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Thermally-induced Migration of Rb and Sr in an Adamellite

by H. BAADSGAARD¹⁾ and O. VAN BREEMEN²⁾

ABSTRACT

Heating of a coarse-grained, two-mica quartz monzonite or adamellite in air produces definite migration and relocation of Rb and Sr without volatilization loss. Rb and Sr^{87r} are lost by muscovite and biotite (in slightly different ways) while normal Sr increases in both minerals. Homogenization of sodic plagioclase and microcline towards a single feldspathic phase makes it difficult to establish the change in Sr^{87r} content in the plagioclase. Apatite definitely gains Sr^{87r} and a little Rb. Migration is detectable only above 800°C, employing a 100-hour heating period, and extensive partial fusion occurs at 1025°C. Microprobe studies indicate no apparent diffusion gradients within mineral grains nor any significant intergrain accumulation of Na, K, or Ca. In this simple experiment the ability of an ion to migrate between silicate lattices is apparently more a function of the chemical-thermodynamic factors involved in mineral transformation than of volume diffusion. When allowable, the migration of an ion appears to proceed relatively rapidly and reversibly.

Introduction

Since the advent of the Nicolaysen (NICOLAYSEN, 1961) Rb-Sr isochron method for determining the Rb-Sr date of rocks and minerals, the whole-rock versus mineral "isochron" methods have become increasingly employed in distinguishing between the time of metamorphism of a rock and its "primary" age of formation. Numerous investigators have found rocks whose Rb-Sr mineral isochron date has been essentially completely reset by metamorphism. A number of investigators have further found situations in which the mineral isochron has not been completely reset (for example, YASHCHENKO et al., 1961; ARRIENS et al., 1966; WHITE et al., 1967) or apparently involves a relatively large scale metasomatic transfer of radiogenic strontium (WASSERBURG et al., 1964; PANKHURST, 1969). Therefore, an attempt was made to experimentally induce and follow the migration of Rb and Sr within a silicate rock under known conditions.

Experimental Material and Procedure

Since the migration and relocation of Sr^{87r} (radiogenic Sr) is perhaps of the greatest interest, an unaltered rock with a relatively high Sr^{87r}-content is desirable as the silicate phase. The so-called Prosperous Lake Granite (actually an adamellite or quartz monzonite) was chosen as the experimental material. A Rb-Sr isochron for

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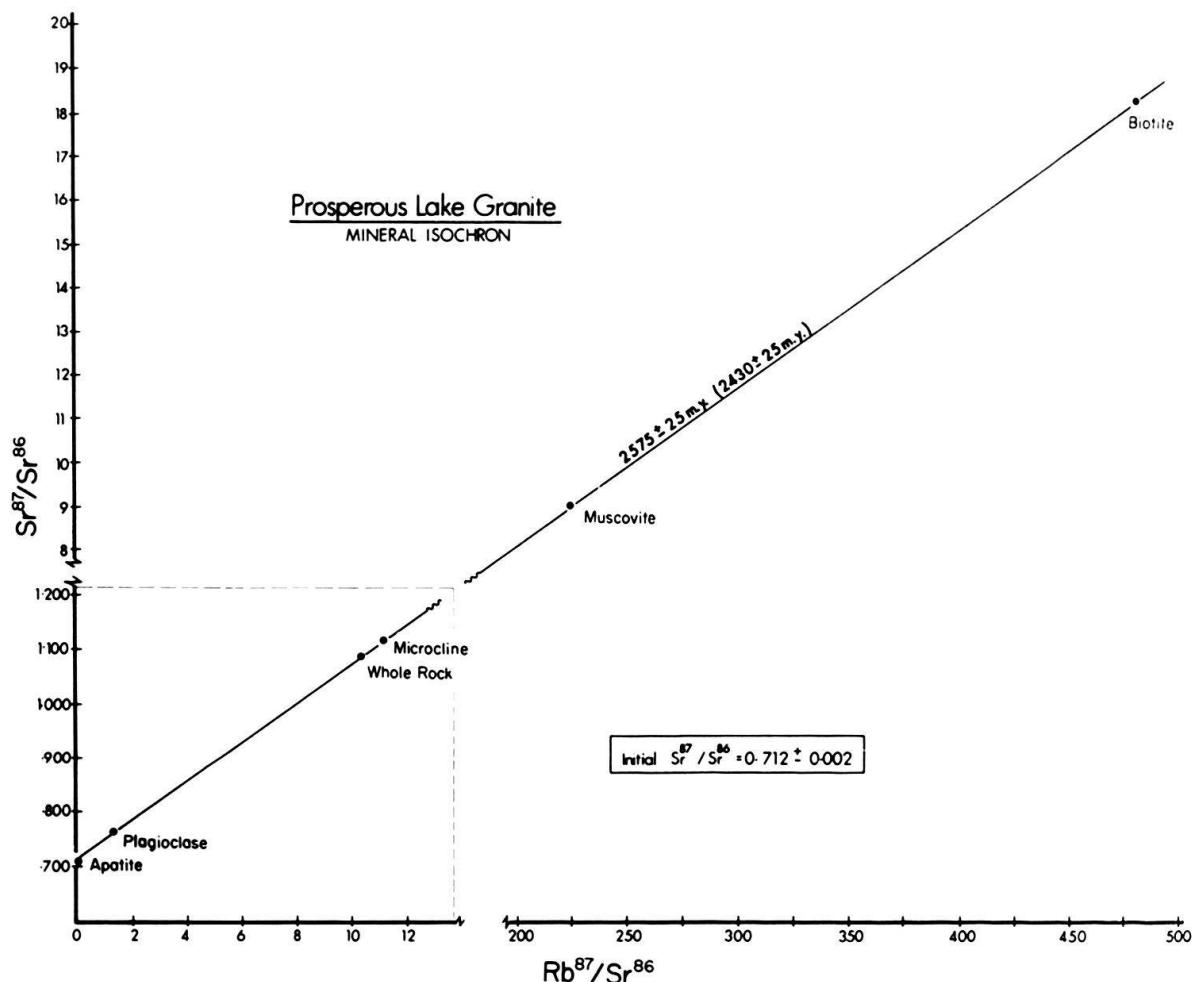


Fig. 1. Mineral isochron for the Prosperous Lake Granite, N.W.T., Canada.

the minerals of this rock is given in Figure 1 and an analysis of the whole rock is given in Table 1 together with the C.I.P.W. norm calculated from the analysis. A brief petrographic description of the rock may be found in the Appendix. Table 2 gives the results of a modal analysis on six slides cut from different orientations in a 120-lb. piece of the rock. The results of the modal analysis reflect the relatively coarse-grained nature of the rock. In order to obtain suitably pure mineral separates for subsequent analysis, this coarse grain size was necessary, though possible diffusion processes would be made more difficult to induce.

