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Professional Paper 1621

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By Marvin A. Lanphere and G. Brent Dalrymple

Professional Paper 1621

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By Marvin A. Lanphere¹ and G. Brent Dalrymple²

ABSTRACT

The accuracy of the ages of primary mineral standards used as fluence monitors is the limiting factor in calculating accurate $^{40}\text{Ar}/^{39}\text{Ar}$ ages. A primary mineral standard can be dated only by measuring its K and radiogenic-Ar contents, using calibrated ^{38}Ar tracers. We describe the analytical procedure for first-principles calibrations of ^{38}Ar tracers in the U.S. Geological Survey (USGS) laboratory in Menlo Park, Calif. Two laboratories in Japan that have made first-principles calibrations of ^{38}Ar tracers report good agreement with the values obtained in the USGS laboratory for the radiogenic- ^{40}Ar content of the SORI biotite standard. The ages of fluence monitors determined in the USGS laboratory and those reported in the literature differ by nearly 2 percent. However, the radiogenic- ^{40}Ar content of the primary mineral standard on which these ages are based has been measured by first-principles Ar calibration in only one laboratory, and that value has never been verified by first-principles measurement in other laboratories.

INTRODUCTION

The mineral standards used in geochronology must be calibrated by using first principles; that is, the contents of radioactive parent isotope and radiogenic daughter isotope in the mineral standard must be measured directly. Once this standard has been dated from its measured isotopic contents, it then can be used for second-order calibrations of isotopic tracers or other mineral standards (fig. 1).

The $^{40}\text{Ar}/^{39}\text{Ar}$ method is a relative dating technique that is a variant of the K-Ar method. The $^{40}\text{Ar}/^{39}\text{Ar}$ method depends on using a mineral standard (also known as a monitor mineral or fluence monitor) of known age to measure the effective fast-neutron dose that an unknown sample receives during

irradiation. The age of the unknown sample is then calculated relative to the age of the standard. At present, some disagreement exists about the ages of certain commonly used mineral standards. Various authors have published papers that contain sets of data on the $^{40}\text{Ar}/^{39}\text{Ar}$ comparison of mineral standards (for example, Roddick, 1983; Hurford and Hammerschmidt, 1985; Lanphere and others, 1990; Baksi and others, 1996; Lanphere and Baadsgaard, 1997, 1998; Renne and others, 1998). Some of these papers are abstracts; complete analytical data have not yet been published. In all of these studies, the principal source of uncertainty is the value adopted for the age of the primary mineral standard against which all the others are compared. The precision of $^{40}\text{Ar}/^{39}\text{Ar}$ ages is substantially better than their accuracy, and so the accuracy of the age of the primary mineral standard is the limiting factor in calculating accurate $^{40}\text{Ar}/^{39}\text{Ar}$ ages, although it does not affect the precision of ages determined relative to that standard.

The straightforward first-principles way to date a primary mineral standard for the K-Ar dating method is to measure the K and radiogenic-Ar contents of the mineral. This primary mineral standard can then be used to calibrate secondary mineral standards by the process of intercalibration, wherein the age of a secondary mineral standard is calculated relative to the known age of the primary mineral standard. A laboratory equipped to do only $^{40}\text{Ar}/^{39}\text{Ar}$ dating cannot make K and Ar determinations and must depend on quantitative measure-

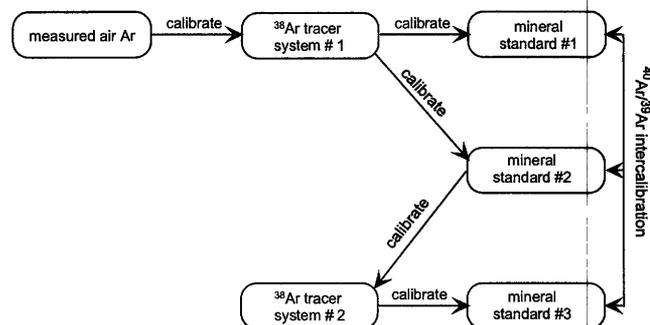


Figure 1.—Flowchart showing procedures for calibrating ^{38}Ar tracer systems and mineral standards.

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ments made in a conventional K-Ar laboratory for the age of the primary mineral standard. In the 1950's and 1960's, many of the laboratories engaged in K-Ar dating made first-principles calibrations of ^{38}Ar tracers; other laboratories depended on calibrating their ^{38}Ar tracers by using mineral standards whose radiogenic- ^{40}Ar content had been measured in some other laboratory that had made first-principles calibrations.

