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# Uranium-Lead Radioactive Dating and Lead Isotope Study on Sphene and K-Feldspar in the Sør-Rondane Mountains, Dronning Maud Land, Antarctica

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## ABSTRACT

Uranium-lead radioactive ages of sphene samples extracted from igneous and metasedimentary rocks of the Sør-Rondane Mountains (Antarctica) are compared with the zircon U/Pb ages, previously obtained, and with some Rb/Sr data. The behaviour of sphene in amphibolite facies metasediments is strikingly different from that of zircon. This latter mineral is predominantly detrital, suffering from partial lead loss during the metamorphism, while sphene, being a metamorphic mineral, should essentially yield the age of metamorphism. Surprisingly, sphene from metasedimentary gneisses constantly shows an age of 540 m.y. which is lower than the zircon age (600 m.y.) of the two geologically younger, cross-cutting intrusives. Therefore, sphene either suffers from extensive lead loss until some threshold point of non-diffusion is reached in the course of the post-metamorphic upheaval, or the investigated granites contain zircon xenocrysts. The first possibility is favoured. Such a zircon-sphene discrepancy is not observed in the case of a younger intrusive (500 m.y. old). In order to obtain sufficient precision on the 206/207 ages, a study of the isotopic composition of the feldspar lead has been necessary. The joint investigation of uranium-lead isotopic relationships in sphene, zircon, K-feldspar, might yield useful petrogenetic information in some cases.

## Introduction

Among the accessories, used for dating by the uranium-lead method, zircon has been the object of many studies, the other minerals (sphene, allanite, monazite, apatite) being comparatively neglected. Specifically the data relative on sphenes remain scarce. They predominantly concern accessory sphene of igneous and meta-igneous rocks (G. R. TILTON and M. GRÜNENFELDER, 1968). Nevertheless, they establish convincingly the great interest of this mineral in the field of radioactive dating. The present study is mainly devoted to a comparison of the results obtained on zircon and sphene extracted from the same rocks, with special attention to metasediments and migmatites. The region chosen for this purpose is sufficiently well understood from the geological and geochronological point of view, and may be considered as illustrating a relatively simple case of regional metamorphism in the almandine-amphibolite facies (upper catazone and mesozone) accompanied by considerable migmatization, grading into the epidote-amphibolite facies.

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### Geological setting and previous geochronological investigations

The Sor-Rondane Mountains are part of the coastal chain of east Antarctica. Geological reconnaissances were carried out in this region on behalf of the Belgian Antarctic Expeditions, and, later on, the Belgian-Netherlands Antarctic Expeditions, since 1958 (E. PICCIOTTO et al. 1960, 1966; J. MICHOT 1962, 1963; T. VAN AUTENBOER et al. 1964; T. VAN AUTENBOER and J. LOY 1966; J. LAVREAU et al. 1967; K. NAERT 1968). A geochronological investigation of the collected material has also been undertaken (S. DEUTSCH et al. 1961; P. PASTEELS and S. DEUTSCH 1963; E. PICCIOTTO et al. 1964; P. PASTEELS and J. MICHOT 1968). The rock samples presently investigated were collected by E. PICCIOTTO and J. MICHOT.

Two geological units, separated by an E-W trending fault zone, can be distinguished in the Sor-Rondane Mountains. The northern complex is composed essentially of a folded, layered sequence of gneisses and migmatites of diverse composition, together with marbles and amphibolites. The gneisses, predominantly of sedimentary derivation, have undergone an upper catazonal or mesozonal metamorphism with, in many places, a more or less pronounced diaphoresis. The southern unit reflects a medium-grade (mesozonal) metamorphism and is composed mainly of mafic gneisses and amphibolites. The relationship between the two units is obscured by local mylonitization accompanied by retrograde metamorphism in epizonal conditions, north and south of the major fault zone.

Various intrusive masses and dykes were emplaced after the folding. The nunataks north of the chain are composed of plutonic rocks: gabbros, monzonites, granodiorites, all clearly intrusive into the gneisses. Granites and syenites also outcrop along the line separating the northern and southern gneisses, exhibiting intrusive relationships with both units. In the central part of the chain, within the northern gneisses, considerable migmatization is observed. Fine-grained microcline-bearing granitic stocks are considered as related to this phenomenon, presumably representing local accumulation of anatectic material. Locally they cross-cut the gneiss structures.

Rb/Sr dating has been limited to biotite, with a few measurements performed on muscovite, microcline and total-rock samples. The Rb/Sr isochron method was not applied systematically to total rocks. Indeed, preliminary determinations of the Rb/Sr ratio by X-ray fluorescence (for which we are grateful to Prof. J. J. Fripiat, of the University of Louvain) indicate very low Rb/Sr ratios, precluding precise age determinations, except for the presumably anatectic granites. For this rock type, the Rb/Sr method was applied successfully to total-rock, microcline, plagioclase, apatite, yielding an age of  $550 \pm 50$  m.y. ( $\lambda^{87}\text{Rb} = 1, 39 \times 10^{-11} \text{ y}^{-1}$ ) with  $(^{87}\text{Sr}/^{86}\text{Sr})_0 = 0.710 \pm 0.002$ . Twenty biotite Rb/Sr ages from diverse rock types range from 460 to 500 m.y., without any connection between the small age differences observed and the relative chronology indicated by field relationships.

