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The lanthanoid content of some minerals  
from the Pyörönmaa pegmatite  
in Kangasala, Finland

by Jaakko Siivola



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THE LANTHANOID CONTENT OF SOME MINERALS  
FROM THE PYÖRÖNMAA PEGMATITE  
IN KANGASALA, FINLAND

BY  
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WITH EIGHT FIGURES AND THREE TABLES IN THE TEXT

GEOLOGINEN TUTKIMUSLAITOS  
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A study was made of the distribution of lanthanoids in the pegmatite minerals of Pyörönmaa. The minerals were analysed with the electron microprobe technique. The results show the light lanthanoids to have been enriched at the beginning of crystallization. The heavy lanthanoids were bound into the minerals during the later stage of crystallization. This criterion might be applied in the determination of the crystallization order of minerals containing rare earth elements.

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## INTRODUCTION

Geochemical researchers have always shown considerable interest in the rare earth elements. The close similarity between the chemical properties of these elements makes it possible to handle them as one, highly coherent group. As a result of improved physical methods of analysis extensive new data have been accumulated on the rare earth content of rocks and minerals. They deal mostly with the distribution patterns of the rare earths in rocks, in certain minerals or mineral groups. The rocks and minerals are commonly selected from a wide geological variety of deposits and from many widely separated localities.

This study represents an effort to determine the distribution of lanthanoids in several cogenetic minerals inside a single deposit. The analysed samples were taken from the Pyörönmaa pegmatite in Kangasala in southwest Finland. This pegmatite is situated inside a late kinematic Svecofennidic granite body, not far from the well-known Varala pegmatite (see, e.g., Lokka 1935). A mineralogical description of the granitic REE pegmatite of Pyörönmaa was given by Vormaa *et al.* (1966). Many in the list of 27 minerals were new to Finland. After the paper was published, the large collection of material called for further study. Later, a new hydrous RE carbonate, lokkaite, found in the material was described by Perttunen (1971). The present work continues the previous studies. With proper standards for electron microprobe analysis, the distribution of lanthanoids in some of the minerals taken from the Pyörönmaa pegmatite have been recorded. Owing to the commonly experienced difficulties in the analysis of rare earths, the results reported here are to be taken as semiquantitative. However, the relative amounts of the lanthanoids in the minerals analysed are quite accurate.

## LANTHANOID ANALYSES

Physical methods of analysis are most frequently used to determine the elements present in rare earth samples and to measure their proportions. No one of the many methods is universally used. Electron microprobe analysis was adopted for this study. The polished thin sections were prepared from the hand specimens collected

TABLE 1  
Analytical conditions

Common	25 kV/60 nA, Mylar window out, PHA level +1.0 V, slit in	
Channel	I	II
Crystal .....	LiF	Mica
Counter .....	sealed	flow
Ctr voltage .....	1760	1700
Recorder .....	punch	pen recorder
Goniometer speed .....	$-\frac{1}{2}^{\circ}$ 2 $\theta$ /min	$-\frac{1}{2}^{\circ}$ 2 $\theta$ /min
	in 10 secs steps	continuously
Range in 2 $\theta$ .....	85°—40°	50°—30°

from the Pyörönmaa pegmatite by Vormaa *et al.* (1966). The standards used were the four synthetic REE silicate glasses prepared and reported by Drake and Weill (1972).

The »rare earths» include the elements scandium (Z 21), yttrium (Z 39) and the lanthanoids (Z 57—71). The main interest in this study was in determining the distribution of the lanthanoids in pegmatite minerals; thus scandium and yttrium (and the unstable lanthanoid promethium, Z 61) were excluded.

For the whole series of analyses, the author used the Geoscan electron probe analyser at the Geological Survey of Finland. The analytical conditions are given in Table 1. The scaler mode of analysing was used to measure the X-ray intensities. The data recorded from channel I were processed with a computer. The computer output consisted of the lists of recorded intensities and the corresponding spectra (Fig.1). The pen recorder spectra received from channel II were used as reference material in the course of the work.

The calculations are based on the net pulse intensities measured from the samples and the standards as well as on the known lanthanoid concentrations in the standards. No other corrections were applied in handling the results. The following X-ray lines were used in the calculations:  $L\alpha_1$ -line for La, Ce, Gd, Tb, Er, Yb and Lu;  $L\beta_1$ -line for Pr, Nd, Sm, Dy, Ho and Tm. The influence of the possible interferences could therefore be reduced to a minimum. The standard spectra and the positions of the analysed lines are shown in Fig. 1. Neither the  $L\alpha_1$ -line nor the  $L\beta_1$ -line could be used for Eu because of the heavy interference of the Pr  $L\beta_2$ , Nd  $L\beta_3$ , Dy  $L\alpha_{1,2}$  lines. These difficulties made the existence or the absence of the element europium impossible to determine by this means.

