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Intercalibration of ^{40}Ar–^{39}Ar age standards NL-25, HB3gr hornblende, GA1550, SB-3, HD-B1 biotite and BMus/2 muscovite

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Abstract

^{40}Ar–^{39}Ar dating requires precise and accurate age data of standard fluence monitors. Besides primary calibration of K and ^{40}Ar concentrations (i.e. the ages are calculated with precise K–Ar dating), intercalibration of the different monitors in use provides important cross-checks of the validity of standard ages and, implicitly, reliability and uncertainty of chronological studies that rely on these standards. Here we report intercalibration of 5 primary international ^{40}Ar–^{39}Ar standards and one internal laboratory monitor. A major goal of this study was to check the ages of the NL-25 hornblende standard and of the muscovite laboratory standard BMus/2 against the international standards HD-B1, GA1550, SB-3 and HB3gr. In replicate measurements of three irradiation series, ^{40}Ar–^{39}Ar ages for the fluence monitors were found which agree within 1σ-error limits with the reported age values of 2660±9 Ma for NL-25, Hb3gr: 1073.6±4.6 Ma, BMus/2: 328.5±1.1 Ma, SB-3: 162.9±0.8, GA1550: 98.79±0.54 and 24.21±0.32 for HD-B1. For the hornblende NL-25 fluence monitor a new – slightly different – ^{40}Ar–^{39}Ar age of 2657±4 Ma is suggested. For the age of the muscovite BMus/2 laboratory standard we recommend 328.5±1.1 Ma, and for biotite HD-B1 a more precise value of 24.18±0.09 Ma.

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1. Introduction

Since 1977 all K–Ar dating techniques use the constants defined in Steiger and Jäger (1977): $\lambda_\beta = 4.962 \cdot 10^{-10}\ a^{-1}$, $\lambda_e = 0.581 \cdot 10^{-10}\ a^{-1}$, ^{40}K/K$= 1.167 \cdot 10^{-4}$, ^{40}Ar/^{36}Ar$=295.5 \pm 0.5$. These decay constants are based on the radioactivity data evaluated by Beckinsale and Gale (1969), while the atmospheric argon abundances are from Nier (1950), and the potassium isotope abundance ratio from Garner et al. (1975). In contrast, the physics community frequently uses the decay constants of Endt and van der Leun (1973). In the last two decades the problem of the accuracy of the constants used for isotopic dating increased (e.g. Min et al., 2000; Begemann et al., 2001), as uncertainties of individual measurements are sometimes dominated by errors in the respective decay constants. Ongoing initiatives (e.g. Min et al., 2000; Begemann et al., 2001; Kwon et al., 2002) to evaluate a new convention of constants for ^{40}Ar–^{39}Ar dating require highly precise and accurate age data of

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specific key events using different isotope chronometric systems (e.g. K–Ar and U–Pb).

In ^{40}Ar–^{39}Ar dating, ages are calculated from argon isotope ratios of individual samples and mineral standards. The ages of these standards are usually given by direct determinations of K and radiogenic argon (^{40}Ar*) contents (e.g. McDougall and Harrison, 1998), so these values have to be known precisely in order to minimize the systematic error of an age calculation. In the last decades some efforts were made to calibrate laboratory and international mineral standards used in ^{40}Ar–^{39}Ar dating (e.g. TCs, HD-B1, FC(T)-2,3, GHC-305, LP-6, SB-3, GA1550, MMhb-1, Hb3gr; e.g. see Roddick, 1983; Samson and Alexander, 1987; Hess and Lippolt, 1994; Baksi et al., 1996; Renne, 1998; Renne et al., 1998; Charbit et al., 1998; Jourdan et al., 2006; Jourdan and Renne, 2007).

The present study focuses on the hornblende standard NL-25, which is frequently used for dating geologically old samples, particular meteorites or lunar samples (e.g. Husain, 1974; Schaeffer and Schaeffer, 1977; Kunz et al., 1995; Pellas et al., 1997; Trieloff et al., 2003b; Bogard and Garrison, 2003; Bogard et al., 2005) and cross-calibrated by Jourdan and Renne (2007) against 4 other mineral standards (common in ^{40}Ar–^{39}Ar dating) using single-grain laser extraction method. Our study is the first cross calibration of multi-grain separates of this standard and also comprises a cross calibration of standards which were not calibrated against each other previously (e.g. HD-B1 against Hb3gr). The use of multi-grain separates eliminates possible grain to grain variations as systematic error source, yields higher signal to noise ratios and allows more precise results. Our results can be applied to the above mentioned ^{40}Ar–^{39}Ar dating studies of extraterrestrial samples which all used multi grain separates (and not single-grain dating). Whether NL-25 is sufficiently homogeneous to be suitable as single grain standard remains to be clarified.

2. Mineral standards

For the calibration study we used the hornblende standard NL-25, the mineral standards HD-B1, GA1550, SB-3 (all three biotites), our laboratory standard BMus/2 (muscovite) and the international standard Hb3gr (hornblende).

2.1. NL-25 hornblende

The NL-25 fluence monitor is a hornblende separate from the Northern Light Gneiss in Minnesota (USA). It was prepared and measured by Hanson et al. (1971) who calculated a K–Ar age of 2610±60 Ma (^{40}Ar* = $(72.50\pm0.40)\cdot10^{-6}$ cm^3 STP/g, K = 0.302±0.006%) of the initially prepared NL-25-1. In lack of NL-25-1 a new separate of the hornblende was made — NL-25-2 (further called only NL-25) with a grain size of 110–150 µm (Husain, 1974). The K-content was determined by Garner and Machlan (National Bureau of Standards, see Schaeffer and Schaeffer, 1977) to 0.3070±0.0008%. Husain (1974) calculated an age of 2668±18 Ma from this K- and a ^{40}Ar*-content of $(72.00\pm0.90)\cdot10^{-6}$ cm^3 STP/g with old decay constants and K-ratios ($\lambda_\beta = 4.72\cdot10^{-10}$ a^{-1}, $\lambda_e = 0.585\cdot10^{-10}$ a^{-1}, ^{40}K/K $= 1.19\cdot10^{-4}$). The recalculated age using Steiger and Jäger (1977) constants is 2650±9 Ma. Schaeffer and Schaeffer (1977) calculate an age of 2660±9 Ma using the ^{40}Ar*-value from Hanson et al. (1971), the K-content of Garner and Machlan and the constants from Steiger and Jäger (1977). This age was confirmed by Renne (2000) with an age of 2649±15 Ma against FC sanidine (with 28.02 Ma). The latest intercalibration of Jourdan and Renne (2007) for five monitors (Hb3gr, GA1550, NL-25, GHC-305 and FC2) lead to a slightly different age of 2635±19 Ma for NL-25 standard, but still agreeing within 2σ error limit. This diverging age values in use by different groups and achieved with different methods (bulk and single grain analysis) make it advisable to redetermine and improve the precision of the age of fluence standard NL-25. The standard material used for the measurements in this study is from bottle 1/7 EE 100–140 mesh.

