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Cameroon Line magmatism: conventional K/Ar and single-crystal laser $^{40}Ar/^{39}Ar$ ages of rocks and minerals from the Hossere Nigo anorogenic complex, Cameroon

J.B. Kamdem [a], M. Kraml [a,b,*], J. Keller [a], F. Henjes-Kunst [b]

[a] *Institut für Mineralogie, Petrologie und Geochemie, Albertstr. 23 b, 79104 Freiburg, Germany*
[b] *Bundesanstalt für Geowissenschaften und Rohstoffe, Stilleweg 2, 30655 Hannover, Germany*

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Abstract

Hossere Nigo is one of several anorogenic plutonic complexes in the north-eastern part of the Cameroon Line (CL), West Africa. It is a dominantly gabbroic ring structure with a core of monzonitic to syenitic intrusive bodies. Previous geochronological work in the CL indicates a time span from 67 to 31 Ma for plutonism and from 42 Ma to Recent for volcanism. No age data for Hossere Nigo were available so far. Therefore, six whole rock samples were dated by the conventional K/Ar method. The model ages obtained range from 82 to 69 Ma but are, however, in conflict with the relative succession of emplacement. We therefore performed $^{40}Ar/^{39}Ar$ laser fusion analyses on single crystals of phlogopite separated from one of the conventionally dated rocks. Their $^{40}Ar/^{39}Ar$ isochron age of 65.0 ± 0.8 Ma (1σ) is about 7 Ma younger than the related whole rock K/Ar date. Elevated whole rock ages are ascribed to hydrothermal overprinting and associated uptake of excess argon. By contrast, the reported Ar/Ar age provides a precise and accurate fix point for an early stage of CL intrusive magmatism.
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Keywords: Cameroon Line; Laser Ar/Ar dating; Anorogenic magmatism; Hossere Nigo

1. Introduction

The continental sector of the Cameroon Line (CL), West Africa is characterized by an alkaline magmatism represented by intrusive anorogenic complexes and volcanic centres (Déruelle et al., 1991). Fig. 1 shows the alignment of these igneous complexes along the CL from Mont Koupé in the SW to Golda Zuelva and Waza at its north-eastern end. Hossere Nigo, one of the major plutonic complexes, is located in the NE part, west of Ngaoundere on the Adamaoua plateau. The present study reports results of K/Ar and Ar–Ar dating of samples taken from four plutonic rocks and two cross-cutting dikes of the Hossere Nigo complex which has not yet been dated.

Present knowledge of the age of CL plutonism is based on a number of Rb/Sr and K/Ar analyses (Table 1). Obtained ages range from 67 ± 2 Ma for a Golda Zuelva granite (Cantagrel et al., 1978) and 66 ± 2 Ma for a granite from Nkogam (Caen-Vachette et al., 1987) to 39 ± 2 Ma for a syenite from Hossere Poli (Lasserre, 1978) (Fig. 1). The 67 Ma whole rock and K-feldspar K/Ar ages of the Golda Zuelva granite (Cantagrel et al., 1978) are corroborated by a convincing seven-point isochron of 66 ± 3 Ma (Jacquemin et al., 1982). By contrast, the 73 Ma isochron for a Mayo Darlé syenite (Nguene, 1982) is only ill-defined and therefore not suitable to set a reliable limit to the beginning of CL magmatism.

The extrusive alkaline volcanism of the CL is younger than the plutonic phase, ranging in age from 42 Ma (Lee et al., 1994) to Recent (Déruelle et al., 1987). A number of high precision $^{40}Ar/^{39}Ar$ analyses (Marzoli, 1996; Marzoli et al., 1999) suggested the onset of silicic volcanic activity in the Western Cameroon Highlands from NE (Oku: 25 Ma) to SW (Sabga: 23 Ma; Bambouto: 18 Ma) as well as younger ages of the compositionally distinct silicic volcanism on the Adamaoua plateau in the Eastern Cameroon Highlands (11-9 Ma). On the basis of reliable Ar/Ar dates Marzoli et al. (1999) conclude that the Cameroon Volcanic Line as a whole may not be

* Corresponding author. Address: Institut für Mineralogie, Petrologie und Geochemie, Albertstr. 23 b, 79104 Freiburg, Germany.

