## Whole-rock analyses, XRF

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## USGS analyses, XRF

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## Analyses:
- Kimura, XRF by Jun Kimura, Shimane University, Japan; USGS, XRF in USGS Denver lab; Coombs, electron-microprobe analyses, at USGS Menlo Park, by Michelle Coombs
- Lipman and Calvert
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Appendix DR2

$^{40}\text{Ar}/^{39}\text{Ar}$ Analytical techniques

Analytical techniques for the submarine Hilo Ridge samples are nearly identical to those reported previously for underwater basalt from Kilauea (Calvert and Lanphere, 2006). Grey, crystalline portions of pillow interiors were separated for dating. Samples were crushed, ultrasonicated and sized to 250-350µm. Dense, clean groundmass was concentrated using a Frantz magnetic separator and careful handpicking under a binocular microscope. For irradiation, 180-190 mg groundmass sample were packaged in Cu foil and placed in a cylindrical quartz vial, together with fluence monitors of known age and K-glass and fluorite to measure interfering isotopes from K and Ca. The quartz vials were wrapped in 0.5 mm-thick Cd foil to shield samples from thermal neutrons during irradiation. The samples were irradiated for two hours in the central thimble of the U.S. Geological Survey TRIGA reactor in Denver, Colorado (Dalrymple et al., 1981). The reactor vessel was rotated continuously during irradiation to avoid lateral neutron flux gradients. Reactor constants determined for these irradiations were indistinguishable from recent irradiations, and a weighted mean of constants obtained over the past five years yields $^{40}\text{Ar}/^{39}\text{Ar}_k = 0.0004\pm0.004$, $^{39}\text{Ar}/^{37}\text{Ar}_c = 0.000406\pm0.000051$, and $^{36}\text{Ar}/^{37}\text{Ar}_c = 0.000281\pm0.000009$. TCR-2 sanidine from the Taylor Creek Rhyolite (Dalrymple and Duffield, 1988) was used as a fluence monitor with an age of 27.87 Ma. This monitor is a secondary standard calibrated against the primary intralaboratory standard, SB-3, that has an age of 162.9 ± 0.9 Ma (Lanphere and Dalrymple, 2000). Data in this study were recalculated to a Fish Canyon sanidine age of 28.02 using $R = 1.00655217\pm0.0005853$. Fluence monitors were analyzed using a continuous CO$_2$ laser system and mass spectrometer described by Dalrymple (1989). Argon was extracted from groundmass and mica separates using a Mo crucible in a custom resistance furnace modified from the design of Staudacher et al. (1978) attached to the above mass spectrometer. Heating temperatures were monitored with an optical fiber thermometer and controlled with an Accufiber Model 10 controller. Gas was purified continuously during extraction using two SAES ST-172 getters operated at 4A and 0A.

Mass spectrometer discrimination and system blanks are important factors in the precision and accuracy of $^{40}\text{Ar}/^{39}\text{Ar}$ age determinations of Pleistocene lavas because of low radiogenic yields. Discrimination is monitored by analyzing splits of atmospheric Ar from a reservoir attached to the extraction line and for these samples $D_{\text{lambda}} = 1.007504 \pm 0.000279$. All isotopic ratios are mass discrimination corrected using $^{40}\text{Ar}/^{36}\text{Ar} = 295.5$ (Steiger and Jager, 1977). A recent determination of atmospheric argon ($^{40}\text{Ar}/^{36}\text{Ar} = 298.56 \pm 0.31$; Lee et al., 2006) is more precise, but acceptance is controversial and that ratio has no impact on this study because normalizing to a different value does not change the age. Typical system blanks including mass spectrometer backgrounds were $1.5x10^{-18}$ mol of m/z 36, $9x10^{-17}$ mol of m/z 37, $3x10^{-18}$ mol of m/z 39 and $1.5x10^{-16}$ mol of m/z 40, where m/z is mass/charge ratio.