2.2. Hb3gr hornblende

Fluence monitor Hb3gr was first analysed by Zartman (1964) (t = 1050±20 Ma) and purified and remeasured by Turner et al. (1971) yielding an age of 1062±10 Ma (^{40}Ar* = $(70.9\pm0.3)\cdot10^{-6}$ cm^3 STP/g, K = 1.247±0.008%, $\lambda_\beta = 4.72\cdot10^{-10}$ a^{-1}, $\lambda_e = 0.584\cdot10^{-10}$ a^{-1}, ^{40}K/K $= 1.19\cdot10^{-4}$; Recalculation with constants by Steiger and Jäger (1977): t = 1072±10 Ma). It was recalibrated by Roddick (1983) to t = 1072±5 Ma (^{40}Ar*/K = 0.08504±0.00425) and verified by Layer et al. (1987) by ^{40}Ar/^{39}Ar laser stepheating (1066±4 Ma, calculated from the measured ^{40}Ar* and a K-value of 1.20±0.02%). Jourdan et al. (2006) calibrated Hb3gr against FC standard and obtained an age of 1073.6±4.6 Ma, which was confirmed by Jourdan and Renne (2007). This age of 1073.6±4.6 Ma was used for age calculations in this study.

2.3. BMus/2 muscovite

BMus/2 muscovite is an in-house standard from the Bärhalde granite in the Black Forrest (Germany), measured by Rittmann (1984) to an age of 326.2±1.1 Ma (^{40}Ar* = 117.7 · 10^{-6} cm^3 STP/g, K = 8.47%) against interlaboratory mineral standard MMHb-1 (using t = 519.5 ± 2.5 Ma, Alexander et al., 1978). The age of BMus/2 standard is 328.5 ± 1.1 Ma against the MMHb value of 523.1 ± 2.6 Ma (MMHb value recalculated by Renne et al., 1998). Monitor from bottle 1, 100–315 μm was used.

2.4. SB-3 biotite

SB-3 biotite is a primary standard proposed by Dalrymple et al. (1981, 1993) and Lanphere et al. (1990) from a quartz diorite in Alaska (USA). The calculated age for this standard is 162.9 ± 0.8 Ma (^{40}Ar* = (49.60 ± 0.25) · 10^{-6} cm^3 STP/g, K = 7.485 ± 0.016). Vial P was used.

2.5. GA1550 biotite

GA1550 is a primary biotite standard from a monzonite in New South Wales (Australia), first introduced by McDougall and Roksandic (1974) by first principle age determination (via isotope dilution), leading to an age of 97.9 ± 0.7 Ma (^{40}Ar* = (1.343 ± 0.007) · 10^{-9} mol/g. corresponding to (30.1 ± 0.1) · 10^{-6} cm^3 STP/g, K = 7.70 ± 0.03%). Baksi et al. (1996), Renne et al. (1998) and Spell and McDougall (2003) remeasured and recalculated the age of the standard to 97.8 ± 0.2, 98.79 ± 0.54 and 98.5 ± 0.8 Ma, respectively, based on different ^{40}Ar*- and/or K-contents (K = 7.70 ± 0.06%, K = 7.626 ± 0.016 and K = 7.645 ± 0.050%, respectively). All ages agree with the given age of McDougall and Roksandic (1974) within error. The age given by Renne et al. (1998) of 98.79 ± 0.54 with a new K content of 7.626 ± 0.016%, with age confirmed by Jourdan and Renne (2007) was taken for age calculation in this study. Split 5/10, 22–60 mesh was used for measurements.

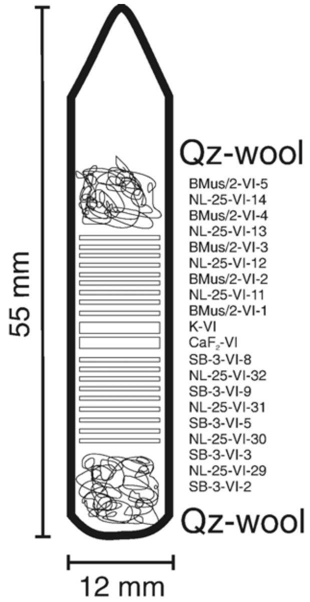

Fig. 1. Packing list of ampoule VI: BMus/2–SB-3–NL-25 in a sealed quartz ampoule as listed in Table 2.

2.6. HD-B1 biotite

The standard HD-B1 is a biotite standard from the Bergell granodiorite (Italy) with a grain size of 200–500 μm (Fuhrmann et al., 1987: t = 24.7 ± 0.3 Ma, ^{40}Ar* = (7.720 ± 0.071) · 10^{-6} cm^3 STP/g, K = 7.985 ± 0.023%). The age of the standard of 24.21 ± 0.32 Ma (^{40}Ar* = (7.536 ± 0.104) · 10^{-6} cm^3 STP/g, K = 7.956 ± 0.051%) is a result of multiple K and Ar analysis determined in several laboratories, with ^{40}Ar–^{39}Ar step heating showing the homogeneous distribution of K and Ar and the absence of any kind of additional excess argon in the standard (Hess and Lippolt, 1994). This age

Table 1
Irradiation parameters for all fluence monitors

Irradiation series	Time [d]	Flux [n/cm^2]	Ampoule	Meas. with spectrometer	Fluence monitors
1	3	3 · 10^{17}	X	GD-150	NL-25, Hb3gr, SB-3, HD-B1
			V	GD-150	NL-25, HD-B1
	10	1 · 10^{18}	VI	GD-150	NL-25, BMus/2, SB-3
			VII	GD-150	NL-25, Hb3gr
2	3	3 · 10^{17}	IX	CH5	NL-25, HD-B1
	9	9 · 10^{17}	XV	CH5	NL-25, Hb3gr, GA1550
3	20	2 · 10^{18}	I	GD-150	Al-foil
			IV	GD-150	NL-25

Table 2
Results of argon measurements for the standard monitors NL-25, Hb3gr, BMus/2, SB-3, GA1550 and HD-B1 – sorted by ampoule number (roman letter); fluence monitors are in the same order as their position in the ampoules – an example is given in Fig. 1 for ampoule VI