Experimental Procedure

It was decided to carry out the simplest procedure and initially study only the isotopic rearrangements induced by heating in air. Accordingly, from 4 to 5 kilograms of unweathered rock in 3 or 4 pieces were placed about a thermocouple in the most evenly-heated zone of a muffle furnace (which was already heated to desired temperature). Small pieces of the source rock were used to keep the sample from touching the bottom and sides of the furnace. Within two to three hours the charge was at the

Table 1. Chemical Analysis of Prosperous Lake «Granite» and the C.I.P.W. Norm.

SiO ₂	74.09	C.I.P.W. Norm	
TiO ₂	0.14	Qz	31.51
Al ₂ O ₃	14.30	Or	30.01
Fe ₂ O ₃	0.18	Ab	30.53
FeO	0.99	An	2.37
MnO	0.02	Co	2.00
MgO	0.31	Hy (En	2.25
CaO	0.82	(Fs	
Na ₂ O	3.61		
K ₂ O	5.08	Mt	0.26
P ₂ O ₅	0.26	Ap	0.62
H ₂ O+	0.40	Ca	----
H ₂ O-	0.04	H ₂ O	0.44
	100.25		100.26

Analysts: L.R. Campbell, A. Rich and
L. Yopik, University of Alberta

Table 2. Modal Analysis, Prosperous Lake «Granite».

Slide Mineral	1	2	3	4	5	6	Average $\pm \sigma$
Quartz	38.45	40.02	33.51	27.00	26.53	37.58	33.85 \pm 5.9
Plagioclase	30.41	30.56	26.80	40.59	39.49	34.33	33.70 \pm 5.5
K-Feldspar	18.82	20.61	20.76	24.55	26.91	21.80	22.24 \pm 3.0
Muscovite	10.22	6.23	17.07	3.88	4.31	4.53	7.71 \pm 5.2
Biotite	1.97	2.14	1.38	3.86	2.59	1.46	2.23 \pm 0.9
Opaque	.11	.40	.44	.09	.15	.29	.25 \pm 0.2
Total %	99.98	99.98	99.96	99.97	99.98	99.99	99.98
Total Count	3071	1829	2003	1621	2575	1500	2100
Area cm ²	3.41	2.03	2.23	1.80	2.86	1.66	2.33
Plag./K-Feld.	1.62	1.48	1.29	1.65	1.47	1.57	

controlled temperature ($\pm 10^{\circ}\text{C}$) and held there for a total period of 100 hours. At the end of the heating period, the samples were removed and allowed to cool in air to room temperature, crushed, sieved to various size fractions and mineral separations carried out. The unheated rock yielded very pure separates of the principle minerals in the 60–80 mesh size fraction (0.177–0.250 mm), but the mineral separations on the heated samples became progressively more difficult with the higher temperatures of heating since chemical transformations had taken place. At 985° and at 1025°C (the temperature at which the rock partially fused), the individual mineral separates of the

Table 3. Isotopic Analytical Data for Mineral Separates.

MINERAL	TEMP., °C	UNHEATED	800	860	920	985	1025
BIOTITE:							
ppm Sr ⁸⁷ r		14.65	15.68	13.87	9.91	8.34	3.39
ppm Sr ^N		8.52	9.26	10.09	11.27	17.17	40.93
ppm Rb	1435.	1496.	1115.	760.	414.	285.	
Sr ⁸⁷ /Sr ⁸⁶	18.27	18.02	14.77	9.70	5.68	1.56	
Rb ⁸⁷ /Sr ⁸⁶	486.	467.	320.	195.	69.7	20.1	
MUSCOVITE:							
ppm Sr ⁸⁷ r		6.76	6.79	5.72	5.60	5.14	3.95
ppm Sr ^N		8.35	9.39	10.48	15.21	23.81	24.96
ppm Rb	652.	662.	620.	588.	507.	391.	
Sr ⁸⁷ /Sr ⁸⁶	8.98	8.11	6.29	4.47	2.919	2.328	
Rb ⁸⁷ /Sr ⁸⁶	225.	204.	171.	112.0	61.6	45.2	
K-FELDSPAR:							
ppm Sr ⁸⁷ r		6.31	6.51	6.30	6.21	3.80*	
ppm Sr ^N		160.	169.	168.	167.	129.9*	
ppm Rb	621.	621.	596.	592.	352.*		
Sr ⁸⁷ /Sr ⁸⁶	1.112	1.107	1.096	1.090	1.010*		
Rb ⁸⁷ /Sr ⁸⁶	11.18	10.63	10.24	10.22	7.85*		
PLAGIOCLASE:							
ppm Sr ⁸⁷ r		0.50	0.77	0.55	0.63		
ppm Sr ^N		99.2	110.	119.	113.		
ppm Rb	48.1	70.5	57.9	64.8			
Sr ⁸⁷ /Sr ⁸⁶	0.762	0.783	0.758	0.768			
Rb ⁸⁷ /Sr ⁸⁶	1.399	1.849	1.405	1.664			
APATITE:							
ppm Sr ⁸⁷ r				0.012	0.123	0.406	1.689
ppm Sr ^N				90.6	99.0	89.4	117.4
ppm Rb				3.72	3.55	4.21	12.73
Sr ⁸⁷ /Sr ⁸⁶	0.709	0.710	0.714	0.724	0.758	0.858	
Rb ⁸⁷ /Sr ⁸⁶			0.119	0.104	0.136	0.313	
WHOLE ROCK							
ppm Sr ⁸⁷ r		2.739				2.641	
ppm Sr ^N		75.9				74.4	
ppm Rb		268.				264.	
Sr ⁸⁷ /Sr ⁸⁶		1.079				1.076	
Rb ⁸⁷ /Sr ⁸⁶		10.22				10.24	

* Values for combined feldspars, 985°C. sample.

feldspars could not be obtained and what had been 'biotite' became very difficult to obtain even as an apparently pure 'mineral' separate. Except for the biotite separates at 985°C (about 90–95% 'pure') and 1025°C (about 85–90% 'pure'), all the other mineral fractions were greater than 95% pure. Isotope dilution analyses were then