Dating with the U/Pb method was applied to zircon only as a first stage. Only after that part of the investigation programme had been completed, was a dating of the sphene also attempted. The zircon data already published (P. PASTEELS and S. DEUTSCH 1963; E. PICCIOTTO et al. 1964–1966; P. PASTEELS and J. MICHOT 1968) are summarized in Figure 1 and Table 3. Since a comparison of zircon versus sphene data appears to be of special interest, it may be worthwhile considering first, briefly, the general

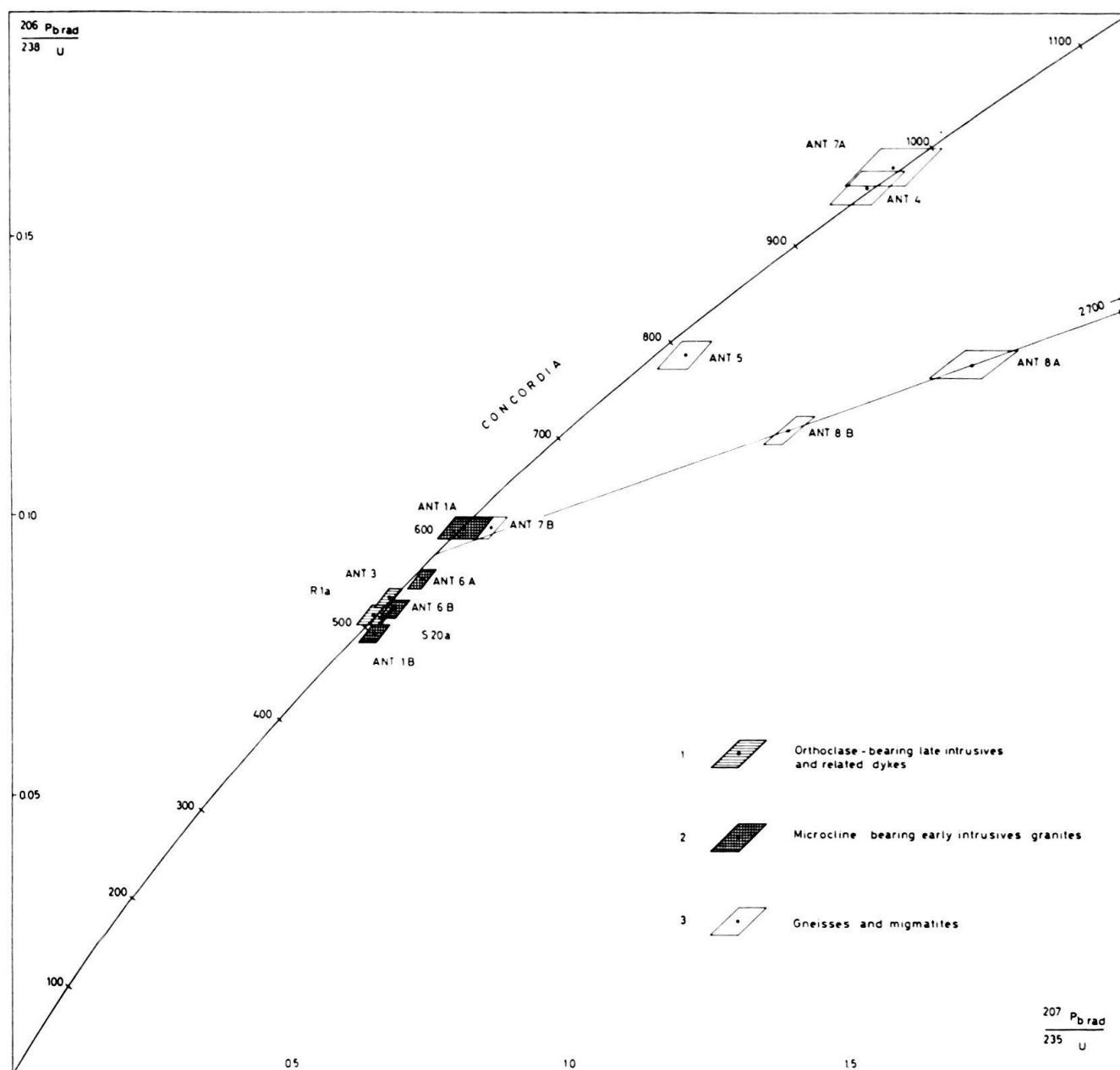


Fig. 1. Concordia diagram of the zircon samples.

conclusions concerning the local chronology based only on the zircon data, as we have formulated them in a previous paper (P. PASTEELS and J. MICHOT 1968).

From these data the following conclusions can be drawn.

1. There are, at least, two distinct post-tectonic intrusive phases.

Three samples representative of the igneous complex of the nunataks (R 1 a, S 20 a, ANT 3) are  $520 \pm 20$  m.y. old. The Lunckeryggen granite, ANT 1, outcropping at the boundary northern gneiss/southern gneiss, is dated at  $600 \pm 30$  m.y., as is ANT 6, one of the stocks of fine-grained granite, presumably anatectic, of the central part of the chain (the same rock was dated at  $550 \pm 50$  m.y. by Rb-Sr measurements).

2. The climax of metamorphism occurred earlier than 600 m.y. ago, but, on geological grounds, it may be thought that it is not much older, presumably 650 to 600 m.y. ago.

3. On the Concordia diagram (Fig. 1), the experimental points relative to ANT 4 (southern gneiss), ANT 7 (northern gneiss), ANT 5 (northern gneiss, migmatic) define a 950–400 m.y. chord. The upper intercept is believed to correspond to the age of the detrital component. The lower intercept has no geological significance. Indeed, microscopic examination of the zircon populations strongly suggest that new formation of euhedral, highly radioactive zircon occurred, resembling those of ANT 6 (anatectic granite) in the gneisses ANT 7 and ANT 5. This zircon neoformation probably occurred during the climax of metamorphism, 650 to 600 m.y. ago, and not at a later time. A subsequent loss of lead must be supposed in order to explain the intercept at 400 m.y.

Part or all of the metasedimentary sequence was thus laid down later than 950 m.y. ago. The northern and southern gneisses belong to a single sedimentary cycle.

4. The two points relative to ANT 8 (northern gneiss) define a 2700–570 m.y. chord. Since this sample and ANT 7 were collected a few meters apart, within a regularly layered sequence, the high age of the detrital fraction found in ANT 8 is unexpected, but it may be supposed that a very ancient basement, as a source of these old zircons, was temporarily emergent at the time of sedimentation. As in the case discussed above, the lower intercept, 570 m.y., must be considered as a minimum figure for the age of metamorphism, because of the possibility of subsequent lead loss.

### **The problem set by the presence of common lead in sphene**

Sphene, being relatively rich in common lead, makes it necessary, at least for “young” samples (600 m.y. old or less), to try to determine the isotopic composition of the common lead initially present in the rock, in order to make the common lead correction, as adequate as possible. We decided to measure the isotopic composition of the lead present in the K-feldspar. It has indeed been shown that K-feldspar leads, in granites of a given age, are of variable isotopic compositions and may deviate considerably from the single-stage model (B. R. DOE 1967).

