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Anomalous Parent-daughter Isotopic Relationships in Rocks Adjacent to the Grenville Front near Chibougamau, Quebec

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ABSTRACT

Anomalously high K-Ar 'ages' have been obtained for biotites from granitic rocks adjacent to the boundary between geologic provinces of the Canadian Shield. This phenomenon is particularly marked in one such area southwest of Chibougamau, Quebec, where the Dauversière Stock outcrops in the Superior Province immediately north of its boundary with the Grenville Province. The stock is roughly circular in configuration having a diameter of approximately 8 miles. Rock samples selected from several localities have yielded concentrates of both biotite and muscovite, thereby providing two mineral indicators within the same rock. In some instances the muscovite K-Ar 'ages' are much lower than those of the associated biotites.

When the results obtained, using both the K-Ar and Rb-Sr methods, are plotted with respect to the distance of the sample sites from the Grenville Front, it is apparent that the anomalies are a function of the proximity to the front. A sample of biotite, containing the greatest quantity of excess radiogenic argon, was selected for special study. The argon was extracted at a series of gradually increasing temperatures in order to ascertain if a portion of the argon could be readily removed thereby leaving a fraction that would provide an indication of the 'true' age of crystallization of the mineral. No evidence of such a component was found; the gas being released regularly as the temperature was increased to the fusion point of the biotite.

The Rb-Sr whole-rock isochron technique has been applied to samples selected from the stock and from the Grenville Province immediately south of the front. The results appear to define a single isochron indicating an age of 2,610 \pm 170 m. y. with an initial ⁸⁷ Sr/⁸⁶ Sr ratio of 0.7011 \pm 0.0015. This evidence is believed to indicate that the rocks on either side of the boundary were formed during the same geological period. Mineral isotopic evidence for those samples now located in the Grenville Province indicates that they were reconstituted during the Grenville orogeny. While the latter event was sufficiently intense to have modified both the 40 K/ 40 Ar and 87 Rb/ 87 Sr isotopic ratios of the constituent minerals, the whole-rock samples appear to have remained as closed systems for Rb and Sr.

In the Superior Province, north of the front, the effects of the Grenville orogeny are strikingly illustrated where the two isotopic systems have responded differentially and anomalously to the thermal gradient, although the lithologic and petrographic character of the rocks has remained unchanged.

Introduction

In the course of the reconnaissance K-Ar dating program undertaken in the Canadian Shield by the Geological Survey of Canada a few samples were encountered that yielded anomalous 'ages'. Characteristically, the biotite results were found to be much too high whereas associated muscovites from the same rock sample contained a much smaller proportion of radiogenic argon. The phenomenon was noted in regions of the Superior Province immediately adjacent to its boundary with the Grenville Province and one such area, near Chibougamau, Quebec has been studied in detail. This paper presents the results of an investigation in which both K-Ar and Rb-Sr dating techniques have been employed. In addition, radiogenic argon was extracted from one biotite concentrate containing anomalously large quantities of radiogenic argon, in order to ascertain if the 'extra' argon could be readily expelled from the crystal structure.

Geological setting

The area studied (see Fig. 1) straddles the Grenville Front at a point approximately 300 miles NNW of Quebec City, Quebec. The first geological reconnaissance of the area was carried out by James Richardson in 1872. His work and subsequent studies by J.OBALSKI (1904), A.P.LOW (1906), H.C.COOKE (1927), C.TOLMAN (1930), J.B. MAWDSLEY and G.W.H.NORMAN (1935 and 1938) have been reviewed by P.-E.IM-BAULT who published a map and report on the area (Queylus) in 1959. Figure 1 is adapted largely from IMBAULT's map and the following rock designations are based on his report. In addition Figure 1 also includes small sections from the adjoining Dollier-Charron Area to the east (E.R.W.NEALE, 1959) and the Rohault Area immediately to the south (J.-E. GILBERT, 1959).

All of the consolidated rocks of the map area (Fig. 1) are Precambrian in age, and the geology is dominated by three major features:

1. A broad zone of weakly metamorphosed rocks typical of the Superior structural Province extending for a great distance to the north and northwest;

2. A marked lithological and structural discontinuity commonly designated as the Grenville Front traversing the area in a generally southwest-northeast direction; and

3. An extensive area of strongly metamorphosed rocks characteristic of the Grenville structural Province to the east and southeast.

Rocks of the Superior Province comprise two main groups: an older group, for the most part low grade meta-volcanics with minor metasediments, and younger granitic rocks and diabase dykes. The volcanic sequence (commonly called Keewatintype rocks) consists mainly of metamorphosed basaltic and andesitic lavas, pyroclastics, apparently concordant sills of gabbro and diorite, and lesser amounts of sedimentary rocks (generally designated as Timiskaming-type), being mainly metamorphosed feldspathic sandstones and greywacke.

The essentially circular Dauversière Stock, the central feature of Figure 1, outcrops over an area of some 60 square miles and underlies all of Lac La Dauversière and most of Lac Le Royer. Its composition varies from granite to granodiorite and its texture from massive and locally porphyritic to distinctly gneissic in proximity to its margins. Generally the rock is a grey to pinkish grey quartz monzonite with biotite and muscovite. The stock is intrusive into the volcanic sequence and, in turn is cut by the later diabasic dykes.

The overall structure of the predominantly volcanic belt north of the Dauversière Stock is synclinal with the fold axis trending in a direction slightly south of east about 1.5 mile north of the northern margin of the stock. Throughout this, and the wider area, these rocks have a general east-west trend and, as far as can be seen, schistosity is everywhere parallel to the bedding. Dips are steep to vertical. Divergencies from the general trend occur in the vicinity of the intruisive Dauversière Stock where, for example, the schistosity curves completely around it, and in the vicinity of the





Fig.1. Generalized geological map of the area straddling the Grenville Front near Chibougamau,

Quebec.

Grenville Front where the trends become northeasterly. Shear zones parallel to the schistosity are numerous throughout the meta-volcanics and increase in abundance southwards towards the stock and the Grenville Front. Faults transecting the general structural trend all strike northeastwards.