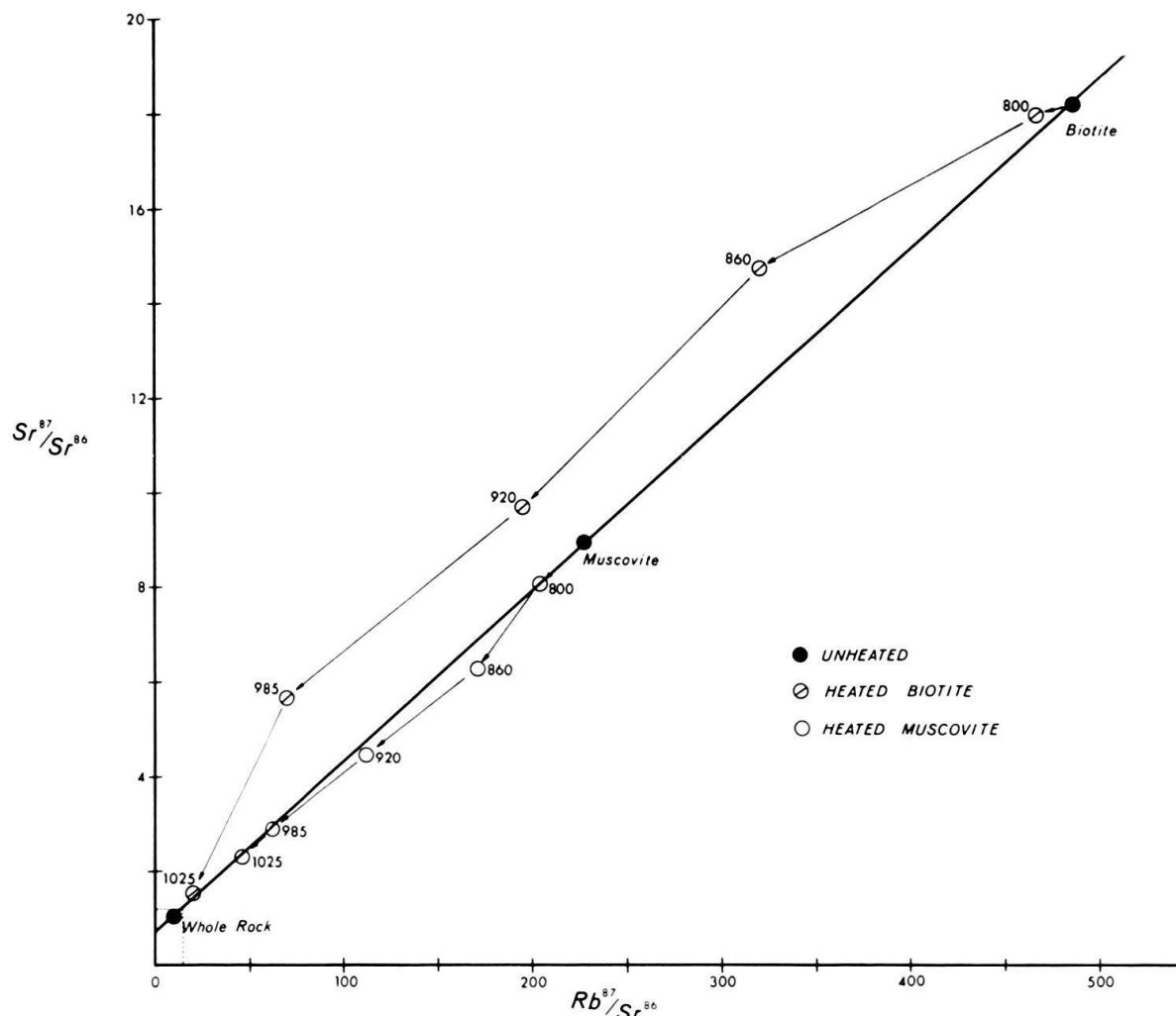


Fig. 2. Isochron plot of unheated and heated mica samples from the Prosperous Lake Granite. The numbers refer to the temperature (°C) of heating.

carried out for Rb and Sr using chemical decomposition and cation exchange separation techniques. Isotope analyses were made on a six-inch single-filament solid source mass spectrometer employing peak switching with a digital voltmeter output. The results are given in Table 3 and plotted in Figures 2 and 3 on an isochron plot.

Figure 2 shows that the heating has effected changes which progressively change the ratios Rb^{87}/Sr^{86} and Sr^{87}/Sr^{86} for the micas with temperature and that the pattern of change is different for biotite and muscovite. Ignoring for the moment possible variations in Sr^N (normal Sr) in these two minerals, it appears that biotite loses Rb and Sr^{87r} relatively more rapidly than muscovite. The K-feldspar (Figure 3) moves only very slightly off the original isochron, but in a direction which indicates some possible Rb and Sr^{87r} loss or Sr^N gain. The plagioclase changes only slightly and erratically while the apatite definitely gains Sr^{87r} but very little Rb. The fused whole rock sample shows that no significant loss of Rb or Sr occurs by volatilization.

Because of the partial melting of the rock at 1025°C, no meaningful feldspar separation could be made. Only incipient fusion occurred at 985°C, and it was possible

