

Cape Cleare was detailed in 1927. This area may prove to be one of extensive uplift if analyzed by comparative surveys of the type used in this study.

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Coupling of Butyl Bromide on Hot Magnesium

Abstract. *A report of the formation of octane when butyl bromide is passed over magnesium turnings at high temperatures should be amended. Such coupling does take place over pure magnesium at more moderate temperatures but yields 3-methylheptane and sec-butyl bromide in addition to octane. Sec-butyl bromide itself forms no coupling product under such conditions but admixed with butyl bromide markedly increases the 3-methylheptane:octane ratio in the product.*

A previous note in *Science* (1) stated that butyl bromide, when passed with helium through a tube containing magnesium turnings at 330°C, formed 10 percent of octane. Using magnesium turnings of high purity (2) we were unable to reproduce these findings, despite many attempts with varying tube lengths, tube geometry, contact times, and temperatures between 275° and 400°C (3). The effluent, condensed in ice and dry-ice traps and then freed of HBr by washing with sodium hydroxide solution, was analyzed by gas chromatography (4) after being dried.

Very small quantities of coupling products were obtained in only one experiment of the many carried out in the range of 300°C. In contrast, when the butyl bromide was passed over pure magnesium at 150° to 200°C, coupling could be effected routinely after a variable induction period. Yields depended on the rate of addition of the butyl bromide and careful maintenance of the experimental conditions.

Analysis of the product by gas chromatography showed the presence of 2.6 percent (of the theoretical

yield) of octane (5), 4.4 percent of 3-methylheptane (5), and 3.6 percent of sec-butyl bromide resulting from a typical experiment when 0.8 ml of butyl bromide per minute was dropped on the magnesium turnings at 150° to 200°C in the presence of 30 ml of helium per minute. Lower boiling products were not investigated; butanes and butenes were present in significant quantities. At an addition rate of 0.02 ml of butyl bromide per minute, yields of 4.6 percent of octane, 11.6 percent of 3-methylheptane, and 2.5 percent of sec-butyl bromide were more typical.

When pure sec-butyl bromide was the feed substance, no coupling on magnesium at 150° to 200°C was detected, even though the analytical method would have shown even a few tenths of a percent of octane, 3-methylheptane, or the possible coupling product, 3,4-dimethylhexane. However, when a mixture of two volumes of butyl bromide to one of sec-butyl bromide was used under the usual conditions with a drop rate of 0.02 ml per minute, 1.0 percent of octane and 8.0 percent of 3-methylheptane were produced.

Although the present investigation has not established the radical or ionic character of the reaction on hot magnesium, any satisfactory mechanism must account for the several findings: the rearrangement of butyl bromide to sec-butyl bromide under our reaction conditions, the nonoccurrence of the reverse process, the production of 3-methylheptane from butyl bromide plus the reactive intermediate derived from sec-butyl bromide, and the lack of formation of 3,4-dimethylhexane from sec-butyl bromide.

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2. Sublimed magnesium was provided through the courtesy of J. F. Pashak of the Wrought Development Section of the Dow Metal Products Company.
3. This negative finding is not disparaging to the results of Turk and co-workers; it points up the complexities of the reaction. Small quantities of metal impurities in the magnesium influence the course of the reaction, and temperatures in the reaction tube may vary widely unless unusually careful control is exerted.
4. A Research Specialties gas chromatograph fitted with a 2-m Apiezon L column (about 10 percent on 60-80 mesh Chromosorb P) and a katharometer detector were used for analytical runs. Quantitative results were verified by calibration with known mixtures.
5. Identity was conclusively established by infrared analysis (Perkin-Elmer 237 spectrophotom-

eter) of macroquantities which were isolated from the product mixture by an Autoprep gas chromatograph (Wilkins Instrument and Research, Inc.) fitted with a 6 m × 0.9 cm column filled with 20 percent SE-30 on 60-80 mesh Chromosorb W.