Other ways have been suggested to calibrate fluence monitors for $^{40}\text{Ar}/^{39}\text{Ar}$ dating. Renne and others (1994) and Hilgen and others (1997) proposed the use of the astronomically calibrated geomagnetic-polarity time scale to date mineral standards for the $^{40}\text{Ar}/^{39}\text{Ar}$ method. Renne and others (1994) pointed out that this method is independent of absolute isotopic measurements. Rial (1999) showed that the climate system of the Earth responds nonlinearly to astronomical forcing by frequency-modulating variations in insolation; he used a simple model to explain the varying durations of the ice ages. Thus, the astronomical method is a model, not a first-principles calibration, that assumes perfect synchronicity between Milankovich forcing, climate change, geomagnetism, and their geologic records—a questionable presumption at the 1- to 2-percent-confidence level. Kunk and others (1994) described a proposal to have the U.S. National Institute of Standards and Technology determine the ^{38}Ar content of an ^{38}Ar tracer system by pressure measurements; this would be a first-principles calibration, but the work has not yet been done. Renne and others (1997) determined an $^{40}\text{Ar}/^{39}\text{Ar}$ isochron age of $1,925 \pm 94$ yr on sanidine from the eruption of Mount Vesuvius in 79 C.E. This measurement would be a first-principles calibration, but the precision of only about 5 percent does not satisfy the requirement for a primary standard.

The art of first-principles ^{38}Ar tracer calibration has largely been lost. In fact, most laboratories in operation today have never done such calibrations. Many of the laboratories that made first-principles calibrations during the 1960's have been closed, and those still in existence, such as the U.S. Geological Survey (USGS) laboratory in Menlo Park, Calif., have not done them for many years. In addition, the calibration procedures were almost never described in detail. From 1964 to 1965, we made several first-principles calibrations of the USGS ^{38}Ar tracer systems; all subsequent tracer systems and mineral standards are traced directly to those first-principles calibrations. We thought it would be useful to describe the calibration procedure we used and to discuss how first-principles calibrations of Ar tracers were done there.

First-principles calibrations of such elements as K, Rb, Sr, U, and Pb are relatively straightforward, though exacting, and rely primarily on weighing, which can be done easily with high precision and accuracy. A solution containing a carefully weighed amount of a standard reference material of known isotopic composition is mixed with a solution containing an isotopic tracer, also of known isotopic composition, to determine the concentration of a given isotope

in the tracer solution. A weighed amount of the tracer solution can then be added to a solution containing a dissolved rock or mineral of unknown age to determine, by comparison with the tracer, the concentrations of the isotopes used in the age calculations.

First-principles calibrations in the K-Ar method, however, are not so straightforward. The K content can be measured by isotope dilution in the same way as for Rb or U. However, measuring the Ar content is inherently more difficult because the Ar cannot be weighed but must be measured in gaseous form at low pressure. Using the various gas laws, we can derive the relationship $n = PV/RT$, where n is the Ar content (in moles), P is the pressure, V is the volume, T is the temperature (in kelvins), and R is the universal gas constant. There are two ways to measure the Ar content of an aliquant of gas. The first way is to isolate a volume of air, whose elemental composition is known, and then remove all the other gases. This way requires that the air be dry; that is, all water vapor must be removed. This step is required because the humidity of air varies widely and is difficult to measure accurately; the measured Ar content of air, 0.934 ± 0.001 volume percent (Hodgman and others, 1969), is for dry samples. The second way is to use commercially purified Ar gas, which can be supplied with impurities of less than 10 ppm. This second way avoids the problem of variation in the humidity of air and does not require the removal of other gases. The Ar-isotopic composition can then be measured to confirm the absence of isotopic fractionation in its preparation. We chose the second way as being simpler and, in principle, more accurate.

ANALYTICAL PROCEDURE

The analytical procedure involves trapping an aliquant of commercially purified Ar gas in a McLeod gauge (Dushman, 1962) and measuring its pressure and volume; the temperature of the Ar gas is measured with an accurate Hg thermometer. Using these measurements and knowing the gas constant, R , we can calculate the Ar content of the aliquant. The McLeod gauge (fig. 2) consists of an open and a closed tube made of precision-bore capillary tubing of uniform cross section and a small reservoir, the volume of which must be accurately known. The aliquant is trapped in the closed tube, and so the volume of the closed tube must also be measured as accurately as possible. At the time of our measurements, McLeod gauges of sufficient precision and accuracy were unavailable commercially, and so we constructed one. We mapped the volume of the closed tube along its length by incrementally filling the capillary tube to various heights with Hg and weighing the Hg (table 1). The height of the Hg column was measured to within ± 0.005 mm with a cathetometer. The capillary tubing must be clean, or else Hg sticking to the wall of the tubing can interfere with the meniscus at the top of the Hg columns. This condition was checked by raising the Hg columns with

Table 1.—Volume measurements with the closed-capillary McLeod gauge.

<i>T</i> (°C)	Hg		vol/cm	Δ vol/cm	<i>h</i> (cm)
	(g)	(cm ³)			
24.25	1.4760	0.10904	0.03410	} 0.03473	3.2
24.50	2.3608	.17442	.03433		5.1
23.65	3.1380	.23180	.03443	} .03475	6.7
24.50	4.2040	.31060	.03457		.03497
25.20	5.0395	.37237	.03465	} .03500	10.70
25.55	6.4143	.47399	.03456		.03424

the system under high vacuum and confirming that the Hg columns in both the closed and open tubes were at the same level.

