Data Repository Material for Chapter 8
Stratigraphy and age of the lower Horse Spring Formation in the Longwell Ridges area, southern Nevada: Implications for tectonic interpretations
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$^{40}Ar/^{39}Ar$ ages reported in this paper were generated at two different labs between 2004 and 2005. We provide the laboratory procedures from each lab below.

Lab Procedures for the Nevada Isotope Geochronology Laboratory
By Terry Spell

Samples analyzed by the $^{40}Ar/^{39}Ar$ method at the University of Nevada, Las Vegas were wrapped in Al foil and stacked in sealed 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 were irradiated at McMaster Nuclear Reactor at McMaster University, Ontario, Canada. The samples were in-core for 7 hours in the 5C position where they are surrounded by fuel rods on all four sides. 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 0.00732 (± 43.45%). Ca correction factors were $(^{36}Ar/^{37}Ar)Ca = 2.87(\pm 4.21\%) \times 10^{-4}$ and $(^{39}Ar/^{37}Ar)Ca = 7.08 (\pm 0.76\%) \times 10^{-4}$. J factors were determined by fusion of 3–5 individual crystals of neutron fluence monitors which gave reproducibility's of 0.09% to 0.2% at each standard position. Variation in neutron flux along the 100 mm length of the irradiation tubes was <4%. The error in age calculations includes J error.

Irradiated crystals together with CaF_2 and K-glass fragments were placed in a Cu sample tray in a 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 et al. (1978) design. Reactive gases were removed by three 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 288.53 ± 0.64% during this work, thus a discrimination correction of 1.02421 (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 2.0 mV for mass 40 and 0.01 mV for mass 36 for laser fusion analyses and 17.99mV for mass 40 and 0.06 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 et al., 1967; Cebula et al., 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).

Cebula, G.T., Kunk, M.J., Mehnert, H.H., Naeser, C.W., Obradovich, J.D., and Sutter, J.F., 1986, The Fish Canyon Tuff, a potential standard for the ^{40}Ar-^{39}Ar and fission-track dating methods (abstract), *Terra Cognita (6th Int. Conf. on Geochronology, Cosmochronology and Isotope Geology), 6*, 139.

Staudacher, T.H., Jessberger, E.K., Dorflinger, D., and Kiko, J., 1978, A refined ultrahigh-vacuum furnace for rare gas analysis, *J. Phys. E: Sci. Instrum.*, *11*, 781–784.

Steven, T.A., Mehnert, H.H., and Obradovich, J.D., 1967, Age of volcanic activity in the San Juan Mountains, Colorado, *U.S. Geol. Surv. Prof. Pap., 575-D*, 47–55.

Wendt, I., and Carl, C., 1991, The statistical distribution of the mean squared weighted deviation, *Chemical Geology*, v. 86, p. 275–285.

NOTE: Check your samples data sheets for the discrimination values used for each sample. Values used during your sample analyses are as follows:

$^{40}Ar/^{36}Ar$	4 AMU discrimination
288.53 ± 0.64%	1.02421
286.00 ± 0.59%	1.03325

Lab Procedures for the New Mexico Geochronological Research Laboratory (NMGRL)

By William McIntosh

Sample preparation and irradiation details

Mineral separates are obtained in various fashions depending upon the mineral of interest, rock type and grain size. In almost all cases the sample is crushed in a jaw crusher and ground in a disc grinder and then sized. The size fraction used generally corresponds to the largest size possible, which will permit obtaining a pure mineral

