



Characterization and calibration of $^{40}\text{Ar}/^{39}\text{Ar}$ dating standards

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Abstract

Because $^{40}\text{Ar}/^{39}\text{Ar}$ ages are based on independent age determinations for standard minerals, it is critical that these standards be well characterized. This includes constraints both on the age of the standards themselves and the ages of the standards relative to one another. In an effort to better characterize standards in common use, we have intercalibrated five of these: the interlaboratory standards MMhb-1 hornblende, 92-176 Fish Canyon Tuff sanidine, and LP-6 biotite, along with two Australian National University intralaboratory standards, 77-600 hornblende and GA-1550 biotite. The results indicate that all standards studied are homogeneous using subsamples of tens of milligrams, indicating that total fusion analyses of such large aliquots will yield reproducible results. However, only 92-176 sanidine and GA-1550 biotite meet the criteria for acceptable standards at the single crystal sampling level, whereas MMhb-1 hornblende, 77-600 hornblende, and LP-6 biotite exhibit inhomogeneity, which compromises their usefulness. Intercalibration R values, which allow a direct comparison of standards, have been derived as follows: $R_{92-176}^{\text{GA-1550}} = 3.575 \pm 0.005$, $R_{\text{GA-1550}}^{\text{MMhb-1}} = 5.996 \pm 0.010$, $R_{\text{GA-1550}}^{77-600} = 4.650 \pm 0.014$, $R_{\text{GA-1550}}^{\text{LP-6}} = 1.305 \pm 0.003$, $R_{\text{LP-6}}^{\text{MMhb-1}} = 4.550 \pm 0.019$, $R_{92-176}^{\text{MMhb-1}} = 21.53 \pm 0.09$, and $R_{77-600}^{\text{MMhb-1}} = 1.289 \pm 0.002$. These values generally agree with those derived in previous studies, although variability between studies significantly exceeds the analytical precision of the $^{40}\text{Ar}/^{39}\text{Ar}$ method, emphasizing the need for a widely accepted set of primary standards of well-established age. Based on a compilation of potassium measurements by flame photometry from 1968 to 2001, an updated K/Ar age of 98.5 ± 0.8 Ma is calculated for GA-1550 biotite, which is used as a primary standard for the intercalibration. Relative to GA-1550, the ages for other standards are derived via the intercalibration R values as follows: 418.3 ± 1.3 Ma for 77-600 hornblende, 523.2 ± 0.9 Ma for MMhb-1 hornblende, 127.5 ± 0.3 Ma for LP-6 biotite, and 28.10 ± 0.04 Ma for 92-176 sanidine.

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1. Introduction

The determination of the age of a sample by the $^{40}\text{Ar}/^{39}\text{Ar}$ method relies upon an independent knowl-

edge of the age of a standard mineral, commonly referred to as the fluence monitor, which is utilized to measure the fast neutron dose received by the sample during irradiation. A fairly large number of such standards are in use by $^{40}\text{Ar}/^{39}\text{Ar}$ dating laboratories worldwide, including interlaboratory standards that have been characterized and distributed, as well as intralaboratory standards that are used dominantly at individual facilities. $^{40}\text{Ar}/^{39}\text{Ar}$ ages are inherently very precise as they are based on isotope ratio

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measurements of a single element; however, the accuracy of the technique is directly tied to the accuracy with which the standard's age is determined. A quantitative comparison of $^{40}\text{Ar}/^{39}\text{Ar}$ ages reported from different laboratories requires that the comparative ages of these standard minerals be well known. Since the work of [Roddick \(1983\)](#), rather few studies specifically aimed at such intercalibrations have been published until relatively recently; these include those by [Baksi et al. \(1996\)](#), [Renne et al. \(1998\)](#), and [Lanphere and Dalrymple \(2000\)](#). More fundamental issues, such as refinement of decay constants ([Min et al., 2000](#); [Villeneuve et al., 2000](#); [Begemann et al., 2001](#); [Kwon et al., 2002](#)), have also been a recent concern.

The characterization and the intercalibration of standards have become more important with the advent of high-sensitivity, low-background, noble gas mass spectrometers and laser fusion systems, which allow rapid analysis of much smaller samples and an increased precision of calculated ages. Quoted analytical precisions for the $^{40}\text{Ar}/^{39}\text{Ar}$ method are now commonly ± 0.1 – 0.2% or better, and in many laboratories, a number of individual single crystals are analyzed to obtain the relevant J factor from a fluence monitor. Despite the high levels of precision being attained, the ages of primary fluence monitors and intercalibrations between fluence monitors are commonly not known to better than ± 1 – 2% . Thus, the accuracy of the ages of standards, tight constraints on their relative ages, and homogeneity at the single crystal level are increasingly important and comprise a major limiting factor on the accuracy and intercomparison of $^{40}\text{Ar}/^{39}\text{Ar}$ ages.

For these reasons, we have conducted analyses on five standard minerals used in $^{40}\text{Ar}/^{39}\text{Ar}$ dating. Several methods have been used to characterize these mineral standards; step-heating $^{40}\text{Ar}/^{39}\text{Ar}$ experiments have been performed on Fish Canyon Tuff sanidine and LP-6 biotite, total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ measurements have been undertaken on all standards studied, and laser fusion $^{40}\text{Ar}/^{39}\text{Ar}$ analyses have been performed on single crystal and multicrystal splits of all standards.

2. Standards investigated

Detailed information on many of the samples studied here has been previously discussed (e.g.,

[Baksi et al., 1996](#); [Renne et al., 1998](#); [Lanphere and Dalrymple, 2000](#)) and is only summarized here. Of the five standards investigated, three are interlaboratory standards in use worldwide (MMhb-1 hornblende, Fish Canyon Tuff sanidine, and LP-6 biotite), whereas two are intralaboratory standards prepared in and used largely at the Australian National University (ANU; 77-600 hornblende and GA-1550 biotite), although GA-1550 has become increasingly utilized as an interlaboratory standard. $^{40}\text{Ar}/^{39}\text{Ar}$ age spectra are well documented for MMhb-1 hornblende ([Harrison, 1981](#); [Lee, 1993](#); [Lee et al., 1991](#); [Rex et al., 1993](#)), 77-600 hornblende ([Tetley, 1978](#); [Harrison, 1981](#)), and GA-1550 biotite ([Tetley, 1978](#); [Lanphere and Dalrymple, 2000](#)) and, with few exceptions, are ideally flat (see below). In contrast, age spectra for LP-6 biotite have proven to be discordant and dissimilar between studies ([Tetley, 1978](#); [Ozima et al., 1979](#); [Baksi et al., 1996](#)), raising concerns as to sample homogeneity. For the Fish Canyon Tuff, there are several published age spectra on biotite ([Hurford and Hammerschmidt, 1985](#); [Baksi et al., 1996](#); [Lanphere et al., 1990](#)) and one for sanidine, the most commonly used phase ([Lanphere and Baadsgaard, 2001](#)).

MMhb-1 hornblende was initially prepared and distributed for use as an $^{40}\text{Ar}/^{39}\text{Ar}$ dating standard by [Alexander et al. \(1978\)](#). Approximately 1.3 kg of this mineral separate was obtained from a syenite in the McClure Mountain Complex, Colorado (see [Olsen et al., 1977](#)), and from its simple geological history, it is expected to contain a homogeneous intracrystalline radiogenic argon ($^{40}\text{Ar}^*$) distribution, one of the requirements for a suitable standard ([Alexander and Davis, 1974](#); [Dalrymple et al., 1981](#)). Supplies of MMhb-1 are becoming limited and the collection and characterization of a new sample from the same location (to be called MMhb-2) are currently underway ([Kunk and Miller, 2002](#); [Kunk et al., 1994](#)). The sample of MMhb-1 used in this study was split 6–16.

The Fish Canyon Tuff is a large-volume ($\sim 5000 \text{ km}^3$) quartz latite ignimbrite erupted in the San Juan Volcanic Field of southern Colorado, USA. Zircon and apatite mineral separates from the Fish Canyon Tuff were initially distributed by the U.S. Geological Survey (USGS) for use as interlaboratory standards in fission-track dating ([Naeser and Cebula, 1978](#)). [McDowell \(1984\)](#) suggested that sanidine from this

unit would be a suitable $^{40}\text{Ar}/^{39}\text{Ar}$ standard and, subsequently, Cebula et al. (1986) proposed the use of hornblende, biotite, and sanidine as $^{40}\text{Ar}/^{39}\text{Ar}$ standards. Sanidine and, to a lesser extent, biotite have now been in use for several years in a large number of laboratories. Because of the simple geological history of the Fish Canyon Tuff, it might be anticipated that there would be concordance of measured K/Ar and Rb/Sr ages on hornblende, plagioclase, biotite, and sanidine, as well as concordance with U/Pb ages on zircon and fission-track ages determined on zircon and apatite. The published results (see below), in fact, are rather more scattered than expected for various reasons. It is worth noting that mineral separates from the Fish Canyon Tuff have not all been obtained from a single sampling and some differences in the preparation of the separates may be present in terms of, for example, purity. The sample used in this study was obtained at the “USGS location,” from outcrops on the north side of Colorado Highway 160 at 9 km southwest of the town of South Fork (sample coordinates $37^{\circ}36.1' \text{ S}$, $106^{\circ}42.0' \text{ W}$), and was processed at the Australian National University (ANU) as sample 92-176. The size fraction utilized for the single crystal measurements was 0.35–0.60 mm.

The preparation and characterization of LP-6 biotite have been described by Ingamells and Engels (1976). The biotite separate was taken from a biotite pyroxenite from the Similkameen composite pluton in Washington State, USA. The separate is reported to be 99.9% pure (Odin et al., 1982). However, Ingamells and Engels (1976) indicated that LP-6 appears to be a mixture of two biotites with ages of ~ 121 and 138 Ma . Because of this inhomogeneity, they have recommended samples of 5 and 50 mg for potassium and argon measurements, respectively, in order to achieve a sampling uncertainty of less than 1%. The sample used here was 6-1-A-3, split 6.

The Australian National University 77-600 hornblende was separated from an amphibolite adjacent to the Cooma Granodiorite in New South Wales, Australia, and was described by Tetley (1978). It was collected in the town of Cooma on Soho Street, at $36^{\circ}14.6' \text{ S}$, $149^{\circ}07.5' \text{ E}$. The hornblende has been prepared at a grain size of 0.13–0.18 mm and is $>99\%$ pure and chemically homogeneous. The sample used in this study was split 14–4.

The Australian National University GA-1550 biotite was separated from the Mt. Dromedary monzonite exposed in a small quarry near Central Tilba, New South Wales, Australia. The coordinates of the locality are $36^{\circ}18.5' \text{ S}$, $150^{\circ}04.4' \text{ E}$. K/Ar age data for this biotite separate were initially reported by McDougall and Roksandic (1974). The sample used in this study was from jar 4, split 5; the size fraction was 0.35–0.85 mm.

3. Analytical methods

Standards analysed by the $^{40}\text{Ar}/^{39}\text{Ar}$ method were irradiated for 192 h in the X33 or X34 position of the HIFAR reactor. Cadmium shielding, 0.2 mm thick, was used to reduce the thermal neutron fluence received by the samples (see Tetley et al., 1980). Corrections for interfering nuclear reactions on potassium and calcium were monitored by including degassed K_2SO_4 and CaF_2 in the irradiation packages. Correction factors thus determined were $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 2.64 (\pm 0.23) \times 10^{-2}$, $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.50 (\pm 0.4) \times 10^{-4}$, and $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 7.86 (\pm 0.01) \times 10^{-4}$. The results are not sensitive to a modest variability of the calcium correction factors (up to 5%), owing to the low calcium content of many of the minerals analyzed. However, owing to the relatively high calcium content in 77-600 hornblende (12.6 wt.% CaO), the data from this mineral are noticeably dependent upon the calcium correction factors being reliable, because up to 30% of the ^{36}Ar is calcium-derived. Although similar proportions of ^{36}Ar are calcium-derived in the case of MMhb-1 hornblende, the very high proportion of radiogenic argon (generally $>99\%$) in the runs means that any modest variability of the calcium correction factors has a negligible effect on the calculated $^{40}\text{Ar}^*/^{39}\text{Ar}_{\text{K}}$ ratios.

Irradiations were performed in two different containers depending on the experiment. The first irradiation geometry was utilized for larger samples to be analyzed by either step-heating or total fusion experiments. This arrangement consists of a set of eight Al cans, which are contained within an Al irradiation vessel with internal dimensions of 34 mm length and 11 mm diameter. Each Al can consists of both an inner and an outer can, with the inner can being

located coaxial within the outer can (see McDougall and Harrison, 1999). The vessel was inverted end-for-end halfway through the irradiation, which reduces lengthwise gradients in the fast neutron flux received to <3%. Note that an advantage of this geometry is that no interpolation is required between positions in order to compare results from different monitors (i.e., two standards being compared at a particular level are expected to receive the same fast neutron fluence). A second arrangement, for single crystal laser fusion experiments, utilized high-purity silica glass tubes with dimensions of 3 mm ID, 5 mm OD, and 28 mm length to contain the samples. Three tubes were contained within the Al irradiation vessel in a triangular fashion and samples, wrapped in Al or Sn foil, were packed in the tubes. Crystals within individual sample packets were restricted spatially to <3 × 3 mm to reduce the possibility of neutron fluence variations within a packet. During irradiation, the vessel was inverted three times in an attempt to reduce both lengthwise and possible radial flux gradients within the vessel, the assumption being that the vessel would be in a random radial orientation each of the four times it was in the reactor. This arrangement was thus designed to ensure that no significant fluence gradients existed within an individual sample packet—a critical concern for single crystal analyses.

Isotopic analyses of aggregate mineral separates and step-heating experiments were performed using a VG MM1200 mass spectrometer operated statically with the ion beam collected on a Faraday cup. Sensitivity and mass discrimination were monitored at weekly intervals by use of atmospheric argon from an on-line gas pipette. During the course of the analyses reported here, sensitivity was $3.2\text{--}3.5 \times 10^{-15}$ mol mV⁻¹ and 1 amu of mass discrimination varied by <0.2%, yielding a correction of 0.99204–0.99376 in favor of heavier masses (i.e., measured ⁴⁰Ar/³⁶Ar ratios of atmospheric argon ranged from 304.9 to 302.9).