In the final presentation the relative amounts of the lanthanoids are used, expressed as  $Ln_2O_3$ . The applied method of analysis was tested with three spectro-chemically analysed samples, namely  $CeF_3$ (synth.), monazite and yttrian spessartine. The results of

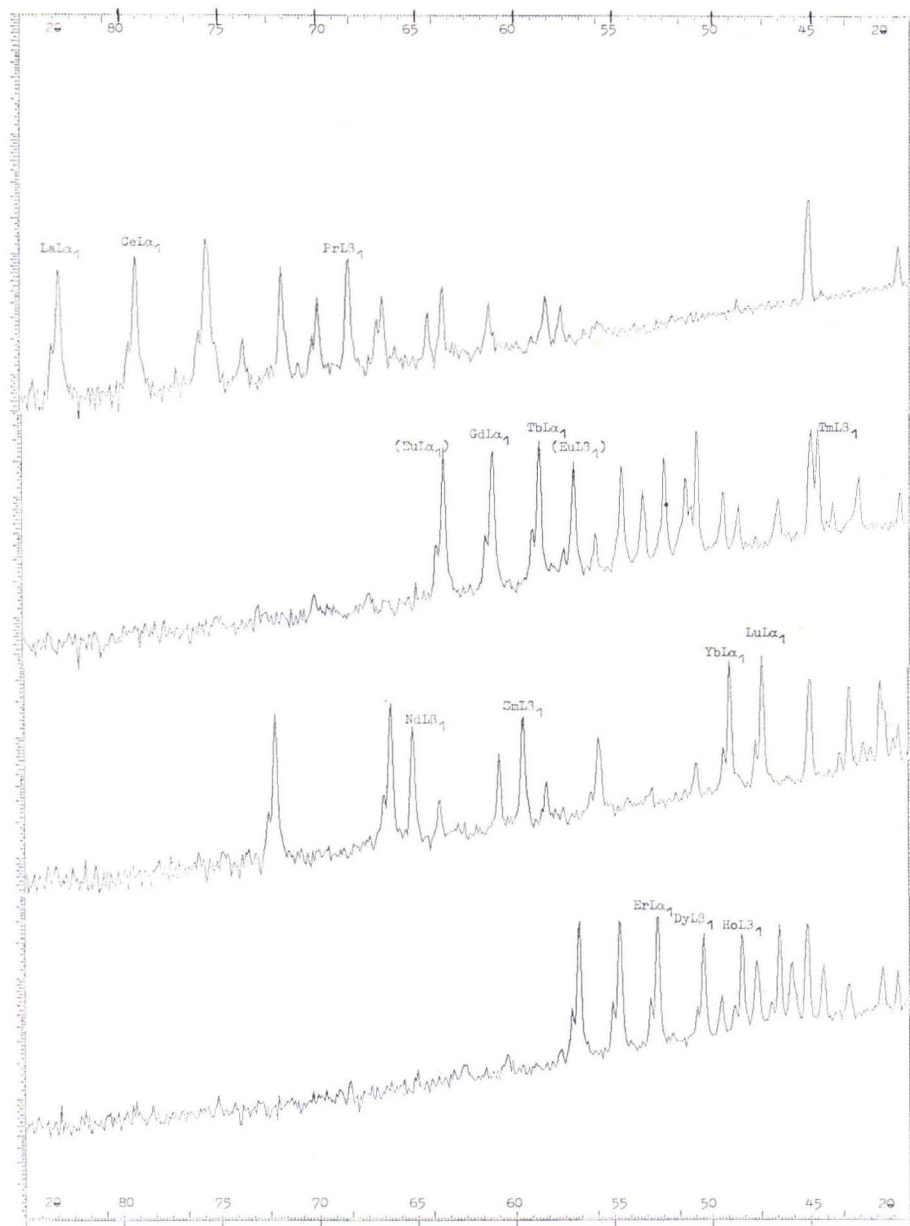


FIG. 1. The standard spectra and the analysed REE X-ray lines.

the tests are shown in Table 2 and in Fig. 2. With a high concentration of lanthanoids, the agreement between these two methods is evident. The correlation is not so good with very low REE contents.