Sample		Weight [mg]	$^{40}Ar/^{36}Ar_{air}$ [10^2]	$^{40}Ar/^{37}Ar$ [10^2]	$^{40}Ar/^{38}Ar_{tot}$ [a] [10^2]	$^{40}Ar_{air}$ [%]	$^{40}Ar^*/^{39}Ar$
Ampoule V (irradiation: 3 days; fast neutron dose: c. $3 \cdot 10^{17}$ n/cm^2)							
HD-B1	V-7	32.5	9.19±0.08	8.58±0.18	4.89±0.04	32.1	7.959±0.072
NL-25	V-24	14.4	107.8±2.4	1.52±0.03	–	2.7	1993±13
HD-B1	V-8	34.8	6.98±0.02	13.02±0.26	5.40±0.02	42.3	7.921±0.032
NL-25	V-25	13.4	116.9±4.2	1.51±0.03	–	2.5	1966±11
HD-B1	V-9	32.6	6.50±0.05	11.95±0.26	5.64±0.05	45.5	7.960±0.076
NL-25	V-26	14.0	95.4±1.2	1.52±0.03	–	3.1	1950±10
HD-B1	V-10	31.3	6.66±0.02	11.56±0.23	5.54±0.02	44.4	7.880±0.041
NL-25	V-27	14.1	86.1±2.0	1.50±0.03	–	3.4	1954±14
HD-B1	V-11	34.7	9.23±0.07	12.02±0.24	4.87±0.02	32.0	7.902±0.039
Ampoule VI (irradiation: 10 days; fast neutron dose: c. $1 \cdot 10^{18}$ n/cm^2)							
SB-3	VI-2	6.9	17.44±0.07	23.59±0.53	8.07±0.04	16.9	21.77±0.13
NL-25	VI-29	8.7	64.1±0.7	0.599±0.011	–	4.6	768.6±1.4
SB-3	VI-3	7.8	16.09±0.15	16.44±0.35	7.95±0.05	18.4	21.52±0.18
NL-25	VI-30	9.3	66.5±2.6	0.593±0.012	368±177	4.4	763.1±1.8
SB-3	VI-5	8.0	16.12±0.07	19.71±0.52	8.03±0.03	18.3	21.50±0.04
NL-25	VI-31	10.3	62.9±0.7	0.594±0.011	–	4.7	763.1±2.5
SB-3	VI-9	4.1	14.32±0.21	18.61±0.43	7.97±0.04	20.6	21.27±0.13
NL-25	VI-32	11.8	67.2±1.8	0.585±0.012	–	4.4	762.0±3.1
SB-3	VI-8	5.9	14.60±0.06	15.26±0.31	7.92±0.02	20.2	21.15±0.07
BMus/2	VI-1	8.9	82.8±1.5	1333±287	24.27±0.30	3.6	44.24±0.12
NL-25	VI-11	8.6	76.7±1.0	0.571±0.011	7.74±0.02	3.8	744.6±2.1
BMus/2	VI-2	10.1	75.2±1.5	1213±140	23.94±0.25	3.9	44.70±0.10
NL-25	VI-12	8.7	42.6±0.3	0.602±0.012	265±147	6.9	750.2±2.4
BMus/2	VI-3	11.2	76.7±1.0	429.9±22.8	24.44±0.21	3.8	43.97±0.08
NL-25	VI-13	10.8	46.6±0.5	0.588±0.012	330±76	6.3	743.1±2.0
BMus/2	VI-4	10.2	77.0±1.2	–	23.63±0.25	3.8	44.17±0.06
NL-25	VI-14	11.2	54.2±0.7	0.579±0.011	–	5.4	738.7±3.3
BMus/2	VI-5	9.5	70.0±0.1	483.7±15.6	23.45±0.41	4.1	43.92±0.07
Ampoule VII (irradiation: 10 days; fast neutron dose: c. $1 \cdot 10^{18}$ n/cm^2)							
Hb3gr	VII-6	3.7	38.9±3.1	0.685±0.014	15.69±0.27	7.6	188.5±1.3
NL-25	VII-6	8.9	25.2±0.2	0.635±0.013	–	11.7	761.8±1.9
Hb3gr	VII-7	4.0	45.2±1.3	0.673±0.013	15.19±0.29	6.5	184.9±0.6
NL-25	VII-7	8.3	29.6±0.4	0.641±0.013	179±59	10.0	772.9±11.7 [b]
Hb3gr	VII-8	3.7	64.4±2.2	0.669±0.013	14.62±0.05	4.6	183.3±0.9
NL-25	VII-9	8.4	57.0±1.5	0.590±0.012	–	5.2	749.5±11.4 [b]
Hb3gr	VII-9	4.1	45.6±0.2	0.624±0.012	13.26±0.10	6.5	182.6±0.7
NL-25	VII-10	9.4	64.2±1.5	0.589±0.012	–	4.6	742.9±1.8
Hb3gr	VII-10	4.9	36.3±0.4	0.651±0.013	13.06±0.02	8.1	179.9±0.6
Ampoule IX (irradiation: 3 days; fast neutron dose: c. $3 \cdot 10^{17}$ n/cm^2)							
HD-B1	IX-1	23.7	9.16±0.06	4.53±0.23	5.30±0.02	32.3	8.709±0.027
NL-25	IX-2	4.4	23.9±0.5	1.69±0.03	109.1±2.3	12.4	2172±38 [b]
HD-B1	IX-2	30.8	5.37±0.04	4.94±0.23	6.83±0.04	55.0	8.648±0.047
NL-25	IX-3	3.8	12.39±0.17	1.87±0.03	61.1±0.8	23.9	2151±12
HD-B1	IX-3	23.3	16.07±0.17	3.30±0.20	4.64±0.02	18.3	8.580±0.022
NL-25	IX-4	4.1	21.1±0.3	1.64±0.02	97.4±1.2	14.0	2135±11
HD-B1	IX-4	23.5	12.12±0.10	3.60±0.22	4.90±0.02	24.4	8.554±0.024
NL-25	IX-5	4.3	15.83±0.10	1.65±0.02	76.0±0.5	18.7	2129±9
HD-B1	IX-5	25.3	10.88±0.14	4.02±0.25	5.01±0.06	27.2	8.525±0.048
NL-25	IX-16	4.5	22.7±0.3	1.65±0.02	102.4±1.1	13.0	2123±7
HD-B1	IX-6	35.8	14.12±0.19	3.96±0.20	4.75±0.06	20.9	8.538±0.040

(continued on next page)

Table 2 (*continued*)