Metamorphism of the rocks of the Superior Province is pervasive throughout, but of generally low grade (Greenschist facies), except in close proximity to the Grenville Front where the meta-volcanics and metasediments become highly schistose. The feldspars of the Dauversière Stock have been altered to epidote, clinozoisite, sericite and albite and this may have resulted from the overall, low grade regional metamorphism. On the other hand, no evidence has been reported for metamorphism of the surrounding country rocks by the stock. In fact, contact phenomena are more noteworthy within the stock where the rock becomes gneissic towards the margins and the development of a variety of hybrid phases produced as a result of contamination by assimilation have been reported.

Rocks of the Grenville Province can be grouped into three principal types, hornblende paragneiss and paraschist, muscovite and biotite paragneiss, and biotite and hornblende orthogneiss. All occur within the area of Figure 1 and all have been highly metamorphosed and derformed. In the map area rocks of the first principal type are mainly hornblende and garnetiferous hornblende schists. These rocks outcrop northeast of Lac Dufresne and west of Lac Malo and both Imbault and Neale report gradational transitions between these and the adjacent Superior Province metavolcanic rocks.

Muscovite and biotite paragneisses are represented in this area by a muscovite gneiss underlying, and extending northeast and southwest of Lac Dufresne. It is a very heavily sheared, cataclastic gneiss in an area of poor outcrop occupied by swampy streams and a series of small lakes. This pinkish grey muscovite gneiss grades eastwards into grey biotite orthogneiss and composite gneisses which cover most of the eastern part of the map area; NEALE considers it to be the cataclastically deformed equivalent of these rocks.

The orthogneisses form a complicated, intergrading mixture of deformed igneous intrusives with composite gneiss, the latter of which is thought to be derived from sedimentary and volcanic rocks particularly to the south where hornblende and complexly interfolded schist become more common. In the map area the predominant rock is biotite orthogneiss of quartz monzonitic to quartz dioritic composition.

Gneissic structures near the northwestern limits of the gneissic terrane are strongly oriented in an overall northeasterly direction and parallel the approximate direction of the Grenville Front. Though the folding is complex, Neale postulates that the orthogneisses in this area form part of a south or southeastward plunging syncline. Faults, where recognized in the gneisses, strike north-northeast parallel to the front.

The grade of metamorphism of the rocks southeast of the Grenville Front is relatively high (amphibolite facies) and is typical of that generally observed in the northern parts of the Grenville Province.

The Grenville Front is one of the major features of the Canadian Shield extending across eastern Canada from the head of Lake Huron to the coast of Labrador. It is the line of demarcation between the Grenville and Superior Structural Provinces, and has been variously interpreted as a fault, or a zone of metamorphic transition. NORMAN (1936 and 1940) has suggested that in the Chibougamau-Lake Mistassini region the front is the locus of northeast trending reverse fault zones along which the southeast (Grenville) side moved up relative to the northwest (Superior) side. In the Lac La Dauversière Area of Figure 1 the front appears to be a zone of shear-modified metamorphic transition. In the swampy valley occupied by Lac Dufresne, through which the front also passes, the muscovite gneiss is strongly sheared and cataclastic textures are very obvious in thin sections of the rock. At this point on the highway the transition from sheared gneiss to low grade meta-sediments appears to be quite abrupt and occurs within a distance of 400 feet.

All shear zones and minor faults in and near the Grenville Front trend northeastward and carbonate-rich zones are common. Locally, the hornblende and garnetiferous hornblende schists have been replaced by more than 50 per cent of rusty weathering iron carbonate.

The main geological features of the front in this area are:

1. The abrupt change in lithology and metamorphic grade between the Grenville and Superior Provinces.

2. The deflection to a northeasterly direction of the generally east-west trends of the Superior Province, and their eventual termination against the front.

3. The presence of intense shearing and more or less faulting of indefinite magnitude within the zone of the front.

4. The possibility, in some places, of tracing rocks of the Superior Province through a metamorphic transition to their high grade equivalents in the Grenville Province.

Isotopic investigations

(a) Previous Results

The first highly anomalous age determinations were obtained for samples collected for C. H. STOCKWELL and reported in WANLESS et al. (1965). Of this group one biotite sample (Sample 9 of Table 1 and Figure 1) was found to have an apparent age of 3300 m.y. whereas the associated muscovite had an apparent age of only 1630 m.y. The sample was collected from the Dauversière Stock, within the Superior Province, slightly more than 2 miles northwest of the Grenville Front. Other samples, also collected from the Dauversière Stock, yielded discordant biotite-muscovite apparent ages but the discrepancy was less marked, and in some instances the muscovite was found to yield an older apparent age than the associated biotite (see, for example, Samples 1 and 5, Table 1 and Figure 1). In marked contrast to these results, samples selected from the region south of the Grenville Front gave ages in the range of 900– 1000 m.y., typical of those generally found in the Grenville Province, although biotites from rocks closer to the Grenville Front yielded apparent ages somewhat above this range (see Samples 20 and 21, Table 1).

Based on the early data STOCKWELL (1963) concluded that muscovite samples from both sides of the Grenville Front gave realistic ages, that the low biotite ages north of the front resulted from a preferential loss of radiogenic ⁴⁰Ar, and that biotites immediately south of the Grenville Front had accumulated excess quantities of radiogenic argon. However, subsequent analyses carried out on Sample 9, Table I,