Part of the common lead present can be removed by washing the sphene samples in hot 6 N nitric acid (A. J. BURGER et al. 1965). However, this common lead may come from four different sources: dissolved mineral impurities, the outside part of the sphene crystals, altered sphene, and laboratory contamination resulting from the various operations of mineral separation. If this fourth source is important relative to the three others, the use of the correction of the lead present in the acid wash is not justified.

### **Analytical procedure**

#### **a) Sample preparation and chemical procedure**

The zircon data having been previously reported, only the details of the analytical procedure for sphene and feldspar will be described here.

From one case investigated by G. R. TILTON and M. GRÜNENFELDER (1968), it appears that the light, uranium-poor sphene of low magnetic susceptibility has a

higher uranium-to-lead ratio, more convenient for dating purposes than the more magnetic, uranium-rich sphene. It was thus decided to separate, whenever possible, a relatively small fraction of low magnetic susceptibility, by means of the Frantz Isodynamic separator, and perform the measurements on this fraction only. In one instance, a small magnetic fraction was also analyzed for comparison purposes, and in this particular case, both fractions had very similar U/Pb ratios but very different U and Pb contents (ANT 7).

Sphene samples were washed in hot distilled  $\text{HNO}_3$  1/1 for half an hour, before analysis. They were, before the acid treatment, at least 95% pure. Some of the impurities present are soluble in  $\text{HNO}_3$ : sulfides, apatite. Other are left undissolved by the mixture of acids used to bring the sphene into solution: zircon, fluorite. In some instances, less than 2% quartz and feldspar impurity was present and analyzed together with the sphene.

The K-feldspar concentrates analyzed were of variable purity (see appendix). Prior to dissolution, they were washed successively in hot distilled  $\text{HNO}_3$  1/1 for half an hour, and in hot distilled HCL 6N for another half hour.

A similar procedure was followed for the sphene and feldspar samples. Between 1 and 3 grams of the sample is placed into a platinum dish and covered with a mixture of concentrated  $\text{HNO}_3$  (about 7 times the sample weight) and HF 40% (about 15 times the sample weight). The dish is placed into a teflon box flushed by a stream of glass-filtered nitrogen, on a hot plate. The dry residue is dissolved in diluted HCL. The uranium (235) and lead spikes (206 for feldspar, 208 for sphene) are added to an aliquot of the total solution. The spiked solution is heated for one hour. Lead and uranium are extracted by the same procedure used for zircon: dithizone extraction for lead, hexone separation for uranium. For the lead purification and extraction, a larger amount of ammonium citrate was used in the case of the feldspar samples than for the sphene. It was not possible, however, to prevent in this way the aluminium hydroxyde from precipitating during the first dithizone extraction, but this precipitation did not hinder a sufficient lead recovery. A further purification of lead by use of an ion-exchange column was not necessary, however except for the sphene acid washes from which, in two instances, lead was separated. In those two cases, a Dowex 1-X resin in 1,5N HCL was used, lead being eluted by triply distilled water.

Several blank experiments were performed, using a 206 spike (Table 1). Though it is uncertain whether the difference between experiments 1) and 2) is not accidental, those reagents corresponding to the lowest lead contamination were used

Table 1. Blank experiments

Conditions	Lead quantity, $\times 10^{-6}$ g
1. Sphene procedure, for 1.3 g of sample, using analytical grade reagents purified in the laboratory	0.47
2. Idem, using Hopkin and Williams "low in lead" $\text{HNO}_3$ , HCl, KCN and Merck "suprapur" HF (other reagents purified in the laboratory)	0.30
3. Feldspar procedure (using more citrate), for 2 grams of sample, with the same reagents as for 2)	0.46



for all feldspar and sphene samples except one (sphene ANT 1, a). In spite of the absence of air filtering in the chemistry laboratory, the lead blank is not prohibitive, since less than 10% of the total common lead detected in the sphene samples can be attributed to laboratory contamination. Concerning the feldspar samples, the lead content has been measured only in one case by stable isotope dilution and found to be of only 7.91 ppm. In this case, laboratory contamination can account for about 5% of the total lead present. But judging from the colour of the second dithizone extract, this particular feldspar (syenite ANT 2) is especially poor in lead, compared to the other analyzed samples, and in all likelihood laboratory contamination contributes less than 5% of the total lead in the other cases.

#### b) Mass spectrometry

The isotopic analyses were performed on an Atlas CH4 instrument. Uranium was loaded on a tantalum filament as uranyl nitrate. Most of the zircon lead measurements were made using the boric acid technique of MARSHALL and HESS (1960). Later on, we adopted the zirconium silicate activator method of AKISHIN et al. (1957). This activator is loaded on a platinum filament. More intense ion beams are obtained with this technique, which was used for some of the zircon leads and all the sphene and feldspar leads. The results on the C.I.T. lead standard for isotopic composition, which we obtained during the period of time when the measurements discussed here were executed, are presented on Table 2.

Table 2. Isotopic analysis of the C.I.T. lead standard

Conditions	206/204	206/207	206/208
1. With boric acid, tantalum	16.64	1.0728	0.4574
2. With Zr SiO <sub>4</sub> , platinum	16.58	1.0729	0.4561
3. With Zr SiO <sub>4</sub> , platinum	16.58	1.0716	0.4577

To some of the isotopic analyses, a correction for the non-linearity of the scale of the recorder was applied, the validity of this correction being checked repeatedly, by recording the same peak at different sensitivities. The mass discrimination factor due to the use of an electron multiplier (together with all other possible systematic mass-dependent effects) was empirically determined by comparison of the uncorrected ratios obtained on the C.I.T. lead standard with the "absolute values" determined for this reference sample by E. J. CATANZARO (1968). The values in Table 2 are corrected. A precision and accuracy of  $\pm 20/00$  on the ratios 206/207 and of  $\pm 40/00$  on 206/204 can be attributed to the common lead measurements presented here. In the case of sphene leads, the 204 peak is smaller and its measurement subject to larger errors. This was taken into account for the error estimates on the 207/206 age values. For all sphene samples it was assumed that the common lead present is similar to that of the corresponding K-feldspar. For comparison purposes, 207/206 age values calculated with an arbitrarily chosen correction lead, and with the lead present in the acid leach in two cases, are also given. For zircon samples, we reproduce here the age values

already published which were calculated using an arbitrarily chosen correction lead. The error estimates, for the zircon ages, are very conservative and admit the possibility that the common lead present actually has an isotopic composition similar to that of the corresponding feldspar lead.