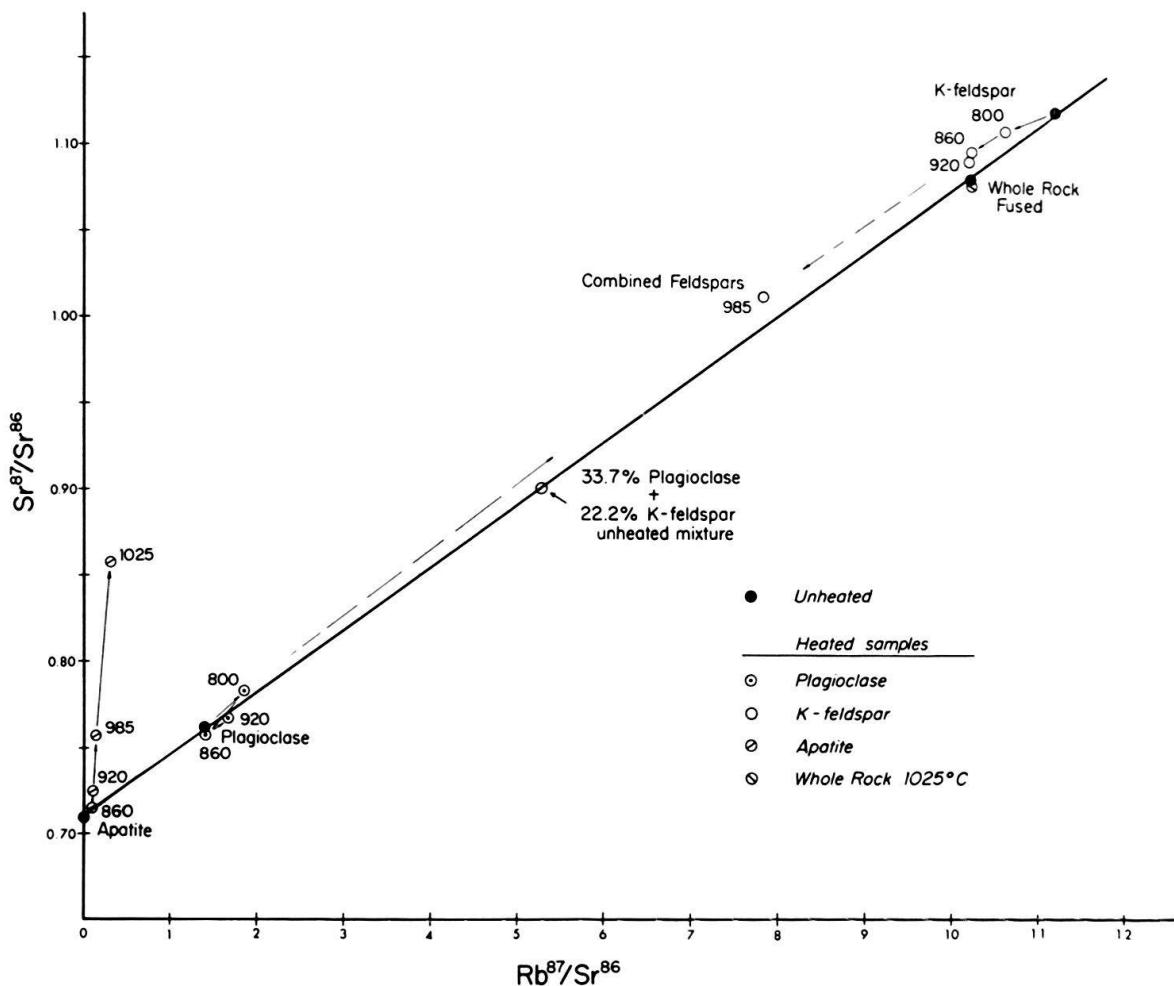


Fig. 3. Isochron plot of unheated and heated apatite, K-feldspar, and plagioclase samples from the Prosperous Lake Granite.

to separate a non-magnetic 'combined' feldspar (60–80 mesh) fraction from quartz. This 'combined' feldspar separate was also analyzed isotopically and is seen to plot clearly above the original isochron at a point approximately between the two feldspar fractions. If one simply mixes the unheated K-feldspar and plagioclase in their modal proportions, the mixture plots at $\text{Sr}^{87}/\text{Sr}^{86} = 0.901$ and $\text{Rb}^{87}/\text{Sr}^{86} = 5.28$. This indicates that the plagioclase has acted as the recipient of most of the Sr^{87r} and Rb lost by the micas and possibly the little from the K-feldspar. The apatite also has gained Sr^{87r} , of course, but since there is so little of it in the rock, its role in accounting for the Sr^{87r} lost from the micas is very small. A material balance for Sr^{87r} and Rb for the essentially unchanged rock (the slight increase in Rb and Sr in the 800°C versus unheated samples is taken to be mainly a reflection of sample inhomogeneity and so the results are averaged) versus the 985°C samples is given in Table 4. This shows that the changes in Rb and Sr^{87r} are accountable well within probable errors (of the modal analysis alone), but show a bias towards extra Rb and Sr^{87r} in the heated feldspars. This is quite likely due to the fact that the separated combined feldspars do not have exactly the same proportion of 'K-feldspar' to 'plagioclase' as given by the

Table 4. Material Balance Calculation on Sr^{87r} and Rb (the mean values of unheated and 800°C samples taken as essentially unchanged sample). Unchanged vs 985°C samples.

MINERAL	MODE per cent	ppm Sr^{87r}		ppm-per cent Sr^{87r}		ppm Rb		ppm-per cent Rb	
		unheated	+ 800	unheated	+ 800	unheated	+ 800	unheated	+ 800
		2	985	2	985	2	985	2	985
BIOTITE	2.2	15.17	8.34	33.4	18.3	1466	414	3225	911
MUSCOVITE	7.7	6.78	5.14	52.2	39.6	657	507	5059	3904
PLAGIOCLASE	33.7	0.64	3.80	21.6	212.4	59	352	1988	19677
K-FELDSPAR	22.2	6.41		142.3		621		13786	
TOTALS				249.5	270.3			24058	24491

Note:

For mixing of unheated feldspars: $\frac{22.2 \times 6.41 + 33.7 \times 0.64}{55.9} = 2.93 \text{ ppm } \text{Sr}^{87r}$; mixed 985°C feldspar has 3.80 ppm Sr^{87r}

modal analysis of the original sample, and arises from the gravity separation procedure and inhomogeneities in the original rock sample. The two feldspars should tend to homogenize at all temperatures above the Ab-Or subsolidus two-feldspar region, and certainly at 800°C and above (BOWEN and TUTTLE, 1950). The gravity separation procedure for the plagioclase and K-feldspar thus biases the separates towards unmodified grains and explains why the plagioclase points in Figure 3 do not show a definite increase in Sr^{87r} and Rb. Obtaining a truly representative sample of the heated feldspars becomes very difficult due to the onset of mixing and resultant change in specific gravity. Thus, the interpretation of the changes in K-feldspar and plagioclase in Figure 3 becomes problematical and at most indicates a slight tendency for change in the K-feldspar.

Microprobe Analysis

To clarify the nature and composition of the mineral samples and investigate changes in the allied major element chemistry, microprobe analyses were carried out. The identical samples used for isotope analysis were analyzed for Ca, K and Na (see Table 5). The 60–80 mesh grains were mounted and 20–50 grains were investigated in five separate places within each grain, depending upon how homogeneous the grains were. Qualitatively, the unheated mineral grains were relatively homogeneous with regard to Na, Ca and K distribution except for the plagioclase, in which the Ca variation was pronounced.

At 985°C the feldspars have mixed extensively, with Na being a little more thoroughly redistributed than K (see Fig. 4 and 5). The Na and K frequency distributions appear to be normal in Figures 4 and 5, but in fact the distribution of 'original' grains of either K-feldspar or plagioclase is skewed toward the distribution in the unheated samples. This is more clearly seen in the plot of K-Na versus Ca in Figure 6. The wide distribution of Ca in plagioclase is shown, and the tail of plotted points towards the unheated K-feldspar region can only be original K-feldspar with more or less Na added and K lost.

