APPENDIX D—

Isotopic Age Methodology

Sample Procedure

Samples analyzed by the $^{40}Ar/^{39}Ar$ method at the Nevada Isotope Geochronology Laboratory at University of Nevada, Las Vegas were wrapped in Al foil and stacked in 6 mm inside diameter Pyrex tubes. Individual packets averaged 3 mm thick and neutron fluence monitors (FC-2, Fish Canyon Tuff sanidine) were placed every 5-10 mm along the tube. Synthetic K-glass and optical grade CaF_2 were included in the irradiation packages to monitor neutron induced argon interferences from K and Ca. Loaded tubes were packed in an Al container for irradiation. Samples irradiated at the Nuclear Science Center at Texas A&M University were in-core for 14 hours in the D3 position on the core edge (fuel rods on three sides, moderator on the fourth side) of the 1MW TRIGA type reactor. Irradiations are performed in a dry tube device, shielded against thermal neutrons by a 5 mm thick jacket of B_4C powder, which rotates about its axis at a rate of 0.7 revolutions per minute to mitigate horizontal flux gradients. Correction factors for interfering neutron reactions on K and Ca were determined by repeated analysis of K-glass and CaF_2 fragments. Measured $(^{40}Ar/^{39}Ar)_K$ values were 0.00 (± 0.0002). Ca correction factors were $(^{36}Ar/^{37}Ar)Ca = 2.90 (\pm 3.89) \times 10^{-4}$ and $(^{39}Ar/^{37}Ar)Ca = 6.66 (\pm 3.23) \times 10^{-4}$. J factors were determined by fusion of 4-5 individual crystals of neutron fluence monitors which gave reproducibility's of 0.17% to 0.38% at each standard position. Variation in neutron flux along the 100 mm length of the irradiation tubes was <4%. An error in J of 0.5% was used in age calculations. No significant neutron flux gradients were present within individual packets of crystals as indicated by the excellent reproducibility of the single crystal flux monitor fusions.

Irradiated crystals together with CaF_2 and K-glass fragments were placed in a Cu sample tray in an high vacuum extraction line and were fused using a 20 W CO_2 laser. Sample viewing during laser fusion was by a video camera system and positioning was via a motorized sample stage. Samples analyzed by the furnace step heating method utilized a double vacuum resistance furnace similar to the Staudacher and others (1978) design. Reactive gases were removed by a single MAP and two GP-50 SAES getters prior to being admitted to a MAP 215-50 mass spectrometer by expansion. The relative volumes of the extraction line and mass spectrometer allow 80% of the gas to be admitted to the mass spectrometer for laser fusion analyses and 76% for furnace heating analyses. Peak intensities were measured using a Balzers electron multiplier by peak hopping through 7 cycles; initial peak heights were determined by linear regression to the time of gas admission. Mass spectrometer discrimination and sensitivity was monitored by repeated analysis of atmospheric argon aliquots from an on-line pipette system. Measured $^{40}Ar/^{36}Ar$ ratios were 289.49 ± 0.18% during this work, thus a discrimination correction of 1.02078 (4 AMU) was applied to measured isotope ratios. The sensitivity of the mass spectrometer was ~6×10^{-17} mol mV^{-1} with the multiplier operated at a gain of 52 over the Faraday. Line blanks averaged 1.96 mV for mass 40 and 0.01 mV for mass 36 for laser fusion analyses and 11.53 mV for mass 40 and 0.04 mV for mass 36 for furnace heating analyses. Discrimination, sensitivity, and blanks were relatively constant

over the period of data collection. Computer automated operation of the sample stage, laser, extraction line and mass spectrometer as well as final data reduction and age calculations were done using LabSPEC software written by B. Idleman (Lehigh University). An age of 27.9 Ma (Steven and others, 1967; Cebula and others, 1986) was used for the Fish Canyon Tuff sanidine flux monitor in calculating ages for samples.

For $^{40}Ar/^{39}Ar$ analyses a plateau segment consists of 3 or more contiguous gas fractions having analytically indistinguishable ages (i.e. all plateau steps overlap in age at $\pm 2\sigma$ analytical error) and comprising a significant portion of the total gas released (typically >50%). Total gas (integrated) ages are calculated by weighting by the amount of ^{39}Ar released, whereas plateau ages are weighted by the inverse of the variance. For each sample inverse isochron diagrams are examined to check for the effects of excess argon. Reliable isochrons are based on the MSWD criteria of Wendt and Carl (1991) and, as for plateaus, must comprise contiguous steps and a significant fraction of the total gas released. All analytical data are reported at the confidence level of 1σ (standard deviation).