Single and multiple crystal ⁴⁰Ar/³⁹Ar analyses utilized a VG3600 mass spectrometer equipped with a Daly photomultiplier detector, with a sensitivity of about 3×10^{-17} mol mV⁻¹. Mass discrimination was monitored at weekly intervals by analyses of atmospheric argon from an online pipette system and averaged 1.00591 ± 0.00161 ($\pm 0.16\%$) per atomic

mass unit. Fusions were accomplished by heating the sample with a 10-W Ar ion laser beam. For dark minerals such as biotite and hornblende, which absorb the Ar ion laser wavelength well, the beam was initially defocused and power ramped up gradually until melting occurred (typically ~ 1–2 W), whereupon the beam was focused and power increased to 2–4 W and held for 2–3 min to ensure complete degassing. For sanidine from the Fish Canyon Tuff, fusions were accomplished by ramping the focused laser to full power within a few seconds.

The ⁴⁰Ar/³⁹Ar ages reported here are based primarily on a reference age of 98.5 ± 0.8 Ma for GA-1550 biotite (see below). All data reported or discussed here are quoted at $\pm 1\sigma$ uncertainty and utilize the decay constants and isotopic compositions recommended by Steiger and Jäger (1977). Uncertainty in decay constants is not included (cf. Renne et al., 1998). Inverse isochrons use the York (1969) regression, and age spectrum plateaus are defined as three or more steps comprising >50% of the total ³⁹Ar released, with ages that are indistinguishable at the 2 σ level. Total gas (integrated) ages are calculated using weighting by the amount of ³⁹Ar released, whereas plateau ages are weighted by the inverse of the variance. Statistics used throughout are arithmetic means (\bar{X}) and sample standard deviations (σ) from which coefficient of variation (CV), a measure of reproducibility in percent, is calculated ($\sigma/\bar{X} \times 100 = \text{CV}\%$).

4. Published ages of the standards

This section discusses primary standard ages (e.g., directly determined by K/Ar or U/Pb dating), which have been reported for the mineral standards studied here. Ages derived from intercalibration with another fluence monitor are treated later. For all the standards utilized here, K/Ar ages are available. The Fish Canyon Tuff has also been dated by the fission-track, U/Pb, Rb/Sr, and U/Th–He methods. These data are summarized in Table 1.

An age of 97.9 ± 0.9 Ma for GA-1550 biotite was reported by McDougall and Roksandic (1974) and has been used at the ANU since that time (see McDougall and Harrison, 1999) (Table 1). A compilation of potassium measurements by flame photometry at

Table 1
Published isotopic age data on $^{40}\text{Ar}/^{39}\text{Ar}$ standards used in this study

Standard	Method	Age (Ma $\pm 1\sigma$)	Reference
GA-1550 biotite	K/Ar	97.9 \pm 0.9	McDougall and Roksandic (1974)
GA-1550 biotite	K/Ar	98.8 \pm 0.5	Renne et al. (1998)
GA-1550 biotite	K/Ar	98.5 \pm 0.8	This study
77-600 Hornblende	K/Ar	414.2 \pm 3.7	Harrison (1980, 1981)
77-600 Hornblende	K/Ar	414.1 \pm 3.9	McDougall and Harrison (1999)
MMhb-1 hornblende	K/Ar	519.4 \pm 3.2	Alexander et al. (1978)
MMhb-1 hornblende	K/Ar	520.4 \pm 1.7	Sampson and Alexander (1987)
MMhb-1 hornblende	K/Ar	513.9 \pm 2.3	Lanphere et al. (1990)
MMhb-1 hornblende	K/Ar	514.5 \pm 3.3	Baksi et al. (1996)
LP-6 biotite	K/Ar	127.8 \pm 0.7	Baksi (1973)
LP-6 biotite	K/Ar	128.9 \pm 1.4	Ingamells and Engels (1976)
LP-6 biotite	K/Ar	127.7 \pm 1.4	Odin et al. (1982)
Fish Canyon Tuff ^a	K/Ar	27.9 \pm 0.4	Steven et al. (1967)
Fish Canyon Tuff ^a	K/Ar	27.4 \pm 0.2	Hurford and Hammerschmidt (1985)
Fish Canyon Tuff ^a	K/Ar	27.9 \pm 0.6	Cebula et al. (1986)
Fish Canyon Tuff ^b	FT	27.8 \pm 1.0	Cebula et al. (1986)
Fish Canyon Tuff ^c	FT	28.0 \pm 1.5	Cebula et al. (1986)
Fish Canyon Tuff ^c	FT	26.8 \pm 2.1	Carpina and Mailhe (1987)
Fish Canyon Tuff ^b	FT	27.9 \pm 1.1	Carpina and Mailhe (1987)
Fish Canyon Tuff ^d	U/Pb	28.4 \pm 0.1	Oberli et al. (1990)
Fish Canyon Tuff ^e	U/Th–He	30.1 \pm 0.5	Reiners and Farley (1999)
Fish Canyon Tuff ^d	U/Pb	28.48 \pm 0.03	Schmitz and Bowring (2001)
Fish Canyon Tuff ^f	K/Ar	27.54 \pm 0.29	Lanphere and Baadsgaard (2001)
Fish Canyon Tuff ^g	Rb/Sr	27.44 \pm 0.08	Lanphere and Baadsgaard (2001)
Fish Canyon Tuff ^h	U/Pb	27.52 \pm 0.05	Lanphere and Baadsgaard (2001)
Fish Canyon Tuff ⁱ	U/Th–He	27.3 \pm 1.1	Reiners et al. (2002)
Fish Canyon Tuff ^d	U/Pb	28.36 \pm 0.03	Oberli et al. (2002)

All ages are calculated using isotopic abundances and decay constants from Steiger and Jäger (1977).

^a Mean of concordant K/Ar ages on sanidine, biotite, plagioclase, and hornblende.

^b Zircon fission-track age.

^c Apatite fission-track age.

^d Zircon $^{206}\text{Pb}/^{238}\text{U}$ age.

^e Titanite U/Th–He age.

^f Mean of concordant K/Ar ages on biotite and hornblende.

^g Feldspar–biotite Rb/Sr isochron age.

^h Zircon U/Pb discordia intercept age.

ⁱ Zircon U/Th–He age.

the ANU between 1968 and 2001 (Table 2) yields a slightly lower mean potassium value of 7.645 (± 0.050) wt.% compared to the original value of 7.70 wt.%. This new average potassium value is derived from multiple analyses ($n=37$) of ~ 15 -mg aliquots over a substantial period of time, and thus should be representative of the biotite separate as well as effectively averaging instrumental operating and calibration variations. Using these more recent data, a K/Ar age of 98.5 ± 0.8 Ma is calculated for GA-1550, based on a radiogenic argon concentration of $1.343 (\pm 0.007) \times 10^{-9}$ mol g^{-1} (McDougall and Roksandic, 1974).

In this study, we utilize the age of 98.5 ± 0.8 Ma based on K/Ar determinations at the ANU, as discussed above. Renne et al. (1998) reported isotope dilution potassium analyses that yield an average of 7.626 (± 0.045) wt.%, which, combined with previous $^{40}\text{Ar}^*$ concentrations, yields a K/Ar age of 98.8 ± 0.5 Ma. We note that these K/Ar ages are statistically indistinguishable.

The age of MMhb-1 has been determined in a number of studies (Table 1). The initial K/Ar age determination of 519.5 ± 2.5 Ma was reported by Alexander et al. (1978) from measurements made at

Table 2
Summary of potassium measurements by flame photometry on GA-1550 biotite from 1968 to 2001 at the ANU

Date	Number of dissolutions	K (wt.%) \pm S.D. (\pm CV)
9/68	2	7.782 \pm 0.042 (\pm 0.54%)
10/68	2	7.639 \pm 0.025 (\pm 0.32%)
10/68	2	7.674 \pm 0.030 (\pm 0.39%)
6/71	2	7.732 \pm 0.036 (\pm 0.46%)
5/72	2	7.745 \pm 0.035 (\pm 0.46%)
10/72	2	7.705 \pm 0.040 (\pm 0.52%)
1/75	6	7.702 \pm 0.051 (\pm 0.66%)
8/77	2	7.673 \pm 0.009 (\pm 0.11%)
11/77	4	7.623 \pm 0.055 (\pm 0.73%)
12/77	6	7.575 \pm 0.045 (\pm 0.60%)
8/78	4	7.639 \pm 0.056 (\pm 0.73%)
9/78	7	7.621 \pm 0.022 (\pm 0.29%)
9/78	7	7.682 \pm 0.098 (\pm 1.27%)
1/79	2	7.597 \pm 0.020 (\pm 0.26%)
5/79	14	7.619 \pm 0.050 (\pm 0.65%)
8/79	4	7.591 \pm 0.019 (\pm 0.24%)
9/79	2	7.669 \pm 0.030 (\pm 0.40%)
9/82	2	7.710 \pm 0.028 (\pm 0.36%)
6/83	2	7.644 \pm 0.023 (\pm 0.30%)
10/84	2	7.597 \pm 0.051 (\pm 0.67%)
2/85	2	7.634 \pm 0.019 (\pm 0.25%)
10/85	2	7.638 \pm 0.045 (\pm 0.58%)
10/90	2	7.679 \pm 0.047 (\pm 0.61%)
10/91	2	7.634 \pm 0.010 (\pm 0.13%)
3/92	2	7.628 \pm 0.046 (\pm 0.60%)
1/95	14	7.665 \pm 0.049 (\pm 0.64%)
2/97	6	7.606 \pm 0.076 (\pm 0.99%)
3/98	2	7.579 \pm 0.063 (\pm 0.83%)
4/98	5	7.638 \pm 0.016 (\pm 0.21%)
5/98	2	7.617 \pm 0.011 (\pm 0.14%)
5/98	2	7.582 \pm 0.054 (\pm 0.71%)
7/99	5	7.572 \pm 0.031 (\pm 0.41%)
9/99	1	7.625
1/00	2	7.654 \pm 0.014 (\pm 0.02%)
7/00	2	7.672 \pm 0.028 (\pm 0.37%)
4/01	2	7.570 \pm 0.090 (\pm 1.19%)
5/01	2	7.661 \pm 0.012 (\pm 0.16%)

Giving each set of readings equal weight, the arithmetic mean ($n=37$) is 7.645% (\pm 0.050), whereas the weighted mean ($n=36$) is indistinguishable at 7.646% (\pm 0.006). Combined with radiogenic argon ($^{40}\text{Ar}^*$) = $134.3 (\pm 0.7) \times 10^{-11} \text{ mol g}^{-1}$, the calculated age is $98.5 \pm 0.8 \text{ Ma}$ (see text). $\lambda^{40}\text{K} = 5.543 \times 10^{-10} \text{ a}^{-1}$.

the U.S. Geological Survey, Menlo Park, CA. Subsequently, Sampson and Alexander (1987) compiled K/Ar data from a number of laboratories worldwide. Weighted means of the reported potassium and radiogenic argon abundances were 1.555 (\pm 0.002) wt.% and $16.26 (\pm 0.05) \times 10^{-10} \text{ mol g}^{-1}$, respectively.

An age of $520.4 \pm 1.7 \text{ Ma}$ was calculated from these data. This age is most commonly used in $^{40}\text{Ar}/^{39}\text{Ar}$ studies that involve MMhb-1. K/Ar ages (which vary widely) from individual laboratories were not presented in Sampson and Alexander (1987); the implications of this are discussed below. It should also be noted that the age obtained for MMhb-1 at the ANU, as reported in Sampson and Alexander (1987) but not attributed to the ANU, was $524.2 \pm 4.0 \text{ Ma}$. Most recently, Lanphere and Dalrymple (2000) reported a K/Ar age of $513.9 \pm 2.3 \text{ Ma}$ for MMhb-1. This age has also been used in some $^{40}\text{Ar}/^{39}\text{Ar}$ dating studies.

A K/Ar age of $414.2 \pm 3.7 \text{ Ma}$ was reported for the ANU hornblende standard 77-600 by Harrison (1980, 1981) (Table 1). This was later adjusted to $414.1 \pm 3.9 \text{ Ma}$ by McDougall and Harrison (1999). This later K/Ar age is based on a potassium content of 0.336 (\pm 0.003) wt.% and a radiogenic argon concentration of $2.712 (\pm 0.008) \times 10^{-10} \text{ mol g}^{-1}$. There is presently some concern regarding the 414.1 Ma age for 77-600 hornblende as there are possible problems with the quantitative extraction of $^{40}\text{Ar}^*$ from this Mg-rich amphibole.

K/Ar ages for LP-6 biotite have been reported in several studies (Table 1). Baksi (1973) gave a K/Ar age of $127.8 \pm 0.7 \text{ Ma}$. This standard was thoroughly characterized by Ingamells and Engels (1976). They provided an age of $128.9 \pm 1.4 \text{ Ma}$. The most recent age reported for LP-6 biotite, and that used in this study, is $127.7 \pm 1.4 \text{ Ma}$ (Odin et al., 1982). This age is based on the potassium content of 8.33 (\pm 0.03) wt.%, as recommended by Ingamells and Engels (1976), and a radiogenic argon concentration of $19.24 (\pm 0.02) \times 10^{-10} \text{ mol g}^{-1}$. The latter is the average of values reported from 12 laboratories, after eliminating data from four laboratories that gave results that were more than 2% from the mean.