Table 5. Microprobe Analytical Data for Ca, K and Na on Mineral Separates*.

MINERAL:	ELEMENT	Temp., °C.	Average counts/10 sec., corrected for background**				
			Unheated	800	860	920	985
Biotite: (contaminant)	Ca		14	7	7	10	26
	Ca					(141)	63
	K		3620	3590	2570	1850	1602
	K					(2557)	1277
	Na		3	70	185	360	421
	Na					(891)	396
Muscovite:	Ca		0	3	8	1	12
	K		4060	4070	3830	3130	2420
	Na		80	105	140	410	630
K-Feldspar:	Ca		1	-2	0	-1	0***
	K		6190	6090	5910	5540	3600***
	Na		90	140	185	280	900***
Plagioclase:	Ca		400	400	410	375	400***
	K		35	95	160	320	3600***
	Na		1939	1870	1800	1800	900***
Apatite:	Ca		11520	11620	11700	11470	11520
	K		1	8	2	4	3
	Na		12	14	13	16	13
							11730
							2
							23

*** Taken from frequency distribution plot in Figure 6.

** Approximate background values: K, 60-70 cts./10 sec.; Na, 12-14 cts./10 sec; Ca, 50-70 cts./10 sec.

* The precision in the results decreases with increasing inhomogeneity of the sample as well as decreasing amount of the constituent, but range from 4 to 19 per cent (1σ) except for Ca in plagioclase. The day to day reproducibility of the microprobe counting rates may be seen in the results for apatite. Quartz gave background count values for all separates.

Microprobe results on the other mineral separates are found in Table 5 and plotted in Figure 7, in which the mobility of sodium is readily apparent. Approximately 90 per cent of the Na in the rock is present in the plagioclase, and it is readily taken up in K-feldspar, muscovite and biotite. Quartz and apatite show no measurable change in Ca, Na, K, Sr or Rb except for the fused apatite, where a small increase in the amount of Sr^N , Na and Rb is detected. Potassium is lost from the K-feldspar and micas and acquired by the plagioclase, while calcium is relatively immobile except at the highest temperatures. Rb variations parallel the changes in K (see Table 3). A plot of the variation in radiogenic strontium in the minerals is shown in Figure 8. The variation for Sr^N in the micas is superimposed on this diagram and shows that Sr^N is gained by the micas as Sr^{87r} is lost. Also, biotite loses its Sr^{87r} much more easily than muscovite.

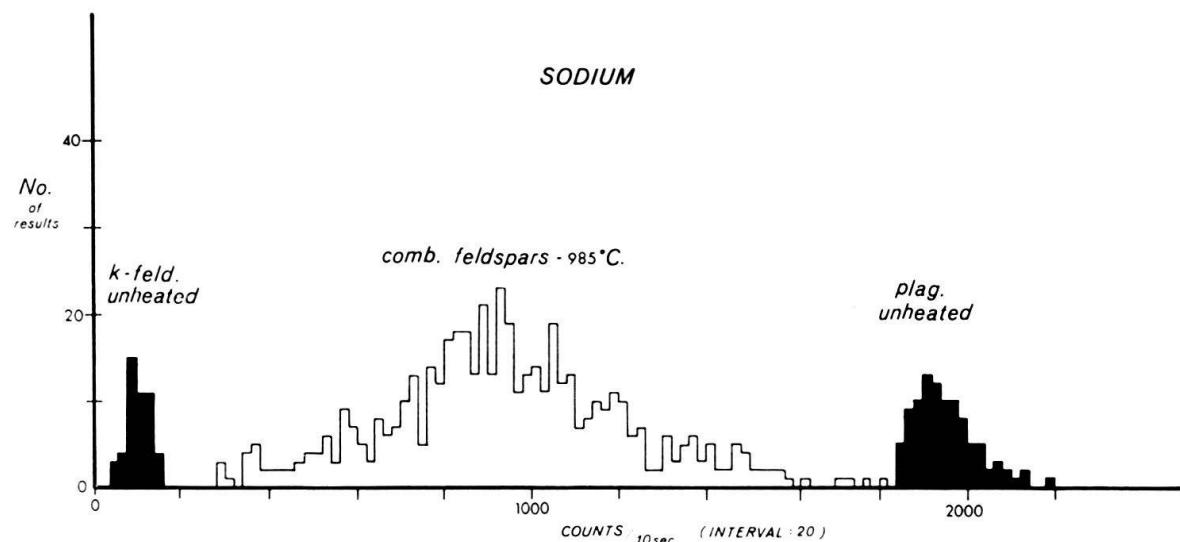


Fig. 4. Frequency distribution plot of the sodium content in the 985°C combined feldspars compared with that for unheated K-feldspar and plagioclase.

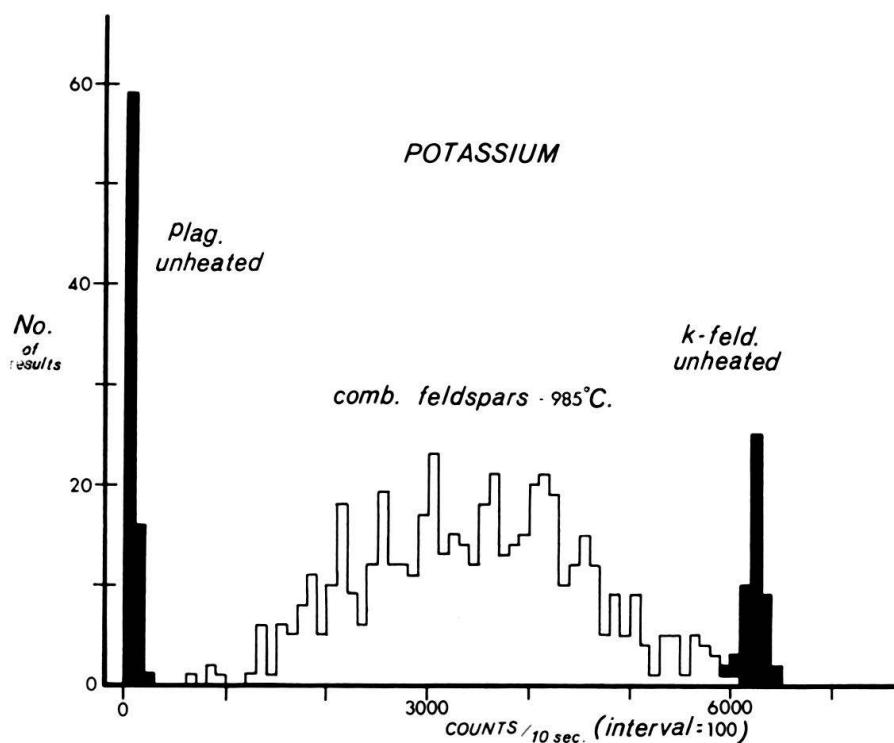


Fig. 5. Frequency distribution plot of the potassium content in the 985°C combined feldspars compared with that for unheated K-feldspar and plagioclase.

