Supplementary text for:

Larick, R., Ciochon, R. L., Zaim, Y., Sudijono, Suminto, Rizal, Y., Aziz, F. Reagan, M. & Heizler, M. (2001) Early Pleistocene $^{40}Ar/^{39}Ar$ ages for Bapang Formation hominins, Central Jawa, Indonesia, *Proc. Acad. Sci. USA*, 10.1073/pnas.081077298

Procedural Details

Samples.

Mineral separates obtained by magnetic, heavy liquid, and hand-picking techniques.

The absolute age of Fish Canyon tuff sanidine is still in debate (1, 2). We use an age of 27.84 Ma based on Cebula *et al*. (3) and recalculated to the monitor MMhb-1 age of 520.4 Ma (4).

Samples and FC-1 irradiated 4 h (D-3 position) at Texas A&M.

Instrumentation.

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

Mo double-vacuum resistance furnace: 7 min steps; reactive gases removal: three SAES GP-50 getters (two at ≈450°C, one at 20°C); W filament exposure at ≈2000° C.

50 W C02 laser furnace: 2-min steps; defocused laser beam; reactive gas removal: 20 min with two SAES GP-50 getters (one at ≈450°C, one at 20°C); W filament exposure at ≈2000°C; cold finger at -140°C.

Analytical parameters.

Electron multiplier sensitivity: furnace averaged 2.31 x 10^{-16} moles/pA; laser averaged 1.78 x 10^{-16} mol/pA (laser).

Total furnace system blanks plus backgrounds: ≈900, 4, 1, 2, 3.5 x 10^{-17} mol at masses 40, 39, 38, 37, and 36, respectively, for temperatures <1300°C.

Total laser system blanks plus backgrounds: 320 ± 5%, 2.74 ± 5%, 0.24 ± 20%, 1.1 ± 10%, 1.4 ± 5% x 10^{-17} mol at masses 40, 39, 38, 37, and 36, respectively.

J-factors determined to ±0.1% precision by $C0_2$ laser-fusion of four single crystals from each of six radial irradiation tray positions.

K-glass and CaF_2-determined interfering nuclear reaction correction factors: $(^{40}Ar/^{39}Ar)_K = 0.0002 \pm 0.0003$; $(^{36}Ar/^{37}Ar)_{Ca} = 0.00028 \pm 0.000011$; and $(^{39}Ar/^{37}Ar)_{Ca} = 0.00089 \pm 0.0003$.

Analytical parameters unique to sample Tjg-2.

Electron multiplier sensitivity averaged 2.09×10^{-16} mol/pA.

Total laser system blanks plus backgrounds: 140, 0.6, 0.2, 0.5, 0.8 x 10-17 moles at masses 40, 39, 38, 37, and 36, respectively.

Blanks were assigned by either linear or cubic regressions of the isotopic blank data collected during the analysis of Tjg-2.

Age calculations.

Total gas ages and errors: by weighting individual steps by the fraction of ^{39}Ar released.

Plateau ages determined by weighting each plateau step by the inverse of the variance (5); errors calculated as per Taylor (6).

If the mean square of weighted deviates (MSWD) value falls above the 95% confidence interval for n-1 degrees of freedom, the error is increased by the square root of the MSWD.

Decay constants and isotopic abundances after Steiger and Jäger (7).

Isochron age determined with York (8) regression method. Regression steps determined by sequential removal of statistical outliers (9) until MSWD falls within the 95% confidence window (10) for n-2 degrees of freedom.

Analytical Methods and Results

Pumice samples from the Bapang Formation of the Sangiran dome SE quadrant were received without indication of sampling site or stratigraphic position. Parenthetical labels listed after the sample site identifications are the blind field labels solely used at the New Mexico Geochronological Research Laboratory (NMGRL). Aliquots of all mineral separates were incrementally heated to obtain $^{40}Ar/^{39}Ar$ plateau ages. Replicate analyses for all of the hornblende samples were conducted to test for sample homogeneity and analytical reproducibility, and to obtain enough data for meaningful statistical analyses. One split of plagioclase from site Bpg B-1 (SP-98-18) was analyzed. Detailed isotopic data for each analysis is given in Table 3.

The analytical protocol evolved in three stages. In the initial experiment, relatively large samples (≈150–200 mg) were heated in a double vacuum resistance furnace. Large samples were chosen to provide a high single to blank ratio, thereby diminishing errors associated with uncertainties in blank values. Furnace heating provided efficient gas cleanup and precise temperature control, with more consistent signal sizes resulting for each step. When uniformly heated, large samples yield better potential age spectrum complexities. Splits from Pcg-2 (99-4-2a) and Gwn D-2 (99-10-4a) were successfully analyzed with this method. Nevertheless, even with long gas cleanup times, the large samples were detrimental to mass spectrometer performance.