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Rubidium-Strontium Isochron Study of the Grenville Front near Lake Timagami, Ontario

Abstract. *Rubidium-strontium isotopic analyses of whole-rock samples and of constituent minerals from a suite of rocks taken across the Grenville Front demonstrate that granitic rocks of the Superior province, with a primary age of approximately 2.4 billion years, and older metasedimentary rocks were reconstituted during Grenville metamorphism, at approximately 930 million years, and now form part of the Grenville province.*

In understanding the evolution of continental masses, the question of the growth of continental bodies through geologic time is of fundamental importance. Parts of the North American continent have been subdivided on the basis of geologic characteristics and apparent age. With regard to the hypothesis of continental accretion it is necessary to determine whether the geologically younger parts of the continental masses represent the addition of new material or are in fact the product of metamorphism of the older pre-existing geologic provinces. The boundaries between old and young provinces are the natural places to study these possible phenomena. Reported herein are some of the characteristics of a portion of the boundary zone between the Grenville and Superior provinces as shown by a study of strontium and rubidium isotopes.

The Grenville Front is the northwestern boundary of the Grenville province of the Canadian Shield (see Fig. 1). For over 800 miles it forms the boundary between the Superior and Grenville provinces (1, 2). These provinces differ in particular in structure and in grade and age of major metamorphism. The easterly trending structures of the Superior province are truncated on the southeast by the northeasterly trending structures of the Grenville.

The Superior province is characterized by widespread low- to medium-grade early Precambrian metamorphism; the Grenville province, by major medium- to high-grade metamorphism which occurred approximately 1 billion years ago. The characteristics of the two provinces are relatively well defined. However, the nature of the boundary between them remains a major problem. It has been claimed that the Front is a major fault, a metamorphic transition, or most probably a combination of the two (3). Correlation of geological units from the one province to the other has been generally unsatisfactory. Although it has often been claimed that rocks of the Superior province have been metamorphosed during the Grenville orogeny and now form part of the Grenville province, in no case has this been demonstrated unequivocally.

Thus, one of the major problems of the nature of the Grenville Front is a geochronological problem in polymetamorphism. Such problems may be amenable to Rb-Sr whole rock and mineral isochron studies, as has been suggested by the work of Compston and Jeffery in Western Australia (4), Fairbairn, Hurley, and Pinson at Sudbury, Ontario (5), and Allsopp (6) in South Africa. Recent work of this kind has been carried out by Long and Lambert (7), and Long (8) in Scotland. The possibilities of this approach have been studied in detail by Lanphere, Wasser-

burg, Albee, and Tilton (9) working in the Panamint Range, California. They showed that redistribution of the isotopes of strontium during metamorphism was not haphazard, but that strontium isotopic equilibration occurred between the constituent minerals of a rock on the scale of the local strontium pool, but not on a regional scale. From the results shown by these several workers it is evident that whole rock isochron studies may allow one to determine the age of the initial isotopic equilibration of strontium in a suite of cogenetic rocks, and that studies of mineral isochrons may reveal the age of the last re-equilibration.

A Rb-Sr isochron study has now been made across a segment of the Grenville Front south of Lake Timagami, Ontario (Figs. 1 and 2). Here, the metamorphic transition by which the Front is defined is unfaulted for over 5 km, outcrop is good, and the area has been studied in some detail (3, 10). In particular it has been possible to tentatively correlate geological units of the Superior and Grenville provinces.

The geology of the Lake Timagami area is outlined in Fig. 2. In the center of the area, early Precambrian metagreywacke and metavolcanics (Kewatin type) occur, intruded successively by quartz diorite and granite. The early metamorphism was probably associated with the emplacement of the granite

(3). To the north these rocks are overlain by a flat-lying blanket of virtually unmetamorphosed Huronian sedimentary rocks, and along with the Huronian rocks are intruded by diabase. To the south the metagreywacke, quartz diorite, and granite underwent later metamorphism which gave rise to a migmatitic terrane in which the altered equivalents of these rocks can be recognized. The lithologies, structures, and grade of metamorphism developed are typical of the northwestern part of the Grenville province, and for this reason the late metamorphism was considered to be of Grenville age. In the west of the area, as shown in Fig. 2, the boundary is a metamorphic transition, corresponding approximately to the southward transition from greenschist to amphibolite facies, while in the east the two provinces are in fault contact. A major implication of the field work (3, 10) is that within the Grenville province there is granite which might be considered to be of primary Grenville age, but which is actually correlative with pre-Huronian granite of the Superior province.

The correlations across the Front are based on factors such as lithology which do not yield unequivocal evidence, and the assignment of Grenville age to the late metamorphism is not axiomatic.

