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^{39}Ar–^{40}Ar DATING: THE IMPORTANCE OF K-FELDSPARS ON MULTI-MINERAL DATA OF POLYOROGENIC AREAS[1]

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ABSTRACT

A comparative ^{39}Ar–^{40}Ar study of two hercynian crystalline segments in southern France is presented in this work. The first area, the Montagne Noire, has been left undisturbed since ca. 300 m.y. while the second, the Eastern Pyrénées, has been severely overprinted by alpine metamorphism about 95 m.y. ago. In the Montagne Noire, all the analyzed minerals (micas and low-temperature K-feldspars) have preserved hercynian age plateaux whereas in the Eastern Pyrénées, hercynian minerals (micas, amphiboles, plagioclases, and low-temperature K-feldspars) have been outgassed to a variable extent. Low-temperature K-feldspars are shown to be a sensitive indicator of tenuous thermomechanical disturbance, even for samples whose biotite is apparently undisturbed. This is considered to result from lowering of the effective grain size of the perthitic crystals by the tectonic event and not from an abnormally high diffusion coefficient for argon. An evaluation of this grain size is proposed which uses the recoil effect of ^{39}Ar production in the neutron irradiation. We conclude that discordant mica ages may be interpreted as cooling ages only after the associated K-feldspars have been shown to have suffered a slight disturbance or none.

INTRODUCTION

The development of ^{39}Ar–^{40}Ar dating on terrestrial rocks of complex history has not resulted so far in a clear chronological methodology as it did for extraterrestrial materials. The stepwise heating method has allowed us to point out some complexities, which the conventional K-Ar technique had overlooked. However, its ability to unravel the apparently simple succession of two orogenic or thermal events on the same sample—a presumed property which has initiated most of the recent developments—still requires extensive experimental investigations.

A large part of the ^{39}Ar–^{40}Ar terrestrial geochronology has been devoted to the study of samples which underwent a simple two-phase history; a definite crystallization time is separated from a subsequent event, either contact metamorphism (Hanson et al. 1975; Berger 1975) or regional metamorphism (Dallmeyer 1975*c*), by a period of thermomechanical quiescence. Even those simple cases did not lead to unambiguous results. Generally speaking, the common minerals which may be taken as undisturbed or thoroughly reequilibrated (e.g., recrystallized) by geological or petrographical evidence, furnish well defined age plateaux, with the exception of low-temperature K-feldspars. In the minerals which have been only partially overprinted by the subsequent event, the presence or absence of age plateaux of geological meaning is still a matter of debate. Berger (1975) has shown that hornblende could exhibit age plateaux in the metamorphic aureole of the Eldora Stock, which have no direct geological significance. Hanson et al. (1975) made the same statement for the biotites for rocks near the contact of the Duluth Gabbro. The latter authors argue that the rate controlling process for natural argon loss is volume diffusion, while for experimental extraction under vacuum, out-

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gassing is controlled by dehydroxylation, thus giving artificial age plateaux of intermediate character. This interpretation of biotite plateaux has been challenged by Berger (1975) and Dallmeyer (1975*c*) who claim that fine scale irregularities can always be detected in the age spectra of partially overprinted biotites. According to Dallmeyer (1975*a*, *b*), the age plateaux observed in metamorphic minerals which indicate ages intermediate between recognized geological events must be interpreted as cooling ages associated with different erosion rates.

The present work was aimed at comparing the ^{39}Ar–^{40}Ar geochronology of two areas in which the orogenic history is different but well controlled by independent observations and geochronological measurements, the hercynian granitoids of the Montagne Noire (Southern Massif Central, France), where no noticeable event has affected the hercynian geochronological pattern, and the hercynian basement of the Eastern Pyrénées where the effects of the alpine orogeny are well characterized. The behavior of various minerals has been investigated, with special emphasis on the micas and low-temperature K-feldspars.

GEOLOGICAL SETTING

1. *The Montagne Noire* (fig. 1).—The Montagne Noire range is the southernmost part of the hercynian Massif Central (France), in which some relic nuclei of previous orogenic cycles have been found. This area has been preserved from any significant effect of the alpine events. A geological account of this region may be found elsewhere (Gèze 1949, Arthaud 1970). Geochronological investigations have been carried out mainly by Hamet and Allègre (1976) using the Rb–Sr method.

The hercynian metamorphism is mostly restricted to the axial zone. Hamet and Allègre (1976) interpreted the age given by the anatexite isochron (330 m.y.) as the top of the metamorphic peak. Mica Rb–Sr ages (282–286 m.y.) are viewed by these authors as cooling ages below approximately 300–400°C.

The granitoids of the Montagne Noire are divided into two distinct series. Early muscovite bearing granites (Anglès, Soulié, Martys, Brousse, Lampy) were emplaced in the axial zone as syn- or late-tectonic intrusions and have yielded a Rb–Sr whole rock isochron of ca. 330 m.y. Calcalkaline, post-tectonic granitoids (Folat and Sidobre) were intruded in the northern zone. Their Rb–Sr whole-rock isochron ages cluster around 285–290 m.y.

2. *The Eastern Pyrénées* (fig. 2).—The Pyrénées may be represented by approximately rectilinear, fan-shaped structures trending WNW–ESE. Complete geological accounts are available elsewhere (Casteras 1933; Ravier 1959; Guitard 1965; Mattauer 1968; Choukroune 1974). We are interested here in the easternmost part of the French Pyrénées.

The present structures of the Pyrénées are the product of two main orogenic cycles. The hercynian orogeny has strongly affected the axial zone and North-Pyrenean Massifs by multiphase folding and metamorphism locally reaching the catazone. During the same episode, two series of granitoids were emplaced, closely following the spatial, temporal and petrographic scheme previously depicted for the Montagne Noire (Vitrac-Michard and Allègre 1975).

The pyrenean (alpine) cycle encompasses two major phases. The first tectonometamorphic event of middle Cretaceous age is centered on the Mesozoic trough between the axial zone and North-Pyrenean Massifs. Recent ^{39}Ar-^{40}Ar and ^{87}Rb–^{87}Sr investigations (Albarède 1976; Albarède and Michard-Vitrac, submitted for publication) performed on metamorphic "pyrenean" minerals have given concordant results in the range 92–104 m.y. This metamorphism is of very low pressure type and the associated deformation is characterized by tight and supple folding. Most of the present structures were created during late Eocene tectonic phase. This latter phase is characterized by shallow deformation and is almost free of thermal anomaly. Finally, tensional faulting was locally active in the easternmost Pyrénées during the late Cenozoic period.