It is difficult to explain how Sr^{87r} leaves a phase when normal Sr is entering the same phase unless we have a mixing process occurring. With increasing temperature the relative amounts of Sr^{87r} lost and Sr^N gained in the biotite are almost directly inverse, but this is not the case for the muscovite.

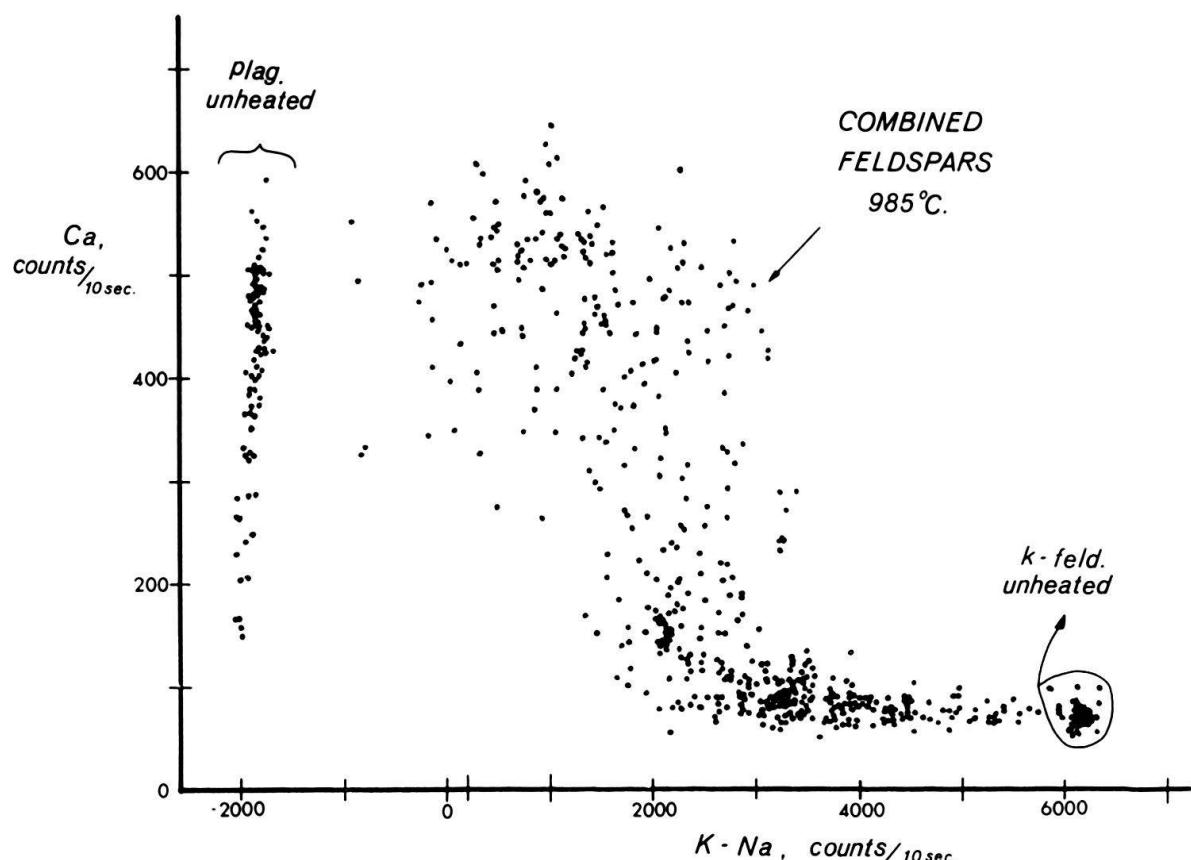


Fig. 6. Plot of K-content minus Na-content vs. calcium-content in 985°C combined feldspars. Unheated K-feldspar and plagioclase are plotted for comparison.

Samples of the unheated and heated whole rock were also examined with the microprobe. Scans of from 500 to 1000 microns were made across a number of mineral grain boundaries in order to examine possible chemical changes in the nature of grain boundary areas. Figure 9 is a faithful tracing of a scan on unheated rock and on the 1025°C rock sample, except that responses due to obvious cracks in the mounted sample have been eliminated. The optically-observed grain boundaries are marked with vertical lines, and the mineral is identified mainly by direct observation of either the grain appearance (micas) or the color of the fluorescent spot produced by the electron beam. The K-feldspar (K?) in the heated sample is not certain and may be a low Ca zone in the plagioclase which has picked up slightly more potassium. (The fluorescent spot color was only faintly changed.) With a scan rate of approximately one inch per minute and a 1.5 second time constant, it can be seen from Figure 9 that the grain boundaries are very sharp in the unheated sample and just as sharp in the 1025°C sample. Moreover, there are no apparent diffusion gradients within the grains. In all the samples scanned, no definite diffusion gradients could be seen, even in 1000 micron scans across large muscovite grains taken from samples at each of the temperatures. Yet the K and Na changed markedly enough for possible gradients to be seen unless the movement of the K and Na into or out of the mineral grains was rapid (compared with 100 hours heating time). No significant intergrain or grain boundary