### Experimental results

The experimental results are given in Tables 3 to 6. The constants used for the age calculations are the following:  $\lambda^{238}\text{U} = 1.537 \times 10^{-10} \text{ y}^{-1}$ ,  $\lambda^{235}\text{U} = 9.72 \times 10^{-10} \text{ y}^{-1}$ ,  $^{238}\text{U}/^{235}\text{U} = 137.8$ .

Sphene ANT 2 illustrates a case where the radiogenic lead to common lead ratio is very small, leading, as a consequence, to an exceedingly large imprecision in the 207/206 and 207/235 ages. It can be observed also (Tables 4 and 5), that the presence of a radiogenic component in the feldspar lead used for the correction, even in very small proportion, becomes a matter of importance in such a case. Indeed, a significant difference is observed in the 206/238 age values calculated respectively with the uncorrected feldspar lead, and with the feldspar lead corrected for radiogenic accumulation of  $^{206}\text{Pb}$  during 550 m.y.

### Discussion

#### 1. Lead isotopic composition

From the studies of other investigators, it appears that the 207/206 age is a reliable indication for sphene, corresponding closely to the zircon age (concordant or extrapolated with the aid of the Concordia diagram), in the case of unmetamorphosed intrusives, while the Rb-Sr whole-rock age (with  $\lambda^{87}\text{Rb} = 1.39 \times 10^{-11} \text{ y}^{-1}$ ) is similar, occasionally lower. On the other hand, the 206/238 and 207/235 ages may be too low, in response presumably to loss of lead, by as much as 10 %.

In the present case, because of the relatively young age of the samples, and the high common lead content of the sphene, the 207/206 age values are, unfortunately, very sensitive of the experimental errors and to the choice of the correction lead. Let us observe first that the 207/206 age, when calculated using the feldspar lead for the correction, is in agreement with the 206/238 age, or slightly higher. This is exactly what may be expected for radiometric ages of sphene.

The correction made with the lead extracted from the acid wash yields in one case (ANT 1) a clearly too low 207/206 age value, as does, in several instances, an arbitrarily chosen "normal" lead of approximately 500 m.y. model age. Another arbitrarily chosen lead would certainly have yielded more acceptable 207/206 ages; however, there is an obvious inconvenience in having the final result depending from an a priori choice. The use of the corresponding feldspar lead is in any case more justified. Indeed, in the case of magmatic rocks, the feldspar lead may be considered as practically identical to the lead originally present in the magma. The same lead was thus also, very probably, incorporated by the sphene. As to the metamorphic rocks, it is possible that all the sphene and most of the K-feldspar were formed in response to metamorphism, and hence incorporated the same lead when they crystallized. If part of the feldspar should be considered as an unmodified detrital component it is far from being



Table 3. Uranium contents and radiometric ages of zircons samples

Nature of the rock, location	Sample ref.	U ppm	t 206/238 in m.y.	t 207/235 in m.y.	t 207/206 in m.y.
a) Intrusive suite of the nunataks					
Granitic dyke, cross-cutting a gabbro Nordtoppen 950	S20a	208	510 ± 20	508 ± 20	500 ± 30
Gneiss xenolith, in a gabbro Nordtoppen 1100	S9	643	555 ± 20	555 ± 55	550 ± 150
Granodiorite, Romnoesfjell	R1a	222	514 ± 20	518 ± 20	540 ± 20 <sup>1)</sup>
Monzonite, Vesthaugen	ANT3	259	526 ± 10	523 ± 16	505 ± 40
b) Granites, central and southern part of the chain					
Anatectic (?) stock, Strandrudfjell	ANT6 A <sup>2)</sup> B <sup>2)</sup>	2770 5230	552 ± 10 521 ± 10	564 ± 17 534 ± 20	610 ± 45 590 ± 65
Plutonic granite cross-cutting a syenite, Lunckeryggen	ANT1 A <sup>2)</sup> B <sup>2)</sup>	473 1710	607 ± 12 494 ± 10	608 ± 25 510 ± 18	610 ± 100 580 ± 60
Gneissic granite					
Erratic boulder collected on the glacier Gunnestadbrean	GB	914	512 ± 20	524 ± 20	575 ± 20 <sup>1)</sup>
c) Gneisses, northern unit					
Embrechite, Vikinghögda	ANT5	1690	790 ± 15	814 ± 25	875 ± 45
Granitic gneiss, Strandrudfjell	ANT7 A <sup>2)</sup> B <sup>2)</sup>	637 1160	979 ± 20 609 ± 12	972 ± 35 639 ± 18	950 ± 70 745 ± 35
Granodioritic gneiss, Strandrudfjell	ANT8 A <sup>2)</sup> B <sup>2)</sup>	645 926	777 ± 15 715 ± 15	1026 ± 35 896 ± 20	1610 ± 55 1378 ± 20
d) Gneiss, southern unit					
Tonalitic gneiss, cross-cut by ANT1, Lunckeryggen	ANT4	179	960 ± 20	953 ± 25	935 ± 50

<sup>1)</sup> Error on t 207/206 reestimated.<sup>2)</sup> Fraction A is of low magnetic susceptibility, fraction B of higher magnetic susceptibility.

Table 4. Isotopic composition of the feldspar leads

Sample number	Rock, location	206/204	207/204	208/204	206/207	206/208
S20a	Granite, Nordtoppen	17.78	15.70	37.94	1.1322	0.4685
ANT1	Granite, Lunckeryggen	17.62	15.50	37.07	1.1364	0.4753
ANT2	Syenite, Lunckeryggen a)	17.62	15.55	37.18	1.1331	0.4740
	Syenite, Lunckeryggen b) <sup>1)</sup>	17.65	15.54	37.27	1.1359	0.4736
	mean	17.64	15.55	37.23	1.1345	0.4738
	mean corrected <sup>2)</sup>	17.37	15.53	37.23	1.1182	
ANT5	Embrechite, Vikinghögda	18.47	15.59	37.50	1.1850	0.4926
ANT6	Anatectic (?) granite, Strandrudfjell	17.77	15.55	37.81	1.1429	0.4700
ANT7	Granitic gneiss, Strandrudfjell	17.03	15.39	36.94	1.1067	0.4610
ANT8	Granodioritic gneiss, Strandrudfjell	17.07	15.42	37.01	1.1069	0.4612

<sup>1)</sup> Repeated chemistry.<sup>2)</sup> U: 0.39 ppm, Pb: 7.91 ppm, assumed age: 550 m.y.