In principle, the measurements are straightforward, but in practice they are not so easy. The goal is to obtain an aliquant of Ar gas similar in volume to an ³⁸Ar tracer, but this procedure requires working near the low-pressure limit of a McLeod gauge. The commercially purified Ar gas was delivered in a

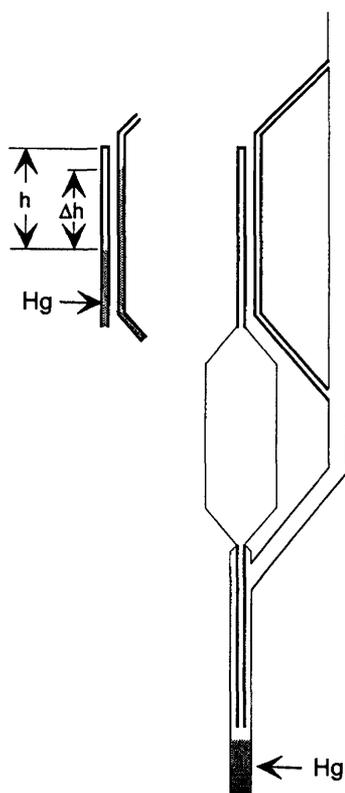


Figure 2.—McLeod gauge. Heavy lines, precision-bore capillary tubing. Right, entire gauge with Hg level (shaded) before trapping volume of Ar gas to be measured. Left, measurements made to determine pressure of volume of Ar gas trapped within gauge after Hg level is raised. *h*, distance between top of Hg column in closed tube and top of closed tube; Δh , distance between tops of two Hg columns.

1-L glass bulb with a breakseal. The manufacturer's specifications are as follows:

Minimum purity	----99.9995 percent
CO ₂	-----max 1 ppm
H ₂	-----max 1 ppm
H ₂ O	-----max 3 ppm
N ₂	-----max 2 ppm
O ₂	-----max 2 ppm
Total hydrocarbons	----max 0.5 ppm

The glass bulb of Ar gas was attached to the apparatus, and the Ar gas was expanded into a calibration system that contained the McLeod gauge, reservoirs, and various gas pipettes. The Ar gas in the glass bulb was at a pressure of 1 atm, and so a gas pipette consisting of two stopcocks connected with a length of Pyrex tubing was used to isolate a suitable volume of the Ar gas for measurement. After the Ar gas was expanded into the calibration system, a column of vacuum-distilled Hg was raised to isolate the Ar gas in the closed tube of the McLeod gauge. The Ar gas in the rest of the system was pumped away. Measurements were then made with the cathetometer of the top of the Hg columns in the closed and open tubes relative to the top of the closed tube of the McLeod gauge. A rubber bulb was used to pressurize the Hg reservoir so that the Hg columns could be moved to different positions. The measurements on a typical aliquant of Ar gas are listed in table 2; a typical calculation of the volume of Ar gas in the aliquant is listed in table 3. All errors cited in this report are standard deviations, or 1σ uncertainties.

After completing these measurements, the Hg column isolating the aliquant of Ar gas in the closed tube was lowered, and the entire aliquant was transferred into a reservoir, using activated charcoal cooled with liquid N₂. The reservoir was a 2-L Pyrex boiling flask whose volume was determined by weighing it empty and full of water. A gas pipette consisting of two 2-mm-bore mercury-seal stopcocks connected with a length of tubing of known volume was attached to the reservoir. The volume of the pipette was determined by weighing the pipette empty, filling the length of tubing with Hg, and weighing the pipette again. The volumes of reservoir and pipette are listed in table 3. Because the volumes of both the pipette and the reservoir were known, the depletion constant of the system, λ , could be calculated from the relation $\lambda = (\text{volume of pipette}) / (\text{volume of pipette} + \text{volume of reservoir})$. The reservoir-pipette system is similar to the Ar bulb tracer system described by Lanphere and Dalrymple (1966). Splits of Ar gas made with the gas pipette were mixed with ³⁸Ar tracers. The gas-tracer mixtures were analyzed with a noble-gas mass spectrometer, and from these results and knowledge of the λ value, the initial tracer quantity (T_0) of the ³⁸Ar bulb-tracer system was calculated.

RESULTS

Five splits of Ar gas from aliquants 7 and 8 were used to calibrate ³⁸Ar tracers from bulb system A. Aliquants 7 and 8

Table 2.—Pressure, volume, and temperature measurements for an aliquant of commercially purified Ar gas.

[h , distance between top of Hg column in closed tube and top of closed tube; Δh , distance between tops of two Hg columns; T , temperature; a , cross-sectional area of closed tube]