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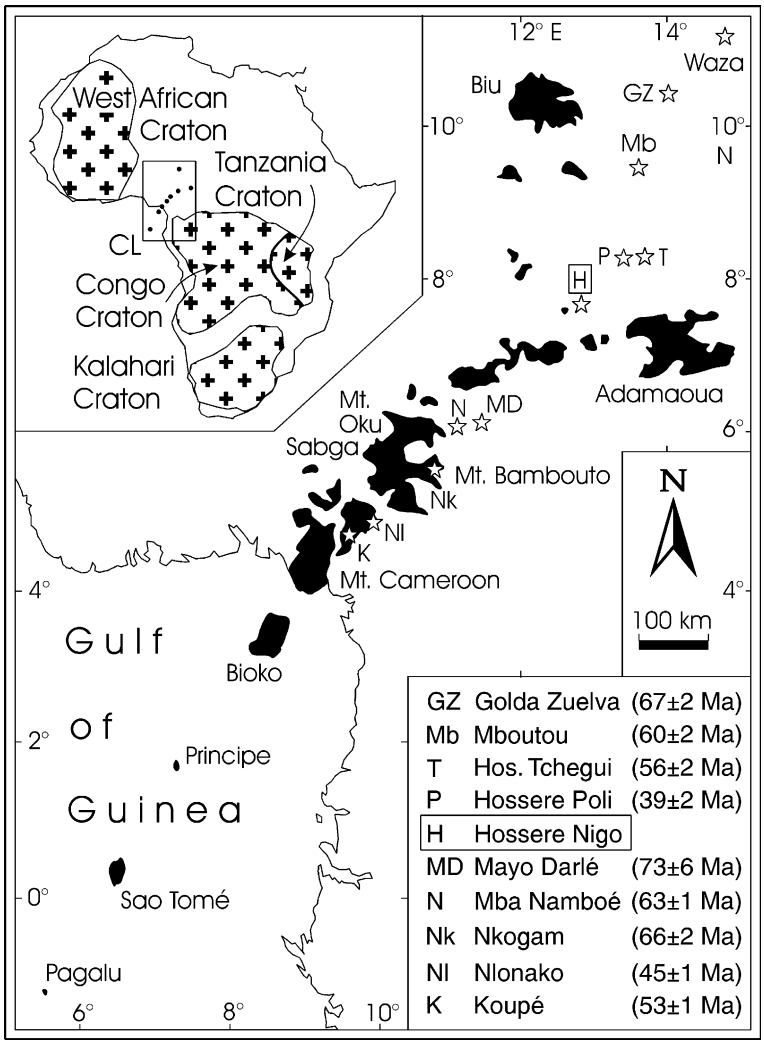

Fig. 1. Location of CL volcanic centres (dark grey) and dated plutons (asterisks) including the studied Hossere Nigo anorogenic complex (slightly modified after Déruelle et al., 1991). Rb/Sr and K/Ar age data given are from Lasserre (1978), Cantagrel et al. (1978), Nguene (1982), Jacquemin et al. (1982), Caen-Vachette et al. (1987), and Lamilen (1989) (see text and Table 1 for details).

interpreted as the surface expression of a simple hot-spot magmatism. This confirms earlier conclusions of e.g. Fitton and Dunlop (1985) and Fitton (1987) drawn from a more restricted data base.