ANALYTICAL TECHNIQUE

The samples have been irradiated in the Osiris reactor at Saclay, France. The total flux average is 1.3×10^{14} neutrons/cm^2/s of which

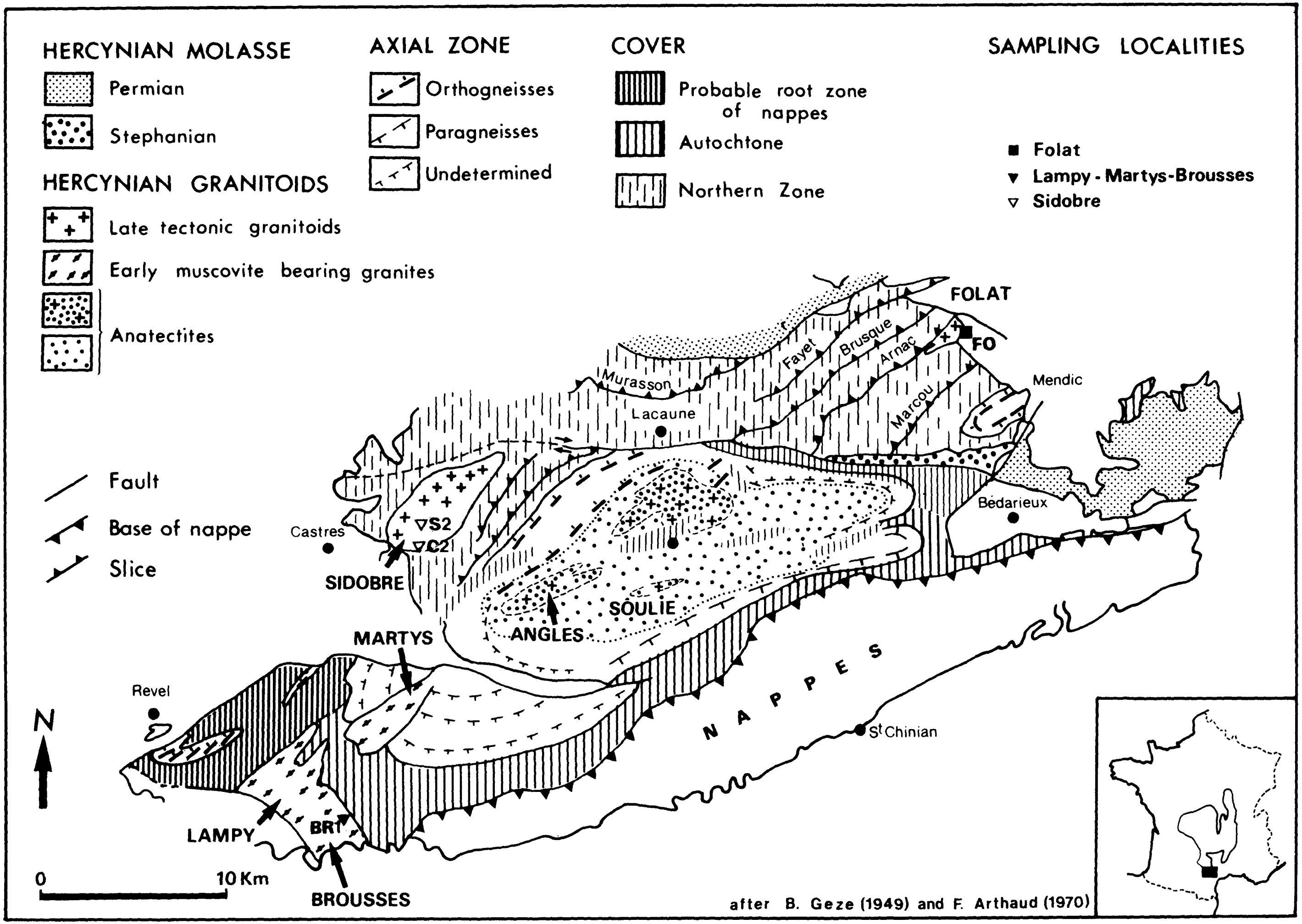

FIG. 1.—Geological sketch map of the Montagne Noire with sampling localities

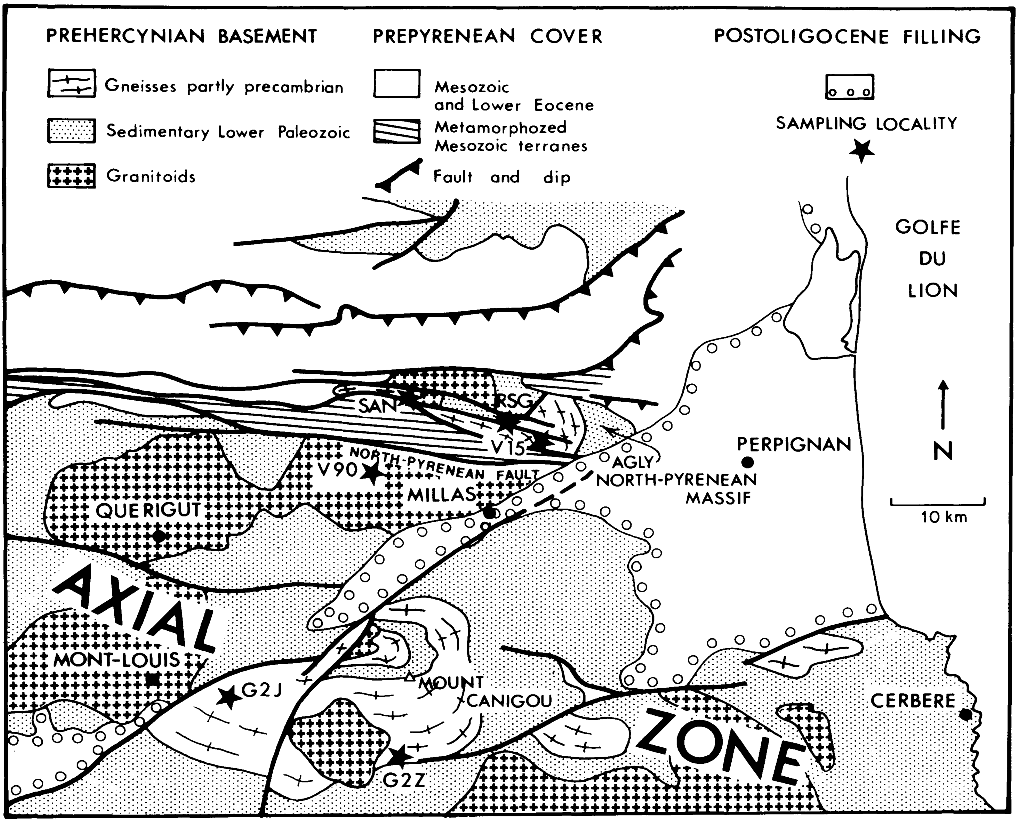

FIG. 2.—Geological sketch map of the Eastern Pyrénées with sampling localities

20% have an energy higher than the absorption threshold of cadium. Five to nine samples were put in each reactor can along with three biotite monitors.

Samples were subjected to an integrated neutron fluence of about 6×10^{17} fast neutrons per cm^2. The temperatures during irradiation were estimated by introducing metallic fuses to be about 100°C.

Fused calcium fluoride was irradiated together with each sample set containing calcium rich minerals in order to insure proper interference corrections.

In the present procedure, argon is extracted from the samples by induction heating in a molybdenum crucible during ca. 50 mn long steps. Temperatures above 800°C are measured to $\pm$ 50°C with an optical pyrometer. Below this temperature, poorer estimations are inferred by extrapolation of the power supply. Active gases are then removed by threefold gettering on titanium, while helium and neon are pumped away.

Argon is introduced in a modified CAMECA THN 205 mass spectrometer equipped with magnetic scanning, peak switching and electron multiplier detection. The data are collected in the static mode. Typical standard deviations on linearly extrapolated isotopic ratios amount to 1%. Absolute amounts are not known to better than 20%.