Determining the age of the Fish Canyon Tuff has been the focus of numerous studies utilizing a variety of techniques. Despite the extensive data set (Table 1), there is no consensus as to the numerical age of this commonly used standard. Reported ages range from 26.8 ± 2.1 to $30.1 \pm 0.5 \text{ Ma}$ (Table 1), a spread of over 10%, and well beyond the analytical accuracy and precision for any of the techniques used. Much of the apparent scatter in reported ages comes from fission-track and U/Th–He measurements, both of which have relatively low precision compared with

K/Ar or U/Pb ages, which fall within a smaller range of 27.4 ± 0.2 to 28.48 ± 0.03 Ma. However, there remains a significant scatter on the order of 4% among these higher-precision data. We note that some reported U/Pb zircon ages of 28.4–28.5 Ma (Oberli et al., 1990; Schmitz and Bowring, 2001) may be an artifact of pre-eruptive residence time (Oberli et al., 2002) and thus overestimate the eruptive age of the Fish Canyon Tuff. For these reasons, along with concerns about quantitative extraction of $^{40}\text{Ar}^*$ during K/Ar analyses, many laboratories have resorted to using an age for the Fish Canyon Tuff derived via intercalibration with another standard mineral, most commonly MMhb-1 hornblende. These issues are discussed further below.

5. $^{40}\text{Ar}/^{39}\text{Ar}$ analysis of aggregate separates

5.1. Step-heating experiments

Conventional step-heating experiments were performed on LP-6 biotite and Fish Canyon Tuff sanidine. GA-1550 biotite, with a reference age of 98.5 ± 0.8 Ma, was used as the fluence monitor for these experiments.

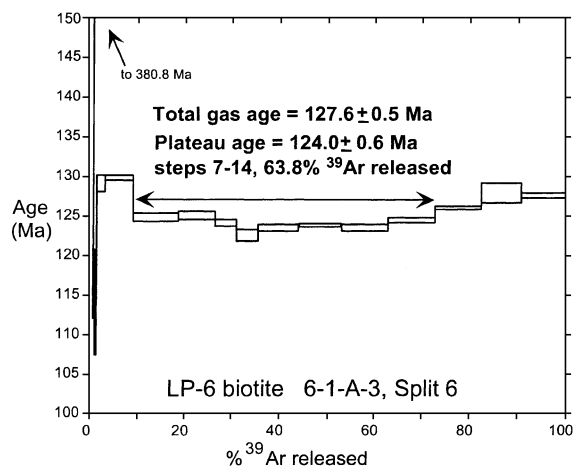


Fig. 1. Step heating age spectrum for LP-6 biotite. Vertical width of individual bars represents $\pm 1\sigma$ analytical errors. Age is calculated using GA-1550, with an age of 98.5 Ma, as a fluence monitor.

5.1.1. LP-6 biotite

The step-heating age spectrum for a split of LP-6 biotite is discordant, exhibiting ages around 130 Ma for the first $\sim 10\%$ of gas released (disregarding some small older and younger steps in the initial $\sim 1\%$ gas released), followed by decreasing ages to ~ 124 Ma for ~ 30 – 60% gas released and increas-

Table 3

LP-6 biotite step-heating $^{40}\text{Ar}/^{39}\text{Ar}$ data—USGS standard, 28.9 mg

Temperature (°C)	^{36}Ar [$\times 10^{-15}$ mol]	^{37}Ar [$\times 10^{-15}$ mol]	^{39}Ar [$\times 10^{-13}$ mol]	^{40}Ar [$\times 10^{-12}$ mol]	$^{40}\text{Ar}^*$ [%]	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$	Cumulative ^{39}Ar [%]	Apparent age [Ma $\pm 1\sigma$]	Ca/K [$\times 10^{-2}$]
500	2.373	0.453	0.144	1.578	55.5	60.846	0.50	383.3 ± 2.6	6.92
550	0.480	0.348	0.057	0.274	48.2	23.306	0.70	156.6 ± 3.4	13.50
600	0.458	0.332	0.076	0.267	49.4	17.268	0.96	117.3 ± 4.5	9.56
640	0.547	0.531	0.129	0.371	56.4	16.263	1.41	110.7 ± 2.5	9.09
680	1.122	0.414	0.459	1.215	72.6	19.226	3.00	130.1 ± 1.0	1.98
720	2.537	1.322	1.722	4.081	81.5	19.324	8.97	130.7 ± 0.3	1.69
750	2.558	0.000	2.790	5.940	87.2	18.555	18.65	125.8 ± 0.6	0.00
780	1.268	0.835	2.213	4.495	91.5	18.594	26.33	126.0 ± 0.5	0.83
810	0.786	1.147	1.357	2.738	91.4	18.444	31.03	125.0 ± 0.5	1.86
850	0.875	1.061	1.224	2.489	89.5	18.200	35.28	123.4 ± 0.8	1.91
890	1.365	1.895	2.474	4.949	91.7	18.349	43.86	124.4 ± 0.4	1.69
920	1.328	1.932	2.613	5.208	92.3	18.407	52.93	124.8 ± 0.2	1.63
950	1.298	1.478	2.891	5.693	93.1	18.344	62.95	124.4 ± 0.4	1.12
980	1.284	1.196	2.832	5.627	93.1	18.507	72.78	125.4 ± 0.3	0.93
1010	1.289	1.190	2.788	5.611	93.1	18.734	82.45	126.9 ± 0.2	0.94
1040	1.236	0.847	2.455	5.043	92.6	19.029	90.96	128.8 ± 1.3	0.76
1300	1.194	3.543	2.605	5.302	93.2	18.977	100.00	128.5 ± 0.3	2.99
Total	22.00	18.52	28.83	60.88		18.841		127.6 ± 0.5	

$\lambda^{40}\text{K} = 5.543 \times 10^{-10} \text{ a}^{-1}$; $J = 3.8984 \times 10^{-3}$; flux monitor—GA-1550 biotite with reference age of 98.5 Ma, all steps = 15 min.

ing ages up to the final fusion step at ~ 127 Ma (Table 3, Fig. 1). K/Ca ratios generally mimic this in an inverse fashion (Table 3), suggesting that there are two phases of differing Ca and K contents contributing to the degassing behaviour in a manner similar to that described by Wijbrans and McDougall (1986) and Lo Bello et al. (1987). This is consistent with the observations of Ingamells and Engels (1976) that LP-6 consists of two distinct biotite components, which they suggested to have ages of ~ 120 and 135 Ma. Steps 7–14 (750–980 °C) are concordant at 2σ and yield a weighted mean plateau age of 124.0 ± 0.6 Ma, comprising 63.8% of the ^{39}Ar released. The integrated (total gas) age is 127.6 ± 0.5 Ma (Table 3) and is similar to ages obtained for individual fusions on single or multiple crystals (see below). Statistically valid isochrons were not obtained from LP-6 step-heating data; however, “errorchrons” obtained do suggest the presence of extraneous argon. An isochron regressed using the initial six heating steps indicates an $^{40}\text{Ar}/^{36}\text{Ar}$ of ~ 570 , whereas data for steps 7–14 yield an atmospheric value within 2σ errors.

5.1.2. Fish Canyon Tuff sanidine

The step-heating age spectrum for the Fish Canyon Tuff (92-176) sanidine is fairly concordant; however,

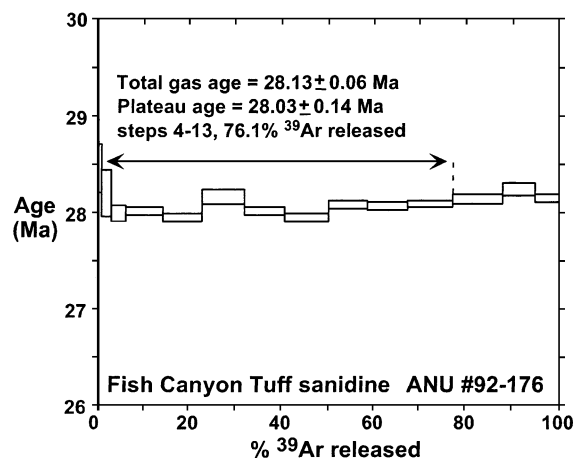


Fig. 2. Step-heating age spectrum for ANU 92-176, sanidine from the Fish Canyon Tuff. Vertical width of individual bars represents $\pm 1\sigma$ analytical errors. Age is calculated using GA-1550, with an age of 98.5 Ma, as a fluence monitor.

some minor structure present (Table 4, Fig. 2). Ages for the initial $\sim 3\%$ gas released appear slightly higher than the total gas age of 28.13 ± 0.06 Ma, although not statistically significant. Some variation in age is apparent during the intermediate temperature steps (e.g., the 1025 °C step) and, overall, there is a suggestion of a slight increase of ages with increasing temperature on

Table 4

Fish Canyon Tuff sanidine step-heating $^{40}\text{Ar}/^{39}\text{Ar}$ data—ANU 92-176, 250–500 μm fraction, 100.2 mg

Temperature (°C)	^{36}Ar [$\times 10^{-16}$ mol]	^{37}Ar [$\times 10^{-15}$ mol]	^{39}Ar [$\times 10^{-13}$ mol]	^{40}Ar [$\times 10^{-12}$ mol]	$^{40}\text{Ar}^*$ [%]	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$	Cumulative ^{39}Ar [%]	Apparent age [Ma $\pm 1\sigma$]	Ca/K [$\times 10^{-2}$]
600	2.580	1.244	0.101	0.135	43.6	5.8516	0.09	38.34 ± 7.59	27.2
670	1.921	0.193	0.261	0.167	65.6	4.1968	0.31	27.58 ± 1.65	1.62
750	1.612	0.597	0.966	0.469	89.3	4.3399	1.12	28.52 ± 0.25	1.36
820	1.386	3.349	2.361	1.062	95.6	4.2988	3.12	28.24 ± 0.24	3.12
870	1.717	4.433	3.847	1.701	96.4	4.2659	6.37	28.03 ± 0.08	2.54
950	2.111	9.008	9.639	4.203	97.9	4.2704	14.51	28.06 ± 0.04	2.06
990	2.026	7.859	9.937	4.319	98.0	4.2606	22.91	27.99 ± 0.04	1.74
1025	0.865	8.139	10.76	4.675	98.9	4.2949	32.00	28.22 ± 0.07	1.66
1050	1.699	7.798	10.38	4.510	98.3	4.2711	40.78	28.06 ± 0.04	1.65
1075	2.046	6.993	11.15	4.839	98.2	4.2615	50.20	28.00 ± 0.04	1.38
1090	1.476	7.280	9.917	4.314	98.4	4.2807	58.58	28.12 ± 0.05	1.61
1110	1.815	7.243	10.5	4.551	98.2	4.2781	67.41	28.11 ± 0.04	1.52
1125	1.829	8.472	11.59	5.044	98.3	4.2814	77.20	28.13 ± 0.03	1.61
1135	1.748	9.166	12.89	5.614	98.5	4.2904	88.09	28.19 ± 0.04	1.56
1140	1.476	5.974	8.291	3.634	98.2	4.3047	95.10	28.28 ± 0.06	1.59
1500	2.230	4.874	5.801	2.570	96.9	4.2912	100.00	28.19 ± 0.05	1.85
Total	28.54	92.26	118.3	51.81		4.2815		28.13 ± 0.06	

$\lambda^{40}\text{K} = 5.543 \times 10^{-10} \text{ a}^{-1}$; $J = 3.6783 \times 10^{-3}$; fluence monitor—GA-1550 biotite with reference age of 98.5 Ma, all steps = 15 min.

the order of $\sim 1\%$. The high initial ages may be related to small amounts of melt inclusion-hosted excess argon (Esser et al., 1997). Despite these small deviations from ideal behaviour, this sample appears to have simple argon release systematics and conforms well to the expectations for a suitable $^{40}\text{Ar}/^{39}\text{Ar}$ standard. Steps 4–13, comprising 76.1% of the total ^{39}Ar released, define a plateau age of 28.03 ± 0.14 Ma (Fig. 2), indistinguishable from the total gas age of 28.13 ± 0.06 Ma (Table 4). Analytical data for 92-176 were too highly radiogenic to yield meaningful isochron results. A more detailed step-heating analysis of this sanidine (25 steps) at the University of Houston yields a very similar age spectrum.

5.2. Total fusion experiments

Results of total fusion analyses on 7.6- to 30.4-mg aliquots of the standards are summarized in Table 5, with the full data set given in Appendix A. Calculated average $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios are based on the mean and standard deviation of two to four aliquots, many of which had multiple gas splits measured. The $^{40}\text{Ar}^*/$

$^{39}\text{Ar}_K$ ratios in Table 5 are calculated by first averaging gas splits for each aliquot, followed by averaging of these resulting numbers. The quoted precisions thus reflect both the reproducibility of the mass spectrometer runs (gas splits) and any variability introduced by sample inhomogeneity (sample splits). The overall reproducibilities range from $\pm 0.06\%$ to 0.65% and average 0.24%. The average reproducibilities for splits of the standards are: MMhb-1 hornblende, 0.16%; GA-1550 biotite, 0.28%; 77-600 hornblende, 0.31%; LP-6 biotite, 0.24%; and 92-176 sanidine, 0.18%. With few exceptions, these levels of precision are within the range of those for replicate analyses of gas splits (± 0.01 –0.38%, averaging $\pm 0.21\%$) and are similar to typical analytical precisions for $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios (averaging $\pm 0.22\%$).