Table	1. Analytic	al Data Mineral Sam	iples.										
Sample	e Distance	Rock Type	Mineral	×	⁴⁰ Ar/ ⁴⁰ K	K-Ar	Reference	Rb	Sr	87 Sr/	⁸⁶ Sr	⁸⁷ Rb/ ⁸⁶ Sr	Rb-Sr
No.	from Gren- ville Front					Age				Mineral	Whole Rock		Age
	miles			%		m.y.		p.p.m.	p p.m.				m.y.
1	7.88 north	Granodiorite	Biotite Muscovite	8.07 6.39	0.2228 0.2619	2085 2295	GSC 63-137 GSC 63-136	123.7	133.3	0.8031	0.7160 ^b)	2.687	2535
7	7.87 north	Granodiorite	Biotite Whole Rock ^a)	7.64	0.2023	1965	I	331.4	8.68	3.7518	0.7193	110.59	1850
3	4.97 north	Quartz monzonite	Biotite Muscovite Whole Rock ^a)	7.07 8.54	0.1364 0.2146	1520 2035	1	595.4 281.3	16.11 29.54	2.0589 1.6830	0.7295	107.0 27.57	845 2375
4	4.97 north	Gneissic quartz monzonite	Whole Rock ^a)								0.7233		
2	4.91 north	Granodiorite	Biotite Muscovite Whole Rock ^a)	7.75 8.04	0.1782 0.2715	1815 2340	GSC 62-153 GSC 62-154				0.7134		
9	3.23 north	Quartz monzonite	Whole Rock ^a)								0.7120		
٢	3.0 north	Granite	Biotite Muscovite Whole Rock ^a)	7.51 8.58	0.3022 0.2127	2485 2025	GSC 63-143 GSC 63-144				0.7353		
8	2.28 north	Gneissic granite	Biotite Muscovite Whole Rock ^a)	8.07 8.01	0.6666 0.1405	3680 1550	11	603.9 291.4	6.07 74.57	4.9824 1.1123	0.7319	288.2 11.31	1000 2415
8			Biotite Muscovite	7.61 8.02	0.7256 0.1678	3825 1745	1 1						
6	2.25 north	Gneissic granite	Biotite Muscovite	7.37 8.62	0.5241 0.1513	3300 1630	GSC 63-146 GSC 63-145						

350

Anomalous Parent-daughter Isotopic Relationships

7.37 0.2724 0.98 0.2513
0.3558 0.3891
0.3231
0.1827
0.1110 0.0830
0.0733
0.0746
0.0823 0.0781
0.1061 0.0733
0.1246 0.0757
I T

^a) Rb-Sr analytical data for whole rock samples given in Table II.

^b) Whole rock ⁸⁷Sr/⁸⁶Sr estimated from X-Ray data. ⁸⁷Rb; $\lambda_{\beta} = 1.47 \times 10^{-11} \text{ yr}^{-1}$; ⁴⁰K; $\lambda_{e} = 4.72 \times 10^{-10} \text{ yr}^{-1}$, $\lambda_{e} = 0.585 \times 10^{-10} \text{ yr}^{-1}$, atomic abundance = 1.19 × 10^{-4}.

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revealed the presence of large excess quantities of radiogenic argon in the biotite 2.25 miles north of the Grenville Front and an apparent age of 1630 m.y. for the associated muscovite. The latter was much lower than one would expect in the Superior Province, and STOCKWELL (1965) therefore modified his earlier conclusions.

In spite of the fact that coarse-grained granitic rocks are considered unsuitable for whole-rock K-Ar age measurement, due to variable indeterminate loss of argon from the constituent feldspars, such determinations were undertaken for several samples from both north and south of the Grenville Front. These analyses were carried out in order to ascertain if the anomalous apparent mineral ages had resulted from redistribution of argon within the rock as a whole (i.e. the whole-rock sample represented a closed system to migration of argon), or if radiogenic argon-40 atoms had in fact migrated over much greater distances than those represented by the wholerock samples. The results (published in WANLESS et al., 1965) indicated that in the majority of the cases studied there had been a net loss of radiogenic argon-40 since all whole-rock apparent ages were lower than those obtained for the constituent biotites. This was especially noticeable for rocks of the Superior Province, whereas wholerock results from the Grenville Province, while low, were generally found to be within 10 per cent of the age determined for the associated biotite.

(b) New Sample Collections

Additional samples were collected in 1966 by WANLESS and STEVENS who traversed the Dauversière Stock and also selected material from areas on either side of the Grenville Front. The object was to verify the existence of the highly anomalous apparent ages, and to collect sufficient quantities of rock to facilitate the preparation of biotite concentrates to be used for controlled temperature experiments. At the same time a Rb-Sr isotopic investigation was planned to provide data for comparison with the K-Ar results, and in an attempt to establish the age of emplacement of the Dauversière Stock.

(c) New Isotopic Results

Analytical data for all samples are given in Table 1 and the apparent ages obtained are plotted on Figures 1 and 2. Appendix II contains sample descriptions, details of the mineral concentrates analysed, and precise sample locations. Analytical techniques employed are discussed in Appendix III.

The primary objective of verifying the existence of the highly anomalous apparent ages was realized. Sample 8, which was collected from a site within one quarter of a mile of sample site number 9, yielded even higher biotite apparent ages of 3680 m.y. and 3825 m.y. based on the analysis of two separate biotite concentrates (numbers 8 and 8'). In addition, it was again observed that the muscovite results were anomalously low at 1550 m.y. and 1745 m.y. respectively. Biotite concentrate number 8' was used for the incremental heating experiments.

Figure 2 displays the isotopic data with respect to the perpendicular map distance (in miles) of the sample sites from the Grenville Front. Since the surface expression of the front is an irregular zone, these distances are subject to interpretational error. However, this error is not believed to be of such a magnitude that it would seriously



Fig. 2 Distribution of apparent isotopic ages near the Grenville Front.

affect the distribution illustrated in Figure 2 or the general conclusions drawn from this study.

The striking feature of Figure 2 is the very high apparent age range obtained for biotites from rocks located in a zone 2 to 3 miles north of the Grenville Front. In addition the apparent biotite ages display an extremely irregular pattern over the complete span of the traverse, from 3 miles south of the front to the western boundary of the Dauversière Stock some 8 miles north of the front. The region of anomalously high biotite apparent ages is flanked by a zone of low apparent ages within the Superior Province and a zone immediately south of the Grenville Front where the apparent ages are higher than those normally found within the Grenville Province. No evidence is available as to the geographical extent of the latter phenomenon but since biotites with high apparent ages have not been previously reported in the Grenville Province it may be a local event of restricted extent.

Muscovite K-Ar apparent ages follow a much more regular progression from typical Grenville Province ages of about 975 m.y. south of the front, through a transition zone of 1600–1800 m.y. values 2 to 3 miles north of the front, to an eventual maximum of approximately 2300 m.y. at the western edge of the stock.

The Rb-Sr mineral results present a strikingly different picture with the biotite samples reflecting typical Grenville Province ages (slightly above 900 m.y.) from a point 2 miles south of the front to a site 5 miles north of the front. From this point westward the apparent ages increase rapidly to maximum of about 1900 m.y., appreciably lower than the K-Ar results obtained for the same mineral concentrates. On the other hand the Rb-Sr muscovite results increase rapidly from Grenville to Kenoran ages (\sim 2500 m.y.) within the 2–3 mile zone immediately north of the Grenville Front.