2. Geological setting

Hossere Nigo, a large anorogenic plutonic complex, is located in the north-eastern segment of the CL, near the Y-shaped bifurcation west of Ngaoundere (Kamdem and Keller, 1995; Kamdem, 1996). Hossere Nigo raises 900 m above the surrounding surface of Panafrican basement and has a diameter of ≈7 km. It forms a gabbroic ring complex with hypersthene bearing monzonite to syenite intrusions in the centre (Fig. 2). The plutonic complex is cut by basaltic and trachytic dikes. The intrusive suite is co-genetic as shown by mineralogy, major and trace element geochemistry and Sr and Nd isotope systematics (Kamdem, 1996). Major element composition, alkali-SiO_2 relationship, and the presence of hypersthene in most of the plutonic and volcanic rocks show a weakly alkaline to transitional character of Hossere Nigo (Kamdem, 1996) in contrast to most of the other anorogenic complexes of the CL. Low $\delta^{18}O$ values of Hossere Nigo magmatic rocks indicate an intense hydrothermal overprint (whole rock $\delta^{18}O_{SMOW} = +2.3‰$ to $+5.9‰$; Kamdem, 1996). The geochemical and mineralogical composition of the dated samples is summarized in Table 2.

3. Analytical procedure

K/Ar and Ar/Ar analyses were performed in the geochronology laboratory of the Federal Institute for Geosciences and Resources (BGR) in Hannover, Germany.

Table 1
Previous Rb/Sr and K/Ar determinations of CL intrusive rocks

Locality	Analysed rock	Method	Age (Ma)	Reference
Golda Zuelva	Granite	Rb–Sr	66 ± 3	Jacquemin et al., 1982
	Granite (total rock)	K/Ar	67 ± 2	Cantagrel et al., 1978
	Granite (K-feldspar)	K/Ar	67 ± 2	Cantagrel et al., 1978
	Granite (K-feldspar)	K/Ar	56 ± 2	Cantagrel et al., 1978
Mboutou	Syenite (two-point isochron)	Rb–Sr	60	Lasserre, 1978
	Syenite (K-feldspar)	K/Ar	57 ± 2	Cantagrel et al., 1978
	Granite (total rock)	K-Ar	52 ± 2	Jacquemin et al., 1982
	Quartz monzodiorite (TR)	K-Ar	60 ± 2	Jacquemin et al., 1982
Hossere Tchegui	Syenite	Rb–Sr	43 ± 6	Lasserre, 1978
	Syenite (biotite)	K/Ar	37 ± 1	Cantagrel et al., 1978
	Syenite (total rock)	K/Ar	34 ± 1	Cantagrel et al., 1978
	Gabbro (total rock)	K/Ar	49 ± 2	Cantagrel et al., 1978
	Gabbro (biotite)	K/Ar	56 ± 2	Cantagrel et al., 1978
	Syenite (total rock)	K/Ar	37 ± 1	Cantagrel et al., 1978
	Syenite (total rock)	K/Ar	33 ± 1	Cantagrel et al., 1978
	Syenite (total rock)	K/Ar	33 ± 1	Cantagrel et al., 1978
Hossere Poli	Syenite	Rb–Sr	39 ± 2	Lasserre, 1978
	Granite (K-feldspar)	K/Ar	31 ± 1	Cantagrel et al., 1978
Mayo Darlé	Granite (K-feldspar)	K/Ar	50 ± 1	Cantagrel et al., 1978
	Granite	Rb–Sr	63 ± 1[a]	Lasserre, 1978
	Syenite	Rb–Sr	73 ± 6	Nguene, 1982
	Rhyolite	Rb–Sr	67 ± 7	Nguene, 1982
	Granite	Rb–Sr	55 ± 1	Nguene, 1982
Mba Namboé	Granite	Rb–Sr	63 ± 1[a]	Lasserre, 1978
	Granite (K-feldspar)	K/Ar	49 ± 1	Cantagrel et al., 1978
Nkogam	Granite	Rb–Sr	66 ± 2	Caen-Vachette et al., 1987
Bana	Granite (two-point isochron)	Rb–Sr	30	Lasserre, 1978
Mont Nlonako	Syenite, granite, rhyodacite	Rb–Sr	43.3 ± 0.3[b]	Lasserre, 1978
	Syenite (total rock)	K/Ar	45 ± 1	Cantagrel et al., 1978
	Granite (total rock)	K/Ar	35 ± 1	Cantagrel et al., 1978
	Micro-granite (total rock)	K/Ar	43 ± 2	Cantagrel et al., 1978
Mont Koupé	Syenite with fayalite	Rb–Sr	50 ± 1	Lamilen, 1989
	Syenite and micro-syenite	Rb–Sr	53 ± 1	Lamilen, 1989
	Syenite	Rb–Sr	46 ± 1	Lasserre, 1978