6. $^{40}\text{Ar}^*/^{39}\text{Ar}$ single and multiple crystal laser fusion experiments

Laser fusion analyses on small sample aliquots, such as single crystals, may allow the detection of sample

Table 5
Average $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio for each sample at each level in irradiations J757 and J791

Irradiation and level (L)	Mineral	<i>n</i>	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm S.D. (\pm CV)]
J757, L3—inner	MMhb-1 Hornblende	2	86.27 ± 0.14 ($\pm 0.16\%$)
J757, L3—outer	GA-1550 Biotite	3	14.37 ± 0.02 ($\pm 0.11\%$)
J757, L4—inner	77-600 Hornblende	2	66.64 ± 0.30 ($\pm 0.45\%$)
J757, L5—inner	GA-1550 Biotite	2	14.42 ± 0.03 ($\pm 0.23\%$)
J757, L5—outer	LP-6 Biotite	2	18.77 ± 0.02 ($\pm 0.09\%$)
J757, L6—inner	MMhb-1 Hornblende	2	85.61 ± 0.05 ($\pm 0.06\%$)
J757, L6—outer	77-600 Hornblende	3	66.39 ± 0.09 ($\pm 0.14\%$)
J757, L7—inner	GA-1550 Biotite	2	14.22 ± 0.05 ($\pm 0.33\%$)
J757, L7—outer	MMhb-1 Hornblende	4	85.03 ± 0.11 ($\pm 0.13\%$)
J757, L8—inner	GA-1550 Biotite	2	14.06 ± 0.03 ($\pm 0.21\%$)
J757, L8—outer	77-600 Hornblende	3	65.53 ± 0.22 ($\pm 0.33\%$)
J791, L1—inner	GA-1550 Biotite	2	15.31 ± 0.10 ($\pm 0.65\%$)
J791, L2—inner	MMhb-1 Hornblende	2	93.09 ± 0.14 ($\pm 0.15\%$)
J791, L2—outer	LP-6 Biotite	3	20.46 ± 0.08 ($\pm 0.39\%$)
J791, L3—inner	92-176 Sanidine	2	4.343 ± 0.006 ($\pm 0.15\%$)
J791, L3—outer	GA-1550 Biotite	3	15.53 ± 0.02 ($\pm 0.13\%$)
J791, L4—inner	92-176 Sanidine	2	4.326 ± 0.013 ($\pm 0.31\%$)
J791, L4—outer	MMhb-1 Hornblende	3	93.14 ± 0.27 ($\pm 0.29\%$)
J791, L5—inner	GA-1550 Biotite	2	15.46 ± 0.05 ($\pm 0.34\%$)
J791, L6—inner	GA-1550 Biotite	2	15.30 ± 0.03 ($\pm 0.21\%$)
J791, L6—outer	92-176 Sanidine	3	4.284 ± 0.004 ($\pm 0.08\%$)

Uncertainty quoted is the sample standard deviation. All measurements on bulk samples, ranging from 7.6 to 30.4 mg in weight, with isotope ratios determined in the VG1200 mass spectrometer using the Faraday collector.

inhomogeneity which is not apparent at the level of tens of milligrams, where the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio is the average value derived from gas released from hundreds of individual crystals. A summary of the results of laser fusion analyses ranging from 1 to ~ 50 individual crystals is given in Table 6, with the full data set given in Appendix A. Analyses were conducted as described above for total fusion runs on large aliquots. The overall reproducibilities range from $\pm 0.16\%$ to 1.46% and average 0.61% . The average reproducibilities for splits of the standards are: MMhb-1 hornblende, 0.82% ; GA-1550 biotite, 0.47% ; 77-600 hornblende, 0.97% ; LP-6 biotite, 1.45% ; and 92-176 sanidine, 0.60% . In contrast to analyses of bulk samples discussed above, some of these reproducibilities are larger than the typical precision of the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ for gas splits (average of $\pm 0.43\%$) and analytical precisions (average of $\pm 0.43\%$) on the VG3600 mass spectrometer used for these analyses, suggesting that

sample inhomogeneity may be contributing to the scatter.

7. Discussion

7.1. Step-heating experiments

Alexander and Davis (1974) and Dalrymple et al. (1981) outlined the requirements for a suitable standard mineral for $^{40}\text{Ar}/^{39}\text{Ar}$ dating and these were reviewed by McDougall and Harrison (1999). One of the requirements is that the crystals of the standard have a homogeneous intracrystalline and inter-crystalline distribution of K and $^{40}\text{Ar}^*$ and, thus, following irradiation, will have a homogeneous distribution of $^{40}\text{Ar}^*/^{39}\text{Ar}_K$. This ensures that errors in the calculation of an $^{40}\text{Ar}/^{39}\text{Ar}$ age for an unknown sample are not introduced via variations in the fluence monitor's $^{40}\text{Ar}^*/^{39}\text{Ar}_K$. Should inhomogeneities exist, they may affect the measurement by introducing a variability from different splits of the standard mineral; this effect would be most pronounced in the case of measurement at the single crystal level. Alternatively, should the age spectrum not be ideally flat, or near to it, incomplete degassing during fusion of the irradiated fluence monitor may bias the measured $^{40}\text{Ar}^*/^{39}\text{Ar}_K$.

One method of assessing the homogeneity of the $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ is through a step-heating experiment with the observation of a flat age spectrum interpreted as signifying homogeneity. However, this approach is only strictly applicable to minerals such as feldspar because hydrous phases such as biotite and hornblende are unstable during in vacuo heating, undergoing structural breakdown and phase changes, thereby obviating the possibility of determining intracrystalline spatial argon distribution by this method (e.g., Gaber et al., 1988; Wartho et al., 1991; Lee, 1993).

The discordant age spectrum documented here for LP-6 biotite (Fig. 1) demonstrates that this mineral is not a suitable fluence monitor, even at the relatively large sampling level of ~ 30 mg. Clearly, incomplete fusion, although unlikely for a biotite, would result in significantly different $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ values than would be found when complete fusion was achieved. This finding confirms that of Baksi et

Table 6
Average $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio for each sample at each level in the three silica glass tubes in irradiation L112

Tube and level (L)	Mineral	<i>n</i>	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm S.D. (\pm CV)]
Tube I, L1	GA-1550 Biotite	4	13.16 ± 0.19 ($\pm 1.46\%$)
Tube I, L2	92-176 Sanidine	5	3.674 ± 0.047 ($\pm 1.28\%$)
Tube I, L3	GA-1550 Biotite	2	12.84 ± 0.04 ($\pm 0.34\%$)
Tube I, L4	MMhb-1 Hornblende	8	75.83 ± 0.62 ($\pm 0.82\%$)
Tube I, L5	GA-1550 Biotite	2	12.73 ± 0.02 ($\pm 0.16\%$)
Tube I, L6	92-176 Sanidine	2	3.557 ± 0.019 ($\pm 0.55\%$)
Tube I, L7	GA-1550 Biotite	1	12.74 ± 0.03 ($\pm 0.22\%$)
Tube II, L5	GA-1550 Biotite	3	13.53 ± 0.04 ($\pm 0.26\%$)
Tube II, L6	92-176 Sanidine	4	3.775 ± 0.014 ($\pm 0.38\%$)
Tube II, L7	GA-1550 Biotite	1	13.52 ± 0.03 ($\pm 0.20\%$)
Tube III, L1	GA-1550 Biotite	2	12.97 ± 0.11 ($\pm 0.83\%$)
Tube III, L2	77-600 Hornblende	8	61.05 ± 0.59 ($\pm 0.97\%$)
Tube III, L3	GA-1550 Biotite	2	13.09 ± 0.02 ($\pm 0.16\%$)
Tube III, L4	LP-6 Biotite	2	17.85 ± 0.26 ($\pm 1.45\%$)
Tube III, L5	GA-1550 Biotite	2	13.12 ± 0.12 ($\pm 0.88\%$)
Tube III, L6	92-176 Sanidine	3	3.705 ± 0.007 ($\pm 0.18\%$)
Tube III, L7	GA-1550 Biotite	1	13.21 ± 0.03 ($\pm 0.22\%$)

Uncertainty quoted is the sample standard deviation. Most measurements for GA-1550 and 92-176 were on one or two crystals; measurements on other minerals involved 1–50 crystals. All minerals were fused by laser beam, and isotope ratios were measured on a Daly collector of the VG3600 mass spectrometer, except for the level 7 GA-1550 biotite, which was measured on a Faraday collector of the VG1200 mass spectrometer.

al. (1996); however, we note that, although age spectra obtained in that study are broadly similar to ours, in detail they differ, giving further evidence for inhomogeneity.

The generally concordant age spectrum for 92-176, Fish Canyon Tuff sanidine, is the expected result for a rapidly cooled volcanic rock containing no xenocrysts (Fig. 2). Aside from concerns about quantitative extraction of $^{40}\text{Ar}^*$, sanidine from a volcanic rock is in many ways an ideal fluence monitor; having a high K-content, a homogeneous intracrystalline $^{40}\text{Ar}^*$ distribution, and being anhydrous, this mineral should exhibit an ideally flat age spectrum, which is representative of the intracrystalline distribution of $^{40}\text{Ar}^*/^{39}\text{Ar}_K$. The age spectrum obtained in this study (Fig. 2) generally conforms to these expectations. We note that the age spectrum presented by Lanphere and Baadsgaard (2001) does not appear to exhibit these older ages with the first few percent gas released, whereas sample 92-176 has this feature reproduced in two separate laboratories (the ANU and the University of Houston). As previously mentioned, there are a number of such individually prepared sanidine separates from the Fish Canyon Tuff in use in $^{40}\text{Ar}/^{39}\text{Ar}$ laboratories worldwide; this illustrates potential differences caused by sample preparation procedures, even though samples may have been collected from the same locality.

7.2. Total fusion experiments

Results for total fusion experiments on large aliquots (7.6–30.4 mg) indicate that no discernable scatter is introduced into the measured $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios by inhomogeneities in the standard minerals studied. Comparison of MSWDs indicates that repeat measurements of splits of the standards are, on average, within the range of reproducibility of mass spectrometer runs on gas splits and are similar to analytical precisions on $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios (Table 5). This suggests that no significant inhomogeneities exist for these standards at these relatively large sample split levels of tens of milligrams (i.e., the variability seen can be accounted for by the precision of mass spectrometer analyses alone). All of these standards are thus suitable for $^{40}\text{Ar}/^{39}\text{Ar}$ dating provided that relatively large aliquots (tens of milligrams) are utilized.

7.3. Laser fusion experiments

In contrast to the large aliquot total fusion analyses, the fusion of aliquots ranging from 1 to ~ 50 single crystals does reveal inhomogeneities in the standards. Detectable sample inhomogeneity is present in MMhb-1 hornblende, 77-600 hornblende, and LP-6 biotite as both individual reproducibilities and average reproducibilities are lower than the precision of the mass spectrometer runs (Table 6). In particular, LP-6 biotite appears to have the highest level of inhomogeneity, yielding a precision of only $\pm 1.45\%$, over three times lower than the reproducibility of mass spectrometer data ($\pm 0.43\%$). Both MMhb-1 hornblende and 77-600 hornblende exhibit a reproducibility of approximately $\pm 1\%$, nearly twice the mass spectrometer precision level. Both LP-6 biotite and MMhb-1 hornblende have been documented as inhomogeneous (e.g., Ingamells and Engels, 1976; Baksi et al., 1996; Renne et al., 1998); our results confirm these earlier studies. However, the relatively poor average reproducibilities for GA-1550 ($\pm 0.47\%$) and 92-176 sanidine ($\pm 0.60\%$) appear anomalous, as these standards are thought to be relatively homogeneous at the single crystal level (Baksi et al., 1996; Renne et al., 1998). We note that in two cases (tube I, L1-GA-1550 biotite; tube I, L2-92-176 sanidine) (Table 6), the level of reproducibility is substantially below that for replicate analyses of gas splits, whereas most others exhibit significantly higher precision. In the case of tube I, L1-GA-1550 biotite, there may have been some contamination by another mineral from an adjacent well in the case of split 114, as evidenced by the anomalously high $^{37}\text{Ar}/^{39}\text{Ar}$ values (see Appendix A). Omission of these analyses results in a CV of $\pm 0.66\%$ and brings the overall average for GA-1550 to $\pm 0.39\%$. The high amount of variability for 92-176 sanidine derives from the large spread in $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios from tube I, L2 for five measurements ($\pm 1.28\%$). Of these, the second split (analyses 2I2a and 2I2b; see Appendix A) yielded anomalously high $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratios. Omission of these measurements results in a CV of $\pm 0.42\%$, and an overall average for 92-176 of $\pm 0.38\%$, although there is no clear indication of contamination as for GA-1550 discussed above. Our experience at the ANU and the UNLV subsequent to this study, involving the routine use of single crystals

Table 7
Summary of $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ ratio between minerals