Blanks are measured between each sample run. Typical blanks between 1,000 and 1,300°C are 4×10^{-13} mole for ^{40}Ar and 2×10^{-15} mole for ^{39}Ar. Within the error bars, the $^{40}Ar/^{36}Ar$ ratio of the blank cannot be distinguished from the air ratio. So, we have lumped ^{40}Ar and ^{36}Ar of the blank with conventional

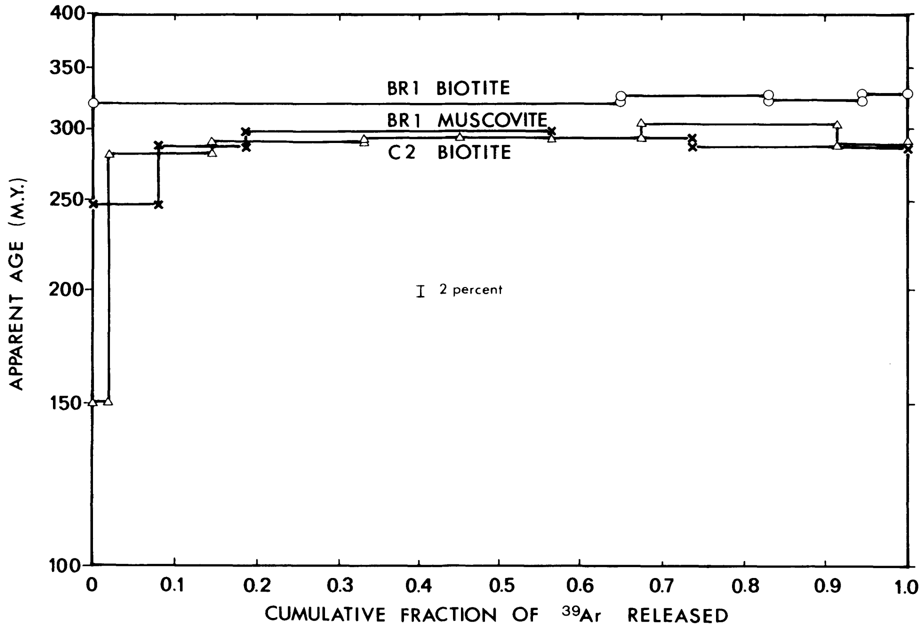

FIG. 3.—Age spectra of Montagne Noire micas

atmospheric contamination. ^{39}Ar was corrected by using the mass ratio 41/39 which equals 2.0 ± 0.3 in the blank.

The monitor is a biotite from the Southern Alps near Lugano. Standard K–Ar determinations by us and Maluski (personal communication) as well as comparison with K–Ar results on neighboring rocks by McDowell (1970), were in good agreement at 310 ± 10 m.y. The Rb–Sr age of this mineral is 300 ± 5 m.y. (Hamet and Albarède 1973).

RESULTS

Results are listed in tables 1 and 2. For some minerals, the ^{37}Ar isotope has only been measured with a poor accuracy. These are, however, potassium rich minerals, for which it is readily verified that interference corrections never significantly alter the results (less than 1%).

For the sake of comparison, Rb–Sr data obtained on the same minerals by Hamet and Allègre (1976) for the Montagne Noire and Vitrac-Michard and Allègre (1975 and unpublished results) for the Eastern Pyrénées have been reported in table 3.

1. *Montagne Noire.*—The age spectrum of the biotite C2 (fig. 3), extracted from a post-tectonic granodiorite (Sidobre), shows within the error bars an age plateau at 292 m.y., in agreement with its Rb–Sr age (292 m.y.) as well as with the whole rock isochron age of the massif (283 m.y.).

The biotite BR1 is a mineral from a syntectonic muscovite bearing granite (Brousses massif). The age plateau (322 m.y.) is in better agreement with the whole rock isochron age by Rb–Sr method (330 m.y.) than with the corresponding Rb–Sr age of the pair biotite-whole rock (278 m.y.). Conversely, the muscovite from the same sample shows a maximum age of 298 m.y. which is intermediate between the whole rock isochron age and the Rb-Sr age of the muscovite-whole rock pair (275 m.y.).

The three analyzed K-feldspars (fig. 4) pertain to the microcline-orthoclase series. The K-feldspar C2 and S2 from the Sidobre granodiorite display different age spectra, even

TABLE 1

Temperature °C	$^{40}Ar_{atm}$ %	$^{36}Ar/^{37}Ar$	$^{40}Ar_{rad}/^{39}Ar$	% ^{39}Ar Cumulated	Apparent Age (m.y.)
S2 K-Feldspar					
600	94.5	n.d	32.0	0.03	275±90
650	64.1	n.d	71.1	0.15	560±30
700	21.2	n.d	41.0	0.90	412±12
750	9.1	n.d	32.1	2.5	275±10
800	5.5	n.d	31.9	7.0	273± 4
920	2.5	n.d	31.8	18.5	272± 4
1050	1.7	n.d	31.5	29.0	270± 4
1120	2.2	n.d.	31.2	40.0	267± 4
1200	1.9	n.d.	31.8	54.5	271± 3
1340	1.2	n.d.	32.5	100.0	278± 3
				Total fusion age:	275
C2 Biotite					
600	66.8	12.0	15.1	0.5	148±10
650	26.5	3.4	15.4	2.8	151± 4
700	3.4	5.8	29.8	14.5	282± 4
770	0.8	5.0	30.8	33.0	291± 4
850	1.9	5.4	31.1	45.0	294± 4
950	1.2	0.7	31.1	67.5	293± 4
1080	0.8	0.5	32.2	91.5	303± 4
1160	3.9	0.25	30.5	99.5	288± 8
1250	35.2	4.5	29.4	100.0	279±16
				Total fusion age:	290
C2 K-Feldspar					
650	21.9	3.2	29.3	1.7	277± 6
750	9.3	0.82	30.3	7.0	287± 4
780	4.9	0.33	30.6	13.5	289± 4
830	4.5	0.19	31.1	19.5	293± 4
850	7.8	0.33	30.8	22.0	291± 4
970	6.6	0.30	30.6	30.0	289± 4
1080	7.5	0.74	30.4	40.0	287± 4
1160	4.5	0.73	31.9	69.5	300± 4
1270	3.1	0.41	32.5	99.5	306± 5
1350	64.5	1.0	31.2	100.0	294±16
				Total fusion age:	297
FO K-Feldspath					
750	31.0	n.d.	33.2	1.5	283± 6
790	22.8	n.d.	33.8	3.5	288± 6
830	10.4	n.d.	33.4	6.0	285± 5
860	5.4	n.d.	32.9	8.0	281± 4
890	9.3	n.d.	33.0	11.0	282± 4
900	6.4	n.d.	32.5	12.5	278± 5
960	3.0	n.d.	32.9	17.0	281± 4
1050	2.6	n.d.	32.6	25.5	281± 4
1150	1.3	n.d.	32.5	32.5	278± 4
1250	2.9	n.d.	31.4	54.0	269± 4
1350	5.4	n.d.	32.6	100.0	279± 6
				Total fusion age:	277

TABLE 1—*Continued*

Temperature °C	$^{40}Ar_{atm}$ %	$^{36}Ar/^{37}Ar$	$^{40}Ar_{rad}/^{39}Ar$	% ^{39}Ar Cumulated	Apparent Age (m.y.)
BR1 Biotite					
980	1.1	0.45	72.5	65.0	320 ± 8
1050	0.7	0.15	73.7	83.0	325 ± 8
1120	0.5	0.06	73.0	94.5	322 ± 8
1300	1.7	0.05	74.2	100.0	327 ± 12
				Total fusion age:	322
BR1 Muscovite					
850	8.4	0.22	55.5	8.0	248 ± 17
940	3.3	0.16	64.5	18.5	287 ± 16
1030	1.3	0.23	67.0	56.5	298 ± 11
1100	2.5	0.21	65.5	73.5	292 ± 11
1300	3.6	0.11	64.1	100.0	286 ± 10
				Total fusion age:	289

NOTE.—Experimental results for analyzed samples from Montagne Noire. ^{37}Ar is corrected for decay. For some samples this isotope was not determined with acceptable accuracy (n.d. = not determined) due to high K/Ca ratio and/or excessive time elapsed after irradiation. Errors on the ages of individual fractions are quoted at $\pm 2\sigma$.