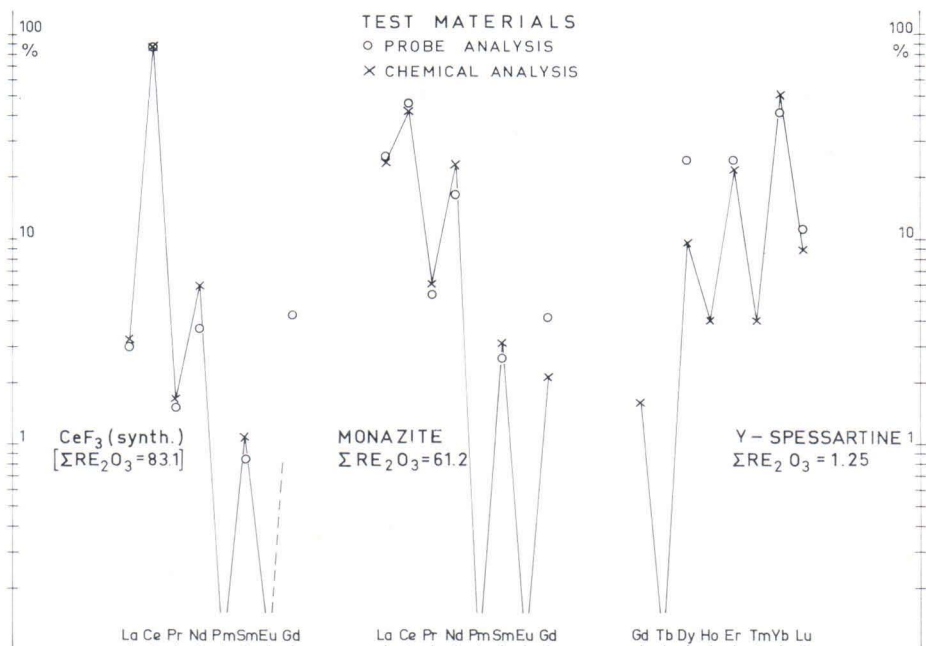


FIG. 2. Comparison between the electron microprobe and the chemical analyses of CeF<sub>3</sub>, monazite and yttrian spessartine.

TABLE 2

Comparison between the probe and spectrochemical analyses of CeF<sub>3</sub> (synth.), monazite and yttrian spessartine. The total sum of Ln<sub>2</sub>O<sub>3</sub> (Σ) has been calculated as 100 %.

Test material	La	Ce	Pr	Nd	Sm	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	Σ	
CeF <sub>3</sub> (synth.) . . .	3.01	86.72	1.52	3.65	0.84	4.25	—	—	—	—	—	—	—	—	probe spect.
	3.25	87.97	1.68	6.02	1.08	—	—	—	—	—	—	—	—	83.1	
Monazite <sup>1</sup> . . . . .	25.25	45.94	5.44	16.62	2.60	4.14	—	—	—	—	—	—	—	—	probe spect.
	23.53	42.16	6.04	23.04	3.10	2.12	—	—	—	—	—	—	—	61.2	
Y-spessartine <sup>2</sup> . .	—	—	—	—	—	—	—	—	—	—	—	—	—	—	probe spect.
	—	—	—	—	—	1.60	—	23.90	4.0	23.77	4.0	41.24	11.08	8.80	

<sup>1</sup> analysis published in Haapala *et al.* (1969)

<sup>2</sup> » » » Vorma *et al.* (1966)

The relative Ln<sub>2</sub>O<sub>3</sub> concentrations in the pegmatite minerals were determined from the calculated X-ray intensities. The results are given in Table 3, where the total sum of Ln<sub>2</sub>O<sub>3</sub> has been taken as 100 %. The list of minerals in Table 3 consists of 21 names. Six of them are still unidentified, namely X<sub>1</sub>, X<sub>3</sub>, inclusion A, Y-Ca silicate, Y silicate and Y-Al silicate. The very small grain size and, partly, the metamict state of the minerals make the X-ray diffraction studies difficult. The mineral rowlandite was not included in the paper of Vorma *et al.* (1966) but was later identified by Vorma (personal communication, see below).

TABLE 3.

The distribution of the lanthanoids in the Pyörönmaa pegmatite minerals.  $\Sigma$  is the total sum of  $\text{Ln}_2\text{O}_3$ , which has been calculated as 100 %.