Irradiation and level	$\frac{(^{40}\text{Ar}^*/^{39}\text{Ar}_K)_{\text{standard 1}}}{(^{40}\text{Ar}^*/^{39}\text{Ar}_K)_{\text{standard 2}}}$	$R_{\text{standard 2}}^{\text{standard 1}}$
J791, level 3—outer and inner	$\frac{15.527 \pm 0.020 (\pm 0.13\%)}{4.343 \pm 0.006 (\pm 0.15\%)}$	$R_{92-176}^{\text{GA-1550}}$ $3.575 \pm 0.007 (\pm 0.19\%)$
J791, level 6—inner and outer	$\frac{15.303 \pm 0.032 (\pm 0.21\%)}{4.284 \pm 0.004 (\pm 0.08\%)}$	$3.572 \pm 0.008 (\pm 0.22\%)$
L112, tube I $\frac{(L1+L3)/2}{L2}$	$\frac{12.998 \pm 0.195 (\pm 1.50\%)}{3.674 \pm 0.047 (\pm 1.28\%)}$	$3.537 \pm 0.070 (\pm 1.97\%)$
L112, tube I $\frac{(L5+L7)/2}{L6}$	$\frac{12.736 \pm 0.034 (\pm 0.27\%)}{3.557 \pm 0.019 (\pm 0.55\%)}$	$3.581 \pm 0.022 (\pm 0.61\%)$
L112, tube II $\frac{(L5+L7)/2}{L6}$	$\frac{13.524 \pm 0.044 (\pm 0.33\%)}{3.775 \pm 0.014 (\pm 0.38\%)}$	$3.583 \pm 0.018 (\pm 0.50\%)$
L112, tube III $\frac{(L5+L7)/2}{L6}$	$\frac{13.166 \pm 0.119 (\pm 0.92\%)}{3.705 \pm 0.007 (\pm 0.18\%)}$	$3.554 \pm 0.033 (\pm 0.92\%)$
Arithmetic mean	\bar{x}_6	$3.567 \pm 0.018 (\pm 0.61\%)$
S.D. mean		$\pm 0.007 (\pm 0.21\%)$
Weighted mean	\bar{x}_6	$3.575 \pm 0.005 (\pm 0.13\%)$
J757, level 3—inner and outer	$\frac{86.27 \pm 0.14 (\pm 0.16\%)}{14.366 \pm 0.016 (\pm 0.11\%)}$	$R_{\text{GA-1550}}^{\text{MMhb-1}}$ $6.005 \pm 0.012 (\pm 0.20\%)$
J757, level 7—outer and inner	$\frac{85.03 \pm 0.11 (\pm 0.13\%)}{14.224 \pm 0.047 (\pm 0.33\%)}$	$5.978 \pm 0.021 (\pm 0.35\%)$
L112, tube I $\frac{L4}{(L3+L5)/2}$	$\frac{75.83 \pm 0.62 (\pm 0.82\%)}{12.786 \pm 0.047 (\pm 0.37\%)}$	$5.931 \pm 0.053 (\pm 0.90\%)$
Arithmetic mean	\bar{x}_3	$5.971 \pm 0.037 (\pm 0.63\%)$
S.D. mean		$\pm 0.022 (\pm 0.36\%)$
Weighted mean	\bar{x}_3	$5.996 \pm 0.010 (\pm 0.17\%)$
J757, level 8—outer and inner	$\frac{65.53 \pm 0.22 (\pm 0.33\%)}{14.064 \pm 0.030 (\pm 0.21\%)}$	$R_{\text{GA-1550}}^{77-600}$ $4.659 \pm 0.018 (\pm 0.39\%)$
J757, $\frac{L4}{(L3(\text{outer})+L5(\text{inner}))/2}$	$\frac{66.64 \pm 0.30 (\pm 0.45\%)}{14.395 \pm 0.036 (\pm 0.25\%)}$	$4.629 \pm 0.048 (\pm 0.51\%)$
L112, tube III $\frac{L2}{(L1+L3)/2}$	$\frac{61.05 \pm 0.59 (\pm 0.97\%)}{13.027 \pm 0.11 (\pm 0.85\%)}$	$4.686 \pm 0.060 (\pm 1.29\%)$
Arithmetic mean	\bar{x}_3	$4.658 \pm 0.029 (\pm 0.62\%)$
S.D. mean		$\pm 0.017 (\pm 0.35\%)$
Weighted mean	\bar{x}_3	$4.650 \pm 0.014 (\pm 0.30\%)$
J757, level 5—outer and inner	$\frac{18.773 \pm 0.016 (\pm 0.09\%)}{14.423 \pm 0.033 (\pm 0.23\%)}$	$R_{\text{GA-1550}}^{\text{LP-6}}$ $1.302 \pm 0.003 (\pm 0.24\%)$
J791, $\frac{L2}{(L1(\text{inner})+L3(\text{outer}))/2}$	$\frac{20.459 \pm 0.079 (\pm 0.39\%)}{15.419 \pm 0.102 (\pm 0.66\%)}$	$1.327 \pm 0.010 (\pm 0.77\%)$
L112, tube III $\frac{L4}{(L3+L5)/2}$	$\frac{17.846 \pm 0.259 (\pm 1.45\%)}{13.104 \pm 0.117 (\pm 0.89\%)}$	$1.362 \pm 0.023 (\pm 1.70\%)$
Arithmetic mean	\bar{x}_3	$1.330 \pm 0.030 (\pm 2.27\%)$
S.D. mean		$\pm 0.013 (\pm 1.31\%)$
Weighted mean	\bar{x}_3	$1.305 \pm 0.003 (\pm 0.23\%)$

Table 7 (continued)

Irradiation and level	$\frac{({}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K)_{\text{standard 1}}}{({}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K)_{\text{standard 2}}}$	$R_{\text{standard 2}}^{\text{standard 1}}$
J791, level 2—inner and outer	$\frac{93.09 \pm 0.14 (\pm 0.15\%)}{20.459 \pm 0.079 (\pm 0.39\%)}$	$R_{\text{LP-6}}^{\text{MMhb-1}}$ $4.550 \pm 0.019 (\pm 0.42\%)$
J791, level 4—outer and inner	$\frac{93.14 \pm 0.27 (\pm 0.29\%)}{4.326 \pm 0.013 (\pm 0.31\%)}$	$R_{92-176}^{\text{MMhb-1}}$ $21.530 \pm 0.092 (\pm 0.43\%)$
J757, level 6—inner and outer	$\frac{85.61 \pm 0.05 (\pm 0.06\%)}{66.39 \pm 0.09 (\pm 0.14\%)}$	$R_{77-600}^{\text{MMhb-1}}$ $1.289 \pm 0.002 (\pm 0.15\%)$

Uncertainties quoted at the level of 1 S.D., combined quadratically as appropriate.

of these two minerals in fluence monitoring, indicates that the reproducibility (typically of three to seven analyses) yields an ${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ averaging about $\pm 0.3\%$, amply confirming the homogeneity at the single crystal level. Thus, for laser fusion analyses, it is clear from this study and previous studies that LP-6 biotite, MMhb-1 hornblende, and 77-600 hornblende are inhomogeneous and are not suitable as fluence monitors at the single crystal level. With the exception of the anomalous analyses discussed above for GA-1550 biotite and 92-176 sanidine, these standards do appear homogeneous at the single crystal level, and are thus suitable as fluence monitors for laser fusion work.

7.4. Intercalibrations between standards

Direct comparisons between standards may be made simply by calculating R values (Table 7), which are relative ${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ ratios of the standards after they have been co-irradiated, thus receiving the same neutron fluence (Renne et al., 1998). An important aspect of this approach is that comparison between results from different studies and different laboratories may be made, essentially independently of assignment of age to any of the standards. And, as pointed out by Renne et al. (1998), the ${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ ratios are derived directly from isotopic ratio measurements, without requiring any knowledge as to the numerical age of a standard. After all measurements among a set of standards are compiled (Table 7), a statistical assessment of the precision of the R values is derived by a quadratic combination of errors on individual

${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ ratios to derive the error in the R value for an individual pair of intercalibrated standards, followed by a calculation of mean and standard deviation, and weighted means of the multiple R values. Both total fusion analyses on large samples (in inner and outer cans at the same level) and laser fusion analyses (where ${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ ratios from the bracketing primary standard are averaged) are combined to derive the final R values. Weighted means and errors are used here, allowing a direct comparison with Renne et al. (1998). This method ultimately allows an assessment of the relative ages between standards as the R value is a multiplying factor in a modified form of the age equation:

$$t_s = \frac{1}{\lambda} \ln[R_p^s (e^{\lambda t_p} - 1) + 1] \quad (1)$$

where t_s denotes the age which is to be calculated for the secondary standard, t_p is the assumed age of the primary standard, R_p^s is the ratio of ${}^{40}\text{Ar}^*/{}^{39}\text{Ar}_K$ values for the secondary and primary standards, and $\lambda = 5.543 \times 10^{-10} \text{ a}^{-1}$. Where R values are not calculated directly in other studies, they can be derived by a rearrangement of Eq. (1) to solve for R , or a multiplication of appropriate reported R values via the following equation with errors propagated by quadratic summation:

$$R_B^A R_C^B = R_C^A \quad (2)$$

R values thus obtained are compared with those from other studies in Table 8. As error estimates for calculated R values reported in Table 8 from other

Table 8

Intercalibration R values calculated from this study compared with those from previously published studies

Reference	$R_{92-176}^{GA-1550}$	$R_{GA-1550}^{MMhb-1}$	$R_{GA-1550}^{77-600}$	$R_{GA-1550}^{LP-6}$	R_{LP-6}^{MMhb-1}	R_{92-176}^{MMhb-1}	R_{77-600}^{MMhb-1}
This study	3.575 ± 0.005	5.996 ± 0.010	4.650 ± 0.014	1.305 ± 0.003	4.550 ± 0.019	21.530 ± 0.092	1.289 ± 0.002
Charbit et al. (1998)	–	–	–	–	4.551 ± 0.035 (C)	–	–
Renne et al. (1998)	3.596 ± 0.004 (P)	5.976 ± 0.007 (C)	–	–	–	21.488 ± 0.008 (P)	–
Baksi et al. (1996)	3.568 ± 0.034 (C)	5.948 ± 0.052 (C)	–	1.321 ± 0.010 (C)	4.504 ± 0.026 (C)	21.238 ± 0.068 (C)	–
Lanphere and Dalrymple (2000)	–	5.955 ± 0.032 (C)	–	–	–	–	–
Lanphere et al. (1990)	–	5.842 (C)	–	–	–	21.417 (C)	–
Cebula et al. (1986)	–	–	–	–	–	21.492 (C)	–
Roddick (1983)	–	–	–	–	4.511 (C)	–	–
Harrison (1981)	–	–	4.644 ± 0.037 (C)	–	–	–	1.277 ± 0.011 (C)

P = as published; C = as calculated.

Where values have been calculated from other studies, error estimates have been obtained as discussed in the text.

studies are quadratic summations of reported errors on ages, they are not necessarily equivalent to those calculated from our data. However, they allow comparisons to be made between studies. In some cases, insufficient data are available to estimate errors; in these cases, they are not reported.

$R_{92-176}^{GA-1550}$: Our data for calibration of GA-1550 biotite relative to 92-176, Fish Canyon Tuff sanidine, are derived from six measurements, two of which are from total fusion analyses on large samples, and four of which are from laser fusion analyses (Table 7). These data give a weighted mean $R_{92-176}^{GA-1550}$ of 3.575 ± 0.005 . This is 0.6% lower than the value of 3.5957 ± 0.0038 reported by Renne et al. (1998) (Table 8). An R value of 3.568 ± 0.034 can be calculated from data reported by Baksi et al. (1996) for GA-1550 biotite relative to biotite (FCT-3) from the Fish Canyon Tuff (Table 4 from Baksi et al., 1996). Assuming that Fish Canyon Tuff sanidine and biotite are equivalent in age, this is 0.1% lower than the value obtained in this study.

$R_{GA-1550}^{MMhb-1}$: Our data for calibration of MMhb-1 hornblende relative to GA-1550 biotite are derived from three measurements, two of which are from total fusion analyses, and one from laser fusion analyses

(Table 7), and give a weighted mean $R_{GA-1550}^{MMhb-1}$ of 5.996 ± 0.010 . This is compatible at the 2σ confidence level, with a value of 5.976 ± 0.007 (0.3% lower) calculated from data reported by Renne et al. (1998) and a value of 5.948 ± 0.052 (0.8% lower) calculated from Table 4 of Baksi et al. (1996) (Table 8). Data reported in Lanphere and Dalrymple (2000) for total fusion and step-heating analyses of GA-1550 using MMhb-1 as a standard can be used to calculate an $R_{GA-1550}^{MMhb-1}$ of 5.955 ± 0.032 (0.7% lower), which is within-error of other R values reported in Table 8. A value of 5.842 (2.6% lower) calculated from relative ages reported by Lanphere et al. (1990) appears to be significantly lower than other values (Table 8).

$R_{GA-1550}^{77-600}$: A weighted mean $R_{GA-1550}^{77-600}$ value of 4.650 ± 0.014 is given by a compilation of two total fusion intercalibrations and one laser fusion intercalibration between 77-600 hornblende and GA-1550 biotite (Table 7). This compares favorably with a value of 4.644 ± 0.037 (0.1% lower) calculated from data reported by Harrison (1981) (Table 8).

$R_{GA-1550}^{LP-6}$: Our intercalibration of LP-6 biotite and GA-1550 biotite is derived from a weighted mean of two total fusion analyses and one laser fusion analysis (Table 7) and gives an $R_{GA-1550}^{LP-6}$ value of 1.305 ± 0.003 . A similar value of 1.321 ± 0.010

(1.2% higher) can be calculated from data reported by Baksi et al. (1996) (Table 8).

R_{LP-6}^{MMhb-1} : A value of 4.550 ± 0.019 for intercalibration of MMhb-1 hornblende with LP-6 biotite is derived from one total fusion analysis (Table 7). This compares with R values of 4.504 ± 0.026 (1.0% lower) calculated from data reported by Baksi et al. (1996) and 4.511 (0.9% lower) derived from data in Roddick (1983) (Table 8).

R_{92-176}^{MMhb-1} : The R_{92-176}^{MMhb-1} value of 21.53 ± 0.09 obtained here for intercalibration of MMhb-1 hornblende with 92-176, Fish Canyon Tuff sanidine, is based on one total fusion analysis (Table 7). This value is in good agreement with a value of 21.4876 ± 0.0079 (0.2% lower) reported by Renne et al. (1998), and values of 21.42 and 21.49 (0.5% and 0.2% lower) calculated from data in Lanphere et al. (1990) and Cebula et al. (1986), respectively. A value of 21.24 ± 0.07 (1.3% lower) calculated from data in Baksi et al. (1996) using FCT-3 biotite is somewhat lower than other R values for this pair of standards (Table 8), assuming that biotite and sanidine from the Fish Canyon Tuff have the same age.

R_{77-600}^{MMhb-1} : The R_{77-600}^{MMhb-1} value of 1.289 ± 0.002 obtained here for intercalibration of MMhb-1 hornblende with 77-600 hornblende is based on one total fusion analysis (Table 7). A value of 1.277 ± 0.011 (0.9% lower) is calculated from data in Harrison (1981).

Through the R values derived in this study, ages can be derived by reference to the preferred or measured K/Ar age of any of the standards. Referenced to GA-1550 biotite at 98.5 Ma, the ages of the other (secondary) standards are: 77-600 hornblende, 418.3 ± 1.3 Ma; MMhb-1 hornblende, 523.2 ± 0.9 Ma; LP-6 biotite, 127.5 ± 0.3 Ma; and 92-176 sanidine, 28.10 ± 0.04 Ma. These compare well with published ages in the case of MMhb-1, LP-6, and 92-176 (Table 1); however, there is a small systematic difference between the published K/Ar age for 77-600 (414.1 ± 3.9 Ma) and its age calibrated at 418.3 ± 1.3 Ma relative to GA-1550 biotite as derived here. Given the well-documented nature of GA-1550 (this study; Baksi et al., 1996; Renne et al., 1998) and concerns regarding quantitative extraction of $^{40}\text{Ar}^*$ from the high-Mg 77-600 hornblende, we recommend that an age of 418.3 ± 1.3 Ma be

assigned to 77-600 through the intercalibration with GA-1550 biotite.