[a] Note that data points from mineralogical and chemical comparable granites of both massifs are used to calculate a common isochron for Mayo Darlé and Mba Namboé.

[b] Note that data points from different plutonic rocks and a rhyodacite are used to calculate an isochron for Mont Nlonako without an independent indication of co-genetic formation.

Conventional K/Ar measurements were performed on a modified MAT CH4 mass spectrometer operated in static mode and a modified CORNING flame photometer with Hamilton dilutor (Seidel et al., 1982). Whole rock samples were crushed, sieved and the ultrasonically cleaned 0.5–1 mm grain size fractions were used for the measurements. Argon was extracted from 600–1200 mg aliquots at temperatures of ≈1600 °C. Ages were calculated using the constants recommended by Steiger and Jäger (1977).

The $^{40}Ar/^{39}Ar$ analyses were performed with the BGR Ar/Ar laser probe. The instrument consists of a 12 W YAG-Nd laser system (Baasel Lasertech), a stainless steel ultra-high vacuum line for gas extraction and purification and a VG Micromass 3600 mass spectrometer (Kraml et al., 1996; Kraml, 1997). Discrimination correction was based on analyses of atmospheric argon of different partial pressures for the different collectors used ($\{^{40}Ar/^{36}Ar\}_{atm} = 285 \pm 3$ for secondary electron multiplier and 300 ± 1 for Faraday cup). Line blanks were measured prior to almost every sample and then used for signal correction.

The selected phlogopite and K-feldspar crystals, generally mm sized, were ultrasonically cleaned, dried at 95 °C, wrapped in Al-foil and filled between two standards in a quartz glass ampoule. The ampoule was evacuated to $\approx 7 \times 10^{-3}$ Pa and sealed.

Neutron irradiation under 1 mm Cd-shielding was performed in the 5 MW research reactor FRG-1 Geesthacht (Germany) for 96 h (fast n-dose of $\approx 10^{18}$ n/cm^2). MMhb-1 hornblende (520.4 Ma; Samson and Alexander, 1987) was used as fluence monitor. This K-poor irradiation standard has a low ^{39}Ar content and shows some heterogeneity at the single grain scale (e.g. Deino et al., 1988; and own results). To reduce the effects of this heterogeneity and to obtain higher ^{39}Ar intensities two grains were fused together in duplicate or triplicate measurements for each standard position. No