To test these tentative correlations in the Lake Timagami area and the age assignment of the late metamorphism, a suite of five granites from a traverse across the Front and a schist from the "Grenville" side of the Front were studied by means of the Rb-Sr technique. The localities of the samples are shown in Fig. 2.

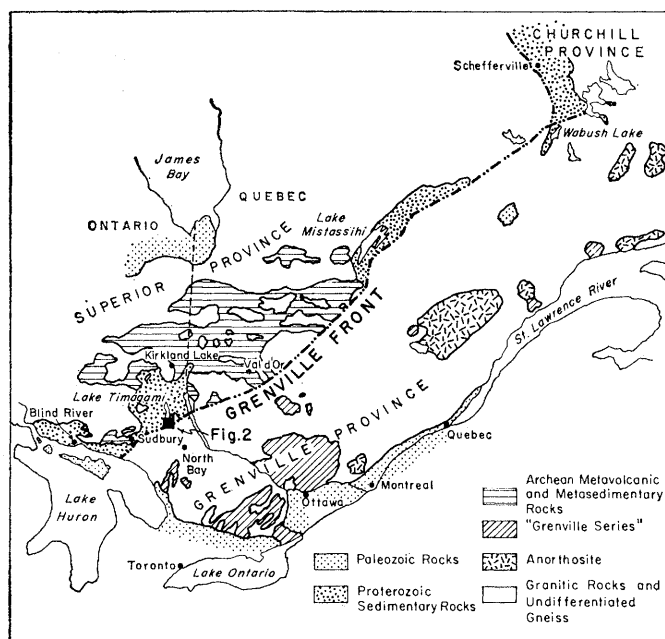
The theory and assumptions underlying the interpretation of Rb-Sr isotopic relationships have been discussed in detail by Lanphere *et al.* (9), who used the strontium evolution diagram of Nicolaysen (11). For a system closed to Rb and Sr one may write the age equation in the form:

$$\left(\frac{\text{Sr}^{87}}{\text{Sr}^{86}}\right)_t = \left(\frac{\text{Sr}^{87}}{\text{Sr}^{86}}\right)_0 + \left(\frac{\text{Rb}^{87}}{\text{Sr}^{86}}\right)_0 (e^{\lambda t} - 1)$$

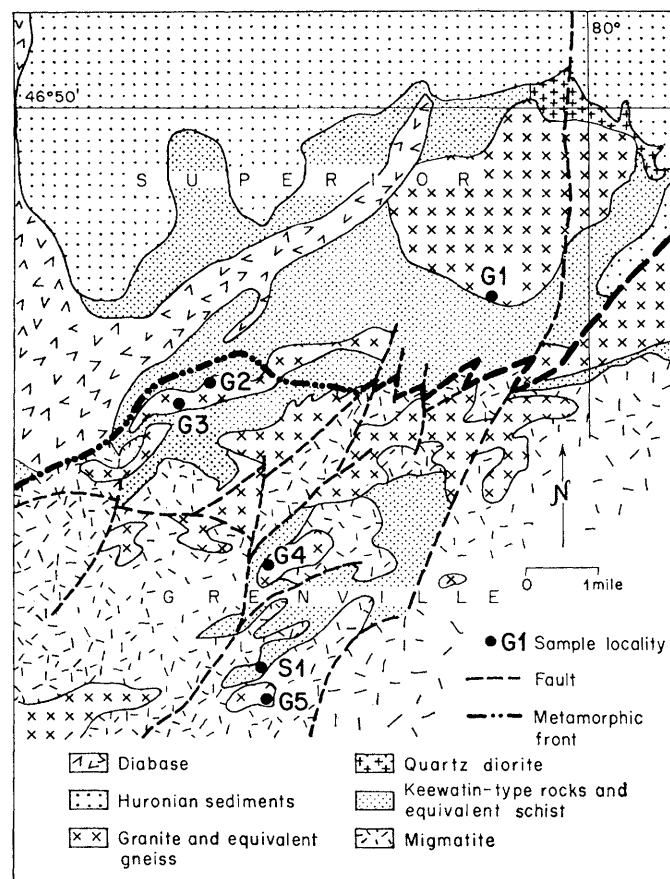
where the subscript *t* refers to the ratio obtained after a time *t* has passed. For such coeval systems, be they whole rocks or minerals, which at the same initial time have the same $\text{Sr}^{87}/\text{Sr}^{86}$ ratio $[(\text{Sr}^{87}/\text{Sr}^{86})_0]$ and differing $(\text{Rb}^{87}/\text{Sr}^{86})_0$ ratios, will define on a strontium evolution diagram a straight line (isochron) whose slope is a function of the time