	La	Ce	Pr	Nd	Sm	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu	$\Sigma$
1. Allanite (pale) . . .	15.89	36.50	5.49	19.86	6.80	6.75	1.05	3.43	0.47	1.67	0.50	0.92	0.67	36.9
2. $X_1$ (?) (near xenotime) . . . . .	5.74	20.55	5.02	12.37	11.30	13.27	4.07	11.26	1.46	7.43	1.70	4.74	1.09	8.87
3. Rowlandite I . . .	0.88	21.48	1.12	3.48	3.71	8.46	2.11	16.62	1.75	17.56	1.12	20.49	1.20	46.9
4. $X_3$ (?) . . . . .	—	13.63	—	4.13	7.40	7.79	3.34	16.58	3.19	20.81	—	20.38	2.75	5.35
5. incl. A in thalenite . . . . .	3.40	8.23	3.64	12.07	9.84	15.13	3.48	18.20	2.19	11.66	1.98	9.11	1.04	11.7
6. Britholite-(Y) . . .	2.34	9.26	2.16	10.72	7.22	13.72	3.26	18.29	2.03	15.85	1.25	12.41	1.47	38.9
7. Gadolinite . . . . .	2.45	8.33	1.95	10.84	7.72	13.16	2.96	20.41	2.11	15.08	1.12	12.49	1.37	35.3
8. Rowlandite II . . .	0.56	4.24	0.91	4.36	5.18	14.38	3.23	24.45	2.40	23.59	1.16	14.06	1.47	53.2
9. Y-Ca silicate . . .	—	2.57	1.52	4.74	6.51	15.09	3.00	27.04	2.31	18.88	1.32	16.42	0.58	42.8
10. Fergusonite (pale) . . . . .	—	2.73	—	4.07	4.68	11.53	3.20	25.77	3.15	21.80	2.56	19.04	1.42	30.9
11. $X_2$ (thorite?) . . .	—	—	—	10.08	12.64	5.74	6.55	21.82	—	13.24	9.35	13.73	6.85	4.68
12. Y silicate . . . . .	—	2.51	—	3.87	5.10	12.16	3.19	22.44	2.88	22.77	1.52	19.74	1.95	43.4
13. Y-Al silicate . . .	—	—	—	1.77	4.31	12.25	4.44	26.51	3.26	26.29	1.78	17.49	1.90	21.9
14. Thalenite . . . . .	1.67	5.91	2.30	6.46	6.29	11.74	2.67	19.80	2.48	19.00	1.37	18.96	1.34	37.0
15. Thorite . . . . .	—	1.67	—	2.99	5.28	9.47	3.62	14.38	3.77	26.24	2.95	26.61	3.01	19.2
16. Zircon (colourless) . . . . .	2.89	2.77	1.89	3.19	4.22	6.75	2.34	14.95	3.14	20.96	2.95	28.16	5.77	11.4
17. Zircon . . . . .	—	1.75	—	—	2.61	5.36	1.45	17.06	2.90	27.08	2.52	34.84	4.44	11.8
18. Xenotime . . . . .	—	—	—	—	2.38	6.47	2.25	21.55	3.33	27.15	1.76	31.63	3.49	32.6
19. Lokkaite . . . . .	1.36	2.69	1.97	7.53	9.36	23.77	4.52	26.03	2.60	13.39	1.23	4.96	0.58	27.5
20. »Carbonate» . . .	1.48	4.75	1.32	7.84	5.64	13.27	3.38	21.20	2.40	23.30	1.06	13.30	1.06	39.9
21. Tengerite . . . . .	—	—	—	1.58	1.36	5.19	2.30	22.48	3.21	29.55	1.92	30.12	2.28	26.1

The same results are presented in Figs. 3, 4, 5 and 6. In the figures, there are logarithmic ordinates (see, Fig. 2) with one decade shift for each distribution pattern. To avoid misunderstandings, the reader should note that the results are only partial analyses of these minerals, which rules out the calculation of the absolute REE percentages. The absence of yttrium from the results does not affect the relative amounts of the lanthanoids.

DISTRIBUTION OF LANTHANOIDS

The reader is first referred to Figs. 7 and 8, which illustrate the occurrence of some of the analysed REE minerals. For more complete information see also Vormaa *et al.* (1966).

The first two distribution patterns in Fig. 3 and in Table 3 show those minerals in which the cerian earths are the predominating lanthanoids. Allanite has the most pronounced cerium assemblage with  $\text{Sm} \sim \text{Gd}$ . X-ray diffraction studies proved the Pyörönmaa allanite to be in a metamict state. The analysis presented here was made on a fresh (pale) domain of this mineral. The other varieties with differing colours from black to reddish were also investigated. The lanthanoid distributions show no remark-

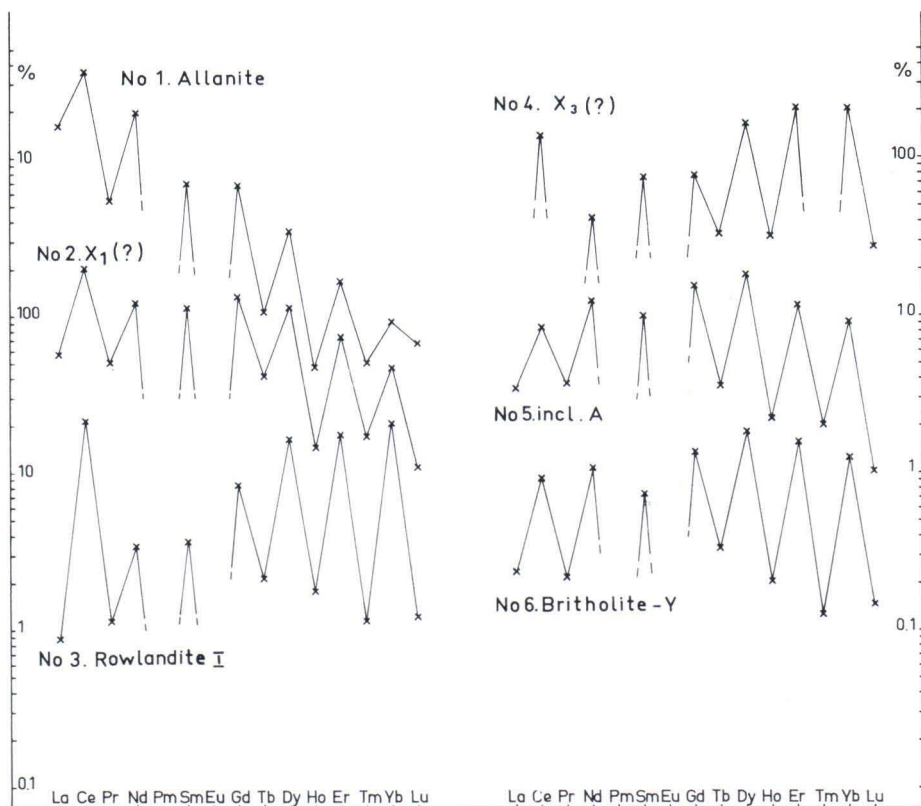


FIG. 3. Lanthanoid distributions in Pyörönmaa pegmatite minerals.

