9951 ± 0.0033 (0.33%; n=290) | 1.0054 ± 0.0032 (0.32%; n=287) | 1.0119 ± 0.0029 (0.28%; n=287) |

Gain factors for different labbooks reported with 1 standard deviation.

*All CO2 experiments are run for 15 cycles with 33s integration time with peak-centering procedure at the start of each run.

7.1.

The bulk tuff samples were crushed or disintegrated and wet sieved over a set of sieves between 63 and 1000 μm . The largest appropriate mineral fraction (125–250 μm or 250–500 μm) was further separated using methylene iodide with densities of 2.54 g/cm^3 and 2.59 g/cm^3 . Samples were subjected to standard magnetic separation techniques (non-magnetic fraction was used), leached with 3% HNO_3 in an ultrasonic bath for 5 min. and finally handpicked under a microscope. We were not able to obtain sanidine from these tuffs (too low K/Ca ratio), but dated a volcanic glass fraction instead. We can exclude plagioclase (density $>2.61 \text{ g}/\text{cm}^3$) or other minerals which have larger densities. There was no alteration on the glass fragments observed. Glass fragments looked very clear, transparent without any sign of alteration.

For the mineral separations approximately 2 kg of each sample was used. Most of the samples had very little material in the range 63 – 250 μm , most of it was $<63 \mu\text{m}$. There was just enough material to get 10 – 20 mg after the separation for irradiation. We did not focus on the accessory mineral fraction, because it does not contain the minerals intended to date with $^{40}\text{Ar}/^{39}\text{Ar}$ method. Nevertheless attempt of heavy mineral analysis was done on this material. From the 2kg of starting material only one slide with heavy mineral was obtained per sample. Those slides did not contain 300 transparent heavy mineral grains for standard modal analysis so this limited data was not used.

Among available materials for dating we chose volcanic glass. Plagioclase has low K and can be compromised by excess argon. Also for plagioclase we should have been dating >10 grains per fusion to obtain sufficient beam sizes on the mass spectrometer and in these type of settings where reworking can be an issue it is preferred to have the amount of grains fused at once as low as possible. So plagioclase wouldn't be our first choice. Unaltered biotite could have been dated, if present, but generally has some recoil issues, making biotite slightly too old. We had biotite in our samples but not in sufficient quantity in samples from Nježić and Laz. With biotite from Jovac and Čučerje we did an extra heavy liquid step. If biotite is lighter than 3.0 g/cm^3 this is often an indication of minor alteration e.g. to chlorite. That was the case with biotite from Jovac so we have not attempted to date biotite from that sample. Sample from Čučerje had suitable biotite but also had transparent grains of volcanic glass so we chose volcanic glass.

The resulting mineral separates and the standard (Fish Canyon tuff sanidine, FCs, KUIPER et al., 2008) were wrapped in aluminum foil and sent for irradiation (12 hours) at the Oregon State CLICIT facility. Sample and standards were loaded in 185 hole trays in 2mm pits, placed in an ultra-high vacuum system and baked in two steps: during 1 day at 250°C in a vacuum system at $<10^{-5}$ mbar, followed by a 1 day bake out in the extraction line at $\sim 125^\circ\text{C}$. Samples were fused using a Synrad 48-5 CO_2 laser and custom made beam delivery system. The released gas was purified in an in-house designed sample clean up line (St172, NP10 and Ti getters) and analyzed on a Helix MC noble gas mass spectrometer.

For part of the samples (labbooks 20141223_185_06a and 20141223_185_06b) the five argon isotopes were measured simultaneously with ^{40}Ar and ^{39}Ar on the H2 and H1-Faraday positions with 10^{12} resistor amplifiers, ^{38}Ar on the AX-CDD, ^{37}Ar on the L1-CDD and ^{36}Ar on the L2-CDD in the first jump and ^{39}Ar on the H2-Faraday in jump 2. Gain factors for different labbooks reported with 1 standard deviation can be found in supplementary material – file 1.