One K-Ar age determination was made on hornblende (Sample 13, Table 1) from a hornblende schist within the area of meta-volcanics south of the Dauversière Stock but approximately 1 mile north of the front (Figure 1). The determined age of 2580 m.y. is in essential agreement with the highest Rb-Sr muscovite age of 2535 m.y. (Sample 1, Table 1 and Figure 2). The latter, however, was only attained at a distance of 8 miles from the front. The superior retentivity of radiogenic argon exhibited by the hornblende sample is in accord with results reported by HART (1964) and ALDRICH et al (1965).

(d) Rb-Sr Whole-Rock Measurements

The rubidium and strontium data for whole-rock samples from both north and south of the front are given in Table 2 and the isochron obtained is plotted in Figure 3. The isochron age, based on 14 samples (9 from the region north of the front and 5 from the south), of 2610 ± 170 m.y. is also plotted in Figure 2. The initial strontium 87/86 ratio was found to be 0.7011 ± 0.0015 . Calculations based solely on the whole-rock samples of the Dauversière Stock from the region north of the front yielded an age of 2666 ± 187 m.y. with a strontium isotope intercept of 0.7007 ± 0.0014 , both of which fall within the limits of the preferred results quoted above for the total isochron. The Rb-Sr isotopic relationship of the whole-rocks north and south of the front is similar to that first reported by GRANT (1964) for rocks near Lake Timagami in Ontario. However the primary age indicated for this region is considerably greater than that determined in the Lake Timagami region (i.e. 2610 m.y. for this area vs. 2210 m.y. for Lake Timagami, when the ages are calculated using the same 87 Rb decay constant of 1.47×10^{-11} yr⁻¹). The initial strontium 87/86 isotope ratios are nevertheless, quite similar.

(e) Controlled Temperature Extractions

Argon was extracted in stages from one of the 'anomalous' biotite samples (Number 8') and the results were compared with those obtained for a similar experiment using a sample of supposedly 'normal' biotite. In both experiments the samples



Fig. 3 Rb-Sr isochron for rocks of the Dauversière Stock and Grenville gneisses. See Table II and Figure 1 for sample locations.

Sample	Distance	Rb	Sr		⁸⁷ Sr/ ⁸⁶ Sr		⁸⁷ Rb/ ⁸⁶ Sr
No.	from Gren- ville Front miles	ppm	ppm	Un- spiked	Spiked	Average	
12	1.35 north	36.64 ± 0.73	392.2 ± 7.8	0.7120 ^a)	0.7111	0.7117 ± 0.0006	0.270 ± 0.008
6	3.23 north	$\textbf{36.41} \pm \textbf{0.73}$	$\textbf{360.5} \pm \textbf{7.2}$	0.7123 ^a)	0.7114	$\textbf{0.7120} \pm \textbf{0.0006}$	0.292 ± 0.009
5	4.91 north	39.05 ± 0.78	$\textbf{378.2} \pm \textbf{7.6}$	0.7134 ^a)	0.7133	0.7134 ± 0.0010	$\textbf{0.299} \pm \textbf{0.009}$
10	1.41 north	$\textbf{34.52} \pm \textbf{0.69}$	$\textbf{283.3} \pm \textbf{5.7}$	0.7157 ^a)	0.7154	$\textbf{0.7156} \pm \textbf{0.0006}$	$\textbf{0.353} \pm \textbf{0.010}$
2	7.87 north	$\textbf{45.00} \pm \textbf{0.90}$	$\textbf{262.1} \pm \textbf{5.2}$	0.7196 ^a)	0.7187	$\textbf{0.7193} \pm \textbf{0.0006}$	0.497 ± 0.015
20	1.30 south	$\textbf{74.93} \pm \textbf{1.50}$	$\textbf{402.4} \pm \textbf{8.0}$	0.7217	0.7230	$\textbf{0.7224} \pm \textbf{0.0010}$	0.539 ± 0.016
4	4.97 north	$\textbf{43.40} \pm \textbf{0.87}$	215.0 ± 4.3	0.7234 ^a)	0.7233	$\textbf{0.7233} \pm \textbf{0.0006}$	0.584 ± 0.018
3	4.97 north	49.63 ± 0.99	196.3 ± 3.9	0.7300 ^a)	0.7285	$\textbf{0.7295} \pm \textbf{0.0009}$	0.732 ± 0.022
8	2.28 north	$\textbf{70.24} \pm \textbf{1.40}$	$\textbf{262.0} \pm \textbf{5.2}$	0.7322 ^a)	0.7313	$\textbf{0.7319} \pm \textbf{0.0006}$	$\textbf{0.776} \pm \textbf{0.023}$
18	0.45 south	$\textbf{56.68} \pm \textbf{1.13}$	$\textbf{205.7} \pm \textbf{4.1}$	0.7361	0.7375	$\textbf{0.7368} \pm \textbf{0.0010}$	0.798 ± 0.024
7	3.00 north	$\textbf{54.84} \pm \textbf{1.10}$	190.2 ± 3.8	0.7353 ^a)	0.7353	$\textbf{0.7353} \pm \textbf{0.0006}$	0.835 ± 0.025
19	0.80 south	$80.24 \pm 1.60^{\mathrm{a}}$)	$\textbf{239.7} \pm \textbf{4.8}$	0.7353	0.7343	$\textbf{0.7348} \pm \textbf{0.0010}$	$\textbf{0.969} \pm \textbf{0.029}$
16	0.10 south	$90.97 \pm 1.82^{\mathrm{a}}$)	$\textbf{177.7} \pm \textbf{3.6}$	0.7594	0.7586	$\textbf{0.7590} \pm \textbf{0.0011}$	1.482 ± 0.044
17	0.30 south	123.4 ± 2.47	$\textbf{184.4} \pm \textbf{3.7}$	0.7754	0.7747	$\textbf{0.7750} \pm \textbf{0.0011}$	$\textbf{1.938} \pm \textbf{0.058}$

Table 2. Analytical Data Whole Rock Samples.