able variations. Some allanite samples were impoverished from the light lanthanoids. As a rule, all the analysed allanites exhibit the cerian-type assemblage. Pattern No. 2 in Fig. 3 presents the lanthanoid distribution in an unidentified small mineral grain  $X_1$ . All the lanthanoids, excluding Pm and Eu, are present with distribution maxima at cerium and gadolinium. The mineral occurs between xenotime and thalenite (Fig. 7), but the lanthanoid distribution is not identical with those of the neighbouring minerals (see below).

Nos. 3 and 4 in Fig. 3 show a unique lanthanoid distribution pattern. In these »spectra», cerium has a pronounced maximum together with another maximum in which  $Dy \sim Er \sim Yb$ . Rowlandite I (No. 3) is a reddish-coloured alteration product of britholite-(Y). Vorma *et al.* (1966) described this mineral as an unidentified Y-Fe silicate. The X-ray diffraction studies performed later by Dr. A. Vorma proved that the crystalline Y-Fe silicate (Fig. 8) produces an X-ray diffraction pattern identical to that obtained from the heated, metamict mineral called rowlandite by Sabina and Traill (1960). — Mineral  $X_3$  (No. 4) occurs in contact with fergusonite. These two minerals differ greatly in the distribution of lanthanoids.

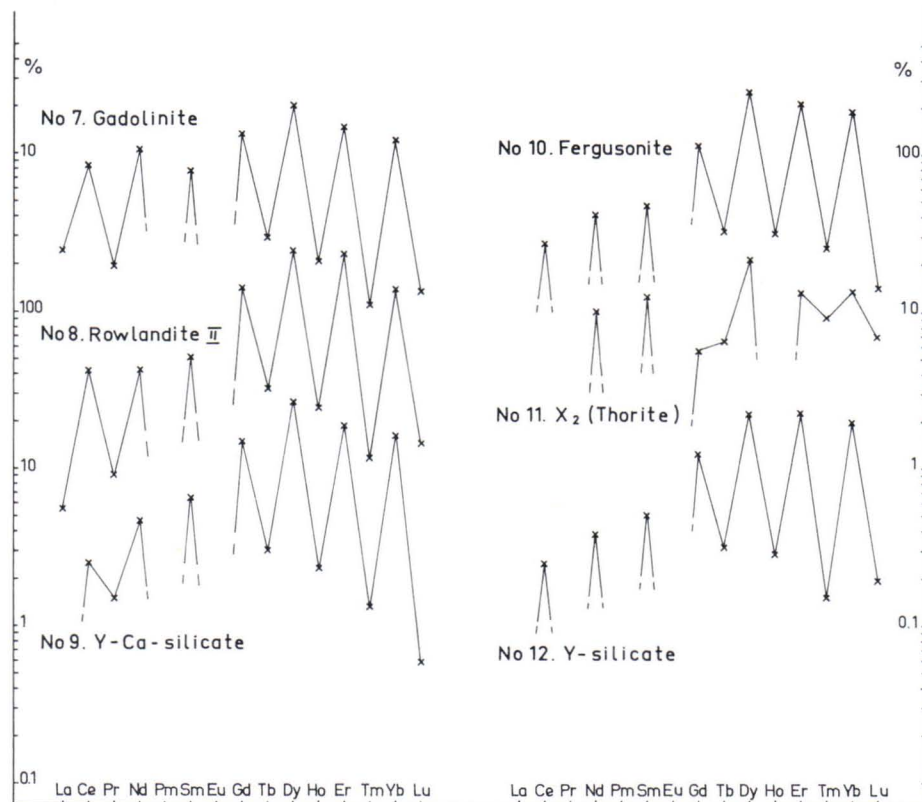


FIG. 4. Lanthanoid distributions in Pyörönmaa pegmatite minerals.

Also the next three distribution patterns, Nos. 5—7, show two maxima. The maximum of the cerium earths occurs at neodymium and that of the yttrium earths at dysprosium with  $Dy > Er > Yb$ . The minerals named rowlandite II and Y-Ca silicate are both alteration products of britholite-(Y). Rowlandite II is colourless compared with the reddish rowlandite I. These two varieties occur intimately together. Further, the unidentified Y-Ca silicate occurs in the alteration rim between britholite-(Y), rowlandites and albite (Fig. 8). The rare earth contents from neodymium to lutetium of these two secondary minerals are quite similar. In the Y-Ca silicate, lanthanum is missing and the cerium content is less than in rowlandite II.

