^{40}Ar-^{39}Ar methodology

The rock samples were ground, sieved, washed with distilled water and rinsed with 99.5% acetone and dried overnight at 60º C. From the fraction between 0.7 to 0.3 mm in size, a concentrate was separated. The rock fragments were examined under the microscope to eliminate phenocrystals by hand, in which case the sample is labeled groundmass otherwise it is named whole rock sample. For the laser fusion experiments conducted with the VG5400 mass spectrometer, aliquots of ~ 0.01 g of sample was used. For the experiments conducted with the MS-10 mass spectrometer, aliquots of 0.5 to 2.0 g were used. All the samples were irradiated in position 5C of the U-enriched, nuclear reactor of McMaster University in Canada. The samples were irradiated in three different groups: CIC-45 (samples DG, AN, DH); CIC-46 (samples DF, AF, DE, AO) and CIC-56 (samples AL2, BV, BZ, BW). Capsules CIC-45 and CIC-46 received a dose of 10 MWH and included as irradiation monitors biotite CATAV 7-4 (89.13± 0.35 Ma: internal standard calibrated with hornblende hb 3gr at the University of Toronto and with hornblende MMhb 1 at the University of Nice) and biotite HD-B1 (24.18±0.09 Ma; Schwarz and Trieloff 2007). Capsule CIC-56 received a dose of 30 MWH using Cd liner and included as irradiation monitor sanidine TCR-2 (28.34±0.28 Ma, Renne et al., 1998). The aliquots of the monitors were distributed evenly between the samples. Upon irradiation the monitors were fused in one-step to calculate the irradiation parameter J. The age of the samples was calculated with the J value obtained from the average of the monitors closest to them. Samples DE and DH, were step-heated with a Modification Ltd Ta furnace on line with an MS-10 mass spectrometer. The rest of the experiments were performed with the VG5400 mass spectrometer using a Coherent Innova 70c argon laser to heat the samples. Only samples DF and AN, were analyzed with both mass spectrometers. These experiments yielded reproducible results. The first step of the data reduction was the blank correction, to do this blanks were routinely measured before each experiment with all the argon masses measured. The blank correction was performed on a mass to mass basis since the blank composition is not atmospheric. The blanks for the MS-10 runs, consisted of a one-step measurement at 1350°C, a temperature dependent factor was applied for the blank correction (F = 0.3 for T< 800°C; F = 0.4 for 800 < T < 1100°C; F = 0.6 T > 1100°C). Typical blanks for mass 40 were ~2.6 E-8 cc STP and ~0.9 E-10 cc STP for mass 36. For the laser step-heating experiments conducted on the VG5400 mass spectrometer, every sample measurement was preceded by a blank run. The blank runs were performed following the same procedure for the sample with the exception of turning on the laser. Blanks for the VG5400 have a typical composition of 1.3 E-12 cc STP for mass 40 and ~6.7 E-14 CC STP for mass 36. Upon blank subtraction the argon isotopes were corrected for mass discrimination using

$(^{40}Ar/^{36}Ar)_{atm}$ = 295.5. At least once a month a sample of zero age basalt was laser fused in one step to determine the discrimination factor for the VG5400 mass spectrometer. The prep-line for the MS-10 mass spectrometer is equipped with a house-made air pipette. Each experiments performed in the MS-10 was followed by a measurement of atmospheric argon pipette to determine the mass discrimination correction. The factors used to correct for calcium, potassium and chlorine neutron induced interference reactions were: $(^{39}Ar/^{37}Ar)_{Ca} = (6.51 \pm 0.31) \times 10^{-4}$; $(^{36}Ar/^{37}Ar)_{Ca} = (2.54 \pm 0.09) \times 10^{-4}$; $(^{40}Ar/^{39}Ar)_{K} = (1.560 \pm 0.040) \times 10^{-2}$ for samples irradiated in capsules CIC-45 and CIC-46. The factors used for samples irradiated in capsule CIC-56 in which a Cd liner was used were: $(^{39}Ar/^{37}Ar)_{Ca} = (6.50 \pm 0.47) \times 10^{-4}$; $(^{36}Ar/^{37}Ar)_{Ca} = (2.55 \pm 0.28) \times 10^{-4}$; $(^{40}Ar/^{39}Ar)_{K} = 0$. Mass 36 was also corrected for chlorine derived ^{36}Ar (^{35}Cl (n, γ) $^{36}Cl \rightarrow {}^{36}Ar + \beta$ with $t_{½} = 3.1 \times 10^{5}$ a). Isotopes ^{37}Ar and ^{39}Ar were corrected for radioactive decay. The constants recommended by Steiger and Jäger 1977 were used in all the calculations while all the straight line, calculations were performed with the equations presented in York et al., 2004. All errors are reported at 1σ level. The errors in the integrated, plateau, isochron and weighted mean age include the uncertainty in the J parameter. Additionally for the plateau, isochron and weighted mean ages, the goodness of fit was included in the age uncertainty whenever the MSWD exceeded 1. The integrated ages were calculated adding the fractions of the step-heating experiments. Plateau ages were calculated with the weighted mean of three or more consecutive fractions, which were in agreement within 1σ errors excluding the uncertainty in J. All the data were plotted in the $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ correlation diagram to determine the composition of the $(^{40}Ar/^{36}Ar)_{i}$ of the samples. Some samples were characterized by poor ^{40}Ar radiogenic content, therefore the data distribution in the correlation diagram did not constrain a reliable isochron age. In Table 1 a summary of the ^{40}Ar-^{39}Ar results is presented, it includes: Code; rock sample name; geographic coordinates; material analyzed; and preferred age. ^{40}Ar-^{39}Ar details for each sample is presented in file **“Electronic supplementary Ar-Ar data.xlsx”**, where the relevant information is presented for all the fractions analyzed. The figures included contain: age spectra; $^{37}Ar_{Ca}/^{39}Ar_{K}$ diagram; and $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ correlation diagram. To facilitate the comparison in the $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ correlation diagrams of the experiments conducted in different irradiations, the argon isotopic ratios were normalized to a fixed J value of 0.001.