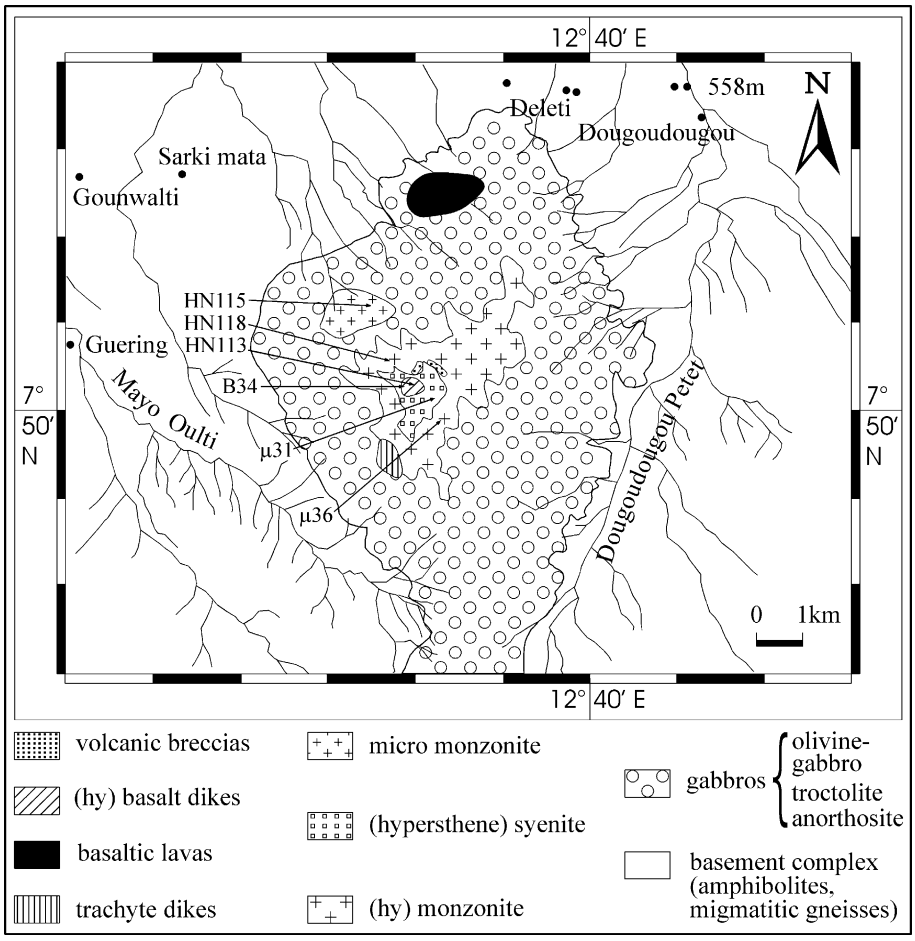

Fig. 2. Geological map of Hossere Nigo magmatic complex with sample locations.

Table 2
Chemical and mineralogical composition of dated samples from Hossere Nigo

No.	HN118	μ36	HN115	B34	HN113	μ31
SiO_2	53.82	58.15	56.09	46.73	49.84	54.42
TiO_2	2.44	1.61	2.19	4.33	1.81	2.45
Al_2O_3	15.28	16.25	15.14	14.45	11.87	15.48
Fe_2O_3	4.10	4.37	4.66	5.46	3.94	5.00
FeO	5.88	3.83	4.95	8.14	8.08	5.19
MnO	0.18	0.17	0.16	0.17	0.15	0.18
MgO	2.84	1.81	2.72	4.46	11.20	2.84
CaO	5.41	4.31	5.17	9.72	9.68	5.45
Na_2O	4.91	5.23	4.45	3.21	2.27	4.85
K_2O	2.05	2.90	2.99	1.01	0.26	2.63
P_2O_5	0.56	0.38	0.57	0.52	0.20	0.57
H_2O	2.18	0.78	0.53	1.53	0.66	0.98
Total	99.65	99.37	99.62	99.73	99.96	100.04
Rock-type	Monzonite (hy-bearing)	Monzonite (hy-bearing)	Micro-monzonite	Basalt	Basalt (hy-bearing)	Syenite (hy-bearing)
Mineralogy	plag, kfsp	plag, kfsp	plag, kfsp	plag, ±kfsp	plag	kfsp ≫ plag
	cpx, opx	cpx, opx	cpx	cpx	cpx, opx	cpx, opx
			qz		ol	qz
	bi		bi			bi
	amph	amph				amph
	Fe–Ti-ox	Fe–Ti-ox	Fe–Ti-ox	Fe–Ti-ox	Fe–Ti-ox	Fe–Ti-ox

plag = plagioclase; kfsp = K-feldspar; cpx = augite; opx = hypersthene; qz = quartz; ol = olivine; bi = biotite/phlogopite; amph = amphibole; Fe–Ti-ox = magnetite/ilmenite.