^a) Average of two determinations.

were given a preliminary outgassing at 150 °C to 200 °C for 18 hours. The temperatures were then raised to the levels indicated in Table 3 and held there for periods of from 45 minutes to 1 hour. The gas released at each temperature was mixed with a volume of argon-38 spike and purified following the procedure routinely employed for K-Ar age determination samples (i.e. passage through hot CuO and treatment over hot titanium sponge). Following the purification of each fraction of gas released the the temperature was raised rapidly to the next level and maintained constant for the prescribed time. The final step involved the complete fusion of the biotite. The percentage of total radiogenic argon released at each stage and the cumulative total per cent released are given in Table 3. Age calculations based on the total radiogenic argon released during the experiments and those based on a routine complete extraction from a second portion of same concentrate are in excellent agreement indicating that all of the argon was released and collected during the course of the experiments.

Sample	Extraction	Radiogenic	⁴⁰ Ar Rele	eased	Age (r	n.y.)
No.	Temp. (°C)	moles/gm. $\times 10^{-11}$	%	Cumulative %	Incremental Extraction	Complete Extraction
8'	200	1.18	0.007	0.007		
	400	32.01	0.18	0.187		
	600	2223.5	12.64	12.83		
	800	6622.8	37.66	50.49		
	900	4897.7	27.85	78.34		
	1000	3105.0	17.65	95.99		
	1100	680.1	3.87	99.86		
	1200	23.97	0.136	99.996		
	+1200	1.28	0.007	100.013		
	Total	17587.54	_		3800	3825
K-Ar 212	300	0.28	0.009	0.009		
	500	38.62	1.20	1.209		
	700	1016.1	31.52	32.73		
	900	1281.3	39.75	72.48		
	1000	887.0	27.52	100.00		
	1300	Lost				
	Total	3223.3	—	-	1490	1550

Table 3. Argon Extraction Experiments.

In Figure 4 the cumulative percentage of gas released is plotted against the measured temperature for each extraction stage. It is immediately apparent that the gas was released from the two samples in an identical fashion and that at least 80 per cent of the radiogenic argon was released between 600 °C and 1000 °C. Thus, within the limits of the experimental procedures used, it has been impossible to detect the separation of an excess argon component from the radiogenic fraction produced in situ.



Fig. 4 Radiogenic argon-40 release from 'anomalous' and 'normal' biotite concentrates.

Discussion

The 2610 m.y. age based on the Rb-Sr whole-rock analyses may represent the time of initial emplacement of the Dauversière Stock, or it may reflect a metamorphic event presumably associated with the early phases of the Kenoran orogeny (STOCK-WELL, 1964, Part II). The low initial ⁸⁷Sr/⁸⁶Sr value of 0.7011 indicates that the time span between the two alternatives is not very long. Since the grade of metamorphism is low it is reasonable to assume that its effect on the original age was minimal, and that the age obtained closely approximates the primary age of emplacement. The indication that the rocks south of the front are the same age and possessed the same initial ratio supports the hypothesis that they are in fact the high grade metamorphic equivalents of the rocks north of the front as suggested by NEALE (1959) and IMBAULT (1959). Independent confirmation of the isochron age is given by the 2580 m.y. K-Ar

hornblende age for the schist found within the Superior Province between the Grenville Front and the southern boundary of the Dauversière Stock. On the basis of this evidence it is suggested that all of the rocks (except the younger diabase dykes) of the map area, in both the Superior and Grenville Provinces, are at least 2600 m.y. old and that the observed variations in apparent mineral age have developed from that level. The high grade metamorphism of the rocks south of the front has produced ages there that are typical of the Grenvillian Orogeny, and the effects of this event are observed in the Superior Province up to 8 miles north of the front.

Conclusions

1. Isotopic systems may be disturbed as a consequence of metamorphic events that were not sufficiently intense to have brought about alterations that are recognizable during routine petrographic examination.

2. Biotites have long been known to have poor retentivity characteristics for argon under metamorphic conditions but this study has revealed evidence for biotites incorporating enormous quantities of excess argon from the immediate environment. In this instance the high apparent ages obtained for biotites are not the consequence of preferential loss of potassium since this element was found to be present in average to high abundance in all samples.

3. Preliminary experiments indicate that the 'excess' argon component cannot be more readily expelled from the crystal lattice than radiogenic atoms produced through in situ decay of potassium-40. The argon release characteristics of this 'anomalous' biotite and a typical 'normal' biotite appear to be identical within the limitations of our experiment.

4. The response of the Rb-Sr isotopic system is in marked contrast to that found for the K-Ar system. The biotite structures have not incorporated additional radiogenic ⁸⁷Sr but on the contrary, at the time of the Grenvillian orogeny, suffered complete expulsion of the in situ radiogenic component up to 5 miles north of the Grenville Front. In contrast, muscovites exhibit superior retentivity for radiogenic ⁸⁷Sr and yield typical Kenoran ages within 2 miles north of the front. No anomalously high muscovite ages were determined.

5. From this study the order of reliability for measurement of primary (premetamorphic) age based on mineral and method is a follows: (1) whole-rock, Rb-Sr isochron; (2) hornblende, K-Ar; (3) muscovite, Rb-Sr; (4) muscovite, K-Ar; (5) biotite, Rb-Sr; and (6) biotite, K-Ar.

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APPENDIX I

Sample Identification Key

This Paper	Field Number	Publication Number ¹	Location
1	SH-54–62	G.S.C63–136, 137	49-36-15 N, 74-26-29 W.
2	WN-3-66		49-36-05 N, 74-26-40 W.
3	WN-10A-66		49-33-45 N, 74-23-44 W.
4	WN-9A-66	_	49-33-45 N, 74-23-45 W.
5	SH-53-62	G.S.C63-153, 154	49-33-45 N, 74-23-45 W.
6	WN-11-66		49-34-46 N, 74-20-22 W.
7	SH-18-63	G.S.C63-143, 144	49-34-50 N, 74-19-25 W.
8	WN-17-66	_	49-32-52 N, 74-20-20 W.
9	SH-17-63	G.S.C63-145, 146	49-32-40 N, 74-20-25 W.
10	WN-19-66	_	49-35-45 N, 74-16-37 W.
11	WN-20-66		49-35-55 N, 74-16-20 W.
12	WN-20A-66		49-35-55 N, 74-16-20 W.
13	WN-13B-66		49-29-55 N, 74-24-57 W.
14	SH-9-59	G.S.C60-107	49-35 N, 74-16 W.
15	SH-55-62	G.S.C62-146, 147	49-35-15 N, 74-15-35 W.
16	WN-25-66		49-34-42 N, 74-15-27 W.
17	WN-46-66	-	49-34-33 N, 74-15-14 W.
18	SH-52-62	G.S.C62-148, 149	49-34-30 N, 74-15-05 W.
19	WN-48-66	—	49-34-18 N, 74-14-43 W.
20	SH-8–59	G.S.C60-108; 61-162	49-34-00 N, 74-14-25 W.
21	SH-58-62	G.S.C63–141, 142	49-32-50 N, 74-11-30 W.