Set	h (cm)	Δh (cm)	T (°C)	$h\Delta h$ (mm ²)	a (mm ²)	$ah\Delta h$ (mm ⁴)
1	6.860	6.638	26.2	4,553.7	3.497	15,924
	6.867	6.670	26.2	4,580.3	3.497	16,017
2	6.370	7.208	26.0	4,591.5	3.475	15,955
	6.362	7.190	26.1	4,574.3	3.475	15,896
3	6.092	7.542	26.1	4,594.6	3.475	15,966
	6.115	7.555	26.1	4,619.9	3.475	16,054
4	7.310	6.240	26.2	4,561.4	3.497	15,951
	7.318	6.250	25.6	4,573.8	3.497	15,995
5	8.092	5.612	25.5	4,541.2	3.500	15,894
	8.088	5.613	25.7	4,539.8	3.500	15,889
6	8.830	5.108	25.5	4,510.4	3.500	15,786
	8.835	5.107	25.5	4,512.0	3.500	15,792
7	6.978	6.598	25.6	4,604.1	3.497	16,101
	6.990	6.570	25.7	4,592.4	3.497	16,060
8	5.694	8.040	25.5	4,578.0	3.475	15,909
	5.712	8.072	25.6	4,610.7	3.475	16,022

were separately prepared volumes of Ar gas. The performance of bulb system A over time was described by Lanphere and Dalrymple (1966). The ^{38}Ar content, T_x , of any given tracer is calculated from the relation

$$T_x = T_0 e^{-\lambda x},$$

where $\lambda = 0.826 \times 10^{-3}$ for this tracer system. The data for the five calibrations are listed in table 4.

Similar calibrations had been made earlier by the first author at the California Institute of Technology (CIT), using commercially purified Ar gas in an apparatus similar to that described above. Aliquants of Ar gas were used to calibrate ^{38}Ar tracers that, in turn, were used to measure the radiogenic- ^{40}Ar content of an intralaboratory biotite mineral standard. This biotite standard, 62ALe-1, was separated from a Jurassic tonalite in the Alaska-Aleutian Range batholith of south-central Alaska. The sample was collected along the road between Pile Bay on Lake Iliamna and Iliamna Bay on the Pacific Ocean (Reed and Lanphere, 1969). The radiogenic- ^{40}Ar content of biotite standard 62ALe-1 is 2.157×10^{-9} mol/g, based on the CIT measurements.

A total of 12 calibrations were made on ^{38}Ar tracers from the USGS laboratory's bulb system A, using the radiogenic- ^{40}Ar content of biotite standard 62ALe-1 biotite measured at CIT (table 5). The difference between the mean value of these

Table 3.—Calculation of the Ar content in aliquant 9.

Given:	Mean temperature = 25.8°C or 298.95K Mean $ah\Delta h = 15,951 \pm 83.3$ mm ⁴ $R = 62.364 \times 10^6$ mm ⁴ /mol-K Volume of reservoir = 2,179.1 cm ³ Volume of pipette = 2.6204 cm ³ (Volume of pipette)/(volume of reservoir + volume of pipette) = 1.201×10^{-3}
Calculation:	$n = ah\Delta h/RT = 0.85557 \times 10^{-6}$ mol Ar gas $^{40}\text{Ar} = 0.996 \times 0.85557 \times 10^{-6} = 0.85215 \times 10^{-6}$ mol

Table 4.—Calibration of ^{38}Ar tracers from bulb system A with splits of Ar gas.

[T_x , ^{38}Ar content of tracer; T_0 , initial tracer quantity]

Aliquant	^{40}Ar content (10^{-11} mol)	Tracer	T_x (10^{-11} mol)	T_0 (10^{-11} mol)
7-2	93.16	A-26	33.340	34.064
7-3	93.05	A-53	32.688	34.150
7-4	92.93	A-9	33.383	33.632
8-3	97.28	A-48	31.624	32.903
8-4	97.17	A-49	31.445	32.743
			Mean	33.498
			σ	.65
			σ_x	.29

Table 5.—Calibration of ^{38}Ar tracers from bulb system A, using biotite standard 62ALe-1.

[Radiogenic- ^{40}Ar content of biotite standard 62ALe-1 was measured at the California Institute of Technology, using a tracer system calibrated with commercially purified Ar gas. T_x , ^{38}Ar content of tracer; T_0 , initial tracer quantity]

Tracer	T_x (10^{-11} mol)	T_0 (10^{-11} mol)
A-2	33.424	33.479
A-12	33.136	33.466
A-13	33.149	33.507
A-24	33.081	33.743
A-35	32.884	33.848
A-50	32.356	33.720
A-77	31.915	34.009
A-124	30.680	33.989
A-160	29.352	33.508
A-184	28.785	33.511
A-221	28.041	33.659
A-222	27.855	33.462
Mean		33.658
σ		.20
σ_x		.06

^{38}Ar tracer calibrations and the mean value of the Ar gas calibrations of the USGS laboratory's bulb system A (table 4) is 0.48 percent. Note that these two sets of calibrations are tied directly to independent first-principles calibrations made in two different laboratories.

A total of 16 different bulb-tracer systems and 1 batch tracer system have been used in the USGS laboratory since the 1960's (table 6). These tracers have been used to calibrate three additional biotite standards, BS-1, SB-2, and SB-3 (tables 7-9), which were separated from the same sample of tonalite that yielded biotite standard 62ALe-1. Each of these biotite standards was treated as an independent standard mineral, and their ages were measured, not assumed. The good agreement in the ages of these standard biotites (table 10) demonstrates that we have been able to fill and refill ^{38}Ar tracer systems without propagating systematic errors. These tracers and standard biotites have been used for K-Ar and $^{40}\text{Ar}/^{39}\text{Ar}$ dating of other minerals that can potentially be used as fluence monitors. The age of a fluence monitor, not just its radiogenic- ^{40}Ar content, must be known. The K contents of biotite standards BS-1 and SB-2 were measured by isotope-dilution mass spectrometry at the U.S. National Bureau of Standards (table 11). The difference between the flame-photometer and isotope-dilution K_2O contents of biotite standard SB-2 is less than 0.05 weight percent. Intercomparisons of flame-photometer values of biotite standards BS-1, SB-2, and SB-3 were used to normalize the K content of biotite standard SB-3 to the isotope-dilution values of biotite standards BS-1 and SB-2.