The R value approach facilitates intercomparison of results from different laboratories, especially in cases where a different age has been used for the fluence monitor (standard). The R values can be calculated from Eq. (1) or Eq. (2), given appropriate data. The age of a sample, relative to a chosen or preferred age for the standard being used, can then be calculated by using a modified form of the age equation:

$$t_r = \frac{1}{\lambda} \ln \left[\frac{(e^{\lambda t_2} - 1)}{(e^{\lambda t_1} - 1)} (e^{\lambda t_0} - 1) + 1 \right] \quad (3)$$

where t_r is the recalculated age for the sample, t_0 is the originally calculated age for the sample, t_1 is the standard age used for t_0 , and t_2 is the intercalibrated standard age used for calculating t_r .

These comparisons serve to illustrate some of the complexities involved in intercalibration of $^{40}\text{Ar}/^{39}\text{Ar}$ standards. In the majority of cases, comparisons of intercalibrations show good agreement at the 95% confidence level; however, some cases yield inconsistent results. From the summary above, these anomalous results appear to be related to standards that are known to be inhomogeneous (e.g., MMhb-1 hornblende and LP-6 biotite). However, even when results are indistinguishable at the 95% confidence level, there may be cases where results vary by significant (i.e., >1%) amounts, as noted by Renne et al. (1998). Such small systematic differences between laboratories may occur in relation to control of discrimination of the mass spectrometer, neutron fluence gradients not being appropriately accounted for in the irradiations, as well as other possible factors. Thus, whereas a single, well-designed intercalibration study may provide R values with precisions of $\ll 1\%$, the accuracy of these values (as shown by variability between studies) may well remain at $\pm 1\%$ or greater.

There has been a trend towards the use of $^{40}\text{Ar}/^{39}\text{Ar}$ fluence monitors that are not primary standards, in that they have not been dated by “first principles” using the K/Ar method in which the ^{38}Ar tracers have been calibrated against measured volumes of argon. Thus, in many examples, standard ages used are secondary, with an age determined by an $^{40}\text{Ar}/^{39}\text{Ar}$ intercalibration with a primary standard. In

some cases, there may be several intermediate $^{40}\text{Ar}/^{39}\text{Ar}$ intercalibrations between a standard and the original first principles standard. These approaches have been used particularly in the case of sanidine fluence monitors, with examples including Taylor Creek sanidine, Alder Creek sanidine, and the widely used Fish Canyon Tuff sanidine. Arguments for this approach commonly cite the difficulties in quantitatively extracting $^{40}\text{Ar}^*$ from viscous sanidine melts during K/Ar dating (e.g., Webb and McDougall, 1967), resulting in inconsistent or anomalously young ages. Thus, sanidine standards are often intercalibrated using other $^{40}\text{Ar}/^{39}\text{Ar}$ standards; most prominent are intercalibrations with MMhb-1 (Hurford and Hammerschmidt, 1985; Cebula et al., 1986; Lanphere and Baadsgaard, 2001; Lanphere et al., 1990; Deino and Potts, 1990). Unfortunately, it is now clear that MMhb-1 is inhomogeneous at small sample sizes (Baksi et al., 1996; Renne et al., 1998; this study).

The age of 520.4 ± 1.7 Ma for MMhb-1, as reported by Sampson and Alexander (1987), appears quite precise and suggests that the age of this hornblende is known to $\pm 0.33\%$ (1σ). This age was based on weighted means of all reported radiogenic argon concentrations and similarly weighted means of reported potassium abundances from 11 laboratories. However, the variation of 11 calculated ages from the individual laboratories (not reported) ranges over 6%, from 507.7 to 538.2 Ma. A mean and sample standard deviation of these individually calculated ages is 522.9 ± 8.6 Ma ($\pm 1.7\%$). Currently, two secondary calibrations for Fish Canyon Tuff sanidine are commonly employed: a widely used age of 27.84 Ma relative to 520.4 Ma for MMhb-1 (Deino and Potts, 1990) (note that intercalibration data are not presented), and an age of 27.55 Ma relative to 513.9 Ma for MMhb-1 (Lanphere et al., 1990). Given that the age of MMhb-1 is not known with a great degree of certainty, and that this standard is known to be inhomogeneous for small sample sizes, the intercalibration of the Fish Canyon Tuff sanidine by this method is of lesser value compared to first principles dating methods.

The current lack of agreement about the ages of $^{40}\text{Ar}/^{39}\text{Ar}$ standards has produced unnecessary confusion in the literature both within and outside of the geochronology community and has undermined confidence in the $^{40}\text{Ar}/^{39}\text{Ar}$ dating method—one of the

most powerful and widely applicable isotopic dating techniques available. Although intercalibration studies are desirable and help quantify the comparison of $^{40}\text{Ar}/^{39}\text{Ar}$ data generated using different standards, they do not address the fundamental problems that include the lack of a widely accepted set of primary interlaboratory standards, a lack of agreement on the ages of standards, inconsistencies in error propagation practices (e.g., Renne et al., 1998), and, most recently, concerns regarding the currently accepted decay constants (Min et al., 2000; Villeneuve et al., 2000; Begemann et al., 2001; Kwon et al., 2002). Until these issues are addressed, the precision of $^{40}\text{Ar}/^{39}\text{Ar}$ ages will remain high; yet the ultimate accuracy of these ages will be no better than ± 1 – 2% .

8. Conclusions

The results of experiments to characterize and intercalibrate the $^{40}\text{Ar}/^{39}\text{Ar}$ standards MMhb-1 hornblende, Fish Canyon Tuff sanidine, and LP-6 biotite, along with two Australian National University intra-laboratory standards 77-600 hornblende and GA-1550 biotite, indicate that all standards studied are homogeneous at a sampling level of tens of milligrams. However, only Fish Canyon Tuff sanidine and GA-1550 biotite meet the criteria for acceptable standards at the single crystal sampling level, whereas the others exhibit inhomogeneity, which compromises their usefulness. A revised K/Ar age of 98.5 ± 0.8 Ma is recommended for GA-1550 biotite, which is used as a primary standard for intercalibration. Relative to GA-1550, the ages for other standards are as follows: 418.3 ± 1.3 Ma for 77-600 hornblende, 523.2 ± 0.9 Ma for MMhb-1 hornblende, 127.5 ± 0.3 Ma for LP-6 biotite, and 28.10 ± 0.04 Ma for Fish Canyon Tuff sanidine.

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Appendix A

Analytical data for total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ measurement of mineral samples used as fluence monitors (irradiation canister J757)

Position	File number	Mineral	Mass [mg]	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-13} mol]
[1IA	304	GA-1550 biotite	9.6	5.292	–	0.4267	15.823	14.246 \pm 0.16%	90.0	8.67]
[1IB	303	GA-1550 biotite	12.2	5.577	–	0.4278	15.962	14.301 \pm 0.17%	89.6	11.59]
[2IA	302	77-600 Hbl	15.0	24.717	–	0.7842	72.203	64.956 \pm 0.24%	89.9	0.50]
[2IB	309	77-600 Hbl	17.4	22.771	–	0.7780	71.434	64.758 \pm 0.20%	90.6	0.73]
[2OA-1	307	MMhb-1 Hbl	31.2	3.128	–	0.2673	86.207	85.266 \pm 0.19%	98.9	2.01]
[2OA-2	308	MMhb-1 Hbl	31.2	3.617	–	0.2647	85.944	84.860 \pm 0.88%	98.7	3.33]
[2OB-1	305	MMhb-1 Hbl	25.8	3.170	–	0.2654	86.274	85.323 \pm 0.15%	98.9	1.72]
[2OB-2	306	MMhb-1 Hbl	25.8	3.535	–	0.2647	86.164	85.105 \pm 0.15%	98.8	2.86]
3IA-1	–	MMhb-1 Hbl	20.6	2.240	2543	–	86.62	86.413 \pm 0.29%	99.5	1.33
3IA-2	–	MMhb-1 Hbl	20.6	2.938	3340	–	86.59	86.327 \pm 0.19%	94.4	2.25
3IB-1	–	MMhb-1 Hbl	24.8	2.928	2559	–	86.58	86.174 \pm 0.19%	99.3	1.28
3IB-2	–	MMhb-1 Hbl	24.8	3.249	2558	–	86.67	86.167 \pm 0.16%	99.2	2.14
3OA-1	–	GA-1550 biotite	21.3	4.051	10.64	–	15.59	14.364 \pm 0.18%	92.2	7.13
3OA-2	–	GA-1550 biotite	21.3	4.053	10.03	–	15.59	14.369 \pm 0.18%	92.2	11.76
3OB-1	–	GA-1550 biotite	24.9	4.124	11.90	–	15.59	14.346 \pm 0.16%	92.0	8.38
3OB-2	–	GA-1550 biotite	24.9	4.102	11.84	–	15.59	14.354 \pm 0.15%	92.1	13.81
3OC-1	–	GA-1550 biotite	25.5	4.258	10.95	–	15.65	14.367 \pm 0.19%	91.8	7.83
3OC-2	099	GA-1550 biotite	25.5	4.257	11.34	–	15.68	14.397 \pm 0.12%	91.8	12.86
4IA	128	77-600 Hbl	15.8	49.276	14,114	0.6792	78.81	66.423 \pm 0.29%	83.3	0.59
4IB	126	77-600 Hbl	19.4	21.748	14,000	0.6827	71.12	66.851 \pm 0.25%	93.0	0.84
5IA	–	GA-1550 biotite	8.0	4.317	17.22	–	15.70	14.400 \pm 0.16%	91.7	7.00
5IB	–	GA-1550 biotite	11.1	4.207	8.336	–	15.71	14.446 \pm 0.18%	91.9	9.38
5OA-1	–	LP-6 biotite	23.7	7.088	6.505	–	20.90	18.782 \pm 0.25%	89.9	8.35
5OA-2	–	LP-6 biotite	23.7	7.193	6.150	–	20.91	18.764 \pm 0.19%	89.7	13.39
5OB-1	–	LP-6 biotite	25.1	7.249	6.600	–	20.92	18.755 \pm 0.17%	89.6	8.35
5OB-2	100	LP-6 biotite	25.1	7.249	6.725	–	20.96	18.791 \pm 0.07%	89.7	13.65
6IA	119	MMhb-1 Hbl	16.4	2.666	2530	0.2555	85.98	85.646 \pm 0.13%	99.4	2.92
6IB-1	120	MMhb-1 Hbl	22.8	3.398	2545	0.2602	86.04	85.489 \pm 0.15%	99.2	1.50
6IB-2	121	MMhb-1 Hbl	22.8	2.880	2548	0.2597	86.05	85.650 \pm 0.12%	99.3	2.50
6OA-1	–	77-600 Hbl	28.9	15.48	13,959	–	69.10	66.673 \pm 0.43%	95.4	0.42
6OA-2	–	77-600 Hbl	28.9	17.05	13,947	–	69.22	66.320 \pm 0.24%	94.8	0.73
6OB	125	77-600 Hbl	24.0	18.157	14,211	0.5095	69.52	66.339 \pm 0.15%	94.4	1.01
6OC	122	77-600 Hbl	26.9	17.479	14,236	0.5091	69.31	66.329 \pm 0.15%	94.6	1.16
7IA	136	GA-1550 biotite	9.2	4.592	13.33	0.2933	15.57	14.191 \pm 0.18%	91.1	7.21
7IB	129	GA-1550 biotite	11.8	4.521	8.565	0.2952	15.62	14.257 \pm 0.29%	91.3	8.16
7OA-1	018	MMhb-1 Hbl	27.0	3.008	2554	0.2214	85.40	84.968 \pm 0.13%	99.3	1.86
7OA-2	019	MMhb-1 Hbl	27.0	2.859	2554	0.2212	85.29	84.900 \pm 0.15%	99.3	3.01
7OB-1	016	MMhb-1 Hbl	30.4	3.145	2534	0.2209	85.13	84.650 \pm 0.18%	99.2	1.95
7OB-2	017	MMhb-1 Hbl	30.4	2.917	2553	0.2194	85.62	85.211 \pm 0.14%	99.3	3.14
7OC-1	014	MMhb-1 Hbl	26.0	2.096	2539	0.2220	85.45	85.279 \pm 0.13%	99.6	1.73
7OC-2	015	MMhb-1 Hbl	26.0	2.838	2546	0.2200	85.35	84.966 \pm 0.15%	99.3	2.89
7OD-1	011	MMhb-1 Hbl	26.1	2.262	2544	0.2205	85.51	85.292 \pm 0.15%	99.5	1.81
7OD-2	012	MMhb-1 Hbl	26.1	2.715	2535	0.2210	85.31	84.957 \pm 0.15%	99.4	2.95
8IA	115	GA-1550 biotite	8.4	4.099	17.58	0.2607	15.321	14.085 \pm 0.13%	91.9	7.80
8IB	114	GA-1550 biotite	9.9	4.801	5.561	0.2621	15.487	14.043 \pm 0.14%	90.7	9.07

(continued on next page)

Appendix (continued)

Position	File number	Mineral	Mass [mg]	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-13} mol]
8OA	118	77-600 Hbl	28.3	21.402	14,117	0.4332	69.94	$65.779 \pm 0.17\%$	93.0	1.15
8OB	117	77-600 Hbl	23.5	23.045	13,943	0.4298	70.05	$65.371 \pm 0.24\%$	92.3	1.03
8OC	116	77-600 Hbl	25.9	17.405	14,158	0.4328	68.41	$65.437 \pm 0.22\%$	94.6	1.15

$^{40}\text{Ar}^*$: radiogenic argon. Irradiation in X33 or X34 position in HIFAR reactor for 8 days, with inversion at 4 days. Cadmium shielding 0.2 mm thick was used. Samples were located in machined Al cans. Each can has an inner (I) can located centrally within the outer (O) can.