Sample		Weight	$^{40}Ar/^{36}Ar_{air}$	$^{40}Ar/^{37}Ar$	$^{40}Ar/^{38}Ar_{tot}$ [a]	$^{40}Ar_{air}$	$^{40}Ar^{*}/^{39}Ar$
		[mg]	[10^2]	[10^2]	[10^2]	[%]	
Ampoule IX (irradiation: 3 days; fast neutron dose: c. 3·10^{17} n/cm^2)							
NL-25	IX-14	4.4	23.4±0.2	1.59±0.02	105.9±1.0	12.6	2139±8
Ampoule X (irradiation: 3 days; fast neutron dose: c. 3·10^{17} n/cm^2)							
HD-B1	X-1	32.2	8.19±0.03	9.61±0.20	5.07±0.03	36.1	7.983±0.036
NL-25	X-19	13.3	102.4±3.6	1.51±0.03	–	2.9	1977±11
Hb3gr	X-1	4.1	55.6±2.1	1.64±0.03	35.2±0.6	5.3	474.8±1.9
SB-3	X-1	7.4	35.3±0.3	36.2±1.0	19.5±0.2	8.4	55.44±0.16
HD-B1	X-2	30.8	7.99±0.03	12.95±0.27	5.15±0.02	37.0	7.885±0.049
NL-25	X-20	14.1	90.0±2.4	1.52±0.03	–	3.3	1973±21 [b]
Hb3gr	X-2	4.0	56.9±2.7	1.63±0.03	37.1±0.8	5.2	478.4±2.5
SB-3	X-4	8.8	37.0±0.4	38.1±0.9	19.3±0.1	8.0	55.24±0.19
HD-B1	X-3	31.9	10.75±0.06	7.11±0.14	4.63±0.02	27.5	7.831±0.032
NL-25	X-21	14.0	87.7±1.4	1.52±0.03	–	3.4	1966±16
Hb3gr	X-3	5.6	54.7±0.5	1.77±0.04	40.0±0.06	5.4	472.6±1.6
SB-3	X-6	9.9	69.3±2.4	47.5±1.5	19.3±0.2	4.3	54.81±0.20
HD-B1	X-4	35.9	10.25±0.09	8.90±0.18	4.70±0.02	28.8	7.836±0.036
NL-25	X-22	13.3	87.9±2.2	1.52±0.03	–	3.4	1965±14
Hb3gr	X-4	4.1	34.7±0.8	1.68±0.03	34.2±0.15	8.5	465.4±2.3
SB-3	X-7	7.1	24.3±0.2	101±10	19.3±0.3	12.2	54.98±0.14
HD-B1	X-5	31.0	10.60±0.14	9.47±0.21	4.65±0.04	27.9	7.780±0.108
Ampoule XV (irradiation: 9 days; fast neutron dose: c. 9·10^{17} n/cm^2)							
Hb3gr	XV-1	4.2	56.1±1.3	0.771±0.017	17.8±0.4	5.3	213.0±0.9
GA1550	XV-1	9.5	25.8±0.3	5.08±0.24	3.73±0.04	11.5	14.70±0.05
NL-25	XV-7	4.4	26.4±0.4	0.736±0.008	92.5±1.2	11.2	870.8±3.4
Hb3gr	XV-2	4.0	54.8±1.3	0.795±0.017	17.9±0.4	5.4	211.7±0.9
GA1550	XV-2	9.8	28.1±0.4	4.98±0.26	3.67±0.04	10.5	14.63±0.08
NL-25	XV-8	4.1	22.2±0.4	0.739±0.008	83.4±1.3	13.3	868.9±3.9
Hb3gr	XV-3	4.1	53.1±0.5	0.764±0.004	17.5±0.1	5.6	211.8±0.3
GA1550	XV-3	11.2	28.3±0.4	4.82±0.25	3.59±0.04	10.5	14.55±0.05
NL-25	XV-10	4.4	21.8±0.4	0.749±0.008	83.4±1.1	13.5	873.7±2.0
Hb3gr	XV-4	3.7	61.8±0.5	0.861±0.003	18.7±0.1	4.8	211.6±0.3
GA1550	XV-4	11.4	29.0±0.5	4.71±0.26	3.59±0.05	10.2	14.60±0.08
NL-25	XV-11	3.9	19.0±0.3	0.762±0.009	73.5±1.2	15.6	867.4±6.0 [b]
Hb3gr	XV-5	3.8	54.3±0.6	0.744±0.003	17.2±0.1	5.4	210.7±0.3
GA1550	XV-5	9.4	22.4±0.4	4.39±0.24	3.65±0.06	13.2	14.52±0.08
NL-25	XV-12	4.9	23.0±0.5	0.742±0.009	84.2±1.5	12.8	870.5±6.5 [b]
GA1550	XV-6	10.2	27.2±0.3	5.19±0.14	3.62±0.08	10.9	14.50±0.19
NL-25	XV-13	6.1	18.5±0.6	0.769±0.011	66.6±2.5	15.9	870.5±12.5 [b]

[a] $^{38}Ar_{tot}={}^{38}Ar_{air}+{}^{38}Ar_{Cl}+{}^{38}Ar_{K}$.

[b] High error due to additional corrections.

was validated by Wijbrans et al. (1995). The samples measured here are from Split 8/4 200–500 μm.

3. Analytical procedure

For the $^{40}Ar/^{39}Ar$ age measurements of the fluence monitors, samples were wrapped in ultra pure aluminium foil (yielding pellets with 1 mm height and c. 8–9 mm diameter) and stacked in evacuated (between 10^{-4} and 10^{-6} mbar) and Cd shielded quartz tubes with c. 12×55 mm size (e.g. see example for ampoule VI in Fig. 1). Evacuation minimizes possible recoil loss of neutron induced argon. For HD-B1 c. 30 mg each were irradiated, GA1550 c. 10 mg, SB-3 c. 7 mg, BMus/2 c. 10 mg, Hb3gr c. 5 mg and for NL-25 c. 5–15 mg (precise weight values see Table 2). The irradiations were performed in three series at the FRG-1 reactor at the Nuclear Research Center Geesthacht (GKSS), Germany, with different irradiation times to exclude uncertainities from systematic errors concerning the irradiation time (e.g. $^{40}Ar^{*}/^{39}Ar$ values). During irradiation the quartz ampoules were rotated, thus the lateral flux gradient is negligible.

The irradiation series are listed in Table 1. The series contain different ampoules with different packing lists for the fluence monitors and were irradiated at different dates. The irradiation time varies from 3 over 9 to 20 days yielding different neutron doses, which are listed in Table 1. The ampoule packing lists for all ampoules listed in Table 1 are shown in Table 2 with the samples listed in the same order as their position was in the ampoules. An example is shown in Fig. 1. for ampoule VI (see Tables 1, 2). Additionally in Table 1 we specified the mass spectrometer used for monitor analyses.

We furthermore performed a check for ^{39}Ar recoil loss during irradiation by filling an individual ampoule with a NL-25 hornblende standard (5.6 mg; third series: ampoule IV, 20 days). To test the purity (and the blank) of the used materials (quartz ampoule, quartz wool, aluminium) we irradiated an empty Al foil (163.4 mg; third series: ampoule I, 20 days). In this third series the highly purified quartz ampoules were evacuated up to 10^{-6} mbar, irradiated and crushed in the spectrometer line for gas analyses as described in Hess and Lippolt (1986).

Before analysis the extraction line was baked out at c. 100 °C. Argon was extracted in one temperature step depending on mineral between 1300 °C and 1600 °C in a double-vacuum resistance-heated tantalum furnace. Extraction blanks (Ta-furnace, line, getters etc.) comprised c. $1 \cdot 10^{-9}$ to $5 \cdot 10^{-9}$ cm^3 ^{40}Ar at lowest temperature up to $5 \cdot 10^{-9}$ to $10 \cdot 10^{-9}$ cm^3 at the highest temperature for both extraction lines, depending on furnace condition. Blanks had atmospheric isotopic composition, containing no ^{37}Ar and ^{39}Ar for the GD-150 and a negligible amount for the CH5 mass spectrometer ($<1 \cdot 10^{-12}$ cm^3/STP for all measurements). The gas was purified in the extraction line with Ta- and SAES-Al-getters. The argon isotope compositions were measured in static mode in two different in-house modified mass spectrometers (MAT GD-150 (with faraday cup) and CH5 (with electron multiplier)) to exclude uncertainties from systematic errors (mass discrimination, background of furnace, extraction line, etc.) induced by one spectrometer. The mass spectrometer detection limit for the GD-150 is about $5 \cdot 10^{-12}$ cm^3/STP for all argon isotopes and for the CH5 $5 \cdot 10^{-14}$ cm^3/STP for isotopes ^{36}Ar to ^{39}Ar and 10^{-12} cm^3/STP for ^{40}Ar. The background of the GD-150 spectrometer is not measurable and for the CH5 it is smaller than $5 \cdot 10^{-10}$ cm^3/STP for ^{40}Ar and $<1 \cdot 10^{-12}$ cm^3/STP for isotopes ^{36}Ar to ^{39}Ar. For more experimental details, see Trieloff et al. (2003a, 2005) and Hess and Lippolt (1986).