Table 5. Isotopic composition of the lead in the leach solutions of the sphene concentrates

Sample number	206/204	207/204	208/204	206/207	206/208
ANT1	20.70	15.79	41.81	1.3113	0.4952
ANT5	37.15	16.70	46.79	2.225	0.7940

Table 6. Experimental data and radiometric ages of the sphene samples

Sample number	U ppm	Pb ppm	Lead isotopic composition 204	Lead isotopic composition		208	Radiometric ages in m.y.				t 207/206 <sup>2)</sup>	t 207/206 <sup>3)</sup>
				206	207		t 206/238	t 207/235	t 207/206	t 207/206 <sup>2)</sup>		
S20a	88.8	13.56	1	151.06	23.22	121.36	498 ± 10	496 ± 15	481 ± 40	516	—	—
ANT1 a)	134.3	27.95	1	86.88	19.53	82.87	547 ± 10	548 ± 20	550 ± 60	509	484	484
b) <sup>1)</sup>	133.6	28.05	1	86.01	19.55	82.15	550 ± 10	558 ± 20	588 ± 60	549	526	526
ANT2	13.76	19.91	1	21.47	15.81	42.14	533 ± 30 <sup>4)</sup>					
							501 ± 30 <sup>5)</sup>					
ANT5	230	28.70	1	183.12	25.09	66.82	539 ± 10	538 ± 15	531 ± 40	524	522	522
ANT7 light	91.8	12.65	1	150.08	23.04	84.83	514 ± 10	516 ± 15	524 ± 40	479	—	—
dark	249	33.6	1	151.26	23.24	76.11	523 ± 10	531 ± 15	563 ± 40	518	—	—
ANT8	127.3	14.15	1	194.40	25.73	53.56	521 ± 10	526 ± 15	548 ± 40	522	—	—

<sup>1)</sup> Repeated chemistry. Spikes evaporated before adding the sample for a), added after sample dissolution for b).

<sup>2)</sup> With a correction lead with the following isotopic ratios: 206/204 = 18.1, 207/204 = 15.6.

<sup>3)</sup> Corrected with the lead isotopic composition of the leach solution.

<sup>4)</sup> Using the mean, corrected feldspar lead i.c.

<sup>5)</sup> Using the mean, uncorrected feldspar lead i.c.

certain that it could have kept, within its lattice, lead very different isotopically from that of the newly formed minerals. Indeed, extensive migration of lead in relation with metamorphism has been observed in other instances (B. R. DOE, G. R. TILTON and C. A. HOPSON 1965, B. R. DOE and S. R. HART 1963). The mobility of lead during metamorphism is further indicated by the close similarity of the leads present in the feldspars of ANT 7 and ANT 8, taken from two gneisses sampled a few metres apart, and which, considering the zircon data, must contain detrital material coming from very different sources. Their similarity in lead isotopic composition is thus not an original feature, but was acquired subsequently, presumably during the metamorphism.

Indeed, the late metamorphic, intrusive granite ANT 6 (regarded as anatectic), collected a few metres apart from ANT 7 and ANT 8 has a K-feldspar lead of very different isotopic composition: the supposed lead migration would have therefore occurred before its emplacement. The granitic material is not derived from the gneisses at the contact, but possibly from some other metasediments. But, contrary to the other intrusives, the late granite S 20a has a feldspar lead which cannot, seemingly, be derived by a mixing process from the country gneiss leads, exemplified by ANT 5, 7 and 8. This situation is illustrated in Figure 2, a  $^{206}\text{Pb}/^{204}\text{Pb}$  versus  $^{206}\text{Pb}/^{207}\text{Pb}$  diagram, where the lead evolution curves corresponding to various chemical milieu indices  $\mu$  and isochrons, have been drawn. This graph illustrates the considerable isotopic variation observed for the metasedimentary feldspar leads, and the less important variation for the feldspar leads of intrusive rocks. Among these latter, sample S 20a corresponds, however, to a relatively high milieu index, which apparently indicates that this 500 m.y. old rock is not derived, or not entirely derived, from the same environment as the 600 m.y. old intrusives. Let us also realize that the model ages of the granite and syenite feldspar leads are close to the real ages, which does not seem to be the general case (B. R. DOE 1967), at least for rocks 500 to 600 m.y. old. Though not intended, initially, the measurements performed on the feldspar leads thus happened to yield independent information of possible genetic significance. In view of the many studies devoted to this subject in the recent years, this was not, in fact, entirely unexpected.

The sphene data obtained on samples representative for the three main groups of rocks investigated, i.e. the late generation of intrusives (520 m.y. old), the older generation of intrusives (about 600 m.y. old, according to the zircon data), and the metamorphic rocks, are discussed below.

## 2. Late intrusives

Sample S 20a is representative of a granite dyke cross-cutting mafic plutonic rocks belonging to the same suite, the younger intrusives dated at  $520 \pm 20$  m.y. by measurements on zircons. The sphene yields concordant ages in excellent agreement with the also concordant ages obtained on the zircon.

## 3. Early intrusives

From the radiometric results obtained on zircon (P. PASTEELS and J. MICHOT 1968), summarized in a previous section, it appears that there is an older generation of post-tectonic intrusives, about 600 m.y. old.