Table 3
Conventional K/Ar data for six whole-rock samples from Hossere Nigo

Sample	Rock-type	K (%) $\pm 1\sigma$	$^{40}Ar^{*}$ (10^{-7} cm^3/g) $\pm 1\sigma$	Model age (Ma) $\pm 1\sigma$
HN 118	Monzonite	1.661 ± 0.007	50.1 ± 0.2	76.1 ± 0.4
μ36	Monzonite	2.402 ± 0.010	67.7 ± 0.3	71.1 ± 0.4
HN 115	Micro-monzonite	2.457 ± 0.011	67.2 ± 0.3	69.1 ± 0.4
B 34	Basalt	0.813 ± 0.005	24.9 ± 0.2	77.2 ± 0.5
HN 113	Basalt	0.213 ± 0.002	7.0 ± 0.1	82.3 ± 0.9
μ31	Syenite	2.144 ± 0.009	61.0 ± 0.3	71.8 ± 0.4

Atmospheric Ar contents were below 10% except for HN113 (15.7%) and HN118 (23.3%).

significant irradiation gradient was observed across the 2 mm vertical distance between the two standards bracketing the phlogopite sample. This was confirmed by analyses of aliquots of the multigrain intra-laboratory biotite standard SN, placed 1.5 mm above and 4 mm below the sample. The *J*-value was determined with a precision of about 1% ($1.465\ (\pm 0.015) \times 10^{-3}$) and its error is included in the internal 1σ analytical uncertainty (Dalrymple et al., 1981). Interfering Ar isotopes from K and Ca were corrected with the following factors: $(^{40}Ar/^{39}Ar)_K$: $1.14\ (\pm 4.51) \times 10^{-2}$, $(^{39}Ar/^{37}Ar)_{Ca}$: $7.52\ (\pm 0.11) \times 10^{-4}$ and $(^{36}Ar/^{37}Ar)_{Ca}$: $2.7\ (\pm 0.7) \times 10^{-4}$. Intensities of the radioactive isotopes ^{37}Ar and ^{39}Ar were corrected for decay.

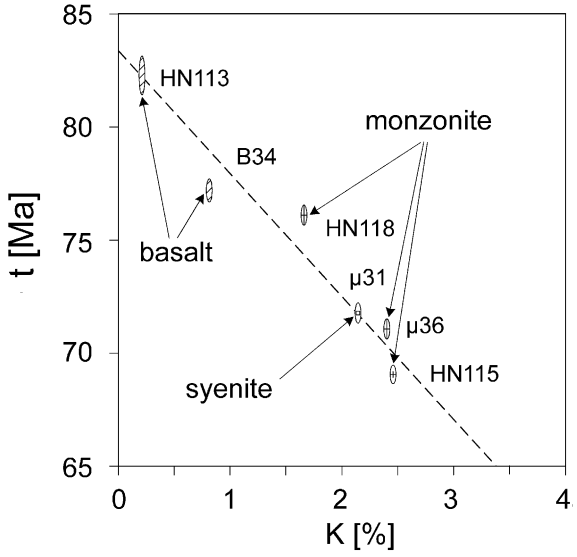

Fig. 3. Negative correlation between conventional K/Ar model ages and K contents of total-rock samples. Note that such a correlation should not exist except for a disturbed K/Ar system where excess argon produces the most striking effect on rocks with low K contents.

4. Results and discussion

K/Ar dating results are reported in Table 3. Ages range from 69.1 ± 0.4 to 82.3 ± 0.9 Ma (1σ). These precise model K/Ar dates are distinctly older than previously published ages for other anorogenic complexes along the CL. However, they are in conflict with the relative succession of emplacement. Basaltic dikes cutting the plutonic complex yield the highest model ages of up to 82 Ma. Furthermore, there is an apparent negative correlation between age values and K content, which means that K-rich rocks give low ages and vice versa (Fig. 3). This indicates that the dates are systematically in error. As even the most pristine among the dated whole rocks are at least partly altered, elevated ages and negative correlations of age and K content may be attributed to a secondary overprint and associated incorporation of excess argon.