For the age calculations, the IUGS recommended constants (Steiger and Jäger, 1977) were used. All data were corrected for blanks (measured before each sample individually), mass discrimination (checked every day by an air pipette), isotope interferences during neutron activation (checked by standard materials in all irradiation series, e.g. CaF$_2$ and sanidine glass) and radioactive decay for ^{37}Ar and ^{39}Ar. The interference for ^{39}Ar and ^{36}Ar produced from Ca are between $(0.930 \pm 0.011) \cdot 10^{-3}$ and $(1.013 \pm 0.025) \cdot 10^{-3}$ for $(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$ and $(4.14 \pm 0.23) \cdot 10^{-3}$ and $(4.75 \pm 0.30) \cdot 10^{-3}$ for $(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$. Those from K are $(1.707 \pm 0.020) \cdot 10^{-2}$ and $(1.866 \pm 0.009) \cdot 10^{-2}$ for $(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}}$ and $(1.41 \pm 0.02) \cdot 10^{-2}$ and $(1.49 \pm 0.28) \cdot 10^{-2}$ for $(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}}$ (see Table 3). The isotope ratios and argon data determined in this study are given with ±1σ precision. All isotope fractions were listed in Table 2 for all measured fluence monitors.

4. Results

4.1. Ampoule experiments

During irradiation, argon nuclei produced from K and Ca via the ^{39}K(n,p)^{39}Ar and ^{40}Ca(n,α)^{37}Ar reactions are recoiled by a certain distance from the initial positions of the respective parent nuclei (e.g. Turner and Cadogan, 1974; Onstott et al., 1995; Villa, 1997). In fine grained or leaky minerals this could result in loss of ^{39}Ar and ^{37}Ar during irradiation. Furthermore, the ampoule can heat up to 100–150 °C during irradiation possibly leading to partial loss of radiogenic ^{40}Ar. Hess and Lippolt (1986) and Paine et al. (2006) have shown that minerals/fluence monitors are variably susceptible for recoil induced argon loss, so both loss of neutron-induced and/or radiogenic argon has to be checked thoroughly for all standards used in ^{40}Ar–^{39}Ar dating.

Table 3
Isotopic interferences during neutron irradiation

Ampoule/Series	n-flux [n/cm^2]/irradiation days	$(^{39}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$ [10^{-3}]	$(^{36}\text{Ar}/^{37}\text{Ar})_{\text{Ca}}$ [10^{-3}]	$(^{38}\text{Ar}/^{39}\text{Ar})_{\text{K}}$ [10^{-2}]	$(^{40}\text{Ar}/^{39}\text{Ar})_{\text{K}}$ [10^{-2}]
VI, VII/1	$1 \cdot 10^{18}$/10	0.951±0.021	4.14±0.23	1.707±0.020	1.41±0.02
V, X/1	$3 \cdot 10^{17}$/3	1.013±0.025	4.51±0.11	1.866±0.009	1.49±0.28
IX/2	$3 \cdot 10^{17}$/3	0.930±0.011	4.21±0.56	n.d.	n.d.
XV/2	$9 \cdot 10^{17}$/9	1.008±0.006	4.75±0.30	n.d.	n.d.

Table 4
Argon data from irradiation loss ampoule experiments for monitors NL-25

Sample (ampoule)	^{36}Ar$_{air}$	^{37}Ar	^{38}Ar$_{tot}$ [a]	^{39}Ar	^{40}Ar	^{37}Ar loss	^{39}Ar loss
	[10^{-8} cm^3 STP]					[%]	
Al foil (I)	0.943 ± 0.008	<0.0004 [b]	0.31 ± 0.10 [c]	<0.0004 [b]	273.1 ± 0.3	<0.01 [b]	<0.01 [b]
NL-25 (IV)	1.016 ± 0.003	<0.0004 [b]	0.22 ± 0.03	<0.0004 [b]	297.8 ± 0.2	<0.01 [b]	<0.16 [b]

[a] $^{38}\mathrm{Ar}_{tot} = {}^{38}\mathrm{Ar}_{air} + {}^{38}\mathrm{Ar}_{Cl} + {}^{38}\mathrm{Ar}_{K}$.

[b] Estimation against mass spectrometer detection limit.

[c] High error due to correction of memory effect in mass spectrometer.

To monitor the ^{39}Ar recoil loss effect for NL-25 hornblende, an ampoule loaded with a single NL-25 sample was irradiated, afterwards placed in the extraction line and cracked online in order to analyse the argon from the ampoule. An Al foil was measured using the same procedure to monitor additional blank effects caused by argon produced during irradiation in and/or released from the Al foil or the quartz ampoule/wool. After removing these two samples from their ampoules they were measured by total fusion like all other standards. The blank level of the crushing equipment and the extraction line was smaller than $1 \cdot 10^{-9}$ cm^3/STP for stable argon isotopes, with atmospheric composition and no masses 39 and 37 present. A background of the GD-150 spectrometer was not detectable. For blanks of the heating procedure, we refer to the 'Analytical procedure' section. Results corrected for line and furnace blanks are shown in Table 4.

For the Al-foil and the NL-25 standard no ^{37}Ar and ^{39}Ar was detected in the ampoule. From the mass spectrometer detection limit (MAT GD-150), we calculated the maximum loss of argon from the NL-25 sample. For ^{37}Ar it is less than 0.01% and for ^{39}Ar less than 0.16% of the total. The better upper limit for ^{37}Ar is due to the higher Ca concentration of the hornblende. However, the loss of ^{39}Ar can be constrained to be much lower than 0.16%, as it should not be higher than for ^{37}Ar, as both are lost via the same mechanism. In contrast, ^{39}Ar loss should be even smaller than 0.01%, maybe at least even twice times lower, due to the different recoil distance for ^{39}Ar (~0.1 μm) and ^{37}Ar (~0.2 μm) (Onstott et al., 1995).

The ^{40}Ar/^{36}Ar and ^{38}Ar/^{36}Ar ratios of the gas released from the ampoules loaded with Al-foil and the NL-25 monitor are 290 ± 3 and 0.33 ± 0.11 and 293 ± 1 and 0.22 ± 0.03, respectively. They are within 2σ-error indistinguishable from the atmospheric values (295.5 and 0.187, Nier, 1950; Steiger and Jäger, 1977) — note that the large uncertainty in ^{38}Ar is due to a spike memory released when the very large amounts of argon from the ampoule entered the GD150 mass spectrometer. Due to the high amount of atmospheric argon left in the ampoule after evacuation, an investigation of the loss of radiogenic ^{40}Ar was not possible.

4.2. Fluence monitor experiments

The fluence monitors were packed in six different ampoules (see Tables 1, 2). The vertical flux gradient of the irradiation for the SB-3, BMus/2 and NL-25 ampoule is shown in Fig. 2 and listed in Table 2, for all other ampoules the packing list is listed in the same way. It can be seen, that the flux gradient derived from the NL-25 standard is nearly linear, but in detail it has an irregular shape (Fig. 2). The ^{40}Ar*/^{39}Ar ratios (and thus the J-value for age calculation) and the corresponding errors of the monitors were calculated from the ^{40}Ar*/^{39}Ar values from the surrounding monitors. The results of all standard measurements are listed in Table 2. A lateral flux gradient has not to be considered: first, the sample container was rotated throughout the irradiation, second, our bulk samples were homogeneously distributed in the Al pellet across the lateral extension of the quartz ampoule.