Table 6.—History of calibration standards and tracer systems used in the U.S. Geological Survey laboratory in Menlo Park, Calif.

[Tracer systems A, C, D, and F had glass pipettes; all other tracer systems had metal pipettes except tracer system OSA, which was a batch tracer]

Year	Calibration standard	Tracer system
1964	Commercially purified Ar gas Biotite ¹ 62ALe-1	A
1965	Commercially purified Ar gas Biotite ¹ 62ALe-1	C
1966	Biotite ¹ 62ALe-1 Biotite BS-1	D
1967	Biotite ¹ 62ALe-1	F
1970	Biotite BS-1	G
1970	Biotite BS-1	H
1971	Biotite BS-1 Biotite SB-2	J
1971	Biotite BS-1 Biotite SB-2	K
1971	Biotite BS-1	OSA
1977	Biotite SB-2	M
1978	Biotite SB-2 Biotite SB-3	N
1978	Biotite SB-2 Biotite SB-3	P
1981	Biotite SB-2 Biotite SB-3	Q
1981	Biotite SB-3	R
1984	Biotite SB-3	S
1985	Biotite SB-3	T
1986	Biotite SB-3	U
1986	Biotite SB-3	WW

¹Radiogenic- ^{40}Ar content measured at the California Institute of Technology, using ^{38}Ar tracers calibrated with commercially purified Ar gas.

DISCUSSION

Other laboratories have made first-principles Ar calibrations and have produced data that provide comparisons with those from the USGS laboratory in Menlo, Park Calif. Shibata (1968) calibrated an ^{38}Ar batch-tracer system at the Geological Survey of Japan, using commercially purified Ar gas and a McLeod gauge. He then used the ^{38}Ar tracers to measure

Table 7.—K and radiogenic-⁴⁰Ar contents of biotite standard BS-1.

[Analysts: K₂O, L.B. Schlocker; Ar, J.C. Von Essen]

K ₂ O content (weight percent)	Radiogenic- ⁴⁰ Ar content (10 ⁻⁹ mol/g)
8.97	2.193
8.83	2.164
8.84	2.174
8.90	2.126
8.87	2.187
8.57	2.149
8.95	2.183
8.85	2.194
8.86	2.177
8.83	2.196
8.84	2.201
8.87	2.208
8.75	2.191
---	2.213
Mean = 8.841	Mean = 2.183×10 ⁻⁹
σ = 0.0984 (1.1 percent)	σ = 0.0233×10 ⁻⁹ (1.08 percent)
σ _x = 0.0273 (0.31 percent)	σ _x = 0.0063×10 ⁻⁹ (0.29 percent)

Table 8.—K and radiogenic-⁴⁰Ar contents of biotite standard SB-2.

[Analysts: K₂O, L.B. Schlocker; Ar, J.C. Von Essen]

K ₂ O content (weight percent)	Radiogenic- ⁴⁰ Ar content (10 ⁻⁹ mol/g)
9.20	2.225
9.18	2.194
9.19	2.214
9.21	2.260
9.18	2.215
9.20	2.224
9.20	2.286
9.19	2.262
9.19	2.260
9.18	2.256
9.19	2.264
9.18	2.277
9.16	---
9.20	---
9.20	---
9.16	---
Mean = 9.188	Mean = 2.245×10 ⁻⁹
σ = 0.014 (0.15 percent)	σ = 0.029×10 ⁻⁹ (1.29 percent)
σ _x = 0.0035 (0.038 percent)	σ _x = 0.0084×10 ⁻⁹ (0.37 percent)

Table 9.—K and radiogenic-⁴⁰Ar contents of biotite standard SB-3.

[Analysts: K₂O, M. Taylor; Ar, J.C. Von Essen]

K ₂ O content (weight percent)	Radiogenic- ⁴⁰ Ar content (10 ⁻⁹ mol/g)
9.03	2.221
9.07	2.209
9.01	2.200
9.01	2.243
9.02	2.207
9.03	2.211
9.02	2.205
9.02	2.219
9.02	2.204
9.00	2.210
8.99	2.218
8.98	---
9.00	---
9.01	---
9.03	---
9.01	---
9.00	---
9.01	---
Mean = 9.014	Mean = 2.213×10 ⁻⁹
σ = 0.019 (0.21 percent)	σ = 0.0011×10 ⁻⁹ (0.51 percent)
σ _x = 0.0045 (0.05 percent)	σ _x = 0.0034×10 ⁻⁹ (0.15 percent)

Table 10.—Calibration of intralaboratory biotite standards used in the U.S. Geological Survey laboratory in Menlo Park, Calif.