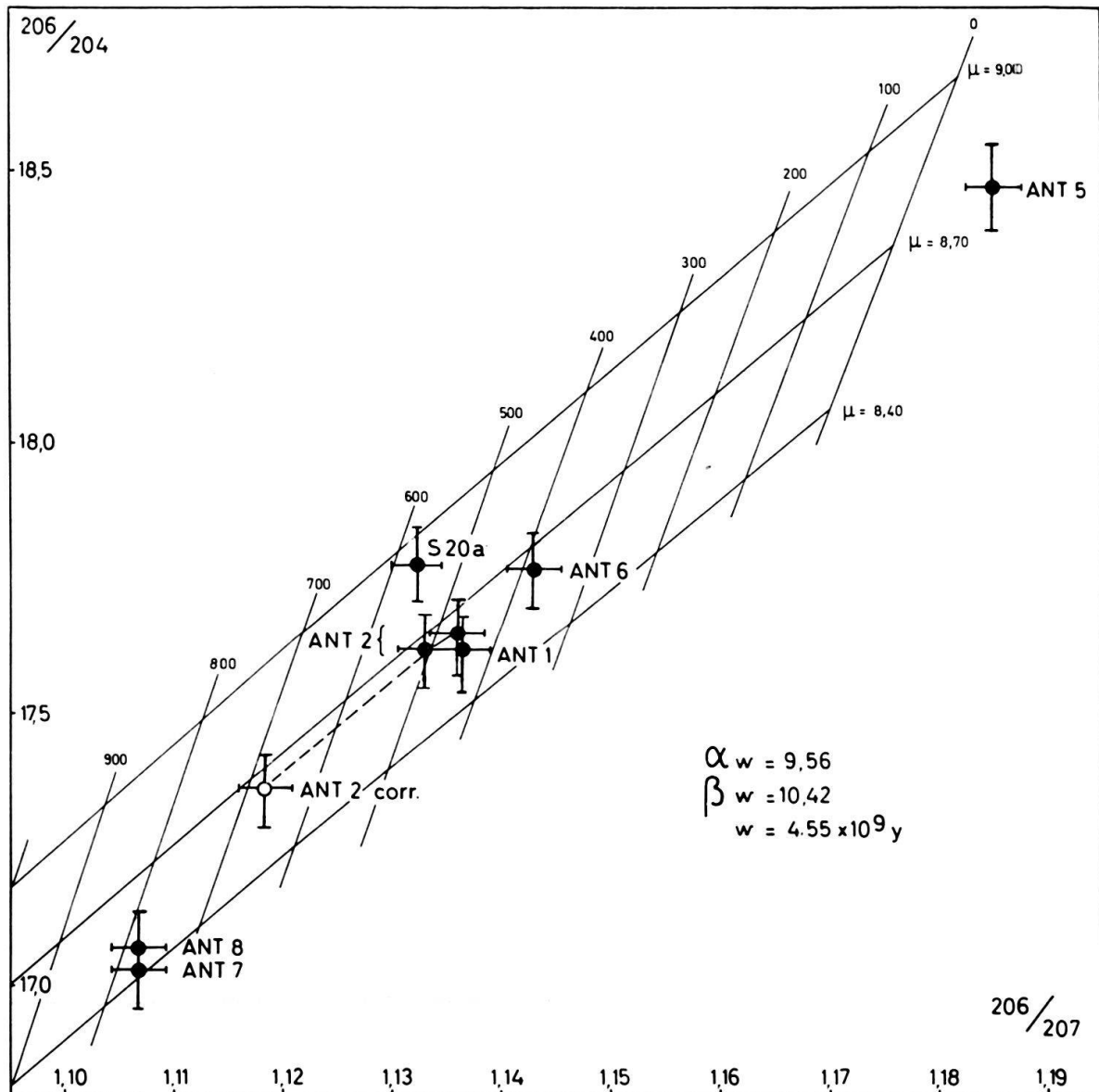


Fig. 2.  $^{206}\text{Pb}/^{204}\text{Pb}$  versus  $^{206}\text{Pb}/^{207}\text{Pb}$  plot for feldspar leads. Lines labelled  $\mu = 9.00$ ,  $\mu = 8.70$ ,  $\mu = 8.40$  are growth curves for these different milieu indices. Lines labelled 0, 100, 200,... are isochrons of 0, 100, 200 m.y....

Point "ANT2 corr." refers to ANT2 feldspar lead, mean corrected for radiogenic accumulation during 550 m.y. It is connected by a dotted line to a point equidistant from the two ANT2 points, uncorrected lead measurements. The crosses give the approximate analytical uncertainties.

The results obtained on the sphene of ANT 1 confirm that this rock, the Lunckeryggen granite, is older than samples S 20a and other rocks of the same suite. However, because of the imprecise  $^{207}\text{Pb}/^{206}\text{Pb}$  age it cannot be decided whether the new results point rather to an age value of 550 m.y. different from the 600 m.y. indicated by the zircon, or else confirm essentially this latter geochronological indication.

#### 4) Metamorphic rocks

Since sphene is formed or recrystallized in the course of metamorphism, it is not surprising that it yields an age indication completely different from that given by the corresponding zircon, in the case of the paragneisses, where this last mineral is partly detrital and not “reset to zero” by metamorphism. The detrital part of the zircon is 950 m.y. old in the case of samples ANT 7 and ANT 5, and some 2,700 m.y. old in the case of sample ANT 8. Nevertheless, sphene in these three rocks yield the same age, i.e.  $540 \pm 30$  m.y. This figure could be interpreted as the age of metamorphism; however, from the zircon data, the climax of metamorphism is dated at 600 to 650 m.y. In order to explain this discrepancy, two possibilities are considered:

- a) The sphene data correctly point to the age of the climax of metamorphism and thus the zircon ages relative to the “early intrusives” (clearly late-metamorphic) are in error, being 50 to 100 m.y. too high.

Such an interpretation is in agreement with the Rb/Sr isochron age of sample ANT 6 ( $550 \pm 50$  m.y.), slightly lower than the age assignment based on zircons. Moreover, too high a zircon age would be readily explained in this case by the presence of zircon xenocrysts, not unexpected in an anatectic granite with local nebulitic texture.

But the case of the Lunckeryggen granite seems more difficult to explain. Indeed, instances are rare where the zircon ages are substantially raised by the presence of xenocrysts and at present there is no independent argument in favour of this. Moreover this explanation fails to account for the 206/238 age value of 607 m.y. measured on zircons of sample ANT 1, fraction A. Incorporation of old radiogenic lead (in the form of zircon xenocrysts or otherwise) cannot raise the 206/238 age value as much as the 207/206 age.