Table 1. Rubidium-strontium analytical data. The data for Sr are normalized to $(\text{Sr}^{86}/\text{Sr}^{88}) = 0.1194$; $(\text{Rb}^{85}/\text{Rb}^{87})$ normal = 2.591; $\lambda \text{Rb}^{87} = 0.139 \times 10^{-10}$ years⁻¹; analytical error in $(\text{Sr}^{87}/\text{Sr}^{86})$ comp. ≤ 0.2 percent; analytical error in $(\text{Sr}^{87}/\text{Sr}^{86})$ calc. ≤ 0.3 percent; analytical error in $(\text{Rb}^{87}/\text{Sr}^{86}) \approx 1$ percent. All analyses were made with Teflon ware (13). Symbols: W, whole rock; A, apatite; P, plagioclase; Mi, microcline; E, epidote; Mu, muscovite; B, biotite.

Sample	Sr^{87} ($\times 10^{-6}$ mole/g)	Rb^{87} ($\times 10^{-6}$ mole/g)	$\text{Sr}^{87}/\text{Sr}^{86}$		$\text{Rb}^{87}/\text{Sr}^{86}$
			Comp.	Calc.	
G1W1	0.06668	0.8621		1.1828	15.29
G1W2	.06715	.8760		1.1850	15.46
G2W1	.1268	.8865		0.9263	6.475
G2W2	.1341	.9002		.9218	6.189
G4W	.2115	.6409		.7828	2.373
G3W	.2937	.5157	0.7456	.7453	1.309
G5W1	.6371	.5360	.7208	.7219	0.6064
G5W2	.5909	.4590	.7207	.7195	0.560
G1A	.16014	.01786	.7805	.7814	.0872
G1P	.03399	.09928		.9748	2.848
G1Mi	.07411	2.235		1.7696	53.3
G1B	.1143	5.142		6.2763	282.3
G2A	.1088	0.00789		0.7195	0.0522
G2B	.1065	5.545		11.339	590.3
G5A	.4859	0.00282	.7138	0.7141	0.0041
G5P	.5276	.09574	.7130	.7150	0.1294
G5Mi	.7303	.9032	.7260	.7272	.8994
G5Mu	.2903	1.186		.7541	3.081
G5B	.7285	2.735	1.3824	1.3789	51.90
S1W	.4300	.4355		0.7284	0.7378
S1E	1.2817	.00800	0.7146	.7158	.0045
S1P	0.5003	.0255		.7202	.3669
S1B	0.02856	1.498		2.3278	122.1



Figs. 1 and 2. Fig 1 (left). Regional map showing the Grenville Front and the location of the area studies. Fig. 2 (right). Geological cartoon of the Lake Timagami area (10).



since the systems had the same value (Sr^{87}/Sr^{86}).

If the correlation of the granites is correct, and if they satisfy the assumptions of closure to Rb and Sr since emplacement and of an original homogeneous strontium isotopic composition, they should define a whole rock isochron whose slope defines the time at which the rocks had a value of $(Sr^{87}/Sr^{86})_0$ given by the intercept at $(Rb^{87}/Sr^{86}) = 0$. If the assignment of a Grenville age to the late metamorphism is correct, and if the minerals of the whole rock samples equilibrated with respect to the isotopes of strontium and subsequently remained closed to rubidium and strontium, then mineral isochrons from the schist (S1) and southernmost granite (G5) should be parallel, both defining a Grenville age for the last equilibration.

The analytical results are given in Table 1, and the granite whole rock data are shown in Fig. 3. Each whole rock point represents a split of about 5 g from about 35 kg of pulverized and thoroughly mixed rock. (The schist is of finer grain-size, and for this whole rock analysis a sample of 2 kg was taken.) In problems such as this, where whole rock and mineral isochrons do

not coincide, it is imperative to obtain good splits of a representative sample; and to check the quality of splitting, three whole rock samples were analyzed in duplicate.