As reported by Vormaa *et al.* (1966) the mineral fergusonite occurs in three different phases in the Pyörönmaa pegmatite. The analysis reported here is of a greenish, completely isotropic fergusonite. This is believed to be the primary fergusonite phase. The earlier analyses (*op. cit.*, pp. 264—268) show that the yttrium content is higher in the greenishbrown, isotropic fergusonite and in the anisotropic phase. This supports the view that these varieties are more advanced recrystallization products. Vormaa reports (personal communication) that the anisotropic material

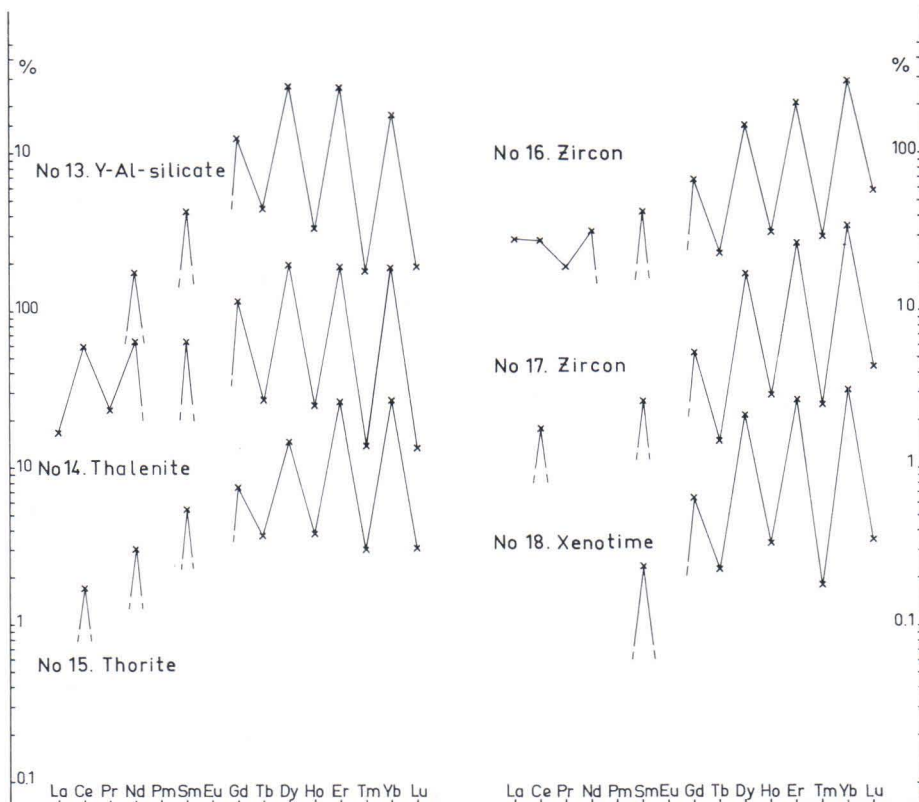


FIG. 5. Lanthanoid distributions in Pyörönmaa pegmatite minerals.

inside the conspicuously hydrated fergusonite is most probably identical with the monoclinic fergusonite synthesized by Wolten and Chase (1967).

The minerals from fergusonite to the Y-Al silicate (Figs. 4 and 5) have lanthanoid distributions with only one maximum with  $Dy > Er > Yb$ . Mineral  $X_2$  (thorite?) shows an abnormal «knick» with  $Gd < Tb$ . This may be due to an analytical error caused by the fairly low REE content of this mineral. Also thalenite (No. 14 in Fig. 5) has a unique lanthanoid distribution. The relative amounts of the even numbered elements is  $(Ce \sim Nd \sim Sm) < Gd < (Dy \sim Er \sim Yb)$ . This is different from the so-called selective lanthanoid assemblage «thalenite type» presented by Goldschmidt and Thomassen (1924).

The distributions observed in the last four samples (Nos. 15—18) in Fig. 5, show a marked increase in the relative amount of the heavier lanthanoids Dy, Er and Yb. Also the odd-numbered elements Ho, Tm and, especially, Lu are enriched in these minerals. The difference between the two zircons is evident. The colourless (pale) variety (No. 16) has  $La \sim Ce < Nd$  whereas in the dark brown zircon (No. 17) only cerium has been detected. Xenotime is completely impoverished from the