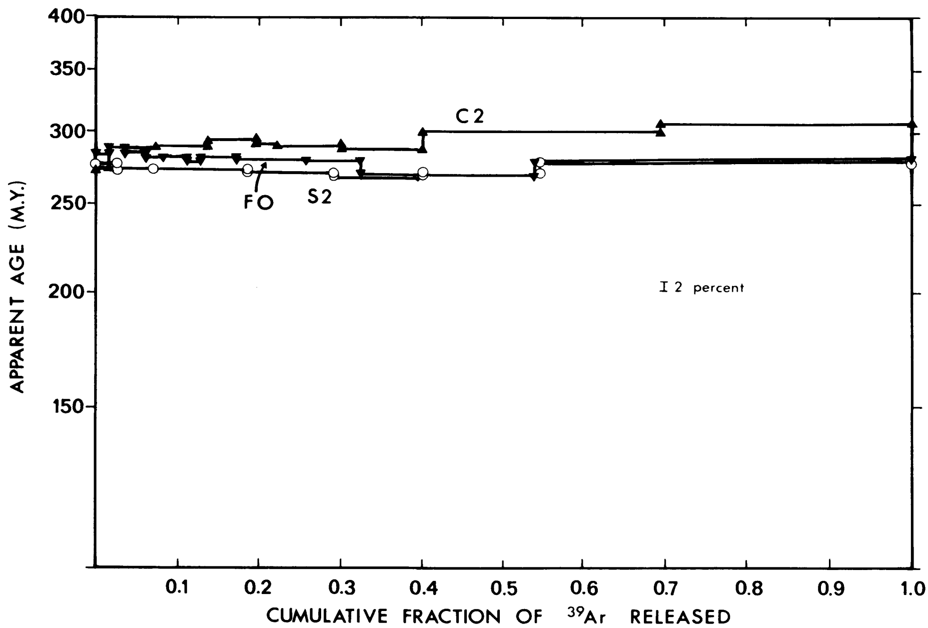

FIG. 4.—Age spectra of Montagne Noire K-feldspars

though they are mineralogically similar. The ages of the successive fractions for C2 show an irregular increase from 287–306 m.y. The spectrum of sample S2 has a slightly saddle shaped structure, but most fraction ages fall in the range 267–278 m.y. The sample F0 is a K-feldspar separated from a microgranite dyke associated with the Folat granodiorite. The same slightly saddle shaped structure is found for this mineral. All but one fraction give ages between 278 and 288 m.y.

2. *Eastern Pyrénées.*—(*a*) Micas (fig. 5).

TABLE 2

Temperature °C	$^{40}Ar_{atm}$ %	$^{36}Ar/^{37}Ar$	$^{40}Ar_{rad}/^{39}Ar$	% ^{39}Ar Cumulated	Apparent Age (m.y.)
G2J Biotite					
700	57.7	n.d.	6.3	2.0	57 ± 12
800	14.5	n.d.	7.64	19.0	69 ± 4
850	5.4	n.d.	7.62	53.5	69 ± 2
900	45.3	n.d.	6.56	55.5	59.5 ± 7
1050	20.5	n.d.	7.01	61.0	63.5 ± 4
1250	8.7	n.d.	7.06	97.0	64.0 ± 3
1400	44.7	n.d.	7.29	100.0	66 ± 9
				Total fusion age:	66
G2J Muscovite					
650	82.6	n.d.	17.7	0.5	155 ± 45
800	36.7	n.d.	13.8	3.0	123 ± 10
850	25.0	n.d.	16.2	5.0	144 ± 10
1050	14.8	n.d.	23.5	42.0	205 ± 7
1200	11.3	n.d.	24.4	74.5	213 ± 4
1400	6.5	n.d.	28.4	100.0	245 ± 7
				Total fusion age:	214
G2Z K-Feldspar					
400	68.1	n.d.	14.4	1.8	280 ± 21
550	35.4	n.d.	21.2	2.1	298 ± 25
660	21.1	n.d.	3.7	6.0	75.5 ± 2.0
690	24.7	n.d.	1.58	11.0	32.7 ± 0.9
720	20.6	n.d.	1.59	17.0	32.9 ± 0.7
780	12.7	n.d.	1.47	23.5	30.5 ± 0.5
930	16.6	n.d.	1.74	36.0	36.0 ± 0.5
1050	18.1	n.d.	2.06	51.0	42.5 ± 0.6
1150	15.1	n.d.	3.09	72.5	63.5 ± 0.9
1230	12.0	n.d.	4.08	93.0	83.5 ± 0.9
1270	13.5	n.d.	4.07	98.5	83.2 ± 1.0
1350	31.2	n.d.	5.03	100.0	102 ± 9
				Total fusion age:	63.0
V90 Biotite					
850	0.9	0.13	72.3	41.5	272 ± 11
1000	0.5	0.074	71.7	78.5	270 ± 10
1070	0.9	0.20	74.6	88.0	280 ± 17
1300	0.9	0.070	74.3	100.0	279 ± 16
				Total fusion age:	273.0
V90 K-Feldspar					
300	95.4	n.d.	3.8	0.06	79 ± 54
380	47.3	n.d.	12.8	0.5	250 ± 14
450	21.6	n.d.	3.08	4.5	63.3 ± 1.1
540	10.4	n.d.	2.66	8.0	54.7 ± 1.0
630	20.7	n.d.	2.28	10.5	47.0 ± 0.8
710	11.8	n.d.	2.48	16.0	51.1 ± 0.7
840	4.0	n.d.	3.59	24.5	73.6 ± 0.7
940	3.1	n.d.	5.28	33.5	107 ± 1
1040	2.2	n.d.	9.89	43.0	196 ± 2
1140	1.5	n.d.	12.2	62.5	238 ± 4
1280	1.3	n.d.	13.9	99.0	271 ± 5
1350	25.8	n.d.	12.7	100.0	249 ± 5
				Total fusion age:	192.0