Correction factors: $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.50 \times 10^{-4}$; $(^{37}\text{Ar}/^{39}\text{Ar})_{\text{Ca}} = 7.86 \times 10^{-4}$; and $(^{40}\text{Ar}/^{39}\text{Ar})_K = 0.0264$.

Samples were fused in resistance furnace. Isotopic analysis of purified argon was done in the VG1200 mass spectrometer, utilizing a Faraday cup. Sensitivity was about 3.3×10^{-15} mol mV $^{-1}$.

Discrimination factor: 0.99204–0.99306 ($\pm 0.1\%$) per mass unit. Results shown in square brackets are not utilized because of technical problems in the measurements (mainly ^{37}Ar data not collected).

Analytical data for total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ measurement of mineral samples used as fluence monitors (irradiation canister J791)

Position	File number	Mineral	Mass [mg]	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-13} mol]
11A	342	GA-1550 biotite	10.5	6.160	9.425	0.6269	17.226	$15.381 \pm 0.14\%$	89.3	9.15
11B	338	GA-1550 biotite	14.8	5.707	10.085	0.6251	16.951	$15.240 \pm 0.15\%$	89.9	12.49
21A-1	333	MMhb-1 Hbl	17.8	2.961	2532.0	0.4819	93.786	$93.378 \pm 0.14\%$	99.4	1.08
21A-2	334	MMhb-1 Hbl	17.8	4.273	2556.6	0.4831	93.798	$93.006 \pm 0.25\%$	99.0	1.82
[21B-1	331	MMhb-1 Hbl	20.8	4.157	2563.5	0.4790	94.198	$93.443 \pm 0.69\%$	99.0	1.26]
21B-2	332	MMhb-1 Hbl	20.8	4.177	2577.2	0.4840	93.749	$92.990 \pm 0.17\%$	99.0	2.14
20A-1	329	LP-6 biotite	16.0	9.357	5.779	0.2004	23.332	$20.541 \pm 0.17\%$	88.0	5.20
20A-2	330	LP-6 biotite	16.0	9.510	5.678	0.2013	23.396	$20.560 \pm 0.15\%$	87.9	8.66
20B-1	327	LP-6 biotite	19.4	9.520	2.415	0.2010	23.230	$20.391 \pm 0.16\%$	87.8	6.10
20B-2	328	LP-6 biotite	19.4	9.432	6.066	0.2006	23.267	$20.454 \pm 0.16\%$	87.9	10.19
20C-1	325	LP-6 biotite	28.6	9.268	5.191	0.2011	23.193	$20.429 \pm 0.15\%$	88.1	9.20
20C-2	326	LP-6 biotite	28.6	9.273	4.634	0.2037	23.147	$20.381 \pm 0.24\%$	88.0	15.08
31A	324	92-176 Sanidine	14.2	0.1651	8.405	0.1819	4.421	$4.347 \pm 0.14\%$	98.3	16.51
31B	323	92-176 Sanidine	20.2	0.1733	8.166	0.1826	4.414	$4.338 \pm 0.12\%$	98.3	23.47
30A-1	322	GA-1550 biotite	23.5	5.240	20.304	0.5123	17.037	$15.465 \pm 0.15\%$	90.8	12.40
30A-2	321	GA-1550 biotite	23.5	5.298	20.637	0.5132	17.138	$15.549 \pm 0.26\%$	90.7	7.44
30B-1	319	GA-1550 biotite	23.6	5.035	28.090	0.5151	17.056	$15.546 \pm 0.19\%$	91.1	7.55
30B-2	320	GA-1550 biotite	23.6	5.070	29.825	0.5145	17.068	$15.547 \pm 0.16\%$	91.1	12.50
30C-1	317	GA-1550 biotite	25.0	5.289	12.168	0.5136	17.104	$15.517 \pm 0.21\%$	90.7	7.50
30C-2	318	GA-1550 biotite	25.0	5.290	12.807	0.5137	17.123	$15.536 \pm 0.15\%$	90.7	12.43
41A	311	92-176 Sanidine	16.7	0.1431	1.997	0.1807	4.404	$4.335 \pm 0.19\%$	98.5	16.79
41B	310	92-176 Sanidine	20.7	0.1700	1.238	0.1804	4.392	$4.316 \pm 0.13\%$	98.3	23.09
40A	316	MMhb-1 Hbl	17.1	3.863	2530.2	0.4255	93.572	$92.896 \pm 0.27\%$	99.1	2.47
40B-1	314	MMhb-1 Hbl	24.6	3.930	2550.8	0.4254	93.944	$93.253 \pm 0.15\%$	99.1	1.50
40B-2	315	MMhb-1 Hbl	24.6	3.544	2551.8	0.4229	94.183	$93.607 \pm 0.16\%$	99.2	2.46
[40C-1	312	MMhb-1 Hbl	22.3	3.781	–	0.4255	94.086	$92.949 \pm 0.19\%$	98.8	1.39]
40C-2	313	MMhb-1 Hbl	22.3	4.531	2554.3	0.4199	93.970	$93.102 \pm 0.15\%$	98.9	2.33
51A	337	GA-1550 biotite	9.6	5.695	14.233	0.6113	17.129	$15.421 \pm 0.16\%$	90.0	7.80
51B	336	GA-1550 biotite	10.0	5.554	23.067	0.6134	17.161	$15.496 \pm 0.16\%$	90.3	8.05
61A	335	GA-1550 biotite	9.8	5.444	20.231	0.6363	16.914	$15.281 \pm 0.16\%$	90.3	8.60
61B	341	GA-1550 biotite	9.9	5.366	7.109	0.6334	16.937	$15.326 \pm 0.14\%$	90.5	8.66
60A	340	92-176 Sanidine	18.4	0.1552	8.784	0.1845	4.354	$4.283 \pm 0.11\%$	98.4	21.99
60B	339	92-176 Sanidine	28.0	0.1038	8.386	0.1838	4.345	$4.288 \pm 0.16\%$	98.7	32.03
60C	344	92-176 Sanidine	23.3	0.1425	7.567	0.1838	4.349	$4.281 \pm 0.12\%$	98.4	26.45
[71A	343	LP-6 biotite	7.6	15.389	6.216	0.2006	24.214	$19.641 \pm 0.18\%$	81.1	6.10]
[71B	347	LP-6 biotite	10.4	11.994	4.355	0.1999	23.705	$20.135 \pm 0.17\%$	84.9	9.28]

Appendix (continued)

Position	File number	Mineral	Mass [mg]	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-13} mol]
7OA	346	92-176 Sanidine	16.4	0.1770	7.132	0.1827	4.335	$4.258 \pm 0.14\%$	98.2	18.03
7OB	345	92-176 Sanidine	25.2	0.1403	8.098	0.1826	4.317	$4.250 \pm 0.13\%$	98.4	29.79
[7OC	299	92-176 Sanidine	11.2	0.6356	–	0.1829	4.500	$4.287 \pm 0.16\%$	95.3	[13.04]
[8IA	300	GA-1550 biotite	11.4	5.640	–	0.5592	16.760	$15.073 \pm 0.15\%$	89.9	[9.64]
[8IB	301	GA-1550 biotite	8.8	5.566	–	0.5670	16.786	$15.121 \pm 0.36\%$	90.1	[7.65]

$^{40}\text{Ar}^*$: radiogenic argon. Irradiation in X33 or X34 position in HIFAR reactor for 8 days, with inversion at 4 days. Cadmium shielding 0.2 mm thick was used. Samples were located in machined Al cans. Each can has an inner (I) can located centrally within the outer (O) can.

Correction factors: ($^{36}\text{Ar}/^{37}\text{Ar}$)_{Ca} = 3.50×10^{-4} ; ($^{37}\text{Ar}/^{39}\text{Ar}$)_{Ca} = 7.86×10^{-4} ; and ($^{40}\text{Ar}/^{39}\text{Ar}$)_K = 0.0264.

Samples fused in resistance furnace. Isotopic analysis of purified argon was done in the VG1200 mass spectrometer, utilizing a Faraday cup. Sensitivity was about 3.3×10^{-15} mol mV⁻¹.

Discrimination factor: $0.99204 \pm 0.1\%$ per mass unit. Results shown in square brackets are not utilized because of technical problems in the measurements. For 92-176 sanidine results 4IA and 4IB, mass 37 was not properly measured, but the effect on the calculated $^{40}\text{Ar}^*/^{39}\text{Ar}_K$ was less than 0.1%.

Analytical data for total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ analysis of mineral samples used as fluence monitors; measurements on single or multiple crystals, fused by heating with a laser beam (silica tube I in irradiation canister L112)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV] (%)	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
112a	200	GA-1550 biotite	2	7.519	5.105	0.3582	15.185	12.938 ± 0.40	85.2	4.10
112b	201	GA-1550 biotite	2	7.472	18.374	0.3586	15.251	13.019 ± 0.32	85.4	2.49
113a	310	GA-1550 biotite	1	11.637	2.077	0.3662	16.515	13.050 ± 0.68	79.0	2.43
113b	311	GA-1550 biotite	1	11.181	8.995	0.3683	16.420	13.090 ± 0.43	79.7	3.98
114a	312	GA-1550 biotite	1	11.738	116.57	0.3465	16.885	13.403 ± 0.85	79.4	1.96
114b	313	GA-1550 biotite	1	11.257	110.16	0.3472	16.783	13.443 ± 0.82	80.1	1.72
115a	315	GA-1550 biotite	1	13.949	9.485	0.3507	17.320	13.173 ± 0.57	76.1	4.94
115b	316	GA-1550 biotite	1	13.890	8.576	0.3519	17.259	13.129 ± 0.40	76.1	4.31
211a	258	92-176 Sanidine	1	0.555	8.672	0.1571	3.844	3.654 ± 0.30	95.1	17.9
211b	259	92-176 Sanidine	1	0.640	10.155	0.1571	3.864	3.649 ± 0.25	94.4	12.7
212a	260	92-176 Sanidine	2	1.118	7.187	0.1590	4.106	3.750 ± 0.39	91.3	13.5
212b	261	92-176 Sanidine	2	1.678	8.019	0.1595	4.280	3.759 ± 0.33	87.8	13.7
213a	262	92-176 Sanidine	3	0.168	6.492	0.1567	3.724	3.648 ± 0.30	98.0	22.8
213b	263	92-176 Sanidine	3	0.239	6.425	0.1567	3.726	3.630 ± 0.34	97.4	14.9
214a	323	92-176 Sanidine	1	0.180	8.910	0.1569	3.726	3.647 ± 0.23	97.9	14.1
214b	324	92-176 Sanidine	1	1.268	11.263	0.1566	4.051	3.651 ± 0.30	90.1	10.0
215a	326	92-176 Sanidine	1	0.106	8.172	0.1571	3.738	3.681 ± 0.47	98.5	25.3
215b	327	92-176 Sanidine	1	0.189	12.008	0.1568	3.751	3.670 ± 0.25	97.8	8.9
311a	214	GA-1550 biotite	2	13.470	3.705	0.3576	16.835	12.828 ± 0.58	76.2	3.54
311b	215	GA-1550 biotite	2	13.464	8.814	0.3580	16.794	12.790 ± 0.41	76.2	2.12
312a	218	GA-1550 biotite	2	8.498	5.286	0.3544	15.389	12.853 ± 0.33	83.5	5.77
312b	219	GA-1550 biotite	2	8.398	5.080	0.3548	15.394	12.887 ± 0.36	83.7	3.43
411a	265	MMhb-1 Hbl	5	5.943	2640	0.3164	77.37	76.02 ± 0.44	98.0	0.98
411b	266	MMhb-1 Hbl	5	5.431	2634	0.3177	77.39	76.19 ± 0.46	98.2	0.96
411c	267	MMhb-1 Hbl	5	6.513	2712	0.3208	77.20	75.69 ± 0.30	97.8	0.67
412a	269	MMhb-1 Hbl	9	5.060	2434	0.3115	77.07	75.94 ± 0.44	98.4	0.87
412b	270	MMhb-1 Hbl	9	4.864	2463	0.3086	77.41	76.35 ± 0.47	98.4	0.85
412c	271	MMhb-1 Hbl	9	4.772	2413	0.3140	77.73	76.69 ± 0.50	98.5	0.59
413a	273	MMhb-1 Hbl	1	5.154	2765	0.3329	76.75	75.66 ± 0.54	98.4	0.68
414a	274	MMhb-1 Hbl	1	2.877	2426	0.2395	75.39	74.91 ± 0.37	99.2	0.83

(continued on next page)

Appendix (continued)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV](%)	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
415a	276	MMhb-1 Hbl	10	5.186	2512	0.3075	77.03	75.88 \pm 0.94	98.3	0.74
415b	277	MMhb-1 Hbl	10	5.924	2565	0.3067	77.43	76.07 \pm 0.65	98.0	0.73
415c	278	MMhb-1 Hbl	10	6.545	2630	0.3102	78.28	76.75 \pm 0.50	97.8	0.50
416a	317	MMhb-1 Hbl	1	6.554	2666	0.3003	76.43	74.90 \pm 0.57	97.8	0.42
417a	318	MMhb-1 Hbl	1	3.568	2892	0.3319	76.95	76.34 \pm 0.36	99.0	0.72
418a	319	MMhb-1 Hbl	1	3.781	2457	0.2558	77.07	76.33 \pm 0.35	98.8	0.64
511a	234	GA-1550 biotite	2	6.833	6.831	0.3051	14.778	12.733 \pm 0.33	86.2	5.80
511b	235	GA-1550 biotite	2	7.099	6.938	0.3018	14.825	12.701 \pm 0.36	85.7	3.48
512a	236	GA-1550 biotite	2	5.394	2.943	0.3450	14.328	12.708 \pm 0.41	88.7	5.90
512b	237	GA-1550 biotite	2	5.417	5.689	0.3499	14.409	12.782 \pm 0.39	88.7	3.58
612b	332	92-176 Sanidine	1	6.007	14.375	0.1562	5.369	3.571 \pm 0.57	66.5	9.56
613a	333	92-176 Sanidine	1	0.386	14.376	0.1556	3.767	3.538 \pm 0.32	96.2	16.1
613b	334	92-176 Sanidine	1	0.319	12.951	0.1555	3.668	3.549 \pm 0.31	96.8	16.1
711	744	GA-1550 biotite	~ 70	7.387	33.548	0.3739	14.948	12.742 \pm 0.22	85.2	416

$^{40}\text{Ar}^*$: radiogenic argon. Irradiation in X33 or X34 position in HIFAR reactor for 192 h, with inversion of the irradiation can at 48, 96, and 144 h. Cadmium shielding 0.2 mm thick was used. Samples located in glass tube I (3 mm internal diameter) with samples stacked longitudinally, each occupying ~ 3 mm height within the tube.