While the main focus of this study was calibrating the NL-25 standard monitor with 5 (primary) ^{40}Ar–^{39}Ar standards, we obtained additional data for intercalibration of the 6 standards. This method calculating ages for each standard against each other minimizes systematic errors concerning one single standard for its given

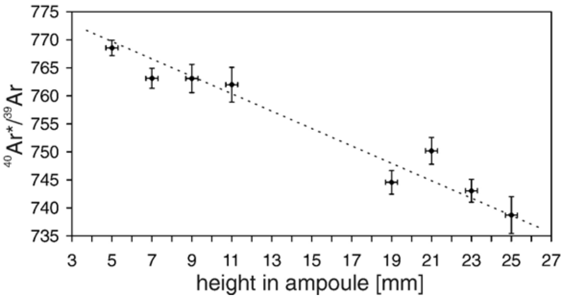

Fig. 2. Vertical flux gradient of standard monitor NL-25 of ampoule VI.

Table 5
Calculated monitor ages (without monitor errors) against the other mineral fluence monitors — GD150: ampoules V, VI, VII, X, CH5: IX, XV

Monitor	NL-25	Hb3gr (old)	Hb3gr (new)	BMus/2 (old)	BMus/2 (new)	SB-3	GA1550	HD-B1
NL-25	–	VII	VII	VI	VI	VI		V
		2637±14	2639±14	2639±8	2648±8	2655±9		2669±10
		2676±22	2679±22	2654±12	2663±12	2654±3		2650±8
		2642±21	2644±21	2649±5	2658±5	2661±9		2642±10
		2643±11	2645±11	2642±7	2650±7	2671±7		2650±10
		X	X			X		X
		2653±13	2656±13			2660±8		2657±10
		2653±15	2656±15			2661±15		2669±15
		2660±15	2662±15			2664±12		2669±11
		2679±11	2682±11			2665±10		2672±11
		IX	IX				XV	IX
		2645±8	2648±8				2648±6	2667±25
		2644±6	2647±6				2652±7	2664±11
		2652±3	2655±3				2663±4	2660±8
		2646±10	2649±10				2653±10	2661±6
		2650±11	2653±11				2663±10	2657±5
							2665±20	2667±5
Hb3gr	VII							
	1094±13							
	1072±8							
	1072±14							
	1085±7							
	1076±6							
	X					X		X
	1070±3					1070±3		1070±6
	1078±4	–	–	n.d.	n.d.	1078±4		1083±5
	1070±3					1074±3		1075±3
	1060±3					1061±3		1066±5
	XV						XV	
	1084±5						1076±5	
	1080±4						1076±4	
	1080±3						1078±2	
	1078±4						1077±2	
	1078±2						1076±2	
BMus/2	VI							
	325.7±1.9							
	331.1±1.3							
	326.5±1.5	n.d.	n.d.	–	–	n.d.	n.d.	n.d.
	330.1±1.0							
	329.3±1.3							
SB-3	VI							
	164.4±1.0							
	163.1±1.4							
	163.6±0.3							
	162.0±1.0							
	163.0±1.0			n.d.	n.d.	–	n.d.	
	X	X	X					X
	165.1±2.3	163.1±0.5	163.4±0.5					163.3±1.6
	162.8±0.6	162.0±0.7	162.3±0.7					164.0±1.0
	162.0±0.6	162.8±0.7	163.1±0.7					162.9±0.6
	162.7±0.5	164.6±0.7	165.0±0.7					164.3±1.0
GA1550	XV	XV	XV					
	99.94± 0.38	98.55±0.41	98.74±0.40					
	99.15±0.61	98.43±0.50	98.62±0.50	n.d.	n.d.	n.d.	–	n.d.
	98.94±0.58	97.92±0.34	98.11±0.34					
	99.25±0.55	98.41±0.51	98.60±0.51					

(continued on next page)

Table 5 (*continued*)

Monitor	NL-25	Hb3gr (old)	Hb3gr (new)	BMus/2 (old)	BMus/2 (new)	SB-3	GA1550	HD-B1
GA1550	XV	XV	XV					
	98.59±0.54	98.13±0.55	98.32±0.55					
HD-B1	V							
	24.11±0.27							
	24.16±0.19							
	24.54±0.25							
	24.37±0.13							
	24.47±0.13							
	X	X	X			X		
	24.36±0.11	24.19±0.28	24.24±0.28			24.34±0.12		
	24.11±0.15	24.04±0.17	24.09±0.16			24.10±0.16		
	24.34±0.31	23.98±0.15	24.03±0.15	n.d.	n.d.	24.05±0.18	n.d.	–
	24.07±0.11	24.34±0.19	24.38±0.19			24.19±0.13		
	23.95±0.33	24.26±0.34	24.31±0.34			24.04±0.34		
	IX							
	24.20±0.43							
	24.11±0.15							
	24.14±0.09							
	24.21±0.07							
	24.19±0.14							
	24.21±0.13							

Monitor ages for NL-25: 2660±9; Hb3gr: old : 1072±5 Ma; new: 1073.6±4.6 Ma; BMus/2: old: 326.2±1.1 Ma; new: 328.5±1.1 Ma; SB-3: 162.9±0.8 Ma; GA1550: 98.79±0.54 Ma; HD-B1: 24.21±0.32 Ma; explanation see ‘Mineral standards’ section.

values for K and/or radiogenic Ar content, weight and irradiation parameters. Table 5 and Fig. 3 show the results of the age calculation for the different standards against each other using the literature monitor values as mentioned in Section 2 (‘Mineral Standards’) and the footnote in Tables 5 and 6. The errors do not include the monitor age uncertainty, because the ages were calculated against only one standard and thus are comparable considering the internal error only, without adding the standard monitor error.

From the data listed in Table 5 it is possible to calculate the weighted mean values of the monitor ages for the investigated standards. To compare the data with the primary measurements and intercalibration of standards of other studies it is necessary to add the monitor error (external error). The results can be seen in Table 6 and Fig. 3, with external error in parentheses.

From these values for one specific standard against several others one can calculate – taking into account the external error, including the respective monitor error – a weighted mean value for the standards used in this intercalibration (see Table 7, Fig. 3). The mean values of all standards well agree within uncertainties (Table 7, columns 4–6) with literature values (Table 7, column 2) which were used as “best” values to perform our cross-calibration. Column 7 in Table 7 shows the age values that result from a least square fitting procedure in order to check the dependency of the results on the choice of the “best” or initial values serving for age calculation of the individual standards. The procedure was performed in the following way: After calculating the mean age of each standard using the “best” values of the cross-calibrating standards according to our measurements, χ^2-deviations are calculated for all standards. For the standard with the largest individual χ^2-deviation, the latter was minimized by fitting the standard’s ‘best initial’ age value. This procedure was repeated for the next standard displaying the largest individual χ^2-deviation, and so on until convergence. In order to check and to reduce possible errors in the initial “best” data set of each fluence monitor, we used various monitor age sets to start with the fitting procedure, particularly by choosing alternative values for NL-25 (2650±9 Ma), Hb3gr (1072±5 Ma) and GA1550 (97.8±0.2 Ma). χ^2-fit results did not differ by more than 0.03%, i.e. small when compared to the uncertainties of 0.1–0.3% in column 4 of Table 7, supporting the reliability of these results.