In the second stage, sample size was decreased to ≈30–50 mg, and fewer heating steps were used. Three replicate splits from Gwn D-2 (99-10-4) were step-heated in five increments. The smaller samples provided better results, but the signal to blank ratio was fairly low and the blank was not as stable as desirable.

In the third stage, 10–20 mg samples were subject to a defocused, flat power profile, 6 mm square CO_2 laser beam. Laser heating provided lowered and more reproducible blanks relative to the furnace. In addition, extracted gas could be passed through a cold finger (-140°C) to trap water evolved from the amphiboles. Disadvantages included minor nonuniform heating of the sample and less predictability in controlling the amount of argon evolved per heating step. Six heating steps were specified, but not all provided measurable gas for each sample. In a few cases [e.g., Gwn D-2a (99-10-4-a) and Pcg-2a (99-4-2-a)], more than six steps were measured and reported.

Four replicate age spectrum runs were conducted on most samples; however, two, three, and five were performed for samples from Bpg B-1 (SP-98-18), Jtr-1 (SP-98-26), and Sbk-1 (99-9-1), respectively. Age spectra for all of the replicate analyses share common features. Initial heating steps are enriched in atmospheric argon. The higher temperature steps generally show an increase in radiogenic yield prior to the highest temperature steps, which reveal a decrease in radiogenic argon (Figs. 5–13 and Table 3). Most of the hornblende K/Ca spectra are uniform and range from ≈0.02–0.03 (Figs. 5–13 and Table 3). The Bpg B-1 (SP-98-18) plagioclase separate had significantly higher K/Ca values (≈0.039) than the two hornblende splits (0.029) (Table 3). The hornblende replicates were assigned weighted mean plateau ages ranging from 0.67–1.57 Ma and are compiled in Table 4. Only 5 of the 34 plateau ages yielded MSWD values above the 95% confidence level (Figs. 5–13 and Tables 3 and 4). The majority of the age spectra were flat for 100% of the ^{39}Ar released. Some analyses, nevertheless, revealed significant spectrum complexities. A few samples gave anomalous old ages for some early and late gas fractions [e.g., Jtr-1c (SP-98-26-c), Fig. 5*c*], whereas a few spectra yielded somewhat undulatory patterns [e.g., (99-2-1-d), Fig. 11*d*]. The

plagioclase age spectrum reveals an age gradient with apparent ages climbing from ≈1 to 4 Ma (Fig. 6*c*).

Pumice from Tjg-2 (99-8-10) was submitted for analysis after the eight others. The hornblende separate was laser step-heated with slightly different analytical parameters (see Procedural Details). Each of four replicate analysis yielded a flat age spectrum with 100% of the ^{39}Ar released defining statistically acceptable plateau ages ranging from 1.05 ± 0.09 to 1.44 ± 0.11 Ma (Fig. 13 and Table 3). The K/Ca spectra each have an initially high value that then step down to a constant value of ≈0.025. Radiogenic yields are generally quite low and are typically 5–10% for the most precise heating steps (Fig. 13 and Table 3).

Discussion

Replicate run plateau ages are summarized in Fig. 14. These probability diagrams do not show perfect gaussian distributions for the replicate analyses and for five of the nine samples the calculated MSWD values for these distributions are higher than predicted based on the assigned analytical error (Fig. 14 and Table 4). This apparent lack of reproducibility in the replicate analyses indicates either an underestimated analytical error (overestimated precision) or geological scatter (population heterogeneity), or both. Geological scatter is considered more likely because four of the nine samples have acceptable MSWD values and all separates have similar age and argon concentrations such that all samples are essentially equally sensitive to lab error propagation.

There are three probable causes for geological scatter of the replicate ages: minor xenocrystic contamination, excess argon within the hornblendes, or plagioclase inclusions and/or argon loss associated with alteration. In addition, old ages associated with initial and late heating steps could be caused by minor excess argon contamination. Isochron analysis provides a partial test for excess argon, subject to the limited number of steps generated for each age spectrum. Additionally, low-resolution laser step-heating age spectrum analysis tends to homogenize thermally distinct argon reservoirs; therefore, if excess argon exists within samples it might not be clearly revealed on isochron plots. Two samples, Jtr-1a (SP-98-26-a) and Bpg B-5c (99-2-1-c), appear to have minor xenocrystic material and apparent ages greater than 20 Ma for the high temperature steps (Table 3).

Despite the scatter of the replicate ages for some samples, eruption dates are assigned to each sample by weighted mean combination of the plateau ages (Fig. 14 and Table 4). The eruption ages define three discrete nodes at ≈1.0, 1.25, and 1.5 Ma on the probability diagram (Figure 15) and these nodes conform with sample stratigraphy (Fig. 16). Consequently, these nodes most likely indicate pulses of volcanism in the vicinity of the Solo basin.

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