TABLE 2—*Continued*

Temperature °C	$^{40}Ar_{atm}$ %	$^{36}Ar/^{37}Ar$	$^{40}Ar_{rad}/^{39}Ar$	% ^{39}Ar Cumulated	Apparent Age (m.y.)
V15 Biotite					
650	71.0	n.d.	7.34	2.5	66 ± 6
750	7.0	n.d.	11.3	24.0	101 ± 2
850	3.2	n.d.	11.3	63.0	101 ± 2
1050	9.2	n.d.	11.1	72.5	99 ± 2
1150	5.6	n.d.	11.0	88.0	99 ± 2
1300	7.4	n.d.	11.0	100.0	99 ± 2
				Total fusion age:	99.0
V15 Muscovite					
700	77.4	n.d.	10.6	0.3	95 ±14
820	16.1	n.d.	12.6	3.0	112 ± 5
860	8.1	n.d.	12.1	8.0	107 ± 4
900	8.6	n.d.	11.9	13.0	110 ± 6
1050	7.7	n.d.	11.6	44.0	104 ± 5
1200	8.3	n.d.	11.5	97.5	103 ± 5
1300	19.0	n.d.	11.6	100.0	104 ±19
				Total fusion age:	104.0
V15 K-Feldspar					
490	62.3	n.d.	9.40	2.0	187 ±38
540	43.1	n.d.	6.80	4.0	137 ± 8
600	7.2	n.d.	4.00	10.5	82 ± 3
670	5.1	n.d.	3.84	15.5	79 ± 2
740	2.6	n.d.	4.60	28.0	94 ± 3
900	3.9	n.d.	6.4	34.5	129 ± 2
970	4.5	n.d.	7.8	47.5	157 ± 3
1110	3.9	n.d.	7.5	73.5	150 ± 3
1200	4.3	n.d.	6.8	91.5	137 ±10
1370	5.7	n.d.	6.6	100.0	134 ± 3
				Total fusion age:	131.0
RSG Phlogopite					
800	12.9	0.4	21.7	9.5	102 ± 5
920	2.2	0.3	22.8	23.5	107 ± 3
1000	5.6	0.4	23.3	33.5	109 ± 2
1060	4.2	0.2	23.6	41.0	110 ± 2
1100	1.9	0.06	22.6	59.5	106 ± 5
1150	2.5	0.2	23.2	74.0	108 ± 2
1200	2.7	0.1	22.8	89.0	106 ± 3
1250	3.8	0.2	23.2	97.0	109 ± 3
1300	11.0	0.1	23.4	100.0	110 ± 3
				Total fusion age:	107.0
RSG Amphibole					
850	16.8	0.028	131.0	7.5	544 ±65
1020	23.3	0.015	47.0	16.5	214 ±23
1180	2.4	0.0019	94.7	62.5	408 ±20
1260	1.9	0.0017	90.4	73.0	392 ±27
1450	1.6	0.0017	100.2	100.0	429 ±37
				Total fusion age:	405.0

TABLE 2—*Continued*

Temperature °C	$^{40}Ar_{atm}$ %	$^{36}Ar/^{37}Ar$	$^{40}Ar_{rad}/^{39}Ar$	% ^{39}Ar Cumulated	Apparent Age (m.y.)
RSG Plagioclase					
780	22.7	0.0073	61.3	7.0	274 ±35
1000	14.4	0.0038	19.5	39.0	92 ± 8
1120	18.7	0.0085	17.7	74.0	83 ± 9
1240	34.1	0.010	24.2	91.5	113 ±18
1340	35.9	0.0072	46.5	97.0	212 ±39
1450	27.8	0.0052	98.1	100.0	421 ±72
				Total fusion age:	122
SAN Biotite					
600	52.6	0.3	16.9	1.0	78 ± 7
700	12.4	0.16	25.2	4.5	115 ± 3
780	1.9	0.16	33.2	12.0	151 ± 2
820	1.0	0.15	34.6	22.5	157 ± 2
860	1.0	0.15	35.0	30.5	159 ± 2
960	1.5	0.12	35.0	39.0	159 ± 2
1060	1.0	0.032	34.5	56.0	157 ± 2
1150	0.4	0.015	34.3	80.0	155 ± 2
1300	0.9	0.008	36.0	100.0	163 ± 3
				Total fusion age:	155
SAN Amphibole					
850	10.6	0.016	52.3	10.5	201 ±23
1000	0.5	0.0014	64.7	26.0	245 ±20
1120	2.2	0.0007	82.6	73.0	308 ±15
1220	2.2	0.0006	77.8	93.0	292 ±24
1420	11.1	0.0021	108.0	100.0	394 ±51
				Total fusion age:	290
SAN Plagioclase					
780	40.2	0.039	43.0	13.5	193 ±37
850	49.4	0.016	23.6	22.5	109 ±28
1070	44.6	0.016	23.5	45.5	108 ±21
1240	21.7	0.017	27.8	79.5	127 ±11
1450	18.2	0.011	110.0	100.0	459 ±23
				Total fusion age:	198

NOTE.—Experimental results for analyzed samples from Eastern Pyrénées. Same legend as table 1.

Three of the analyzed biotites (V90, V15, and SAN) show well defined high temperature plateaux. The plateau age at 273 m.y. of the biotite V90 (late granodiorite of Millas) is in good agreement with the Rb-Sr whole rock isochron age of this intrusion (285 ± 12 m.y.) determined by Vitrac-Michard and Allègre (1975). This age is significantly higher than the corresponding Rb-Sr age of the biotite-whole rock pair (140 m.y.). Biotite V15, extracted from a paragneiss of the North Pyrenean Agly massif, displays a well defined age plateau at 99 m.y. The Rb-Sr mineral age of this fraction is lower (88 m.y.) but still compatible when difficulty of defining the initial strontium ratio is taken into account. The two chronometers do not seem to date significantly different events. Biotite SAN was separated from a quartz diorite inclusion in the late granodiorite of St Arnac (Agly massif). A plateau age of 155 m.y. is indicated by the data which show moderate scatter (less than 6%). The apparent Rb-Sr age of 174 m.y. for this mineral is higher in this case.

The biotite G2J (orthogneiss from the Canigou area, axial zone) shows the effect of a more recent event. The relative scatter (10%) of the

TABLE 3

APPARENT ^{87}RB-^{87}SR AGES OF ANALYZED OR RELATED MINERALS

Sample	$^{87}Rb/^{86}Sr$	$^{87}Sr/^{86}Sr$	Apparent Age[1] (m.y.)
Montagne Noire (Hamet and Allègre, 1976)			
C2 Biotite	14	1.09	292
γB Biotite[2]	106	1.12	280
BR1 Biotite	544	2.82	278
BR1 Muscovite	29	0.825	275
Eastern Pyrenees (Vitrac-Michard and Allègre 1975, and unpublished results)			
G2Z Biotite	112	0.968	160
G2J Biotite	154	1.071	160
G2J Muscovite	16.1	0.781	275
V90 Biotite	477	1.67	140
V15 Biotite	130	0.891	88[3]
V15 Muscovite	70	0.900	200[3]
RSG Phlogopite	35.2	0.760	102
SAN Biotite	132	1.028	174

NOTE.—[1] Calculated with the whole rock results given by the same authors, excepting RSG and SAN which have been calculated using an initial strontium ratio of 0.710.

[2] This sample pertains to the Folat granodiorite, of which the FO microgranite is an associated dyke.

[3] Large uncertainty due to the high $^{87}Sr/^{86}Sr$ of the whole rock (about 10%).

individual ages around an average value of 66 m.y. precludes a confident chronometric use of this result. Moreover, this result is strongly discordant with the Rb-Sr age of the biotite-whole rock pair, 160 m.y.

Sample RSG is a separate from a metadiorite (Agly massif) made up of dominant phlogopite (attributable to the pyrenean-alpine-phase?), and of minor deformed biotite of presumed hercynian age. Despite this character, the sample shows a well defined age plateau of 107 m.y. only slightly older than the 102 m.y. Rb-Sr age.

Two muscovites have been studied (fig. 5). In the paragneiss V15 (Agly massif), this mineral displays a small age decrease to a high temperature plateau at 104 m.y., a slightly older age than for the associated biotite (99 m.y.). The Rb–Sr age this mineral is largely discordant at 200 m.y. Muscovite G2J (Canigou area) shows steadily increasing ages up to 245 m.y., a figure which clearly underestimates the crystallization time taken as its Rb–Sr age (275 m.y.). To be noted is that the associated biotite shows the youngest apparent age observed for the area.