Part of the samples (labbook 20150122_T0002) are measured with the five argon isotopes simultaneously with ^{40}Ar on the H2-Faraday position with a 10^{12} resistor amplifier, ^{39}Ar on the H1-CDD, ^{38}Ar on the AX-CDD, ^{37}Ar on the L1-CDD and ^{36}Ar on the L2-CDD.

All samples, blanks, standards and pipettes are measured during 33s integration time for 15 cycles per analysis. Apart from blanks all analyses started with a peak centering routine on L2 (labbooks 20141223_185_06a and 20141223_185_06b) or H2 (labbooks 20150122_T0002). Blanks use the peak positions of the preceding measurements.

We used the regular software controlled gain procedure for the 10^{12} Ohm amplifiers, using a stable reference current to determine the gain calibration factor between the H2 and H1 amplifiers. This approach is not applicable to the CDDs and we therefore developed a different approach.

Gain calibration for the CDDs was done by peak jumping a CO_2 reference beam (mass 43.989829) on all detectors (5 jumps). CO_2 is present in as background in the mass spectrometer and is measured in dynamic mode yielding an intensity of $\sim 2\text{--}4$ fA of CO_2 . Baseline correction is done by subtracting the off peak data for each collector. Ratios of $[\text{CO}_2]_{\text{H2}}/[\text{CO}_2]_{\text{L2}}$, $[\text{CO}_2]_{\text{H1}}/[\text{CO}_2]_{\text{L2}}$, $[\text{CO}_2]_{\text{AX}}/[\text{CO}_2]_{\text{L2}}$, $[\text{CO}_2]_{\text{L1}}/[\text{CO}_2]_{\text{L2}}$ are calculated for each cycle and repeated in blocks of 15 cycles. After each block of 15 cycles, a new block starts with a peak centering routine. Uncertainty for Faraday gain corrections is relatively large ($\sim 1\%$) due to the noise of the Faraday cups compared to the baseline.

For data reduction we used the ArArCalc software (KOPPERS, 2002), although it is not fully compatible with the demand of multi-collection–set-up. For unknowns and standards we performed our initial data reduction in ArArCalc. We exported the uncorrected intercept values for blanks, unknowns and standards to an Excel spreadsheet. Gain correction was done in Excel and intercept values of corrected blanks, unknowns and standards were copied back in the corrected ArArCalc.age file. As a next step the mass discrimination factor based on simultaneous measurement of ^{40}Ar on the H2 Faraday and ^{36}Ar on the L2 CDD is applied. Also these intensities are corrected for gain based on CO_2 measurements. In our calculations we used FCs of 28.201 Ma (KUIPER et al., 2008), MIN et al. (2000) decay constants and the atmospheric air value of LEE et al. (2006).

8.1.

| | Jovac | Laz | Čučerje | Nježić |
|---|-----------------|------------------|------------------|---------------------------------------|
| Min. & max. age | 14.87-15.80 Ma | 14.04 – 18.31 Ma | 14.53 – 15.72 Ma | 14.30 – 16.18 Ma |
| Weighted mean age with data points excluded until MSWD < t-test statistic | 15.10 ± 0.06 Ma | 15.42 ± 0.15 Ma | 14.81 ± 0.08 Ma | 14.40 ± 0.03 Ma or 15.21 ± 0.08 Ma |
| Weighted mean age with all data included (except outliers in fig X) and standard error multiplied by square root MSWD | 15.57 ± 0.08 Ma | 15.68 ± 0.16 Ma | 15.05 ± 0.11 Ma | 14.59 ± 0.11 Ma |
| Mean and standard deviation of all data excluding outliers | 15.36 ± 0.27 Ma | 15.82 ± 0.83 Ma | 15.15 ± 0.32 Ma | 14.95 ± 0.61 Ma |

*Errors are ±1σ analytical errors