Biotite standard	Radiogenic- ⁴⁰ Ar calibrations	Age (Ma) ¹
BS-1	6 with D bulb tracers 8 with F bulb tracers	162.7±2.5
SB-2	2 with OSA batch tracers 2 with D bulb tracers 8 with K bulb tracers	162.1±2.1
SB-3	8 with N bulb tracers 3 with Q bulb tracers	162.9±0.9

¹λ_g=0.581×10⁻¹⁰ yr⁻¹, λ_β=4.962×10⁻¹⁰ yr⁻¹, ⁴⁰K/K=1.167×10⁻⁴ mol/mol.

Table 11.—K contents of biotite standards BS-1 and SB-2 determined by isotope-dilution mass spectrometry.

[Analysts: E.L. Gamer and L.A. Machlan. Uncertainty is estimated error limit and includes an allowance for possible effects of trace impurities in measurement of ³⁹K/⁴¹K ratio]

Biotite standard	K content	K ₂ O content
BS-1	7.3942	8.9069
	7.3937	8.9063
	Mean = 7.3940±0.0185	Mean = 8.9066±0.0223
SB-2	7.6306	9.1917
	7.6311	9.1923
	Mean = 7.6308±0.0191	Mean = 9.1920±0.0230

Table 12.—Ar analyses of the SORI biotite in different laboratories.

Laboratory	Radiogenic- ⁴⁰ Ar content (10 ⁻⁶ cm ³ /g)	Reference
Geological Survey of Japan	24.90±0.15	Uchiumi and Shibata (1980).
Okayama University, Japan	25.23±0.22	Nagao and others (1984).
	25.05±0.35	Nagao and others (1996).
U.S. Geological Survey, Menlo Park, Calif.	24.98±0.13	K. Uto (written commun., 1998).

the radiogenic-⁴⁰Ar content of a biotite standard, separated from the SORI granodiorite in Gumma Prefecture, Japan; results of the analyses are listed in table 12 (Uchiumi and Shibata, 1980). Keisuke Nagao calibrated an ³⁸Ar bulb-tracer system at Okayama University, Japan, using Ar gas separated from air, and used these tracers to measure the radiogenic-⁴⁰Ar content of the SORI biotite standard (table 12; Nagao and others, 1984, 1996). Finally, Kozo Uto of the Geological Survey of Japan analyzed the SORI biotite standard in the USGS laboratory in Menlo Park, Calif, using ³⁸Ar tracers from bulb tracer-system U (table 12). The close agreement of the results on the SORI biotite standard from all three laboratories shows that the first-principles calibrations of ³⁸Ar tracers, when done carefully, yield concordant results.

The ages of the fluence monitors used for ⁴⁰Ar/³⁹Ar dating in various laboratories differ by nearly 2 percent. Renne and others (1998) made a detailed intercalibration of six minerals used as neutron-fluence monitors. They adopted biotite standard GA1550 prepared at the Australian National University (ANU) as their primary neutron-fluence monitor, from which they determined the ages of the other fluence monitors. McDougall and Roksandic (1974) reported a K-Ar age of 97.9±0.9 Ma for biotite standard GA1550, based on a K content of 7.70±0.03 weight percent and a radiogenic-⁴⁰Ar content of (3.01±0.01)×10⁻⁵ cm³/g (at standard temperature and pressure). They stated that a set of ³⁸Ar tracers were accurately calibrated against known amounts of atmospheric Ar gas, but no details of the calibration procedure were given. The K content of biotite standard GA1550 was determined by flame photometry. Wellman (1971) gave a slightly more detailed description of the ANU calibration procedure. Capillary tubes of known volume were filled with dry air and sealed with a blob of molten glass; how the air was dried was not specified. The amount of Ar in the tubes was calculated by using the temperature and pressure of the laboratory. The air samples were mixed with tracers on an extraction line, and, after purification, the composition of the Ar mixture was measured on a mass spectrometer. In 1970, we attempted tracer calibrations by using this procedure but obtained widely varying (scattered) results. Wellman noted that this tracer calibration gave ages 1 to 2.5 percent older than previously

published Australian Cenozoic K-Ar ages, but the reason for the difference was unknown. Renne and others (1998) redetermined the K content of biotite standard GA1550 by isotope-dilution mass spectrometry and obtained a K value of 7.626±0.016 weight percent, about 1 weight percent lower than the flame-photometry value of McDougall and Roksandic (1974). Renne and others (1998) did not measure the radiogenic-⁴⁰Ar content of biotite standard GA1550. Using the new K content and the radiogenic-⁴⁰Ar content of McDougall and Roksandic (1974), Renne and others (1998) calculated an age of 98.79±0.54 Ma for biotite standard GA1550.