Correction factors: $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.50 \times 10^{-4}$; $(^{37}\text{Ar}/^{39}\text{Ar})_{\text{Ca}} = 7.86 \times 10^{-4}$; and $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}} = 0.0264$.

Samples fused using argon ion laser beam, except for file number 744 for which the sample was fused using a resistance furnace. Isotopic analyses were done on purified argon in the VG3600 mass spectrometer, utilizing a Daly detector. Sensitivity was about 1.3×10^{-17} mol mV^{-1} .

Discrimination factor: $1.00591 \pm 0.16\%$ per mass unit, based on repeated air argon measurements made over the 6-week analysis period. Run number 744 on GA-1550 biotite was measured on the Faraday collector of the VG1200 mass spectrometer with a sensitivity of about 3.3×10^{-15} mol mV^{-1} and a discrimination factor of $0.9918 \pm 0.10\%$ per mass unit (key to position, e.g., 311a—level 3 in tube I, aliquot 1 of sample, measured on equilibrated gas split a).

Analytical data for total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ analysis of mineral samples used as fluence monitors; measurements on single or multiple crystals, fused by heating with a laser beam (silica tube II in irradiation canister L112)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
1111a	204	GA-1550 biotite	2	5.693	31.03	0.3444	14.708	13.003 \pm 0.36%	88.4	3.33
1111b	205	GA-1550 biotite	2	5.465	33.92	0.3459	14.798	13.160 \pm 0.42%	88.9	3.27
1112a	206	GA-1550 biotite	2	9.857	1.025	0.3536	15.971	13.032 \pm 0.53%	81.6	4.09
1112b	207	GA-1550 biotite	2	9.990	3.442	0.3536	15.997	13.019 \pm 0.36%	81.4	2.49
3111a	220	GA-1550 biotite	2	5.137	7.349	0.3306	14.812	13.268 \pm 0.36%	89.6	4.25
3111b	221	GA-1550 biotite	2	5.304	12.657	0.3303	14.868	13.275 \pm 0.30%	89.3	2.56
3111c	222	GA-1550 biotite	2	5.033	5.674	0.3307	14.882	13.369 \pm 0.21%	89.8	4.53
3112a	224	GA-1550 biotite	2	5.475	8.847	0.3454	14.954	13.311 \pm 0.37%	89.0	3.81
3112b	225	GA-1550 biotite	2	5.220	12.264	0.3436	14.991	13.423 \pm 0.46%	89.5	2.31
5111a	239	GA-1550 biotite	2	6.655	17.677	0.3807	15.508	13.517 \pm 0.49%	87.2	4.71
5111b	240	GA-1550 biotite	2	6.798	17.156	0.3772	15.528	13.495 \pm 0.39%	86.9	4.68
5112a	241	GA-1550 biotite	2	6.314	3.847	0.3701	15.425	13.533 \pm 0.37%	87.7	5.06
5112b	242	GA-1550 biotite	2	6.199	1.530	0.3728	15.455	13.597 \pm 0.26%	88.0	5.57
5113a	244	GA-1550 biotite	1	9.275	2.100	0.3524	16.253	13.486 \pm 0.31%	83.0	4.87
5113b	245	GA-1550 biotite	1	9.067	1.121	0.3522	16.213	13.508 \pm 0.36%	83.3	4.80
6111a	343	92-176 Sanidine	1	0.0959	3.040	0.1580	3.825	3.770 \pm 0.21%	98.6	11.58
6112a	344	92-176 Sanidine	1	0.1538	9.593	0.1576	3.831	3.760 \pm 0.24%	98.2	11.96
6113a	345	92-176 Sanidine	1	0.0704	9.153	0.1572	3.823	3.777 \pm 0.27%	98.8	13.82

Appendix (continued)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
6II4a	346	92-176 Sanidine	1	0.0369	5.466	0.1577	3.844	$3.807 \pm 0.47\%$	99.0	17.24
6II4b	347	92-176 Sanidine	1	0.0977	9.158	0.1578	3.837	$3.782 \pm 0.28\%$	98.6	12.13
7III	745	GA-1550 biotite	~ 60	6.871	3.381	0.3792	15.579	$13.522 \pm 0.20\%$	86.8	750.9

$^{40}\text{Ar}^*$: radiogenic argon. Irradiation in X33 or X34 position in HIFAR reactor for 192 h, with inversion of the irradiation can at 48, 96, and 144 h. Cadmium shielding 0.2 mm thick was used. Samples located in silica glass tube II (3 mm internal diameter) with samples stacked longitudinally, each occupying ~ 3 mm height within the tube.

Correction factors: $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.50 \times 10^{-4}$; $(^{37}\text{Ar}/^{39}\text{Ar})_{\text{Ca}} = 7.86 \times 10^{-4}$; and $(^{40}\text{Ar}/^{39}\text{Ar})_K = 0.0264$.

Samples fused using argon ion laser beam, except for file number 745 for which the sample was fused using a resistance furnace. Isotopic analyses were done on purified argon in the VG3600 mass spectrometer, utilizing a Daly detector. Sensitivity was about 1.3×10^{-17} mol mV $^{-1}$.

Discrimination factor: 1.00591 ± 0.161 per mass unit, based on repeated air argon measurements made over the 6-week analysis period. Run number 746 on GA-1550 biotite was measured on the Faraday collector of the VG1200 mass spectrometer with a sensitivity of about 3.3×10^{-15} mol mV $^{-1}$ and a discrimination factor of $0.9918 \pm 0.10\%$ per mass unit (key to position, e.g., 5IIIa—level 3 in tube II, aliquot 1 of sample, measured on equilibrated gas split a).

Analytical data for total fusion $^{40}\text{Ar}/^{39}\text{Ar}$ analysis of mineral samples used as fluence monitors; measurements on single or multiple crystals, fused by heating with a laser beam (silica tube III in irradiation canister L112)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [\pm CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
1III1a	209	GA-1550 biotite	2	8.277	2.142	0.3921	15.482	$13.010 \pm 0.43\%$	84.0	4.43
1III1b	210	GA-1550 biotite	2	8.754	4.218	0.3938	15.483	$12.870 \pm 0.45\%$	83.1	2.67
1III2a	211	GA-1550 biotite	2	6.432	3.275	0.4112	14.958	$13.031 \pm 0.38\%$	87.1	6.54
1III2b	212	GA-1550 biotite	2	6.751	6.462	0.4110	15.077	$13.056 \pm 0.32\%$	86.6	3.98
2III1a	279	77-600 Hbl	20	19.475	15518	0.7349	64.58	$61.15 \pm 0.45\%$	93.5	0.72
2III2a	280	77-600 Hbl	20	22.476	13840	0.6695	65.18	$60.61 \pm 0.54\%$	92.0	0.61
2III3a	281	77-600 Hbl	21	17.764	13670	0.7168	63.90	$60.69 \pm 0.67\%$	94.0	0.59
2III4a	288	77-600 Hbl	~ 50	19.419	13743	0.7005	64.21	$60.52 \pm 0.32\%$	93.2	1.01
2III4b	289	77-600 Hbl	~ 50	19.676	14051	0.7046	64.69	$60.98 \pm 0.49\%$	93.2	1.04
2III4c	290	77-600 Hbl	~ 50	19.291	13710	0.6959	63.83	$60.17 \pm 0.53\%$	93.2	0.72
2III5a	291	77-600 Hbl	7	36.782	14550	0.8045	67.22	$58.49 \pm 0.97\%$	86.0	0.16
2III6a	293	77-600 Hbl	4	26.031	14640	0.6493	63.42	$57.88 \pm 1.22\%$	90.2	0.09
2III7a	294	77-600 Hbl	3	42.132	13597	0.6797	68.04	$57.58 \pm 2.08\%$	83.7	0.06
2III8a	295	77-600 Hbl	~ 50	18.300	12407	0.6152	65.30	$61.74 \pm 0.51\%$	93.6	0.96
2III8b	296	77-600 Hbl	~ 50	17.134	12322	0.6122	65.33	$62.12 \pm 0.33\%$	94.2	1.00
2III8c	297	77-600 Hbl	~ 50	18.383	12422	0.6705	65.05	$61.48 \pm 0.41\%$	93.6	0.69
2III9a	306	77-600 Hbl	~ 12	26.968	14736	0.7389	67.88	$62.13 \pm 0.79\%$	90.5	0.25
2III10a	307	77-600 Hbl	~ 17	26.823	15950	0.6950	66.32	$60.78 \pm 0.67\%$	90.5	0.29
2III11a	308	77-600 Hbl	~ 12	20.845	13103	0.7259	64.92	$60.72 \pm 0.73\%$	92.6	0.32
3III1a	226	GA-1550 biotite	2	7.199	2.053	0.3564	15.279	$13.125 \pm 0.40\%$	85.9	5.25
3III1b	227	GA-1550 biotite	2	7.537	5.533	0.3561	15.285	$13.032 \pm 0.35\%$	85.3	3.16
3III1c	228	GA-1550 biotite	2	7.260	2.918	0.3574	15.325	$13.152 \pm 0.28\%$	85.8	5.63
3III2a	230	GA-1550 biotite	2	4.548	14.416	0.3530	14.492	$13.123 \pm 0.43\%$	90.6	4.03
3III2b	231	GA-1550 biotite	2	4.308	18.556	0.3534	14.400	$13.103 \pm 0.34\%$	91.0	3.96
3III2c	232	GA-1550 biotite	2	4.757	24.845	0.3531	14.421	$12.992 \pm 0.40\%$	90.1	2.74
4III1a	251	LP-6 biotite	8	17.738	9.594	0.1685	23.54	$18.27 \pm 0.44\%$	77.6	4.34
4III1b	252	LP-6 biotite	8	18.105	7.992	0.1685	23.17	$17.79 \pm 0.41\%$	76.8	4.33
4III1c	253	LP-6 biotite	8	18.153	4.668	0.1690	23.42	$18.03 \pm 0.47\%$	77.0	2.99
4III2a	254	LP-6 biotite	7	28.573	32.921	0.1719	25.97	$17.50 \pm 1.28\%$	67.4	1.15
4III2b	255	LP-6 biotite	7	27.225	12.198	0.1698	25.85	$17.78 \pm 0.82\%$	68.8	1.84
4III2c	256	LP-6 biotite	7	27.293	16.869	0.1698	25.80	$17.71 \pm 0.78\%$	68.6	2.00

(continued on next page)

Appendix (continued)

Position	File number	Mineral	Number of crystals	$^{36}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{37}\text{Ar}/^{39}\text{Ar}$ [$\times 10^{-3}$]	$^{38}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}/^{39}\text{Ar}$	$^{40}\text{Ar}^*/^{39}\text{Ar}_K$ [± 1 CV]	$^{40}\text{Ar}^*$ [%]	^{39}Ar [10^{-15} mol]
5III1a	246	GA-1550 biotite	2	7.261	–	0.3773	15.356	$13.184 \pm 0.35\%$	85.9	4.78
5III1b	247	GA-1550 biotite	2	7.195	1.804	0.3766	15.370	$13.218 \pm 0.34\%$	86.0	4.78
5III2a	248	GA-1550 biotite	1	5.261	14.505	0.3375	14.660	$13.081 \pm 0.56\%$	89.2	1.30
5III2b	249	GA-1550 biotite	1	5.547	–	0.3354	14.661	$12.996 \pm 0.36\%$	88.6	2.19
6III1a	248	92-176 Sanidine	1	0.0374	8.890	0.1567	3.748	$3.711 \pm 0.19\%$	99.0	15.6
6III2a	349	92-176 Sanidine	1	0.9331	18.397	0.1575	4.005	$3.705 \pm 0.89\%$	92.5	1.87
6III3a	350	92-176 Sanidine	1	0.1485	9.221	0.1562	3.767	$3.698 \pm 0.22\%$	98.2	15.6
7III1a	746	GA-1550 biotite	~ 50	7.720	14.008	0.3773	15.518	$13.212 \pm 0.22\%$	85.1	534

$^{40}\text{Ar}^*$: radiogenic argon. Irradiation in X33 or X34 position in HIFAR reactor for 192 h, with inversion of the irradiation can at 48, 96, and 144 h. Cadmium shielding 0.2 mm thick was used. Samples located in glass tube III (3 mm internal diameter) with samples stacked longitudinally, each occupying ~ 3 mm height within the tube.

Correction factors: $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}} = 3.50 \times 10^{-4}$; $(^{37}\text{Ar}/^{39}\text{Ar})_{\text{Ca}} = 7.86 \times 10^{-4}$; and $(^{40}\text{Ar}/^{39}\text{Ar})_K = 0.0264$.

Samples fused using argon ion laser beam, except for file number 746 for which the sample was fused using a resistance furnace. Isotopic analyses were done on purified argon in the VG3600 mass spectrometer, utilizing a Daly detector. Sensitivity was about 1.3×10^{-17} mol mV^{-1} .

Discrimination factor: $1.00591 \pm 0.16\%$ per mass unit, based on repeated air argon measurements made over the 6-week analysis period. Run number 746 on GA-1550 biotite was measured on the Faraday collector of the VG1200 mass spectrometer with a sensitivity of about 3.3×10^{-15} mol mV^{-1} and a discrimination factor of $0.9918 \pm 0.10\%$ per mass unit (key to position, e.g., 2III1a—level 2 in tube III, aliquot 1 of sample, measured on equilibrated gas split a).

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