The errors for the χ^2-fit (Table 7, column 7) are remarkably lower than that of the calculated mean ages (Table 7, coloumn 2). This is a result of the fit procedure which minimizes the differences between the different mineral standards and thus their uncertainties. For this reason we do not recommend these errors (shown in Table 7 column 7), but we recommend the (rounded) errors of the calculated mean age in column 2. Thus we recommend the values and errors listed in Table 7,

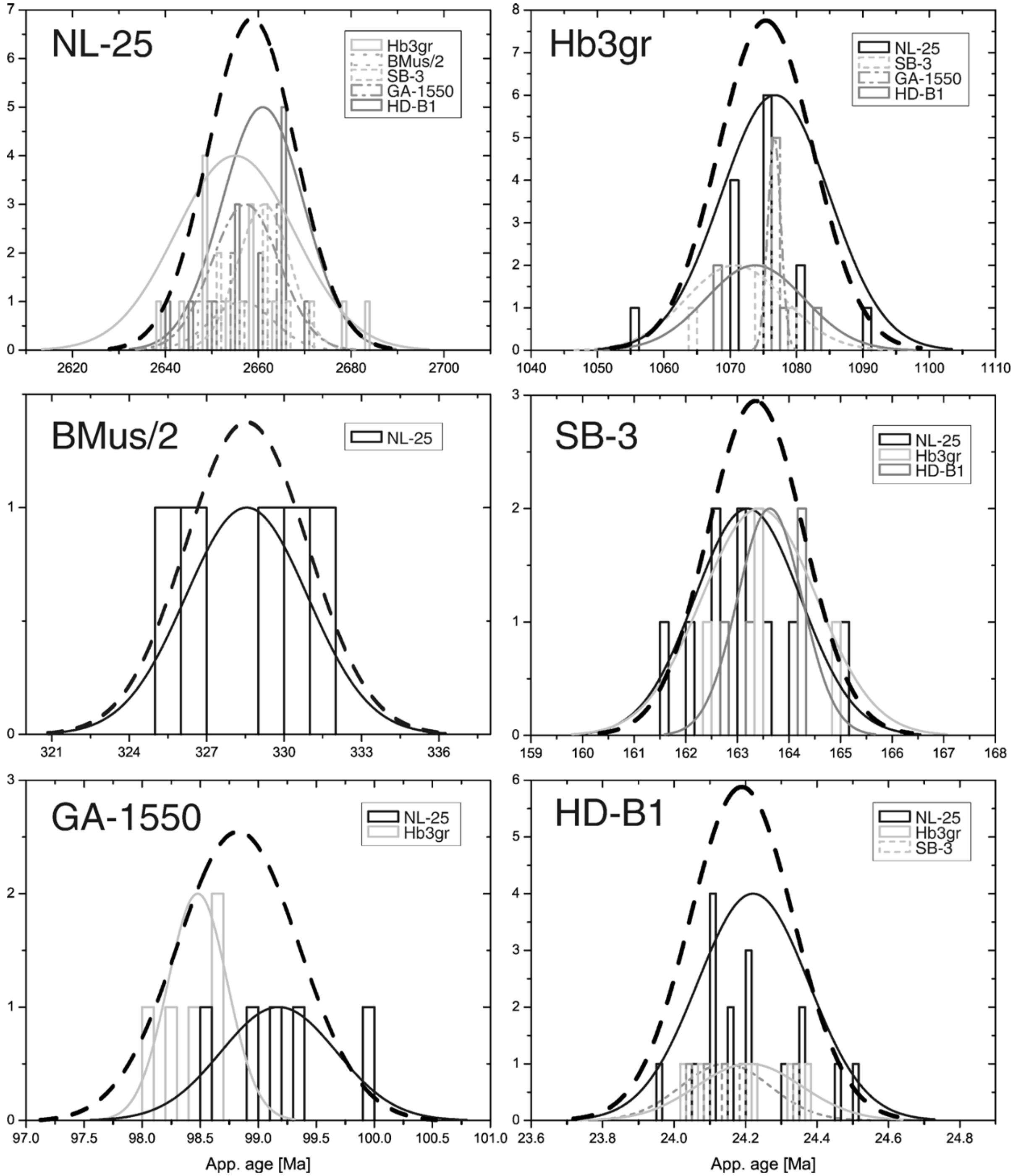

Fig. 3. Histogram for all investigated standards with a Gaussian standard deviation plot for each monitor (solid line) and the total (dashed line).

column 8 for NL-25: 2657 ± 4 Ma; HD-B1: 24.18 ± 0.09 Ma and BMus/2: 328.5 ± 1.1 Ma.

The errors, particular for NL-25 appear quite low, but several reasons explain the high level of precision of the NL-25 age result: First, the error is given as age error, not as error in the $^{40}Ar^*/^{39}Ar$ ratio. In case of the NL-25 monitor, which has a very high age of 2.6 Ga, our reported 1.5 permil error in age corresponds to a 2.5 permil error in the $^{40}Ar^*/^{39}Ar$ ratio (due to the exponential character of the decay curve). 2.5 permil

Table 6
Mean values of Table 5 for all standards — in parentheses additional monitor error

Monitor	NL-25	Hb3gr (old)	Hb3gr (new)	BMus/2 (old)	BMus/2 (new)	SB-3	GA1550	HD-B1
NL-25	–	2652.3± 3.1 (9)	2653.7± 2.3 (8)	2645.8± 3.4 (6)	2656.4± 3.5 (6)	2658.0± 2.3 (8)	2658.0± 2.9 (8)	2660.4± 2.2 (19)
Hb3gr	1075±6 (8)	–	–	n.d.	n.d.	1071± 6 (7)	1077± 2 (5)	1075± 5 (12)
BMus/2	329.2±0.9 (2.2)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
SB-3	162.6±0.3 (1.1)	163.1± 0.5 (1.1)	163.4± 0.5 (1.0)	n.d.	n.d.	–	n.d.	163.5± 0.3 (2.1)
GA1550	99.31±0.25 (0.67)	98.25± 0.20 (0.65)	98.44± 0.20 (0.56)	n.d.	n.d.	n.d.	–	n.d.
HD-B1	24.22± 0.03 (0.16)	24.11± 0.09 (0.15)	24.16± 0.09 (0.16)	n.d.	n.d.	24.20± 0.07 (0.14)	n.d.	–

Monitor ages for NL-25: 2660±9; Hb3gr: old : 1072±5 Ma; new: 1073.6±4.6 Ma; BMus/2: old: 326.2±1.1 Ma; new: 328.5±1.1 Ma; SB-3: 162.9±0.8 Ma; GA1550: 98.79±0.54 Ma; HD-B1: 24.21±0.32 Ma; explanation see 'Mineral standards' section.

uncertainty are comparable to other studies (e.g. Fish Canyon monitor by Jourdan and Renne (2007) is reported with a 3.5 permil error). Second, we used multi-grain, milligram-size separates: they yielded large amounts of sample gas and at the same time we avoided sample heterogeneities. Third, we calibrated many NL-25 samples against 4 other primary international standards. The overall accurate result is due to using all primary K- and ^{40}Ar-determinations of different primary standards (not only those of one standard). The error given for all standards includes all analytical and systematic errors related to the apparature (mass discrimination — partial pressure dependence and day to day variation, blank correction, etc.), irradiation (interfering isotopes, flux gradient, etc.). Furthermore systematic errors due to measurements at two different spectrometers are taken into account (CH5: constant acceleration voltage, variable magnet field to separate isotopes, electron multiplier to determine isotope abundance ratios; GD150: variable acceleration voltage, permanent magnet, Faraday cup to measure isotope abundance ratios). The only (systematic) error which is not included is the uncertainty of the decay constants derived from the Beckinsale and Gale (1969) compilation – they were added and listed in parentheses in the further text – keeping in mind, that a redetermination of the ^{40}K decay parameters is necessary (e.g. Min et al., 2000; Begemann et al., 2001).