Multigrain samples of hornblende standard MMhb-1 were used as fluence monitors in an intercalibration experiment involving the USGS laboratories in Menlo Park, Calif., and Reston, Va.; this experiment included biotite standard GA1550 and several other standards. Hornblende standard MMhb-1 was developed as a dating standard by E.C. Alexander, Jr., and G.M. Mickelson. The initial analyses of hornblende standard MMhb-1 were made at the USGS laboratory in Menlo Park, Calif.: K by flame photometry, and Ar by isotope-dilution mass spectrometry. From these measurements, a K-Ar age of 519.5±2.5 Ma was calculated (Alexander and others, 1978). The Ar measurements were made on a newly developed multiple-collector mass spectrometer (Stacey and others, 1981). Only about 20 percent of a typical gas sample was introduced into the mass spectrometer. An isotopic analysis of Ar took only 5 to 10 minutes to complete, and so it was common to analyze two or three splits of Ar gas and average the results. This procedure was followed for the Ar measurements of hornblende standard MMhb-1 reported by Alexander and others (1978). Subsequently, we noticed that the isotopic compositions of second and third splits of gas did not agree with the composition of the first split as well as they should have. In addition, the isotopic composition of the second and later splits of gas changed in the direction of the composition of the sample analyzed previously, indicating a memory effect (Dalrymple and Lanphere, 1969) in the unbaked sample manifold of the mass spectrometer that affected analyses of the second and later splits of the current sample. If only the first splits of Ar gas are used in calculating

the K-Ar age of hornblende standard MMhb-1, the age changes from the 519.5±2.5 Ma reported by Alexander and others (1978) to 514.9±2.2 Ma. The original Ar analyses plus additional Ar analyses have yielded the currently accepted K-Ar age of 513.9±2.3 Ma for hornblende standard MMhb-1 (table 13). A mean ⁴⁰Ar/³⁹Ar age of 513.8±2.6 Ma was determined on hornblende standard MMhb-1, using biotite standard SB-3 as the fluence monitor (table 14). During the late 1980's, while Scott Samson and E.C. Alexander, Jr., were compiling analytical data on hornblende standard MMhb-1, we informed them about the new data from the USGS laboratory in Menlo Park, Calif. However, they chose to include only the original data (Alexander and others, 1978) for hornblende MMhb-1 in their compilation (Samson and

Alexander, 1987). This choice has caused unnecessary confusion about the mean K-Ar age of 513.9±2.3 Ma determined in the USGS laboratory in Menlo Park, Calif.

⁴⁰Ar/³⁹Ar ages were measured on biotite standard GA1550 as part of an intercomparison experiment between the USGS laboratories in Menlo Park, Calif., and Reston, Va. Three splits each of biotite standard GA1550 and several other mineral standards were irradiated, using hornblende standard MMhb-1 as the fluence monitor. Total-fusion ⁴⁰Ar/³⁹Ar ages were determined on two splits of each mineral, and an incremental-heating experiment was made on the third split. The two total-fusion ages of biotite standard GA1550 were 97.49±0.62 and 97.05±0.60 Ma (table 15). An incremental-heating experiment yielded a weighted-mean plateau age of 97.00±0.31 Ma for

Table 13.—K-Ar ages and analytical data for hornblende standard MMhb-1.

[Analysts: K₂O, S.T. Neil; Ar, J.C. Von Essen]

K ₂ O content (weight percent)	Radiogenic- ⁴⁰ Ar content		Calculated age (Ma)
	(10 ⁻⁹ mol/g)	(percent)	
1.867	1.611	86.2	515.9±3.3
1.881	---	---	---
1.876	1.599	94.8	512.4±3.2
1.877	---	---	---
1.875	1.613	89.9	516.4±3.2
1.881	---	---	---
1.874	1.604	95.2	513.9±4.4
1.872	---	---	---
1.869	1.594	91.7	511.0±4.4
1.873	---	---	---
1.867	---	---	---
1.872	---	---	---

Mean = 1.874±0.005

Table 14.—⁴⁰Ar/³⁹Ar total-fusion ages and analytical data for hornblende standard MMhb 1.

[Analysts: J.Y. Saburomaru and J.C. Von Essen]

⁴⁰ Ar/ ³⁹ Ar ratio	³⁷ Ar/ ³⁹ Ar ratio	³⁶ Ar/ ³⁹ Ar ratio	Radiogenic ⁴⁰ Ar (pct)	³⁹ Ar/ _{Ca} (pct)	³⁶ Ar/ _{Ca} (pct)	Age (Ma)
88.51	2.504	0.1321	56.1	0.2	0.5	511.3±4.6
58.94	2.500	.0297	85.4	.2	2.3	517.4±4.6
48.65	2.424	.005584	97.0	.2	11.5	512.4±4.5
48.77	2.447	.004590	97.6	.2	14.5	516.3±4.5
66.72	2.472	.005584	97.8	.2	11.7	511.4±2.6
72.83	2.453	.02657	89.5	.2	2.4	513.9±2.8

Table 15.— $^{40}\text{Ar}/^{39}\text{Ar}$ total-fusion ages and analytical data for biotite standard GA1550.