The whole rock data for the five granites (Fig. 3) approximately define an isochron, and are considered proof that the granites conformed closely to the postulates that at one time they had the same strontium isotopic composition, and that since then they have remained as systems closed to rubidium and strontium. The best-fit isochron thus defined yields an age of 2.35 billion years and $(Sr^{87}/Sr^{86})_0 = 0.703 \pm 0.003$. Isochrons for 2.5 billion years and 2.2 billion years are drawn for comparison on the same figure. The interpretation of these data is that the five granites from north and south of the Grenville Front were comagmatic intrusions with a primary age of 2.35 ± 0.15 billion years, proving the tentative correlation.

To confirm that the age of the late metamorphism was indeed Grenville, mineral data from the southernmost granite (G5) and the schist (S1) were obtained.

Sample G5 is a slightly foliated, medium- to coarse-grained, inequigranular quartz monzonite, with about 4

percent biotite showing negligible chloritization. This sample is from a particularly homogeneous mass in the migmatite with which it is generally conformable. Apatite, plagioclase, microcline, muscovite, and biotite were analysed, and the corresponding five points and the two whole-rock points all lie within analytical error on an isochron. This isochron yields an age of 920 million years and a value of 0.713 for $(Sr^{87}/Sr^{86})_0$.

Sample S1 is a biotite-quartz-plagioclase schist of fine to medium grain, with about 4 percent epidote. The biotite is not chloritized. This rock is tentatively correlated with Keewatin-type metagreywacke north of the Front, and has been metamorphosed to the amphibolite facies. Epidote, plagioclase, and biotite and a whole-rock split were analyzed. The data for plagioclase, biotite, and the whole rock split define an isochron within analytical error, the epidote point lying close to, but below, this line. The isochron yields an age of 920 million years and a value of 0.719 for $(Sr^{87}/Sr^{86})_0$. This isochron and the mineral isochron of granite G5 are virtually parallel (Fig. 4).

These results confirm that strontium isotopic re-equilibration between the

minerals constituting granite G5 was virtually complete and that equilibration between the minerals of schist S1 was nearly complete at 920 to 940 million years—that is, during Grenville metamorphism. This age is on the young side of the Grenville time band, and it may be thought that this is due to slight loss of daughter Sr^{87} . The excellence of the mineral isochrons of G5 and S1 militates against this: the determined age is apparently a true age for the last equilibration. The value of $(\text{Sr}^{87}/\text{Sr}^{86})_0$ corresponding to the last equilibration for the schist S1 is different from the value obtained from the granite G5. From this it may be inferred that while the schist and granite individually came to virtually complete local strontium isotopic equilibration at the same time, this equilibration did not occur on a regional scale despite the fact that the metamorphism here was to amphibolite facies.

Thus Grenville metamorphism is confirmed in the south of the Lake Timagami area. But it is possible that the effects of this metamorphism can be traced beyond the macroscopic limit, the Grenville Front, for it has been shown (9, 12) that the Rb-Sr technique may yield evidence of metamorphism in rocks where this was not evidenced in the field. Granite G1 comes from a pluton north of the Front, and is a medium-grained massive leucocratic quartz monzonite, with about 1 percent biotite, partly intergrown with chlorite, but on a coarse scale which permits good mineral separation. This rock shows no macroscopic features definitely attributable to the late metamorphism and is typical of the Superior province.

Apatite, plagioclase, microcline, and biotite from this rock were analysed, and the results are shown in Fig. 5. It is obvious that this rock has in fact been disturbed. The minerals yield a linear array which deviates markedly from the whole rock isochron. The mineral points do not fall within analytical error on a straight line as did the mineral points of granite G5; plagioclase lies above the biotite-whole rock join, that is, the line joining the points representing the analyses of the biotite and whole rock samples, apatite and microcline lie below. The apparent ages derived from the biotite-, plagioclase-, and microcline-whole rock joins are 1360, 1200, and 1100 million years, respectively. The deviation of the mineral points from colinearity

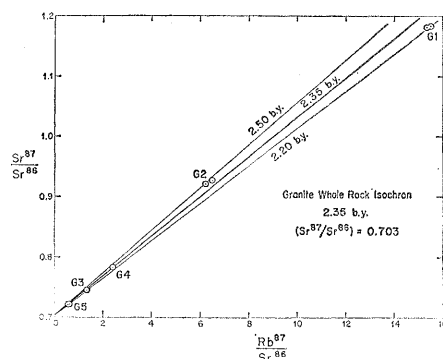


Fig. 3. Strontium evolution diagram for granite whole rock samples.