separate. Following sizing, the sample is washed and dried. For plutonic and metamorphic rocks and lavas, crystals are separated using standard heavy liquid, Franz magnetic and hand-picking techniques. For volcanic sanidine and plagioclase, the sized sample is reacted with 15% HF acid to remove glass and/or matrix and then thoroughly washed prior to heavy liquid and magnetic separation. For groundmass concentrates, rock fragments are selected which do not contain any visible phenocrysts. The NMGRL uses either the Ford reactor at the University of Michigan or the Nuclear Science Center reactor at Texas A&M University. At the Ford reactor, the L67 position is used (unless otherwise noted) and the D-3 position is always used at the Texas A&M reactor. All of the Michigan irradiations are carried out underwater without any shielding for thermal neutrons, whereas the Texas irradiations are in a dry location which is shielded with B and Cd. Depending upon the reactor used, the mineral separates are loaded into either holes drilled into Al discs or into 6 mm I.D. quartz tubes. Various Al discs are used. For Michigan, either six hole or twelve hole, 1 cm diameter discs are used and all holes are of equal size. Samples are placed in the 0, 120 and 240° locations and standards in the 60, 180 and 300° locations for the six hole disc. For the twelve hole disc, samples are located at 30, 60, 120, 150, 210, 240, 300, and 330° and standards at 0, 90, 180 and 270 degrees. If samples are loaded into the quartz tubes, they are wrapped in Cu foil with standards interleaved at ~0.5 cm intervals. For Texas, 2.4 cm diameter discs contain either sixteen or six sample holes with smaller holes used to hold the standards. For the six hole disc, sample locations are 30, 90, 150, 210, 270 and 330° and standards are at 0, 60, 120, 180, 240 and 300°. Samples are located at 18, 36, 54, 72, 108, 126, 144, 162, 198, 216, 234, 252, 288, 306, 324, 342 degrees and standards at 0, 90, 180 and 270 degrees in the sixteen hole disc. Following sample loading into the discs, the discs are stacked, screwed together and sealed in vacuo in either quartz (Michigan) or Pyrex (Texas) tubes.

Extraction Line and Mass Spectrometer details

The NMGRL argon extraction line has both a double vacuum Mo resistance furnace and a CO_2 laser to heat samples. The Mo furnace crucible is heated with a W heating element and the temperature is monitored with a W-Re thermocouple placed in a hole drilled into the bottom of the crucible. A one inch long Mo liner is placed in the bottom of the crucible to collect the melted samples. The furnace temperature is calibrated by either/or melting Cu foil or with an additional thermocouple inserted in the top of the furnace down to the liner. The CO_2 laser is a Synrad 10W laser equipped with a He-Ne pointing laser. The laser chamber is constructed from a 3 3/8″ stainless steel conflat and the window material is ZnS. The extraction line is a two-stage design. The first stage is equipped with a SAES GP-50 getter, whereas the second stage houses two SAES GP-50 getters and a tungsten filament. The first stage getter is operated at 450°C as is one of the second stage getters. The other second stage getter is operated at room temperature and the tungsten filament is operated at ~2000°C. Gases evolved from samples heated in the furnace are reacted with the first stage getter during heating. Following heating, the gas is expanded into the second stage for two minutes and then isolated from the first stage. During second stage cleaning, the first stage and furnace are pumped out. After gettering in the second stage, the gas is expanded into the mass spectrometer. Gases evolved from samples heated in the laser are expanded through a cold finger operated at -140°C and directly into the second stage. Following cleanup, the

gas in the second stage and laser chamber is expanded into the mass spectrometer for analysis. The NMGRL employs a MAP-215-50 mass spectrometer, which is operated in static mode. The mass spectrometer is operated with a resolution ranging between 450 to 600 at mass 40 and isotopes are detected on a Johnston electron multiplier operated at ~2.1 kV with an overall gain of about 10,000 over the Faraday collector. Final isotopic intensities are determined by linear regression to time zero of the peak height versus time following gas introduction for each mass. Each mass intensity is corrected for mass spectrometer baseline and background and the extraction system blank. Blanks for the furnace are generally determined at the beginning of a run while the furnace is cold and then between heating steps while the furnace is cooling. Typically, a blank is run every three to six heating steps. Periodic furnace hot blank analysis reveals that the cold blank is equivalent to the hot blank for temperatures less than about 1300°C. Laser system blanks are generally determined between every four analyses. Mass discrimination is measured using atmospheric argon, which has been dried using a Ti-sublimation pump. Typically, 10 to 15 replicate air analyses are measured to determine a mean mass discrimination value. Air pipette analyses are generally conducted 2–3 times per month, but more often when samples sensitive to the mass discrimination value are analyzed. Correction factors for interfering nuclear reactions on K and Ca are determined using K-glass and CaF_2, respectively. Typically, 3–5 individual pieces of the salt or glass are fused with the CO_2 laser and the correction factors are calculated from the weighted mean of the individual determinations.