[Analysts: J.Y. Saburomaru and J.C. Von Essen]

$^{40}\text{Ar}/^{39}\text{Ar}$ ratio	$^{37}\text{Ar}/^{39}\text{Ar}$ ratio	$^{36}\text{Ar}/^{39}\text{Ar}$ ratio	Radiogenic ^{40}Ar		$^{39}\text{Ar}_{\text{Ca}}$ (pct)	$^{36}\text{Ar}_{\text{Ca}}$ (pct)	K/Ca ratio	^{39}Ar (pct)	Age (Ma)
			(10^{-10} mol)	(pct)					
19.01	0.01440	0.009403	1.966	85.4	<0.01	0.04	34.0	100	97.49±0.62
19.22	.01303	.007571	2.0766	88.3	.01	.05	37.6	100	97.05±0.60

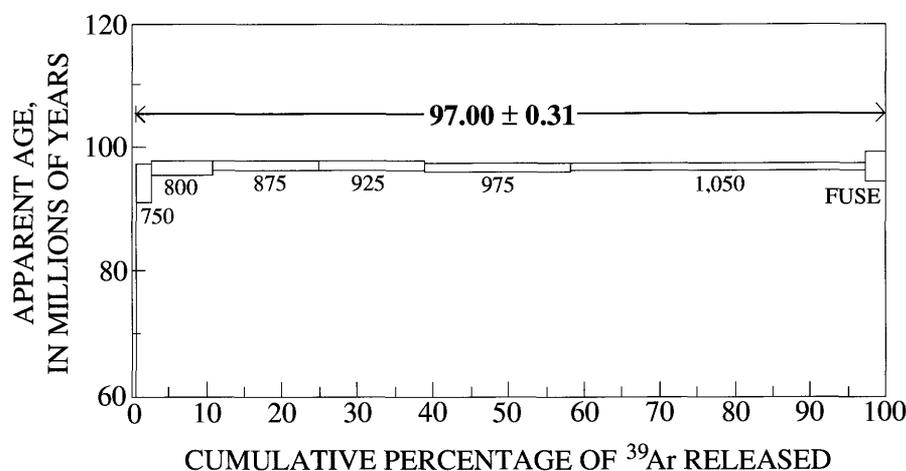
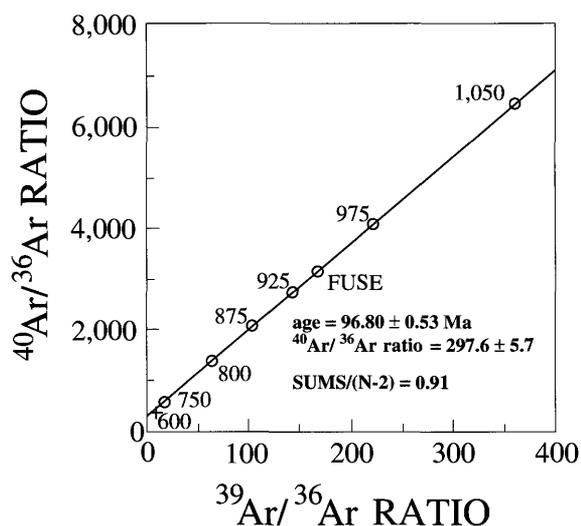
Figure 3.—Age-spectrum diagram for biotite standard GA1550. Vertical dimensions of boxes is estimated analytical precision of increment ages at $\pm 1\sigma$. Numbers are temperatures (in degrees Celsius) at which Ar was extracted.

Figure 4.—Isotope-correlation diagram for biotite standard GA1550. Numbers are temperatures (in degrees Celsius) at which Ar was extracted. Circles, points used in isochron fit; plus sign, point not used in isochron fit.

biotite standard GA1550 (fig. 3), an isochron age of 96.80 ± 0.53 Ma (fig. 4), and an isochron intercept of 297.6 ± 5.7 . The difference between our $^{40}\text{Ar}/^{39}\text{Ar}$ plateau age and the age for biotite standard GA1550 adopted by Renne and others (1998) is 1.85 percent.

Minerals from the Fish Canyon Tuff of Colorado have been dated in several laboratories. Renne and others (1998) calculated an age of 28.02 ± 0.16 Ma for sanidine from the Fish Canyon Tuff on the basis of their intercalibration with biotite standard GA1550. Lanphere and others (1990), Lanphere and Baadsgaard (1997, 1998), and Kozo Uto (written commun., 1998) suggested an age of 27.5 Ma for sanidine and biotite from the Fish Canyon Tuff. The difference between the ages of 28.02 and 27.5 Ma is 1.89 percent, consistent with the difference in age of biotite standard GA1550.

The $^{40}\text{Ar}/^{39}\text{Ar}$ ages suggested by Renne and others (1998) for six mineral standards depend completely on the radiogenic- ^{40}Ar content of biotite standard GA1550 measured at the ANU. That value has never been verified by first-principles measurements in any other laboratory, and so the $^{40}\text{Ar}/^{39}\text{Ar}$

ages suggested by Renne and others (1998) are of questionable accuracy, though highly precise. The age of any primary mineral standard must be measured by first-principles calibration of the radiogenic- ^{40}Ar content in more than one laboratory. Because the $^{40}\text{Ar}/^{39}\text{Ar}$ ages suggested by Renne and others (1998) fail this criterion, we consider it unwise to adopt them for general use.

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