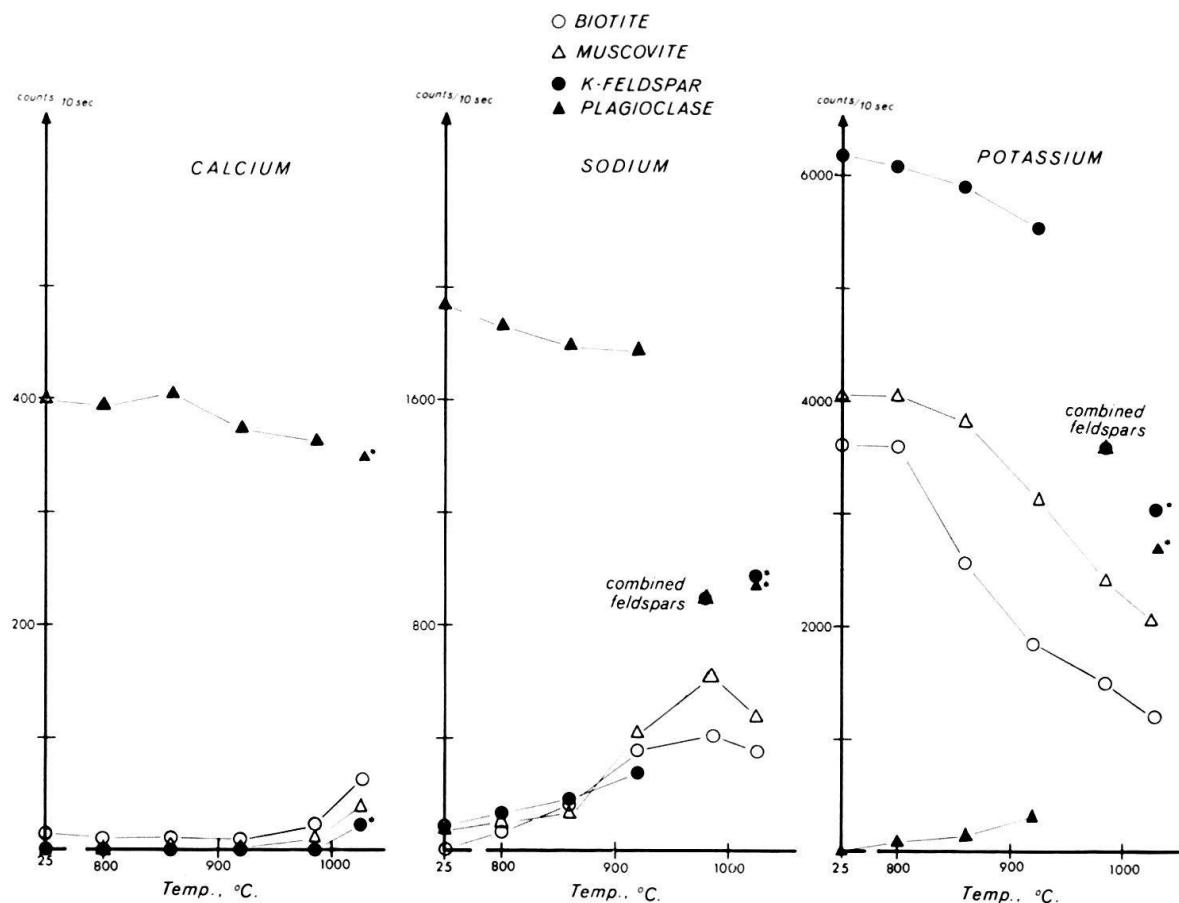


Fig. 7. Change in Ca, Na and K-content of minerals of the Prosperous Lake Granite with increasing temperature of heating (100 hour heating period). Sample points marked with * are estimates from microprobe scanning of whole rock samples.

accumulations or changes were observed. In view of this, it is perhaps reasonable to infer that the dynamics of initial migration of the various constituents observed above is mainly controlled by the degree to which a mineral phase becomes unstable and changes its composition to a new phase. This migration and transfer is in turn modified by the existing structure and chemical nature of the phase insofar as gaining new constituents is concerned. Thus, quartz and apatite are little affected even when one of the contiguous mineral phases is fused, the feldspars mix relatively quickly and the micas change rapidly because they are the least stable under the new conditions and are going over rapidly to new minerals. The conversion of the micas begins with biotite being rather completely oxidized at temperatures where the bound water is not yet completely driven off (see Table 6). Even at 920°C a small amount of the original bound water yet remains in the micas. By 985°C all the H₂O is gone. It is interesting to note that the oxidation of the mica does not seem to immediately promote Rb or K losses and this may be due to the fact that the other phases have not time or stringent enough conditions to accept this K or Rb. When conditions are suitable for both loss and acceptance of a constituent, then it apparently migrates readily.

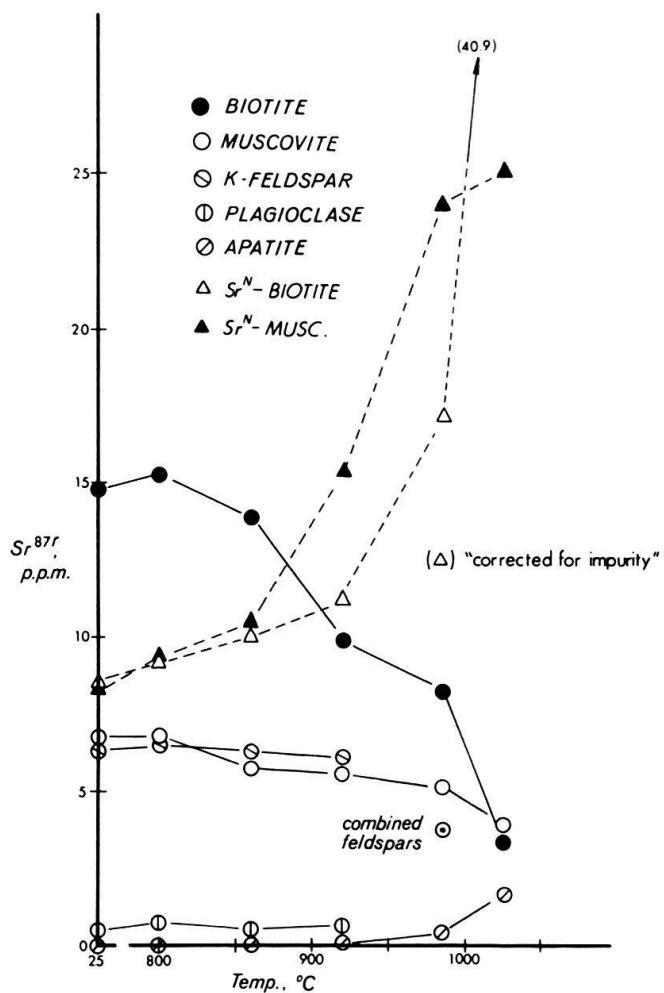


Fig. 8. Changes in the radiogenic strontium content of minerals from the Prosperous Lake Granite with increased temperature of heating. The normal strontium-content changes are also plotted and are indicated by the dashed lines.

The actual movement of the constituents in this experiment is still probably diffusion through a lattice, perhaps somewhat along a grain boundary, and into another lattice where it is more or less acceptable. Although 80% of the water in the micas is lost by 800°C, very little ion migration has taken place. This would seem to indicate that water plays a relatively slight role (in this case) in the changes that have taken place upon progressive heating.