¹) See G.S.C. Papers 61–17 to 64–17 inclusive.

APPENDIX II

Sample Descriptions²)

Sample 1. SH-54-62: Biotite muscovite granodiorite. 49-36-15 N, 74-26-29 W.

This is a medium-grained, massive, grey, granodiorite consisting of quartz, oligoclase, biotite, muscovite, and accessory apatite, sphene, and zircon. The feldspar is considerably altered to epidote and sericite but the micas are fresh. The muscovite is euhedral against biotite.

Biotite Concentrate: Clean concentrate of brown biotite. Biotite flakes contain a few inclusions of zircon and quartz. Chlorite content less than 1%.

Muscovite Concentrate: Impure concentrate of muscovite. Most of the muscovite flakes are intergrown with feldspar, quartz, biotite, and epidote. Some flakes are stained buff. Impurities consist of traces of quartz, K-feldspar and plagioclase.

Sample 2. WN-3-66: Biotite granodiorite. 49-36-05 N, 74-26-40 W.

The rock is a medium-grained, massive, grey granodiroite consisting of albite, quartz, ragged anhedral biotite, lesser K-feldspar and epidote, and minor muscovite, apatite and sphene. Occasional rounded zircons have been detected.

Biotite Concentrate: A relatively clean concentrate of unaltered medium-brown biotite. About 60% of the mica flakes contain strong pleochroic haloes surrounding colourless inclusions. The concentrate contains about 5% hornblende impurity.

Sample 3. WN-10A-66: Biotite quartz monzonite. 49-33-45 N, 74-23-44 W.

A medium to coarse-grained, massive, whitish-grey quartz monzonite consisting of approximately

²) Rock samples with numbers prefixed SH- were described by C.H.STOCKWELL in G.S.C. Papers 61-17 to 64-17 inclusive. Rock samples with numbers prefixed WN- were described by R.D.STEVENS.

equal oligoclase, K-feldspar and quartz, with lesser biotite and minor muscovite, epidote, sphene and zircon. Both varieties of feldspar are heavily charged with sericite and epidote. The quartz grain boundaries are distinctly sutured and probably indicate some recrystallization.

Biotite Concentrate: This is a clean concentrate of unaltered dark olive-green biotite with about 1% muscovite impurity. Some 40% of the biotite flakes are so dark as to be almost opaque.

Muscovite Concentrate: Relatively clean concentrate of very slightly brown-stained muscovite with about 5% biotite and a trace of quartz as impurities.

Sample 4. WN-9A-66: Quartz monzonite gneiss. 49-33-45 N, 74-23-45 W.

This is a very coarse-grained, grey gneissic rock consisting of heavily altered, zoned plagioclase of indeterminate composition, strained, shattered and recrystallized quartz, relatively fresh K-feldspar, fresh biotite, granular epidote aggregates, and accessory sphene, apatite, sericite and very fine amphibole in the plagioclase alteration products.

Sample 5. SH-53-62: Biotite muscovite granodiorite. 49-33-45 N, 74-23-45 W.

The rock is a light grey, medium-grained, massive granodiorite composed of quartz, oligoclase, microcline, biotite, muscovite, and a little carbonate, epidote and sphene. The micas are primary and fresh and the rock shows no visible evidence of metamorphism.

Biotite Concentrate: Clean concentrate of brown biotite. Some biotite flakes contain inclusions of quartz and are slightly altered to chlorite. The total chlorite content is about 4%

Muscovite Concentrate: Impure concentrate of muscovite. About 40% of the muscovite flakes are clean. About 60% of the flakes are intergrown with feldspar and contain inclusions of quartz, epidote, apatite and zircon. The total chlorite, quartz, and epidote content is less than 1%. The plagioclase content is about 10%.

Sample 6. WN-11-66: Biotite muscovite quartz monzonite. 49-34-46 N, 74-20-22 W.

This a medium to coarse-grained, massive to very slightly gneissic, grey rock composed of approximately equal quartz and zoned oligoclase-albite, with lesser orthoclase, biotite, muscovite, epidote, and accessory sphene, apatite and rare rounded zircons. Both varieties of feldspar contain abundant inclusions of epidote and very fine muscovite.

Sample 7. SH-18-63: Biotite muscovite granite. 49-34-50 N, 74-19-25 W.

This is a medium-grained, grey, gneissic granite composed of quartz, feldspar, biotite, muscovite, and accessory sphene. The feldspar is considerably altered to sericite and epidote and the biotite to chlorite but the muscovite is fresh. Both micas are slightly bent.

- *Biotite Concentrate:* Reasonably clean concentrate of olive-brownish biotite. About 40% of the biotite flakes are partly bleached and locally altered to pale green chlorite and epidote. Total chlorite content 20%.
- *Muscovite Concentrate:* Reasonably clean concentrate of muscovite. Most of the muscovite flakes contain small inclusions of quartz and feldspar, while some flakes have attached specks of partly altered biotite and are stained yellow. Total chlorite content 2%.

Sample 8. WN-17-66: Gneissic muscovite granite. 49-32-52 N, 74-20-20 W.

This rock is a medium-grained, moderately to strongly gneissic, pale pinkish-brown granite composed of medium and fine grained strained and recrystallized quartz in mosaic texture, coarse to medium K-feldspar (some microcline) with very fine muscovite, epidote and rare zircon inclusions, a few coarse plagioclase (albite-oligoclase) grains with similar inclusions, ragged biotite and lesser muscovite and epidote.