Code DG: groundmass sample VB 104, Figure A1 DG

Three laser step-heating experiments were performed on groundmass fragments from the sample. The two, three and four fractions collected respectively on the step-heating experiments yielded reproducible results. The bulk of the ^{39}Ar was released at high temperature. This sample is characterized by very small $^{40}Ar^*$ (radiogenic ^{40}Ar) content, the data plot very close to the ordinate axis of the $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ correlation diagram and therefore do not constrain a reliable isochron age (t_c = 166 ± 265 Ka; $(^{40}Ar/^{36}Ar)_i$ = 292 ± 8, MSWD = 0.67 for n = 9). The preferred age of 78 ± 56 Ka (MSWD = 0.11 for n = 4) was obtained from the weighted mean of the fractions that represent the bulk of the ^{39}Ar.

Code DF: whole rock sample VB 48, Figure A2 DF

Three laser step-heating experiments were conducted, the argon isotopes were analyzed with the VG5400 mass spectrometer plus two step-heating experiments with the temperature controlled Ta-furnace, the argon isotopes for these experiments was analyzed with the MS-10 mass spectrometer. The results for both mass spectrometers are reproducible. A slight saddle shape is displayed in the age spectra, but the middle section clusters about 80 Ka. Furthermore, a segment with an age of 73 ± 62 Ka is defined by 66.91% of the ^{39}Ar released with MSWD = 0.04 for n =2. The preferred age t_c = 81 ± 35 Ka; $(^{40}Ar/^{36}Ar)_i$ = 299 ± 1, MSWD = 1.36 for n = 22 is the isochron age, calculated with all but one of the fractions obtained in the five experiments conducted.

Code AL2, whole rock sample VB 137, Figure A3 AL2

Two VG5400 laser fusion experiments were performed with this sample: one-step fusion and a step-heating run. The results obtained were reproducible. These yielded low radiogenic argon content. For the step-heating experiment, the bulk of the ^{39}Ar (81%) defines a weighted mean age of 109 ± 45 Ka (MSWD = 0.6 for n =2), this is the preferred age for sample AL2. This age is statistically indistinguishable from the isochron age of 123 ± 94 Ka $(^{40}Ar/^{36}Ar)_i$ = 295 ± 13, MSWD = 4.1 for n = 5 calculated with all the data.

Code AF, groundmass sample VB 60, Figure A4 AF

Two laser step-heating experiments plus one-step laser fusion were performed with a groundmass concentrate of sample VB 60. The data yielded an isochron age of 300 ± 83 Ka $(^{40}Ar/^{36}Ar)_i$ = 297 ± 5, MSWD = 0.02 for n = 5. The preferred age is taken from the weighted mean age 315 ± 36 Ka MSWD = 0.02 for n = 5.

Code BV, groundmass sample VB 8b, Figure A5 BV

A laser step-heating experiment and a one-step laser fusion were. Four fractions were collected during the step-heating experiment. Consecutive fractions representing 63.5% of the ^{39}Ar released, define the weighted mean age 882 ± 189 Ka MSWD = 0.97 for n = 3. The distribution of the data does not allow to obtain a reliable isochron age, the straight line displayed in the $^{36}Ar/^{40}Ar$ versus $^{39}Ar/^{40}Ar$ correlation diagram, was calculated forcing the Y-intercept through ($^{40}Ar/^{36}Ar$) atmospheric value and is presented only for illustrative purposes.