4.2.1. NL-25

The age of the hornblende standard NL-25 of 2657±4 Ma agrees very well within the 1σ-error with the age values given in Schaeffer and Schaeffer (1977) and Husain (1974) of 2660±9 Ma and 2650±9 Ma, respectively, and is higher than the value given by Jourdan and Renne (2007) of 2635±19 Ma, but indistinguishable on the 2σ error level. Including the error of the decay constants would increase the standard error to 6 Ma.

Table 7
Monitor ages calculated using values of Table 6

Monitor	Calculated mean ages [Ma]	Lit. age [Ma]	Difference calc. age−lit. age [%]	Error calc. ages [%]	Error lit. ages [%]	Ages obtained by iterative least square fitting of initial values [Ma]	Recommended ages [Ma]
NL-25	2656.6±3.6	2660±9	−0.12	0.13	0.3	2657.4±1.9	2657±4
Hb3gr	1074.9±3.5	1073.6±4.6	+0.12	0.3	0.4	1075.4±2.2	1075±4
BMus/2	329.2±2.2	328.5±1.1	+0.21	0.7	0.3	328.6±1.0	328.5±1.1
SB-3	163.1±0.7	162.9±0.8	+0.12	0.4	0.5	162.8±0.4	
GA1550	98.79±0.43	98.79±0.54	±0	0.4	0.5	98.89±0.22	
HD-B1	24.20±0.09	24.21±0.32	−0.04	0.4	1.3	24.18±0.04	24.18±0.09

Values in column 7 were obtained by least square fitting: The standard causing the largest χ^2-deviation was minimized by fitting its age, the procedure was repeated until convergence.

4.2.2. Hb3gr

The Hb3gr standard result of 1075±4 (6) Ma differs by only 0.12% compared to the literature age of 1073.6±4.6 Ma given by Jourdan et al. (2006) and confirmed by Jourdan and Renne (2007) and agrees within 1σ-error limit.

4.2.3. BMus/2

Using the new MMhb-1 age of 523.1±2.6 Ma (see 'Mineral standards BMus/2' section) the recalculated age of Rittmann (1984) of the laboratory muscovite standard BMus/2 of 328.5±1.1 Ma. This is nominally almost identical with our preferred value of 328.6±2.2 (2.4) Ma. Hence, we do not recommend the value obtained in this study, as the standard age of 328.5±1.1 Ma seems still reliable.

4.2.4. SB-3

Our age for biotite standard SB-3 (Table 7) agrees also very well with its literature value of 162.9±0.8 Ma (e.g. Lanphere et al., 1990). Lanphere and Baadsgaard (2001) found for this SB-3 value a FC sanidine and biotite age of 27.57±0.18 Ma which is different to the proposed value for FC sanidine in Jourdan and Renne (2007) of 28.03±0.08 Ma, only concordant at the 3σ-error level. This difference may be due to an underestimated error for SB-3 as discussed in Jourdan and Renne (2007). In this respect it should also be considered that the Jourdan and Renne (2007) and Lanphere and Baadsgaard (2001) calibration studies did not directly compare SB-3 with the GA1550 and Hb3gr standards. These standards were calibrated against Fish Canyon minerals, but while Jourdan and Renne (2007) used the BGC sanidine split, Lanphere and Baadsgaard (2001) used also other sanidine and biotite splits. They give only one age value for BGC sanidine split (measured by total fusion), yielding an age of 27.70±0.07 Ma. The age value of 27.57±0.09 Ma is a mean of certain ^{40}Ar–^{39}Ar stepheating data from different sanidine and biotite splits, while all K–Ar and Ar–Ar total fusion analyses yield 27.57±0.18 Ma, i.e. the values depend on data selection. Finally, the Jourdan and Renne (2007) and Lanphere and Baadsgaard (2001) calibrations were performed in two different laboratories and using two different irradiation setups.

On the contrary, our calibration – that confirms the age value of 162.9 Ma – directly compared SB-3 with the NL-25, Hb3gr and HD-B1 standards and was performed using identical splits of all standards, identical irradiation setups and the same mass spectrometer, thus eliminating a number of possible systematic errors. To conclude we finally mention that even if the SB-3 age would indeed differ by 1.5%, this would lead to a change for the other standards of less than 0.05% of their calculated ages (including the χ^2-fit results).

4.2.5. GA1550

The age value for the fluence monitor GA1550 of 98.79±0.54 (Renne et al., 1998) is also in agreement with our results and that in Jourdan and Renne (2007) against FC sanidine, so we do not recommend refined values here, but note that a value for GA1550 as low as 97.8±0.2 Ma (Baksi et al., 1996) seems not to be consistent.

4.2.6. HD-B1

For HD-B1, our results confirm its age on a higher confidence level, so we recommend 24.18±0.09 (0.10) Ma instead of 24.21±0.32 Ma (Hess and Lippolt, 1994).

5. Summary

The age of the hornblende standard NL-25 was redetermined by the use of 5 other laboratory and interlaboratory mineral standards for ^{40}Ar–^{39}Ar dating with total fusion analysis of multi-grain separates. By multiple measurements it is shown, that the standards HD-B1, SB-3, Hb3gr, GA1550 and NL-25 agree within their 1σ-error limits, and the difference to the literature age values is smaller than 0.2%. This also indicates homogeneity on the chosen weight level.

We recommend a more precise value for the hornblende NL-25 fluence monitor of 2657±4 Ma and confirm the age of our laboratory standard BMus/2 of 328.5±1.1 Ma. For Hb3gr we obtained 1075±4 Ma, confirming a value of 1073.6±4.6 Ma (Jourdan et al., 2006; Jourdan and Renne, 2007). The SB-3 age of 162.9±0.8 Ma (Lanphere et al., 1990), GA1550 of 98.79±0.54 Ma (Renne et al., 1998) are confirmed here with recalculated ages of 162.8±0.4 (163.1±0.7) and 98.89±0.22 (98.79±0.43) Ma. A lower age value for GA1550 of 97.8±0.2 (Baksi et al., 1996) is only hardly consistent. For HD-B1 we recommend 24.18±0.09 Ma instead of 24.21±0.32 Ma (Hess and Lippolt, 1994). Our results also support an age of 523.1±2.6 Ma for the MMHb-1 monitor.

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