In the incremental-heating experiments, the extraction line is isolated from pumping systems and the sample is heated to a specified temperature for 10 minutes, cooled for 3-5 minutes, and transferred to an isolated mass spectrometer. The gas is exposed to getters during the entire extraction. Isotopic ratios are measured and corrected for instrumental blanks, mass discrimination and interfering isotopes generated in the
reactor. In these experiments we separated and loaded enough material to do 12-18 steps on each unknown in order to carefully characterize the argon release. The incremental heating data are plotted both as an age spectrum diagram and as an isotope correlation (isochron) diagram. For the age spectrum, apparent ages are calculated assuming that non-radiogenic Ar is atmospheric \(^{40}\text{Ar}/^{36}\text{Ar} = 295.5\) in composition and are plotted against the cumulative \(^{39}\text{Ar}\) released during the experiment. In cases with several contiguous steps yielding ages within analytical error, we calculate and report plateau ages by weighing individual ages by the inverse of their analytical error. Most groundmass age experiments do not yield identical ages across the entire spectrum due to minor alteration, recoil of \(^{39}\text{Ar}\) during irradiation or modest excess \(^{40}\text{Ar}\). Generally accepted criteria for a meaningful incremental heating age are: (1) well-defined plateau (horizontal age spectrum) for more than 50% of the \(^{39}\text{Ar}\) released; (2) well-defined isochron for the plateau gas fractions; (3) concordant plateau and isochron ages; and (4) \(^{40}\text{Ar}/^{36}\text{Ar}\) isochron intercept not significantly different from 295.5.

For isochron plots, data are not corrected using an atmospheric ratio. Isochron ages include plateau steps on well-behaved samples or a subset of data that yield a reasonable goodness of fit. We show normal isochron plots for these low-radiogenic rocks because the data are easier to visualize. Inverse isochron results are indistinguishable. Isochron ratios are particularly vulnerable to mobilization of argon isotopes during irradiation, particularly in fine-grained volcanic rocks. \(^{39}\text{Ar}\) produced from \(^{39}\text{K}\) in the reactor recoils ~0.1\(\mu\)m causing different degassing rates during analysis (Huneke and Smith, 1976). Recoil moves ratios along the x-axis of isotope correlation plots with low-T steps moved to lower values (loss of \(^{39}\text{Ar}\)) and high-T steps moved toward higher values (deeply implanted \(^{39}\text{Ar}\)). In this case, the isochron line becomes shallow, yielding a high \(^{40}\text{Ar}/^{36}\text{Ar}\) ratio and young isochron age. We interpret this as an irradiation artifact. The most reliable results generally include gas from the middle of the release spectrum with consistent K/Ca ratios and concordant isochron data with \(^{40}\text{Ar}/^{36}\text{Ar}\) intercepts within error of air. Ages and isotopic ratios reported below are 1\(\sigma\).

References


### Hilo Ridge \(^{40}\text{Ar}^{39}\text{Ar}\) tabulated data

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#### K215-6 Basalt

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**Notes:**
- \(^{40}\text{Ar}^{39}\text{Ar}\) is radiogenic argon, isotopes in volts (6.8e-14 moles/volt), corrected for blank, background, discrimination, and decay
- Calculated K2O = 0.29%wt., Calculated CaO = 7.6%wt., Calculated C = 0.3ppm
- Total Gas Age = 1465 ± 67 ka
- Weighted Mean Plateau Age = 1158.5 ± 33.4 ka (±1 sigma, including J), 68.2% 39Ar released
- Weighted Mean Plateau Age = 1158.5 ± 33.4 ka (A priori, without J)
- MSWD = 0.93 (Good fit, MSWD < 2.11)
- Steps 10 of 12 (550,650,700,750,800,850,900°C)
- Calculated K2O = 0.29%wt., Calculated CaO = 7.6%wt., Calculated C = 0.3ppm
- Total Gas Age = 1510 ± 208 ka
- Weighted Mean Plateau Age = 1138.5 ± 34.2 ka (±1 sigma, including J), 73.9% 39Ar released
- Weighted Mean Plateau Age = 1138.5 ± 34.2 ka (A priori, without J)
- MSWD = 0.96 (Good fit, MSWD < 2.29)
- Steps 8 of 12 (550,600,650,700,750,800,850,900°C)
- J = 0.000338231±0.00001687 (A Priori)
- J = 0.000338231±0.00001687 (A Priori)
- J = 0.000338231±0.00001687 (A Priori)
Total gas age = 1465 ± 67 ka

WMPA = 1158.5 ± 33.4 ka (68.2% 39Ar released; MSWD = 0.93)

Age = 925.6 ± 144.6 ka
MSWD = 1.29
40Ar/36Ar = 298.6 ± 4.3 (±2s)

Age = 1318 ± 549 ka
MSWD = 21.43
40Ar/36Ar = 294.9 ± 16.6 (±2s)
Total gas age = 1510 ± 208 ka

WMPA = 1138.5 ± 34.2 ka (73.9% 39Ar released; MSWD = 0.96)

Age = 1024.2 ± 285.1 ka
MSWD = 1.10
40Ar/36Ar = 297.2 ± 9.3 (±2σ)

Age = -812 ± 402 ka
MSWD = 9.04
40Ar/36Ar = 324.5 ± 12.4 (±2σ)