Because alteration can seriously affect the K/Ar system, we decided to perform $^{40}Ar/^{39}Ar$ laser fusion analyses on ten phlogopite crystals (Table 4 and Fig. 4) and four K-feldspar grains (Table 5 and Fig. 5) separated by

Table 4
Results of laser $^{40}Ar/^{39}Ar$ analyses on ten individual phlogopite crystals from hypersthene syenite μ31

No. of grain	^{39}Ar (%)	$^{40}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$	$^{40}Ar^{*}/^{39}Ar_K$	$^{40}Ar^{*}$ (%)	Date $\pm 1\sigma$ (Ma)
1	28.46	25.835	0.00297	24.957	96.6	64.8 ± 0.9
2a Rim	0.96	28.414	0.01026	25.381	89.3	65.8 ± 1.5
2b Core	7.10	26.205	0.00218	25.562	97.5	66.3 ± 1.3
3a Rim	5.09	26.468	0.00492	25.015	94.5	64.9 ± 1.3
3b Rim	6.23	26.370	0.00409	25.161	95.4	65.3 ± 1.3
3c Core	5.93	25.647	0.00203	25.048	97.7	65.0 ± 1.3
4–10	46.23	26.485	0.00406	25.285	95.5	65.6 ± 1.0
Mean age:						65.3 ± 0.8

Grains 1 and 4–10 were analysed using a Faraday cup and grains 2 and 3 using a secondary electron multiplier. Note that ^{37}Ar was below detection limit.

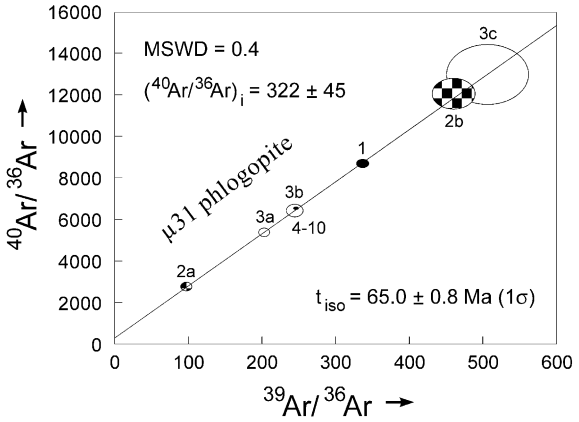

Fig. 4. $^{40}Ar/^{39}Ar$ isochron (calculated according to Kent et al., 1990) for phlogopite crystals separated from hypersthene syenite μ31. Numbers above/below data points refer to analysed grains.

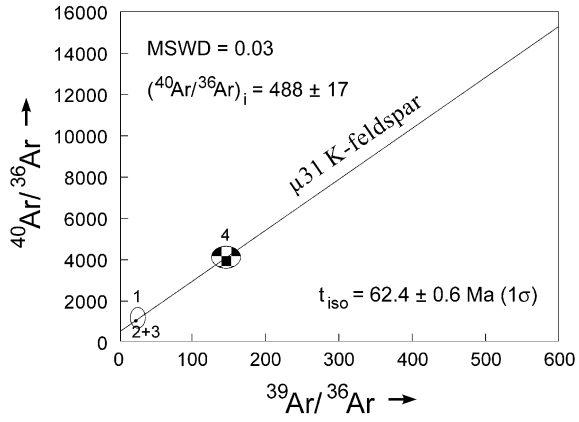

Fig. 5. $^{40}Ar/^{39}Ar$ isochron diagram for K-feldspar crystals separated from hypersthene syenite μ31. Numbers above/below data points refer to analysed grains. Unusual small MSWD is due to the low number of data points.