Biotite Concentrate # 1: Relatively clean concentrate of unaltered olive biotite with about 3% hornblende contamination.

Biotite Concentrate # 2: A clean concentrate of unaltered khaki biotite with about 1% hornblende contamination. A few of the mica flakes contain colourless blebs surrounded by strong pleochroic haloes.

Muscovite Concentrate # 1: Relatively clean, slightly greenish muscovite with traces of free chlorite and biotite impurities.

Muscovite Concentrate # 2: This is a mixture of two similar concentrates of clear to slightly stained muscovite with a few included quartz blebs. Impurities consist of biotite and quartz (5%) and a trace of K-feldspar.

Sample 9. SH-17-63: Gneissic biotite muscovite granite. 49-32-40 N, 74-20-25 W.

This is a light grey, medium-grained, gneissic granite composed of quartz, orthoclase, biotite, muscovite, and accessory sphene and zircon. The feldspars are considerably altered to sericite and

epidote. Feldspars and quartz show a recrystallized cataclastic structure but the micas are fresh and undeformed.

Biotite Concentrate: Clean concentrate of brown biotite. Biotite flakes contain a few zircon inclusions surrounded by dark brown pleochroic haloes. Some flakes are slightly bleached and contain inclusions of quartz.

Muscovite Concentrate: Reasonably clean concentrate of muscovite. Some muscovite flakes have attached specks of biotite, quartz, feldspar and epidote.

Sample 10. WN-19-66: Hornblende biotite quartz monzonite. 49-35-45 N, 74-16-37 W.

This is a medium-grained, dark grey, slightly gneissic rock composed of altered orthoclase, lesser altered plagioclase, abundant biotite partly to completely altered to brownish green chlorite, green fibrous chlorite, green to blue-green hornblende, epidote and opaque oxides. Accessory apatite, sphene and zircon were observed. Both feldspars are heavily charged with minute prismatic crystals in great abundance. Identification is doubtful, but this appears to be a mixture of clinozoisite/ epidote and tremolite.

Biotite Concentrate: This is a relatively clean concentrate of slightly altered, bleached olive biotite with about 3% chlorite and 1% quartz contamination.

Sample 11. WN-20-66: Altered diabase. 49-35-55 N, 74-16-20 W.

This rock is a heavily altered, massive, fine-grained, dull black diabase consisting of laths of labradorite embedded in a pseudomorphous, aggregated, and in part amorphous mass of chlorite, very fine amphibole, red-brown biotite, powdery iron oxides, 'talc' and alkali feldspar. The rock has been very deeply altered and the relatively abundant (2%) biotite and powdery oxides are related to this alteration. Primary iron oxides occur as larger grains and skeletal crystals.

Sample 12. WN-20A-66: Biotite quartz monzonite. 49-35-55 N, 74-16-20 W.

This is a medium to coarse-grained, greenish grey, slightly gneissic, rather deeply altered rock consisting of epidotized K-feldspar and oligoclase, strained and recrystallized quartz, fine biotite, and fine to coarsely aggregated yellow epidote. Accessory minerals include opaque oxides, apatite and rare zircons. The fine biotite and the epidote replacing both feldspars appear to be secondary in origin. *Biotite Concentrate* # 1: Relatively clean, unaltered khaki biotite with about 3% hornblende con-

tamination. About 30% of the mica flakes contain apatite inclusions surrounded by pleochroic haloes, and about 5% contain opaque blebs.

Biotite Concentrate # 2: Clean concentrate of olive biotite with about 2% hornblende contamination. Sample 13. WN-13B-66: Hornblende schist. 49-29-55 N, 74-24-57 W.

The rock is a medium to fine-grained, very dark grey to black, strongly lineated schist composed of about 80% elongated, perfectly oriented hornblende rods with fine mosaic quartz in deformed lenticular areas between the amphiboles, and lesser opaque oxide grains and stringers parallel to the lineation. Very thin and discontinuous quartz veins cut the lineation at high angles.

Hornblende Concentrate: Clean, pleochroic blue-green to yellow-green hornblende with tiny colourless inclusions and a trace of quartz contamination.

Sample 14. SH-9-59: Gneissic biotite granodiorite. 49-35 N, 74-16 W.

The granodiorite is a medium-grained, grey, foliated rock composed chiefly of quartz, oligoclase, and biotite. Sericite and epidote are abundant alteration products of feldspar, and epidote is closely associated with the biotite. Although discrete grains of chlorite occur here and there, the biotite is unaltered and may have formed later than the chlorite.

Biotite Concentrate: Biotite flakes are ginger-brown, some slightly bleached. Some flakes contain minute inclusions. Chlorite not detected.

Sample 15. SH-55-62: Paraschist. 49-35-15 N, 74-15-35 W.

The sample is a medium-grained, grey paraschist that has been derived from arkose and forms a steeply dipping bed from 1 foot to 2 feet thick in fine-grained, dark green, hornblende schist of volcanic origin. As seen microscopically, the paraschist consists of closely spaced irregular fragments of feldspar and a few of quartz lying in a fine-grained matrix of the same minerals together with metamorphic flakes of biotite and muscovite which wrap around the fragments. Minor constituents include chlorite, zircon, apatite, and pyrite.

Biotite Concentrate: Reasonably clean concentrate of brown biotite. Biotite flakes contain small inclusions of quartz, a few opaque grains and a few zircons surrounded by dark pleochroic haloes. About 5% of the biotite flakes are partly bleached and slightly altered to chlorite. Impurities, totalling about 5%, consist of chlorite and a few flakes of muscovite.

Muscovite Concentrate: Reasonably clean concentrate of muscovite. Muscovite flakes contain inclusions of feldspar, quartz, and small adhering fragments of pale brown biotite, as well as small euhedral crystals of dark-brown biotite. Quartz content is about 1%. Feldspar content is less than 5%. Chlorite not detected.

Sample 16. WN-25-66: Quartz feldspar muscovite gneiss. 49-34-42 N, 74-15-27 W.