- b) More likely, the paroxysm of metamorphism is well dated at 600–650 m.y., sphene being subject to the well-known post-metamorphic rejuvenation effect, as are other minerals which are datable by the Rb-Sr and K-Ar methods, namely the micas. Thus, during the early stages of the slow cooling and uplift following the metamorphic climax, a considerable part of the radiogenic lead produced was not retained within the sphene lattice. In order to affect the 207/206 ages to a great extent, this loss must indeed have been practically complete, and have ceased rather abruptly. This does not imply, however, “instantaneous” episodic loss of lead, for it is quite compatible with the data that several tens of million years elapsed from the time the mineral started to retain part of the radiogenic lead, to the time where lead loss ceased completely. The 540 m.y. figure would then fix a date intermediate between these two events (beginning of lead retention and end of lead loss).

Two distinct ways of lead escape are, in fact, conceivable. The first possibility is that the cooling and uplift have been progressive, undisturbed, and that lead loss from sphene is essentially temperature-dependent. A threshold temperature of non-diffusion was reached some 540 m.y. ago. The second possibility is that the late stages of the metamorphic evolution of the tectonic segment considered here were far from being quiet. A distinct episode of retrograde metamorphism



(accompanied with tectonic movements) would be responsible for the episodic lead escape 540 m.y. ago. Though we do not rule out this second possibility there are not many positive arguments in favour of it. Sphene appears as primary in these rocks. It was not formed, nor has it visibly reacted, during a second metamorphism. Concerning the existence of a distinct episode of retrograde metamorphism on a regional scale, the geological observations are not conclusive; the observed facts may also be interpreted in terms of local deformations, not all contemporaneous, having allowed here and there the achievement of the mineral equilibrium at lower P, T conditions, in the course of very slow uplift.

To some extent, the behaviour of sphene can thus be compared to that of the micas. However, in the investigated region, the biotite clock started 480 m.y. ago ( $\lambda^{87}\text{Rb} = 1.39 \times 10^{-11} \text{ y}^{-1}$ ), and thus at a much later stage in the tectonic evolution of the area. The uplift has been very slow in any case, having a total duration of about 150 m.y.

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### APPENDIX

#### Short description and location of the samples

- R I a** Romnoesfjelled granite.  $71^{\circ}27' \text{ S}$ ,  $23^{\circ}57' \text{ E}$ .  
Coarse-grained porphyroblastic granite of nearly granodioritic composition. Phenocrysts of orthoclase, matrix composed of quartz, biotite and oligoclase. Described by J. MICHOT (1962). Among the accessories, ilmenite and zircon are abundant. No sphene is present. The morphology of these zircons has been described by G. HOPPE (1964).
- S20a** Granite dyke cross-cutting a massive gabbro-diorite. Nunatak Nordtoppen 950.  $71^{\circ}26' \text{ S}$ ,  $25^{\circ}20' \text{ E}$ .  
Homogeneous, fine-grained, pink granite. Oligoclase, quartz, microcline, biotite, accessories among which abundant zircon and sphene. J. MICHOT (1962), G. HOPPE (1964). Feldspar concentrate: microcline more than 99% pure.
- S 9** Gneiss xenolith as inclusion in gabbro-diorite. Nunatak Nordtoppen 1100.  $71^{\circ}27' \text{ S}$ ,  $25^{\circ}17' \text{ E}$ .  
Xenolith, 2 to 3 m in size, with well defined contacts. Banded gneiss, the sample being, taken in a lightly coloured part, granodioritic in composition. Andesine, microcline, pyroxene, amphibole, biotite, accessories.
- G B** Coarse-grained granite with a distinct preferential orientation of the dark minerals. Erratic found near Romnoesfjell, likely to have come from Gunnestadbreen. Microcline, quartz, oligoclase, biotite, hornblende, allanite, accessories. Zircons described by G. HOPPE 1964.
- ANT1** Lunckeryggen granite.  $72^{\circ}03' \text{ S}$ ,  $24^{\circ}38' \text{ E}$ .  
Medium-grained massive granite. Microcline, oligoclase, quartz, amphibole, biotite, accessories (sphene, epidote, allanite, opaques, apatite, zircon). Described by J. LAVREAU et al. 1967. Feldspar concentrate: microcline more than 99% pure.

- ANT2 Lunckeryggen syenite. 72°01' S, 24°40' E.  
Intruded by the Lunckeryggen granite ANT1. Medium to coarse-grained rock, with local parallelism of the large feldspars. Microcline (about 75% of the rock), augite, richterite, biotite, sphene, apatite. Described in detail by T. VAN AUTENBOER et al. 1964 and J. LAVREAU et al. 1967. Feldspar concentrate: more than 99% pure microcline.
- ANT3 Vesthaugen "monzonite" 71°43' S, 23°43' E.  
This rock occurs as intrusive lenses, dyke-like, but concordant with the gneiss structures, exhibiting weak foliation. Its composition is variable, being locally that of a diorite, but elsewhere monzogabbroic or monzonitic (with hypersthene and mesoperthite) (J. MICHOT 1963, T. VAN AUTENBOER and J. LOY 1966). Sample ANT3 is composed of andesine, diopside, biotite, quartz, with some orthoclase and myrmekite, opaques, apatite, zircon.
- ANT4 Gneiss of the southern unit, Lunckeryggen 72°05' S, 24°30' E.  
Cross-cut by the Lunckeryggen granite. Oligoclase-andesine, quartz, actinolite, greenish biotite, chlorite, epidote, opaques, apatite, zircon. Chlorite and epidote seem to be primary (J. LAVREAU et al. 1967).
- ANT5 Embrechite, Vikinghögda 72°04' S, 23°28' E.  
Augen-gneiss of granitic composition. Microcline, quartz, oligoclase, biotite, epidote, allanite, sphene, zircon. Secondary minerals: chlorite (from biotite), carbonate, opaque inclusions in chlorite. Feldspar concentrate: about 60% microcline, 30% plagioclase, 10% quartz.
- ANT6 Fine-grained microcline granite, Strandrudfjell 71°53' S, 25°38' E.  
Intrusive body of moderate size, in some places concordant, elsewhere clearly cross-cutting the gneisses. Microcline, acidic oligoclase, quartz, biotite, myrmekite, accessories (opaques, allanite, scarce apatite and zircon) (J. MICHOT 1963). Feldspar concentrate: about 50% microcline, 40% plagioclase, 10% quartz.
- ANT7 Granitic gneiss, Strandrudfjell.  
Collected a few metres apart from the contact with the Strandrudfjell granite, and also at very close distance from the sampling place of this latter (ANT6). The country rock of the granite consists of a layered sequence of gneisses, granitic, granodioritic and dioritic in composition. Quartz, oligoclase, microcline, biotite, muscovite, carbonate, opaques, allanite, sphene, zircon. Feldspar concentrate: about 85% microcline and 15% plagioclase.
- ANT8 Granodioritic gneiss, Strandrudfjell.  
Collected a few metres apart from ANT7 and ANT6. Layered rock varying from granitic to dioritic composition. Quartz, oligoclase, microcline (abundant in some layers, absent in others), hornblende, biotite, epidote, allanite, sphene, apatite, carbonate, zircon. Feldspar concentrate: about 96% microcline, 4% plagioclase.