(*b*) Amphiboles (fig. 6).

This mineral has been extracted from two samples. RSG is a metadiorite (Agly massif) and shows a high temperature plateau at 400 m.y. Amphibole SAN comes from a quartz diorite inclusion in the hercynian St. Arnac granodiorite (Agly massif). Only two fractions at about 300 m.y. (comprising 65% of the ^{39}Ar released) are concordant with the geologically recognized age of the intrusion.

(*c*) Plagioclases (fig. 6).

The two analyzed plagioclases were extracted from the same samples as the above amphiboles, and both display a U-shaped age spectrum. The age at the minimum of the spectrum is 83 m.y. for RSG, and 108 m.y. for SAN.

(*d*) K-feldspars (fig. 7).

The three analyzed pyrenean K-feldspars pertain to the low temperature series, are perthitic and appear to have suffered extensive argon losses. At low and intermediate temperature, the spectra display a saddle shaped structure which has already been described (Lanphere and Dalrymple 1971; Albarède et al. 1973; Berger 1975). At high temperature, no acceptable plateau is found and no memory of hercynian ages has been retained, excepting perhaps two fractions of sample V90, whose associated biotite presents an age plateau of 275 m.y.

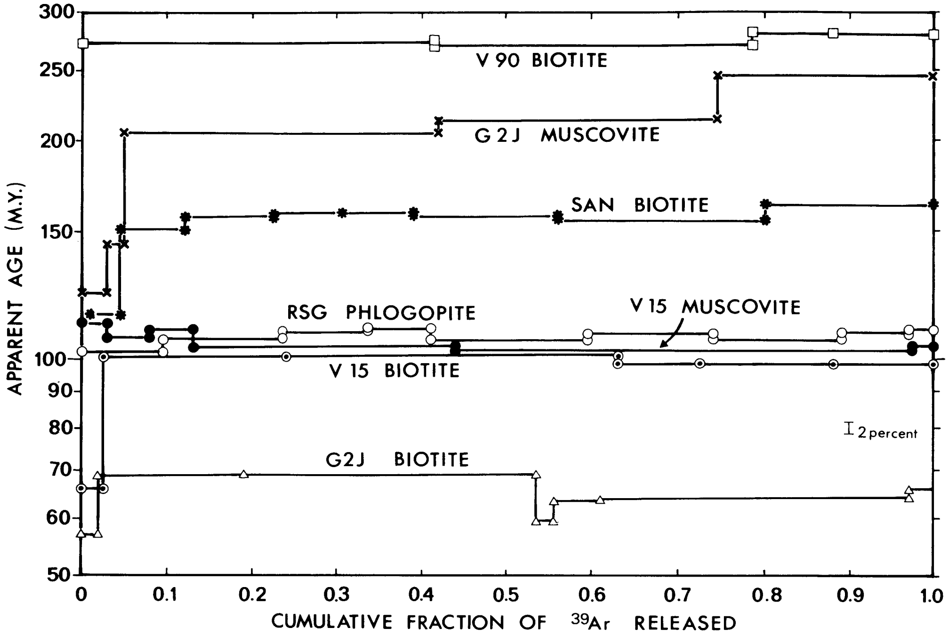

FIG. 5.—Age spectra of Eastern Pyrénées micas

DISCUSSION

At first glance, the behavior of the $^{39}Ar-^{40}Ar$ chronometer appears drastically different if one compares the results for both investigated areas. Hercynian ages characterize the Montagne Noire as they do for Rb–Sr results (Hamet and Allègre 1976), while an extreme chronological confusion seems to arise from our results on the Eastern Pyrénées. Detailed analysis of these results is attempted below in order to show that valuable information may be obtained on the regional chronology and our understanding of the $^{39}Ar-^{40}Ar$ chronometer.

(*a*) Micas.

All the micas from the Montagne Noire have kept the record of their crystallization during the hercynian orogeny, thus bearing out the absence of any significant orogenic event since this time.

In the Eastern Pyrénées, the only mica which has retained the record of hercynian age is the biotite V90 (Millas granodiorite, axial zone), thus indicating the prominent effects of the subsequent alpine disturbances on a regional scale. In the North-Pyrenean massif of Agly, biotite and muscovite V15, phlogopite RSG present plateau ages in the range of 99–107 m.y. These ages are in general agreement with the ages obtained by Rb–Sr method on the biotite V15 and the phlogopite RSG (table 3). It may be accepted that these ages reflect the effects of the middle Cretaceous metamorphism which has been dated in that area in the range of 94–104 m.y. (Albarède 1976; Albarède and Michard-Vitrac, submitted for publication). Conversely, the age plateau (155 m.y.) of the biotite SAN (quartz diorite inclusion in the hercynian granodiorite of St. Arnac, Agly massif) cannot be related to any known geological event, even though the fraction ages scatter by no more than 6%. This conclusion is strengthened by its discordant Rb–Sr age.

In the easternmost part of the axial zone, the biotite G2J has suffered a recent argon loss (younger than ca. 65 m.y.) but in the absence of an acceptable plateau, no definite conclusion may be drawn. The associated muscovite, however, has a steadily increasing age spectrum reminiscent of the argon loss models calculated by Turner (1968). This feature is unique for the samples we have analyzed. In spite of the

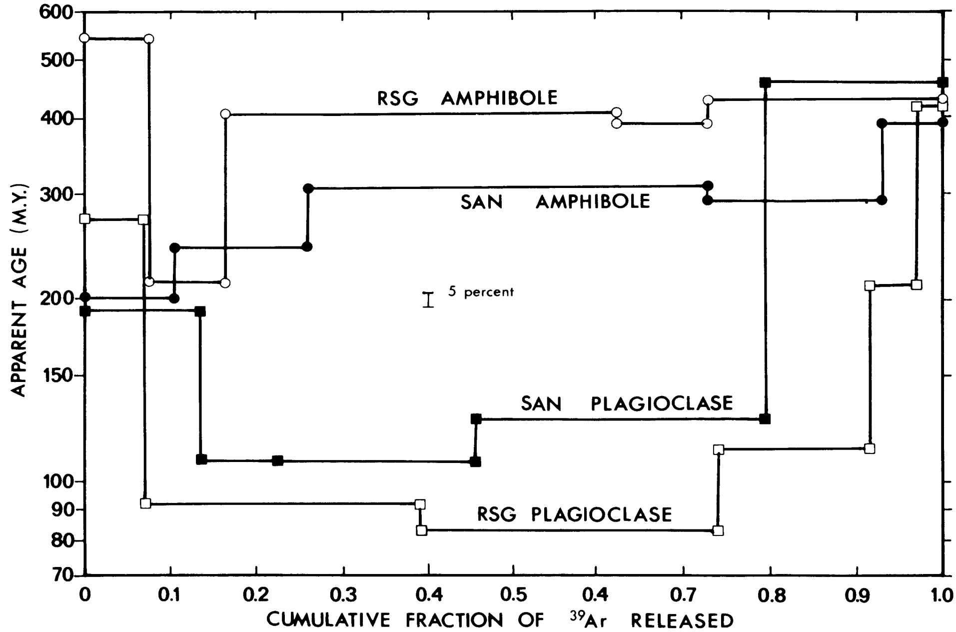

FIG. 6.—Age spectra of Eastern Pyrénees amphiboles and plagioclases

extensive degassing of this mineral, an extrapolation of the high temperature part of the spectrum could give a result (250–270 m.y.) not conflicting with the expected crystallization age which we consider to be recorded by the Rb–Sr age of this mineral (275 m.y.).