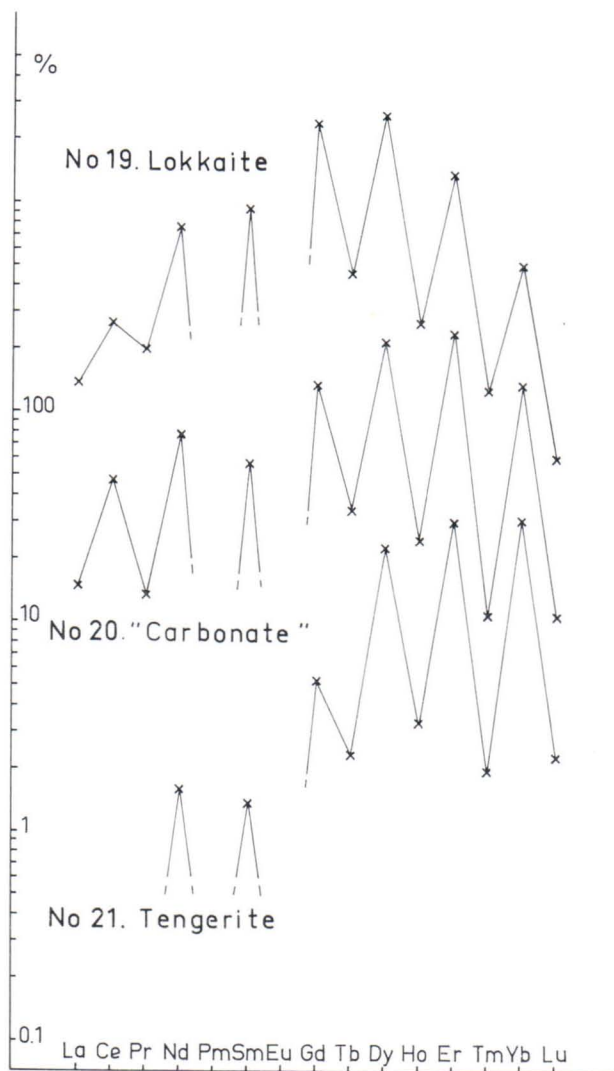


FIG. 6. Lanthanoid distributions in three carbonates from Pyörönmaa pegmatite.

cerian earths except from samarium. The lanthanoid maximum has shifted to ytterbium with  $Yb > Er > Dy$ .

In the course of this work, three different types of carbonates were analysed. The carbonates are rich in heavy lanthanoids. (Fig. 6). In each of them, the lanthanoid distribution is different from the others. No. 19 in Fig. 6, labelled as Lokkaite, displays only one clear maximum with  $Gd \sim Dy$ . No. 20 has a maximum with  $Er > Dy > Gd$  ( $\sim Yb$ ) and a secondary maximum at neodymium. The carbonate (No. 21), tentatively named tengerite, shows a different lanthanoid assemblage. Lanthanum and

cerium are lacking and neodymium prevails over samarium. The maximum has moved to the heavier elements with  $Yb \geq Er > Dy$ . The carbonates (Nos. 19 and 21) are rich in yttrium containing about 30 weight-%  $Y_2O_3$  (see  $\Sigma$  in Table 3 in this paper and p. 68 in Perttunen 1971). The third carbonate (No. 20) contains  $Y_2O_3$  only approximately half of that value. In the distribution of lanthanoids, these carbonates differ markedly, from bastnaesite and synchysite-(Nd), which were reported by Vorma *et al.* (1966) to be rich in cerium earths.

