Annex 3. ⁴⁰Ar/³⁹Ar step-heating experiments

Our criteria for the plateau and mini-plateau ages are as follows: plateaus and mini-plateaus must include at least 70% and 50% of ³⁹Ar released, respectively, distributed over a minimum of 3 consecutive steps and a probability of fit of at least 0.05. In this study they are both given the same validity. Plateau and mini-plateau ages are calculated using the mean of all the plateau steps, each weighted by the inverse variance of their error. Integrated ages (that compare with total fusion ages) are calculated using the total gas for each Ar isotope.

1. Step heating experiments

1.1 Fish Canyon plagioclase

The largest aliquots of nine of the fourteen fractions were step-heated (Fig. A3.1 and Table 1). Seven out of nine step-heating experiments yielded plateau ages, ranging between 28.42 ± 0.20 and 23.6 ± 3.0 Ma. In general, plateau ages for the fractions between 125 and 28 µm are in good agreement with the total fusion age obtained on other aliquots from the same fraction. The 125-85µm and 74-63µm fractions failed to produce any plateau and yield much older integrated ages (31.04 ± 0.30 and 29.14 ± 0.22 Ma, respectively) compared to the total fusion ages. The reason why these fractions are perturbed is not clear. The integrated (equivalent to total fusion) ages obtained on fraction that produced a plateau age are in good agreement with the single-step total fusion ages obtained on these fractions.

From 22 to 5 μ m, three plateau ages ranging from 23.6 \pm 3.0 to 28.10 \pm 0.34 Ma were obtained (Fig. A3.1). Similarly, to the total fusion ages obtained on these fractions (Fig. 1), the plateau ages are not correlated with the fraction size, with the youngest age being obtained for the 22-15 μ m fraction. In addition, the plateau age does not necessarily correspond to the integrated age of the same experiment (The 10-5 μ m fraction) or to the weighted-mean total fusion ages obtained on the same aliquots (the 22-15 μ m fraction). No spectra were generated for the grain size below 5 μ m due to the low amount of material available for this fraction.

1.2 Hb3gr hornblende

The relatively low amount of material available for each fraction prevented us to carry stepheating measurements on most of the fractions. Only the 53-38 µm and 10-5 µm fractions have been step-heated and yielded plateau ages of 1068 ± 3 Ma (91% ³⁹Ar released; P = 0.35) and 1076 ± 4 Ma (71% ³⁹Ar released; P = 0.30), respectively (Fig. 5). These ages are indistinguishable from the nominal age of Hb3gr with respective Δ_{Hb3gr} of $-0.7 \pm 1.1\%$ and $-1.8 \pm 1.1\%$. On the other hand, the plateau age obtained for the 10-5 µm fraction is distinctly younger than the ³⁹Ar recoil-corrected age of Hb3gr with a Δ_{Hb3gr} (corr.) of -1.8 ± 1.1 % (Fig. 4). Integrated ages of the 53-38 µm and 10-5 µm fractions are slightly younger than their equivalent plateau ages with apparent ages of 1066 ± 3 and 1069 ± 4 Ma, respectively although plateau, integrated and total fusion ages all overlap within uncertainty (Table 2). As for the plateau age, the 10-5 µm fraction has an integrated age distinct from the ³⁹Ar-recoil corrected age of Hb3gr with Δ_{Hb3gr} (corr.) = -2.5 ± 1.1 %. The difference between the plateau and integrated ages is caused in both cases by younger steps in high-temperature parts of the age spectra.



Fig A3.1. ${}^{40}Ar/{}^{39}Ar$ spectra plotted versus the cumulative percentage of ${}^{39}Ar$ released for FC plagioclase samples. Errors are quoted as 2σ and do not include systematic errors. Both plateau (and mini-plateau) and integrated ages are indicated. MSWD and P statistical tests are provided with plateau and mini-plateau ages.



Figure A3.2. ${}^{40}Ar/{}^{39}Ar$ and $[Ca/K]_{Ar}$ spectra plotted versus the cumulative percentage of ${}^{39}Ar$ released for Hb3gr hornblende samples. Errors are quoted as 2σ and do not include systematic errors. Both plateau (and mini-plateau) and integrated ages are indicated. MSWD and P statistical tests are provided with plateau and miniplateau ages.

2. Discussion of the step heating data

2.1 FCp

The fractions of FCp that yielded a plateau age do not show any particular structure in their age spectra (Fig. A3.1). Only the 15-10 μ m fraction shows a composite spectrum with the first 15% of the spectrum showing ages around 18 Ma, whereas the other 85% yielded an older plateau age indistinguishable from the ³⁹Ar recoil-corrected age of FCp (Δ FCp(corr.) = -1.0 ± 1.2 %). For a given fraction, plateau and integrated ages are similar to the weighted mean age of the total fusion experiments, except for the 22-15 μ m fraction where the total fusion ages are older (Table 1).

2.2 Hb3gr

Only two step-heating experiments were carried out on Hb3gr, for the 53-38 μ m and 10-5 μ m fractions (Fig. A3.2). None of these spectra shows a well-defined structure although they both exhibit lower ages for high-temperature extraction steps. In addition, the Ca/K ratio spectrum obtained for the 10-5 μ m fraction shows many steps, where the Ca/K is within error of 0 suggesting loss of ${}^{37}Ar_{Ca}$. The integrated (total fusion) ages are both lower than the age expected in the case of ${}^{39}Ar$ recoil loss alone (negative Δ Hb3gr(corr.)) and corroborate the information obtained by total fusion experiments.