Table 5
Results of laser $^{40}Ar/^{39}Ar$ analyses on four K-feldspar crystals from hypersthene syenite μ31

No. of grain	^{39}Ar (%)	$^{40}Ar/^{39}Ar$	$^{36}Ar/^{39}Ar$	$^{40}Ar^*/^{39}Ar_K$	$^{40}Ar^*$ (%)	Date ± 1σ (Ma)
1	0.97	46.546	0.04575	33.026	71.0	85.2 ± 14.3
2 + 3	60.98	48.042	0.04922	33.497	69.7	86.4 ± 2.0
4	38.05	27.973	0.00806	25.592	91.5	66.4 ± 1.5
Mean age:						78.8 ± 1.4

All grains were analysed utilizing secondary electron multiplier. Note that ^{37}Ar was below detection limit. Grains 1–3 were cloudy and showed homogeneously distributed inclusions of alteration products whereas grain 4 was transparent and did not exhibit any signs of alteration (see text for further discussion).

handpicking from the conventionally dated hypersthene syenite μ31. The analysed micas did not show any signs of alteration and the original stoichiometry with unaltered high K contents (8.2% K) was shown by microprobe data (Kamdem, 1996).

Fused grains as well as selected zones of single crystals reproducibly yielded values ranging from 64.8 ± 0.9 to 66.3 ± 1.3 Ma (1σ). The calculated weighted mean age of 65.3 ± 0.8 Ma (1σ) is in excellent agreement with the isochron age of 65.0 ± 0.8 Ma (1σ). Given this reliable date, it is possible to calculate an excess Ar content of 5.9×10^{-7} cm^3/g (= 9.7% of non-atmospheric argon) present in the μ31 total rock. Assuming that the other samples have incorporated equivalent amounts of gas, the corrected dates for the plutonic rocks are 67 Ma (HN118), 65 Ma (μ36), and 63 Ma (HN115). Thus, an cooling age of ≈65 Ma is suggested for the entire Hossere Nigo plutonic complex.

To localize the siting of excess argon we fused four K-feldspar grains showing different grades of alteration (Table 5), ranging from pristine to completely decomposed.

The grains yielded apparent ages between 66.4 ± 1.5 and 86.4 ± 2.0 Ma. The calculated weighted mean age of all K-feldspar crystals is 78.8 ± 1.4 Ma. The lowest date (66.4 ± 1.5 Ma) was obtained from the unaltered grain no. 4 (Table 5) and it agrees well with the phlogopite results. A three point isochron (Fig. 5) corresponds to an age of 62.4 ± 0.6 Ma which is identical to the phlogopite date at 2σ confidence interval. Also the high ratio of the initially trapped argon ($[^{40}Ar/^{36}Ar]_i = 489 \pm 17$) suggests that excess argon, responsible for the high apparent ages, is present in the altered K-feldspars. The K-feldspar and phlogopite isochrons have the same slope (i.e. same age), indicating that the excess Ar is homogeneously distributed in the K-feldspars.

Generally, an alteration event resets the K/Ar chronometer towards younger ages because of complete or partial Ar loss (e.g. Goll et al., 1995). The anomalously high ages at Hossere Nigo are explained not only by uptake of excess argon but also by a net potassium loss from the total rock during hydrothermal alteration. In a further paper we will explain this rarely described feature of excess argon combined with potassium loss in greater detail.

5. Conclusions

Compared to known emplacement ages of other anorogenic complexes along the CL, the 65 Ma old Hossere Nigo complex represents one of its earliest

magmatic events. This observation reinforces the evidence that the magmatic record of the CL extends back to the K/T boundary (e.g. Déruelle et al., 1991).

All conventionally dated total rocks from Hossere Nigo showed more or less elevated K/Ar ages, depending on their K-content. Hydrothermal fluids are made responsible for changing both the argon isotopic composition and the potassium balance. As also described by McConville et al. (1994) and Goll et al. (1995) microscopically fresh, mm sized phlogopite crystals are not significantly affected by this process.

Systematic Ar/Ar studies on mineral separates from other plutonic complexes are needed to reveal the influence of this age-modifying effect along the entire CL. This would improve our knowledge of the ages of individual anorogenic complexes and enable us to decide, whether the plutonic activity of the CL shows any systematic migration in space and time.

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