The rock is coarse-grained, light greyish pink, strongly gneissic with schistose shear planes, and small scale augen texture. In thin-section it is a strongly sheared gneissic rock with elongated lenticular areas of strained, recrystallized, drawnout quartz and feldspar (20%) orthoclase, 10% bent and fractured oligoclase). The lenticular areas are sheathed by finely recrystallized, schistose textured zones rich in fine, oriented muscovite, granular to almost euhedral epidote and minor irregular carbonate patches. Accessory minerals include tiny opaque grains, occasional zircons, and unidentified acicular rods.

Muscovite Concentrate: A clean concentrate of very slightly ironstained muscovite with a trace of hornblende inpurity.

Sample 17. WN-46-66: Quartz feldspar muscovite gneiss. 49-34-33 N, 74-15-14 W.

The sample is a strongly gneissic, medium to coarse-grained, greyish pink rock consisting of quartz, K-feldspar (including microcline), albite, strongly oriented muscovite, granular to subhedral epidote, and accessory apatite, opaques, sphene and zircons.

Muscovite Concentrate: This is a mixture of two similar concentrates of very slightly brown-stained muscovite with quartz inclusions. Total quartz contamination amounts to less than 5%.

Sample 18. SH-52-62: Micaceous gneiss. 49-34-30 N, 74-15-05 W.

The sample is a medium-grained light grey gneiss which is strongly sheared with the development of plentiful, parallel flakes and shreds of muscovite and lesser amounts of biotite. Other constituents include abundant quartz, some feldspar and a little epidote, titanite, apatite, and pyrite.

Biotite Concentrate: Reasonably clean concentrate of brown biotite. Minor impurities consist of green chlorite and epidote. Total chlorite content is about 6%.

Muscovite Concentrate: Reasonably clean concentrate of muscovite. About 15% of the muscovite flakes contain inclusions of feldspar, quartz and a few specks of epidote. The total chlorite, quartz, and epidote content is less than 1%. The plagioclase content is about 5%.

Sample 19. WN-48-66: Quartz microcline biotite gneiss. 49-34-18 N, 74-14-43 W.

The rock is a fine-grained, strongly gneissic, equigranular reddish pink gneiss consisting mainly of quartz, microcline and other K-feldspars, oriented biotite, minor plagioclase, muscovite and epidote, with accessory apatite, opaques, sphene and possible zircon.

Sample 20. SH-8-59: Orthogneiss. 49-34-00 N, 74-14-25 W.

The orthogneiss is a medium-grained, grey, quartz-feldspar-biotite rock with some muscovite, and the rock is permeated with stringers of coarse, pinkish grey, muscovite granite. The feldspar includes oligoclase and lesser amounts of orthoclase and microcline. The biotite is brown, clear, and unchloritized and both it and the muscovite are in roughly parallel flakes. Epidote and apatite are minor constituents.

Biotite Concentrate: About 5% muscovite is present. Biotite flakes vary in colour from brown to olive-green. Chlorite not detected.

Muscovite Concentrate: Contains about 85% clean muscovite flakes, 10% muscovite intergrown with quartz, and 5% muscovite intergrown with biotite. Only a trace of chlorite is present.

Sample 21. SH-58-62: Granitic gneiss. 49-32-50 N, 74-11-30 W.

The granitic gneiss is a medium-grained rock in which grey and pink layers alternate. It consists of quartz, oligoclase, biotite muscovite, epidote, and accessory apatite, sphene, and zircon. Most of the biotite is fresh but a few crystals are interleaved with chlorite and a dark opaque material. The muscovite crystals are euthedral against biotite.

- Biotite Concentrate: Reasonably clean concentrate of dark olive-greenish biotite. Some flakes are almost opaque. About 15% of the biotite flakes are in part altered to chlorite and contain small inclusions of epidote. Total chlorite content 8%.
- Muscovite Concentrate: Reasonably clean concentrate of muscovite. Some muscovite flakes are intergrown with green biotite and contain inclusions of quartz and red crusts of hematite. Total chlorite content 4%.

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APPENDIX III

Experimental Techniques

a) K-Ar Analyses

The experimental procedures developed have been discussed in the annual age determination reports of the Geological Survey of Canada. Recent modifications include the application of an A.E.I. MS10 mass spectrometer operated in the static mode for argon analyses, and the use of digital recording of ion currents. Samples are placed in molybdenum crucibles for fusion in vacuo and the released gases are purified by passage through CuO at 450 °C and over titanium sponge at 900 °C. Isotope dilution techniques are used for potassium determination in hornblendes whereas X-ray fluorescence techniques are employed for micaceous and whole rock samples. b) Rb-Sr Analyses

The rubidium and strontium concentrations of whole rock samples (-200 mesh) were determined using isotope dilution techniques (WANLESS and LOVERIDGE, 1969). The analyses were carried out on a 10 inch radius, 90 degree, Nier-type mass spectrometer. Prebaked triple filaments (tantalum side filaments, rhenium centre filament) were used throughout, and samples were loaded on the side filaments as Rb_2SO_4 and $Sr(NO_3)_2$. Ion currents were detected with a 9 stage electron multiplier, the output of which was amplified by a vibrating reed electrometer. A correction proportional to the square root of the isotopic mass was applied to all spectra to compensate for electron multiplier discrimination. The earlier analyses of this project were recorded on a strip chart recorder and required a correction for the non linear response of this instrument to a maximum of 0.5% of the recorded peak height. The later analyses were measured by means of integrating digital voltmeter and did not require this correction.

All strontium analyses were normalized to a 86 Sr/ 88 Sr ratio of 0.1194; directly for unspiked analyses, and through the use of a 84 Sr spike for spiked analyses. Six analyses of the MIT SrCO₃³) isotopic standard solution yielded a 87 Sr/ 86 Sr ratio of 0.7085 \pm 0.0004 when normalized to a 86 Sr/ 88 Sr ratio of 0.1194. The uncertainty in a single determination of the 87 Sr/ 86 Sr ratio is in the order of 0.14 per cent (i.e. 0.7000 \pm 0.0010). This uncertainty has been reduced for some samples through the use of replicate analyses. The uncertainty in Rb and Sr elemental determinations is in the order of 2 per cent yielding an uncertainty in the 87 Rb/ 86 Sr ratio of \pm 3 per cent. The isochron slope and intercept were calculated using the computer program developed by YORK (1966).

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