If we have succeeded to a certain extent to interpret our results on micas in terms of the *known* geological history of both regions, it is clear that such a method has no predictive property in an area of complex geological history. On a regional scale, there is no clear cut result, neither an intrinsically meaningful character of plateau age, nor a relationship between biotite and muscovite ^{39}Ar–^{40}Ar plateau ages, or between ^{39}Ar–^{40}Ar and Rb-Sr ages. Both the "primary" crystallization age and overprinting event (about 100 m.y.) have been recognized and emphasized by the present data, but no criterion could be found for deciding *a priori* if a dating experiment on micas alone is reliable or not.

We have gained no new compelling evidence concerning the physical significance of biotite age plateaux from the present results. The meaningless age plateau of biotite SAN could support the contention of Hanson et al. (1975) who state that the argon loss during a metamorphic perturbation and in the extraction device are physically unrelated due to the lack of gas pressure monitoring in ^{39}Ar–^{40}Ar experiments. As a matter of fact, the underlying problem of "true" or "false" plateaux is the absence of a reliable identification test for identifying age plateaux at a requested confidence level. As already pointed out by Dalrymple and Lanphere (1974) and Fleck et al. (1977), such a test is difficult to design since it should encompass the number and the extent (in terms of ^{39}Ar released) of compared individual fraction ages which define the plateau and hence appears strongly dependent on the heating schedule.

(*b*) Amphiboles.

Amphibole is usually taken as the most retentive of the common minerals used for K-Ar dating. This remains true for the two analyzed samples from the Agly massif, even in the metadiorite RSG in which phlogopite appears to have completely recrystallized and its age reset during the middle Cretaceous event. The significance of high temperature plateaux, however, must be cautiously assessed, especially

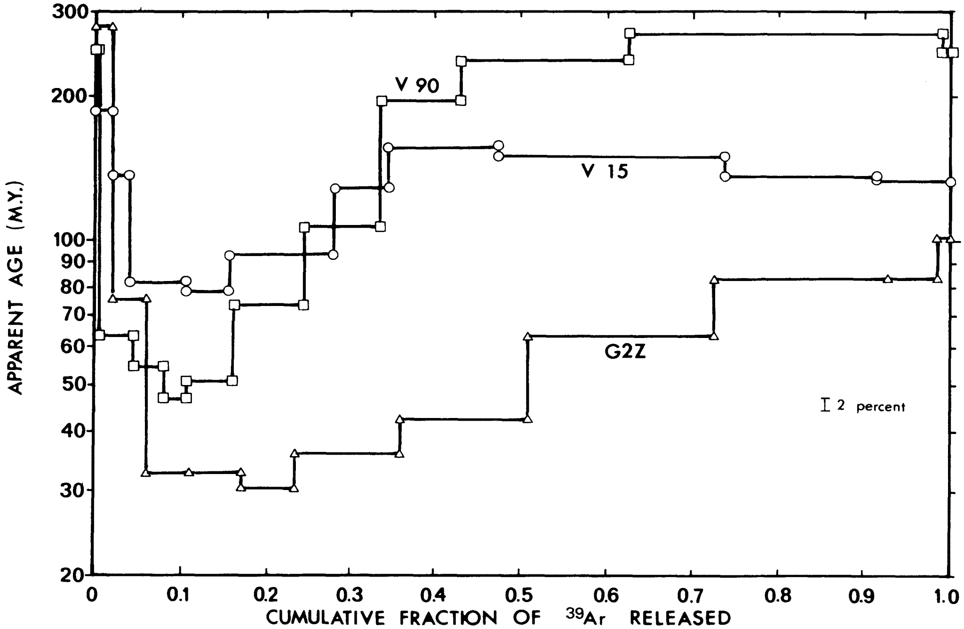

FIG. 7.—Age spectra of Eastern Pyrénées K-feldspars

for such isolated results, taking into account the difficulty of achieving a perfect separation from discordant biotite (Engels 1971).

(*c*) Plagioclases.

Contrary to lunar plagioclases which turned out to be very reliable ^{39}Ar–^{40}Ar chronometers (e.g., Turner et al. 1972; Stettler et al. 1973; Horn et al. 1975), the present data seem rather disappointing. Such U-shaped spectra were also obtained on terrestrial plagioclases by Lanphere and Dalrymple (1971, 1976) who assert that it could be a diagnostic of excess argon. The main feature which distinguishes terrestrial from lunar plagioclases is the lower anorthite content of the former and also their crystallization in a water rich environment. The part that excess argon and possible irradiation artifacts play in generating the U-shaped plagioclase spectra still has to be more rigorously analyzed.

(*d*) K-feldspars.

Although low temperature K-feldspars are believed to be unreliable K–Ar chronometers (e.g., Dalrymple and Lanphere 1969), the data we have obtained on the Montagne Noire samples are quite consistent with the ^{39}Ar–^{40}Ar results on micas, Rb–Sr geochronology and geological evidence.

The spread of the whole set of individual fraction ages of K-feldspars from that area does not greatly exceed 10%. It is well centered on the Rb–Sr ages obtained both from whole rocks and micas (280–295 m.y.) of the same intrusions. The most important aspect which can be drawn from these K-feldspar data deals with its argon retentivity. It is evident that the analyzed K-feldspar samples from the Montagne Noire suffered no significant argon loss since the end of the hercynian orogeny, i.e., the time of the last recognized perturbation. We therefore concur with Foland's (1974) conclusion, from independent evidence, that it is not justified to suspect the low-temperature K-feldspars to have intrinsically poor argon retentivity due to high diffusion coefficient at low temperature.

The situation is more complicated for K-feldspars from the Eastern Pyrénées. The saddle shaped spectra of all samples, even for Millas granodiorite (axial zone) in which the biotite is apparently undisturbed, preclude any confident chronological use of either the low temperature minimum or the high temperature part of the spectra. However, comparison with the above data on the Montagne Noire K-feldspar suggests that these minerals are the most sensitive indicators of tenuous thermomechanical disturbances (Albarède et al. 1973, Berger 1975) and that no part of the investigated area has really been unaffected by the post-hercynian events. It is our contention that low-temperature K-feldspars only can insure the reliability of a regional K–Ar or ^{39}Ar–^{40}Ar chronology on micas and amphiboles in pointing out eventual disturbances which could have been overlooked by other methods.

A comprehensive interpretation of the K-feldspar age spectra must account for both the high sensitivity—in terms of argon retention—of this mineral to mild perturbations and the age decrease at low temperature. Perthitisation (Sardarov 1957; Foland 1974; Berger 1975) alone is not by itself the relevant mechanism since the K-feldspars from the Montagne Noire, while perthitic, are almost free of minimum in their age spectrum.

Transfer of a radiogenic argon rich component from high retentivity to low retentivity sites such as cracks, fissures, grain boundaries, has been advocated (Fitch et al. 1969, Berger 1975) as an explanation of the low temperature age decrease. However, this requires the sites of low thermal retentivity to be able to retain significant amounts of argon for long periods of time, and a physical model agreeing with those rather divergent properties seems difficult to conceive. Moreover, it would remain to be found, why such properties are restricted to disturbed low-temperature K-feldspars.