Code AN, groundmass sample VB 131, Figure A6 AN

Three laser step-heating experiments were performed with the VG5400 mass spectrometer and two more with the temperature controlled Ta-furnace connected on line with the MS-10 mass spectrometer. The sample yielded reproducible results. The age spectra have slight saddle shape, with a wide flat central region. The isochron age 949 ± 37 Ka was calculated combining the data from the five experiments $(^{40}Ar/^{36}Ar)_i$ = 301 ± 4 with MSWD = 1.76 for n = 22.

Code DE, whole rock sample VB 34-1, Figure A7 DE

Two step-heating experiments were performed with the temperature controlled Ta-furnace. The argon isotopes were measured with the MS-10 mass spectrometer. The isochron age of 505 ± 527 Ka with $(^{40}Ar/^{36}Ar)_i$ = 306 ± 9 with MSWD = 0.21 for n = 9 In spite of an acceptable MSWD, their distribution, do not constrain the X-intercept therefore the isochron age is not considered reliable. The preferred age 995 ± 120 Ka MSWD = 0.23 for n =4 was obtained from the weighted mean of the central fractions from each experiment, representing 56.66% and 44.19% of the ^{39}Ar released.

Code DH, groundmass sample VB 105, Figure A8 DH

Two step-heating experiments were performed with the temperature controlled Ta-furnace. The argon isotopes were measured with the MS-10 mass spectrometer and yielded reproducible results. The distribution of the data constrain the isochron age of 1049 ± 81 Ka with $(^{40}Ar/^{36}Ar)_i$ = 303 ± 2 MSWD = 0.24 for n = 11.

Code AO, groundmass sample VB 132, Figure A9 AO

Six one-step laser fusion experiments plus one laser step-heating experiment were performed on the groundmass concentrate. The one-step experiments were plotted as a pseudo-age spectrum, where each single fusion is shown as an individual fraction. On top of these data the step-heating experiment is presented. Two fractions representing 57.1% of the ^{39}Ar released from the step-heating experiment yield a weighted mean age of 1137 ± 90 Ka MSWD = 0.009. The preferred age obtained with five one-step experiments plus the two central fractions of the step-heating experiment, is the isochron age of 1095 ± 105 Ka with $(^{40}Ar/^{36}Ar)_i$ = 295 ± 6 and MSWD = 1.71 for n = 7.

Code BZ, whole rock sample VB 140, Figure A10 BZ
One laser step-heating and a laser one-step fusion experiments were performed. The two experiments yielded similar ages. The data define an isochron age of 1414 ± 129 Ka, with a $(^{40}Ar/^{36}Ar)_i$ = 285 ± 5 MSWD = 1.5 for n =4. Because the $(^{40}Ar/^{36}Ar)_i$ obtained is below the atmospheric value of 295.5, the isochron age is not considered reliable. The preferred age for this sample is taken from the step-heating experiment, it was selected from the fraction with the highest radiogenic yield, which is represented by a segment with 45.66% of the ^{39}Ar released indicating an age of 1322 ± 100 Ka.

Code BW, whole rock sample VB 10b, Figure A11 BW
A laser step-heating and a one-step laser fusion experiments were performed. The data yield a poorly constrained (MSWD = 5.5 for n = 4) isochron age of 1511 ± 206 Ka with a $(^{40}Ar/^{36}Ar)_i$ = 333 ± 34. The bulk of the ^{39}Ar (84.77%) was released in the three middle fractions of the step-heating experiment. The weighted mean of their ages yields the preferred age 1628 ± 56 Ka with MSWD = 1.4 for n = 3.

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Table 1

Summary of ^{40}Ar-^{39}Ar ages

CODE	Rock sample	Material dated	Age in Ka
DG	VB 104	gms	78 ± 56
DF	VB 48	wr	81 ± 35
AL2	VB 137	wr	109 ± 45
AF	VB 60	gms	315 ± 36
BV	VB 8b	gms	881 ± 189
AN	VB 131	gms	949 ± 37
DE	VB 34-1	wr	995 ± 120
DH	VB 105	gms	1049 ± 81
AO	VB 132	gms	1095 ± 105
BZ	VB 140	wr	1322 ± 100
BW	VB 10b	wr	1511 ± 206

Complete data for all the experiments performed follows. Decay constants used to calculate the ages: $\lambda_e = 0.581 \times 10^{-10}$ a^{-1}; $\lambda_\beta = 4.963 \times 10^{-10}$ a^{-1}, errors are 1 σ