## REFERENCES

- AKISHIN, P. A., NIKITIN, O. T., and PANCHENKOV, G. M. (1967): *A New Effective Ionic Emitter for the Isotopic Analysis of Lead*. Geochimiya USSR engl. transl. 5, 500–505.
- BURGER, A. J., VON KNORRING, O., and CLIFFORD, T. N. (1965): *Mineralogical and Radiometric Studies of Monazites and Sphene Occurrences in the Namib Desert, South-West Africa*. Min. Mag. 35, 519–528.
- CATANZARO, E. J. (1968): *Absolute Isotopic Abundance Ratios of Three Common Lead Reference Samples*. Earth and Planetary Sci. L. 3, 343–346.
- DEUTSCH, S., LEDENT, D., and PASTEELS, P. (1965): *Datation par les méthodes Sr/Rb au laboratoire de géochronologie du Service de Géologie et Géochimie Nucléaires*. Université Libre de Bruxelles, 166 pages.
- DEUTSCH, S., PICCIOTTO, E. E., and REINHARZ, M. (1961): *Age Measurements on Antarctic Rocks (Queen Maud Land)*. Nature 191, 1286–1287.
- DOE, B. R. (1967): *The Bearing of Lead Isotopes on the Source of Granitic Magmas*. J. Petrogr. 8, 51–83.
- DOE, B. R., and HART, S. (1963): *The Effect of Contact Metamorphism on Lead in Potassium Feldspar Near the Eldora Stock, Colorado*. J. Geophys. Res. 68, 3521–3530.

- DOE, B. R., TILTON, G. R., and HOPSON, C. A. (1965): *Lead Isotopes in Feldspar from Selected Granitic Rocks Associated with Regional Metamorphism*. J. Geophys. Res. 70, 1947–1968.
- HOPPE, G. (1964): *Morphologische Untersuchungen als Beiträge zu einigen Zirkon-Altersbestimmungen*. N. Jb. Miner. Abh. 102, 89–106.
- LAVREAU, J., MICHOT, J., and SOUCHEZ, R. (1967): *Géologie et Géomorphologie des Sør-Rondane occidentales, Terre de la Reine Maud (Antarctique)*. Ann. Soc. géol. Belg. 90, B 119–159.
- MARSHALL, R. R., and HESS, D. C. (1960): *Determination of Very Small Quantities of Lead*. Anal. Chem. 32, 960–966.
- MICHOT, J. (1962, 1963): *Etude pétrologique des échantillons prélevés dans les Monts Sør-Rondane (Antarctique)*. Expédition Antarctique Belge 1957–1958. I. *Les roches à caractère intrusif*. Ann. Soc. géol. Belg. 85/1, B 87–99 (1962); II. *Le complexe métamorphique*. Ann. Soc. géol. Belg. 85/5, B 151–174 (1963).
- NAERT, K. (1968): *Contribution à l'étude pétrologique des Sør-Rondane (Terre de la Reine Maud), Antarctique*. Ann. Soc. géol. Belg. 91/1, B 111–126.
- PASTEELS, P., and DEUTSCH, S. (1963): *Age Measurements on Antarctic Rocks (Queen Maud Land)*. Nature 199, 996–997.
- PASTEELS, P., and MICHOT, J. (1968): *Nouveaux résultats géochronologiques obtenus par la méthode U-Pb sur des zircons des Monts Sør-Rondane (Antarctique)*. Ann. Soc. géol. Belg. 91, 283–303.
- PICCIOTTO, E., DEUTSCH, S., and PASTEELS, P. (1964): *Isotopic Ages from the Sør-Rondane Mountains, Dronning Maud Land*, in: R. J. Adie (ed.), *Antarctic Geology*, Proc. 1st symp. on Antarctic Geology, Cape Town 16–21 sept. 1963 (North Holland Publ. Co.), p. 579–589.
- PICCIOTTO, E., MICHOT, J., DEUTSCH, S., and PASTEELS, P. (1966): *Expédition Antarctique Belge 1957 à 1958. Résultats scientifiques*, Vol. 7, Géologie.
- PICCIOTTO, E., MICHOT, J., and MICHOT, P. (1960): *Reconnaissance géologique et pétrographique des Monts Sør-Rondane (Terre de la Reine Maud)*. Bull. Soc. belge géol. 69/2, 1–13.
- TILTON, G. R., and GRÜNENFELDER, M. H. (1968): *Sphene: Uranium-lead Ages*. Science 159, 1458–1461.
- VAN AUTENBOER, T., and LOY, J. (1966): *The Geology of the Sør-Rondane, Antarctica. Data Report: Central Part of the Range*. Centre National des Recherches Polaires de Belgique, 61 pages.
- VAN AUTENBOER, T., MICHOT, J., and PICCIOTTO, E. (1964): *Outline of the Geology and Petrology of the Sør-Rondane Mountains, Dronning Maud Land*, in: R. J. Adie (ed.), *Antarctic Geology*, Proc. 1st symp. on Antarctic Geology, Cape Town 16–21 sept. 1963 (North Holland Publ. Co.), p. 501–514.