Summary

It should be born in mind that the separated mineral samples are not necessarily representative of the total mineral in the heated samples since obtaining a sufficiently pure mineral separate involved the discarding of much of the original mineral. In particular, the heated plagioclase and K-feldspar separates were somewhat biased as to average composition because of the gravity separation procedure.

Notwithstanding this problem, simple heating of an adamellite in air for 100 hours causes migration of Rb, Sr, K, Ca, Na between various mineral constituents. Oxidized

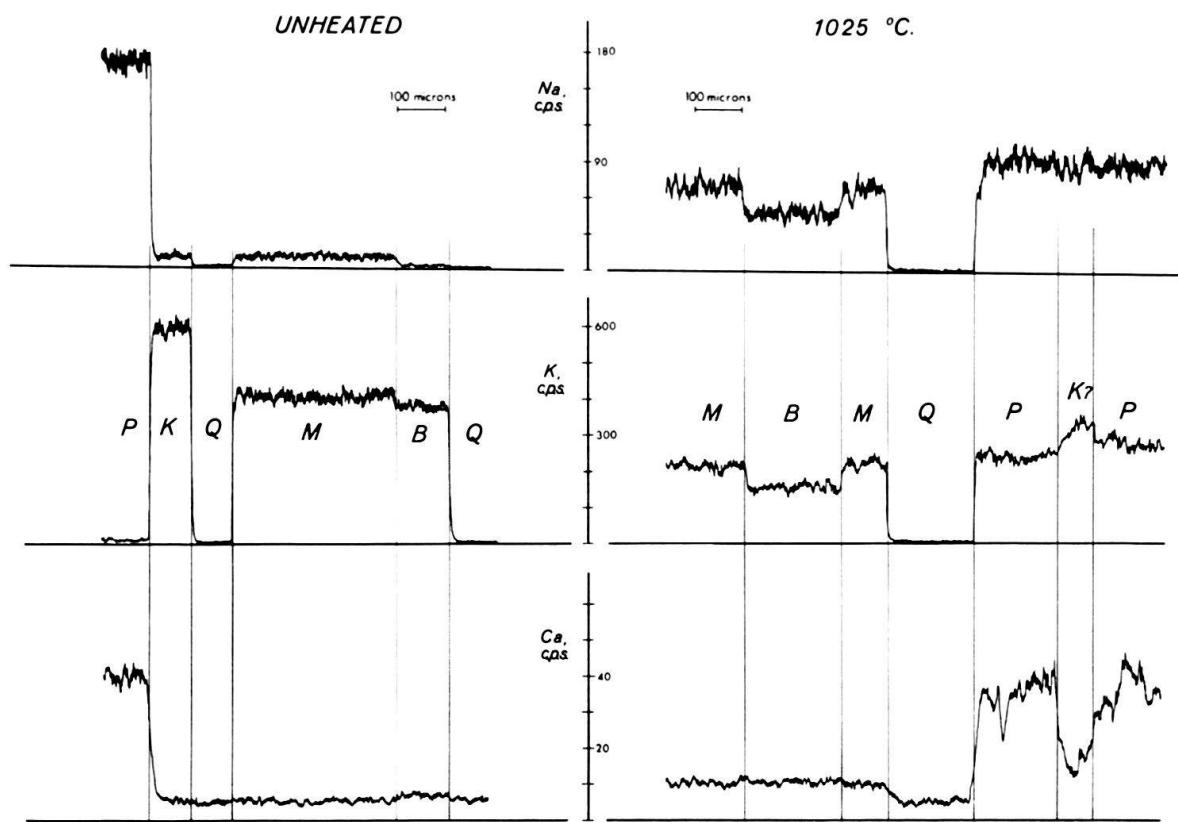


Fig. 9. Electron microprobe scans recording Na, K and Ca response from sections of the Prosperous Lake Granite heated to 1025°C and unheated. P = plagioclase, K = K-feldspar, Q = quartz, M = muscovite and B = biotite.

Table 6. H_2O and $\text{FeO-Fe}_2\text{O}_3$ Determinations on Micas From the Prosperous Lake Granite.

Mineral	Temp., °C	Unheated	800	860	920
Biotite:					
% H_2O		4.60	0.70	0.54	0.36
% FeO		19.82	<0.02	<0.02	<0.02
% Fe_2O_3		5.39	27.42	27.67	27.74
Muscovite:					
% H_2O		4.84	1.22	0.91	0.32

biotite loses Rb and Sr^{87r} more easily than muscovite, while both minerals gain Sr^N . Based on the original $(\text{Sr}^{87}/\text{Sr}^{86})_0$, intermediate (800–900°C) heating for 100 hours thus produces a biotite with too high an apparent age while muscovite yields too low

an apparent age. Plagioclase gains Sr^{87r} in greater relative quantity than Rb to yield higher apparent ages, while the K-feldspar loses relatively more Rb than Sr^{87r} to produce also high apparent ages.

The ability of an ion to migrate between silicate lattices once the mineral phase is unstable enough to be able to release it seems to be more a function of the chemical and thermodynamic factors involved in mineral transformation and formation than of pure volume diffusion. Under the condition for this experiment, a local transient equilibrium is apparently established between mineral grains at temperatures (and with heating times) that do not allow complete chemical equilibration of the whole rock system. The migration of a constituent (such as potassium), once allowed by the instability or transformation of a donor and acceptor mineral phase, apparently proceeds relatively rapidly in either direction between the acceptor and donor phase. Thus, the Sr^{87r} can be lost from the micas at the same time the micas gain Sr^N .

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APPENDIX

The rock is a light brownish gray (5 YR 7/1), medium-grained, hypidiomorphic granular muscovite-biotite quartz monzonite (adamellite). The average grain size is approximately 2 mm, with a maximum (muscovite and microcline) of 4 mm. No foliation is visible megascopically in outcrop or hand specimen. The plagioclase is zoned from sodic oligoclase to albite, with the more calcic cores clouded with a small amount of fine-grained hematite and sericite. The sutured grain boundaries, together with the unstrained anhedral quartz grains indicate the absence of significant dynamic metamorphism. The biotite is pleochroic from light brown to dark reddish brown with haloes around minute radioactive inclusions. There is very little chloritization of the biotite. The muscovite is overgrown by a slight amount of finer-grained muscovite and contains occasionally very fine interlayers of biotite. The microcline is anhedral and poikilitically encloses plagioclase, quartz and biotite.

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