may be due to some or all of the minerals having been open to some extent to Sr or Rb during the evolution of the rock after the last equilibration, but there is no evidence to support this hypothesis. It is much more plausible, and indeed to be expected, that G1 did not attain complete strontium isotopic equilibration during Grenville metamorphism, for the granite and the surrounding rocks show little macroscopic evidence of this metamorphism. If this is the case, and if the minerals tended to equilibration with the average $(\text{Sr}^{87}/\text{Sr}^{86})$ of the rock, then at the time of approximate equilibration, minerals to the left of the whole rock (plagioclase and apatite) and those to the right (microcline and biotite) would approach the new zero isochron from below and above, respectively. If the system were quenched prior to complete equilibration and if the equilibration paths did not cross the zero isochron or the whole rock isochron, then all the mineral-whole rock joins would give ages higher than the true age of the event. Thus the minimum age of 1100 million years, derived from the microcline-whole rock join of G1, is probably a maximum for the true age of this partial equilibration. The conclusion is

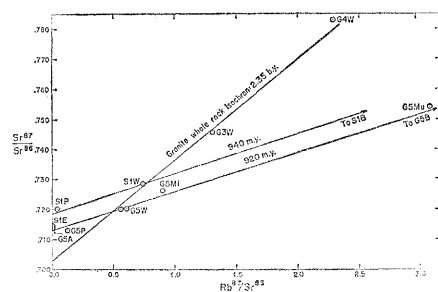


Fig. 4. Strontium evolution diagram for granite G5 and schist S1 from Grenville province.

that the Rb-Sr technique indeed allows one to trace the effects of metamorphism beyond the limits of macroscopic evidence, and that this granite, north of the Grenville Front and in the Superior province, approached strontium isotopic equilibration at $(\text{Sr}^{87}/\text{Sr}^{86})_0 \approx 0.947$ during Grenville metamorphism at a time less than 1100 million years ago.

An additional analysis of biotite from granite G2 was made to further document the northward effects of Grenville metamorphism. This is a medium-grained, slightly foliated leucogranite adjacent to the Front as defined in the metagreywacke. It contains about 2 percent biotite intergrown with chlorite. The biotite-whole rock joins from this sample and from the northernmost granite (G1) and from the southernmost granite (G5), yield the following apparent ages: G5, 920 million years; G2, 1270 million years; and G1, 1360 million years. This suggests the northward decrease in the effectiveness of Grenville metamorphism in resetting the Rb-Sr clock.

This decrease is also evidenced by data for accessory "common strontium" minerals. Apatite from G5 virtually equilibrated with the other minerals of the rock at 920 million years. Epidote from S1 did not equilibrate so completely with the remainder of the rock, and the epidote-whole rock join yields an apparent age of 1.22 billion years. Contrasted with this are data for apatites from G1 and G2 to the north. The apatite-whole rock joins from these rocks yield the following apparent ages: G1, 1.87 billion years, and G2, 2.31 billion years. These are markedly closer to the age of 2.35 billion years derived from the whole rock isochron than to the age of Grenville metamorphism. It may be noted that the apatite-whole rock join from G2 yields a higher age than does the same join for G1, but the significance of this is not apparent. Obviously, the apatites in these two rocks were much less affected by Grenville metamorphism than were the associated biotites. It has been demonstrated (9) that some common strontium minerals are affected by redistribution of strontium isotopes, although not as markedly as other strontium-bearing minerals, and therefore cannot be considered axiomatically to yield the initial $(\text{Sr}^{87}/\text{Sr}^{86})_0$ for the rock in which they occur.

Thus, with the Rb-Sr technique, it is possible to detect evidence of Grenville metamorphism in rocks north of the Grenville Front, although these

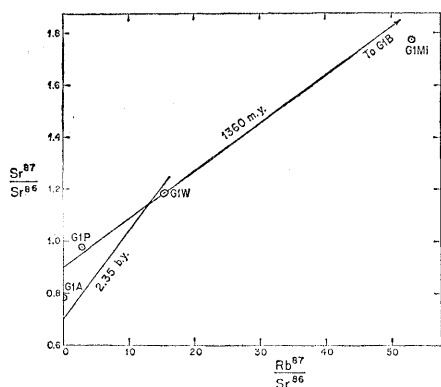


Fig. 5. Strontium evolution diagram for granite G1.

rocks are macroscopically unaffected by this event. It remains to be determined how far the overprint of disturbed strontium isotopic systems can be traced into the interior of the Superior province.