Alternatively, it may be anticipated that ^{39}Ar–^{40}Ar age spectra similar to those of low-temperature K-feldspars could be produced by simple irradiation artifacts. It has been shown (Brereton 1972; Turner and Cadogan 1974; Huneke and Smith 1976) that the recoil effect of the ^{39}K $(n, p)^{39}$Ar reaction can induce ^{39}Ar migrations in the order of 0.1 μm, resulting in a net ^{39}Ar loss for small K-rich crystals if they are intimately mixed with a K-poor phase acting as a ^{39}Ar catcher. Our interpretation is that the coherency between perthites and host K-feldspar lattice is lost during the tectonic disturbance(s) which has affected the perthitic K-feldspar from Pyrénées. The K-feldspars were shattered at a micron size, allowing a reduction of the effective grain size of the subgrains together with the appearance of a discrete albitic phase as a ^{39}Ar catcher. This is a more formal basis for our previous proposal (Albarède et al. 1973) according to which K-feldspars are very sensitive indicators of tenuous thermomechanical disturbances. Extrapolating down the data of Foland (1974) on the ^{40}Ar diffusion in orthoclase, one can calculate a closure temperature of about 150°C for a 2 micron-sized crystal. This low figure can explain, by itself the common lack of reliability of K–Ar dating by low-temperature K-feldspars in most apparently undisturbed areas.

How could the effect of ^{39}Ar recoil be appreciated in a more quantitative way? Turner and Cadogan (1974) have calculated that for a grain the superficial ^{39}Ar depletion can be described by the equation:

$$^{39}\mathrm{Ar}\ (x) = {}^{39}\mathrm{Ar}\ (\infty) \quad [1 - 0.5 \exp(-x/x_0)] \tag{1}$$

where ^{39}Ar (x) represents the amount of this isotope at the distance x from the surface and x_0 is a characteristic length which they calculated to be 0.082 micron. Experimental evidence of the recoil phenomena has been obtained by Huneke and Smith (1976) who came to the following conclusions:

—the near surface part of the K-rich minerals are ^{39}Ar depleted to an extent adequately described by eq. (1).

—K-poor adjacent to K-rich minerals are able to catch the ejected argon in very retentive sites, thus lowering the age of high temperature fractions of the mixture [a conclusion which was already drawn from independent observations by Horn et al. (1975)].

From geometrical considerations and use of eq. (1), Huneke and Smith (1976) found that the fraction of ^{39}Ar lost by recoil f_L could be expressed as:

$$f_L = 0.5 x_0 \ (A/V) \tag{2}$$

where A/V is the surface to volume ratio of the grain. For most common geometries (cube and sphere) it is convenient to define the "size" D of

TABLE 4

COMPARISON OF MODEL GRAIN SIZE OF K-FELDSPAR SAMPLES AS CALCULATED FROM EQ. (5) AND (4) WITH THEIR TOTAL FUSION AGE

	MONTAGNE NOIRE			PYRENEES		
Sample	S2	C2	FO	G2Z	V90	V15
Model grain size (μm)	50	—	120	0.45	4.6	3.7
Total fusion age (m.y.)	275	297	277	63	192	131

the volume as:

$$D = 6\ V/A \quad (3)$$

which may be used to define an apparent (model) grain size of the mineral population from:

$$D = 3x_0/f_L \quad (4)$$

In the case of ^{39}Ar loss by recoil from fine grained crystals mixed with a K-poor phase, an upper limit of the fragment size may be estimated in the following way. Taking the $(^{40}Ar_{rad}/^{39}Ar)_{min}$ ratio of the minimum on the age spectrum as the true isotopic ratio of low temperature steps, the fraction f^i_L of the ^{39}Ar lost for the ith temperature fraction is readily shown to be:

$$f^i_L = f^i \left[\frac{(^{40}Ar_{rad}/^{39}Ar)_i}{(^{40}Ar_{rad}/^{39}Ar)_{min}} - 1\right] \quad (5)$$

where the subscript i refers to the observed isotopic ratio and f^i stands for the fractional amount of ^{39}Ar released for this step. Summing up for all the low temperature fractions below the minimum and inserting into eq. (4) one gets the desired fragment size.

Using the appropriate values, the model grain sizes reported in table 4 were computed. The pyrenean K-feldspars display model grain sizes in the micron range, while for Montagne Noire samples those sizes are of the same order of magnitude as what can be measured under the microscope (50–200 μm). It is clear that, the larger the loss of radiogenic argon (as measured by the total fusion age), the smaller the model grain size appears. The consistency of this relationship with what could be expected from a volume diffusion behavior may be taken as evidence of the importance of recoil phenomena in generating the age spectra characteristic of K-feldspars.

The importance of K-feldspars for ^{39}Ar–^{40}Ar dating deserves some additional illustration by two examples taken outside the present field area. Dealing first with the sample 22500 from the metamorphic aureole of the Eldora Stock (Berger 1975), the biotite gives a nicely defined age plateau at 1,230 m.y. while U–Pb and Rb–Sr dating as well as hornblende ^{39}Ar–^{40}Ar age plateau agree at about 1,400 m.y. Berger interprets this plateau as significant of a 1,230 m.y. old thermal event or of a cooling age, but remains aware of the difficulty to justify his choice. It is worth noting that the associated K-feldspar 22500 shows the evidence of a significant argon loss of radiogenic argon (about 25% relative to the biotite age) along with the common saddle shaped structure in the low temperature part of its spectrum (model grain size of 2 microns). These observations may be reconciled if one admits a slight but general argon loss for all minerals of sample 22500, including biotite, while there is no measurable variation nor scattering of the ages of the high temperature fractions. A simple explanation would be to admit that the maximum distance at which the mica K–Ar ages have been affected by intrusion of the Eldora Stock was underestimated both by Hart (1964) and Berger (1975).

The same observation can be made for the sample MM43 from the Marble Mountains (southern California) which Lanphere and Dalrymple (1971) analyzed. When compared with U–Pb and Rb–Sr control ages, the biotite plateau indicates an age too young by about 20%. In that case, also, the associated microcline shows evidence of radiogenic argon loss and an age minimum at low temperature leading to a model grain size of 0.4 microns.

This lack of reliability of biotite age plateaux where K-feldspars are partially overprinted favors, to a certain extent, the contention of Hanson et al. (1975) who rejected systematic confidence in those plateaux. As suggested by Dallmeyer (1975*c*), this is not justified for severely overprinted micas and the suspicion must be restricted to the effects of mild disturbances which could generate some confusion if the plateau concept is taken as a magic geochronological tool.

(*e*) The significance of cooling ages.

It is a widely held practice (see, for instance, Jaeger et al. 1967; Clark and Jaeger 1969; Dallmeyer 1975*a*, *b*; Dallmeyer et al. 1975, 1976) to use regional age variations on minerals as a measure of variable cooling rates during the post-orogenic erosional discharge. It is necessary, however, to demonstrate that these age variations do not result from a mild disturbance which has been overlooked by field and petrographic observations. We propose, as an independent check of plateau reliability, to verify the undisturbed character of the age spectrum of associated low-temperature K-feldspars. For undisturbed areas (as in the Montagne Noire), these minerals should display almost flat age spectra, whose age is expected to match approximately the high temperature plateaux of biotites and amphiboles. These weakly disturbed patterns should be correlated with large model grain size of K-feldspar calculated, according to the aforementioned procedure, from the recoil hypothesis. Conversely, the presence of significantly discordant biotite-K-feldspar pairs, along with small model grain sizes, calls for a careful check for subtle disturbances.

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