

Sample preparation

Samples were trimmed to remove any weathering/staining using a diamond water saw. Samples were split using a hydraulic rock splitter to reduce the sample to manageable fragments for the jaw-crusher. The jaw-crusher is used to reduce the split sample to a size suitable for sieving. They were then passed over with a strong hand magnet to remove any metal particles introduced during crushing. Dust was removed by washing the samples in deionized water in an ultrasonic bath for 10 minutes. Samples were leached in 3N HNO_3 for 20 minutes in an ultrasonic bath at 50°C. This was repeated until a clear solution was obtained. Samples were then rinsed with deionized water and then washed with deionized water in an ultrasonic bath for 10 minutes. Biotite was then handpicked under a binocular. For each sample 1 g of groundmass separate was attained.

Methods

Samples and neutron flux monitors were placed in aluminum discs and stacked in quartz tubes. The relative positions of wells in the discs were precisely measured for later reconstruction of neutron flux gradients. The sample package was irradiated in the Oregon State University reactor, Cd-shielded facility. Alder Creek sanidine (1.2056 ± 0.0019 (1 σ) Ma, [1]) was used to monitor ^{39}Ar production and establish neutron flux values (J) for the samples. Gas was extracted from samples via step-heating using a mid-infrared (10.6 μm) CO_2 laser with a non-gaussian, uniform energy profile and a 3.5 mm beam diameter. The samples were housed in a doubly-pumped ZnS-window laser cell and loaded into a copper planchette containing four 2.6 cm^2 square wells. Liberated argon was purified of active gases, e.g., CO_2 , H_2O , H_2 , N_2 , CH_4 , using three Zr-Al getters; one at 16°C and two at 400°C. Data were collected on a GV instruments ARGUS V multi-collector mass spectrometer using a variable sensitivity faraday collector array in static collection (non-peak hopping) mode [2,3]. Time-intensity data are regressed to t_0 with second-order polynomial fits to the data. Mass discrimination was monitored on a daily basis by comparison to running-average values of an air standard. The average total system blank for laser extractions, measured between each sample run, was 2×10^{-15} mol ^{40}Ar , 1×10^{-17} mol ^{39}Ar , 2×10^{-17} mol ^{36}Ar . All data are blank, interference and mass discrimination corrected using the MassSpec software package (MassSpec, version 8.058, authored by Al Deino, Berkeley Geochronology Center, Version 8.058).

Any use of trade, firm, or product names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

References

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2. Mark, D.; Barfod, D.; Stuart, F.; Imlach, J. The ARGUS multicollector noble gas mass spectrometer: Performance for $^{40}\text{Ar}/^{39}\text{Ar}$ geochronology. *Geochemistry, Geophysics, Geosystems* **2009**, *10*.
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