In the south of the Lake Timagami area, rather than being an overprint, Grenville metamorphism has profoundly reconstituted rocks of the Superior province and has incorporated them into the Grenville province. But to the southeast of this area no primary ages significantly older than Grenville have been found so far. However, the only detailed study of this particular problem is that of Krogh (13) in the vicinity of the Hastings Basin, some 300 km to the southeast. He studied only granitic rocks, using the Rb-Sr method, and reports no evidence of primary ages significantly older than Grenville. Thus the question is still open within this 300 km as to how far southeast the old granites extend and how far northwest from the Hastings region primary Grenville granite can be found. An associated problem is that of the metasediments within the Grenville province. Within the Lake Timagami area, schist considered equivalent to the Keewatin-type metagreywacke predates the granite on geological grounds. From the single sample analyzed it is not possible to determine the age of deposition of the original sediment, particularly because the corresponding $(\text{Sr}^{87}/\text{Sr}^{86})_0$ is unknown. A probable minimum limit for this value is 0.700, yielding a maximum age of 2.7 billion years. If the metasedimentary rocks of the Hastings region—the so-called Grenville Series—were deposited only a short time before the emplacement of the Grenville granites, then an unconformity separating them from the Archean rocks to the northwest should exist between the

Hastings region and the Lake Timagami area. This would represent the margin of the old Superior craton. However, on the basis of present knowledge, it is entirely possible that the metasedimentary rocks in the Hastings region were deposited contemporaneously with the Keewatin-type rocks north of the Grenville Front.

Finally, the question may be posed whether this reconstitution of rocks of the Superior province during the Grenville orogeny represents merely the welding of a new portion of the continent to the old Superior craton, or if a major part of the Grenville province represents reconstitution of the ancient continent.

There is evidence of metamorphism of intermediate age (1700 ± 150 million years) in the Sudbury area (5) and at the southern end of the Labrador Trough (2). Ages within this range have been found locally in the vicinity of the Front between these two regions (2, 14). This has given rise to speculation that an orogeny of intermediate age extended completely around the old Superior craton. However, these workers (2, 14) attribute the intermediate ages not to a definite metamorphic event of that age, but to incomplete resetting of clocks during Grenville metamorphism. Nor is there any evidence for such a middle-age spread in the Lake Timagami area. While it is certainly possible that metamorphism of intermediate age did occur in the vicinity of the Front, it would be most difficult to resolve such an event where later (Grenville) metamorphism was superimposed upon it. To prove the presence of such an event one would require rocks with real or apparent primary ages corresponding to the event, and no such rocks are known between Sudbury and the Labrador Trough.

The granite suite taken across the Grenville Front yields whole-rock data which define an isochron: this implies close conformity to the postulate that these rocks had initially the same strontium isotopic ratio. The value of this ratio, $(\text{Sr}^{87}/\text{Sr}^{86})_0 = 0.703 \pm 0.003$, is compatible with a primary magmatic origin for these rocks. The granites are therefore correlated as comagmatic intrusions aged 2.35 ± 0.15 billion years. Mineral data from the southernmost granite and an adjacent schist define two parallel but distinct isochrons indicating virtually complete strontium isotopic re-equilibration between the constituent minerals of each rock at ap-

proximately 930 million years. This confirms Grenville metamorphism in the south of the area. Mineral data from the northernmost granite suggest that Grenville metamorphism effected partial re-equilibration even north of the macroscopic Front.

These data demonstrate that granitic rocks of the Superior province, with primary ages of approximately 2.4 billion years, and older metasedimentary rocks were reconstituted during the Grenville orogeny and now form part of the Grenville province. This study provides strong support for the hypothesis that, on the scale of the local strontium reservoir, strontium isotopic re-equilibration between minerals is not a rare phenomenon but a common accompaniment of metamorphism, and provides a most sensitive approach to problems of polymetamorphism.

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