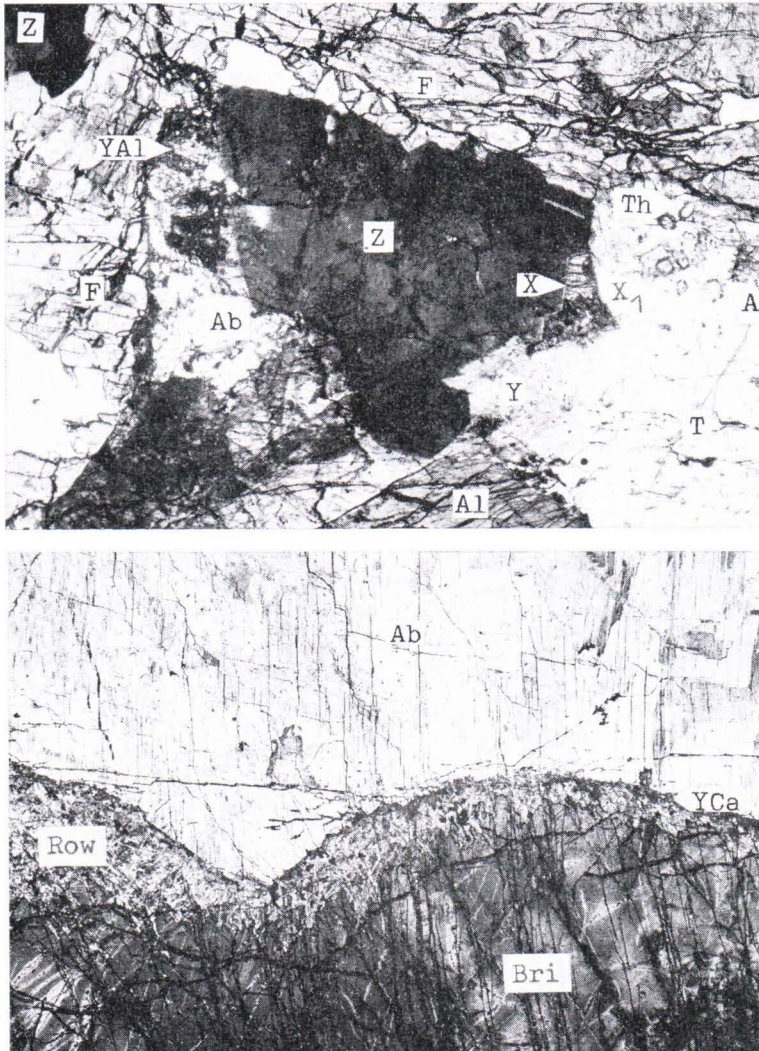


FIG. 7. Partial magnification of Fig. 10 from Vorma *et al.* (1966). Zircon (Z), fergusonite (F), thalenite (T), xenotime (X), unknown mineral ( $X_1$ ), albite (Ab), thorite (Th), allanite (Al), unknown Al-Y silicate (A), unknown Y-Al silicate (YA1), unknown Y silicate (Y). Magn. 5 x. Photo: Erkki Halme. FIG. 8. Britholite-(Y) (Bri) replaced by rowlandite (Row) and an unidentified Y-Ca silicate (YCa). Upper part of the figure is albite (Ab). Magn. 7 x. Photo: Erkki Halme. See also Fig. 9 in Vorma *et al.* (1966).

## CONCLUSIONS

The analyses show that the distribution of lanthanoids varies considerably in the Pyörönmaa pegmatite minerals. The following features appear when the observations made of the thin sections and of the lanthanoid distribution patterns are combined.

Allanite has a normal cerian composition. This is typical of allanite if the paragenesis contains phosphates (K. K. Zhirov *et al.*, 1961). According to Vormaa *et al.* (1966), the Ce-rich silicate, allanite, is the first RE mineral to be crystallized in the Pyörönmaa pegmatite. Allanite is older than the Y-rich silicates and fergusonite. The lanthanoid distribution patterns of allanite and fergusonite seem to show that the older mineral, allanite, has a higher cerium earth content than the younger mineral, fergusonite. Yttrium-rich allanite has not been found among the mineral samples taken from the Pyörönmaa pegmatite.

The distribution pattern of rowlandite I and the unknown mineral X<sub>3</sub> with maxima at cerium and ytterbium (erbium) is basically identical to that of »type IV gadolinites» established by Vainshtein *et al.* (1960). The unidentified mineral X<sub>3</sub>, No. 4 in Fig. 3 and in Table 3, with a high cerium content may have crystallized before fergusonite. Compared with the entirely anhydrous fergusonite, this mineral shows a subhedral to euhedral habit.

A characteristic feature of the distribution curve of gadolinite is the presence of two maxima, at neodymium and at dysprosium with Dy > Nd, respectively. Vainshtein *et al.* (1960) reported that gadolinite from granite pegmatites with rare earth mineralizations exhibits this type of distribution.

Britholite-(Y) has a lanthanoid distribution almost identical to that of gadolinite. This supports the idea advanced by Vormaa *et al.* (1966) that these minerals have crystallized simultaneously. The alteration products of britholite-(Y) differ greatly from each other. Rowlandite I shows the unique pattern with two maxima where Ce ~ Yb. The other two secondary minerals, rowlandite II and Y-Ca silicate, are impoverished in the cerian earths with respect to britholite-(Y). This additional fractionation of the lanthanoids has taken place during alteration processes. A slightly different example of the fractionation of cerium in rhabdophane has been given by Adams (1969). The system of nomenclature for RE minerals by Levison (1966) requires that the secondary minerals rowlandite I and rowlandite II should be called rowlandite-(Ce, Yb) and rowlandite-(Dy).

The mineral inclusion A in thalenite has obviously crystallized before thalenite. Its lanthanoid distribution also tends more towards the light lanthanoids than that of thalenite. As pointed out, the lanthanoid content in thalenite with (Ce ~ Nd ~ Sm) < Gd < (Dy ~ Er ~ Yb) differs strikingly from the classic »thalenite type».

The colourless (pale) zircon occurs partly inside the dark variety. Also the pale zircon has a more cerian lanthanoid content than the dark variety. Xenotime with a distribution rich in heavy lanthanoids is probably one of the latest minerals to be crystallized in the Pyörönmaa pegmatite.



Yttrian spessartine was used as a calibration material in this work. The lanthanoid distribution pattern of this mineral (Fig. 2) — compared with those of the other minerals present in the Pyörönmaa pegmatite — shows the most pronounced maximum at ytterbium.

The carbonates seem to be of secondary origin. They have been formed through replacement (secondary reactions) from the primary minerals or crystallized as supergene precipitations on other minerals and in fractures between and in mineral grains. As was reported by Vormä *et al.* (1966), this is the last stage of evolution of the minerals containing rare earth elements in the Pyörönmaa pegmatite.

### SUMMARY

In the intermediate zone of the Pyörönmaa pegmatite, there occur silicates, carbonates, oxides and phosphates containing rare earth elements. The distribution of lanthanoids in some of these minerals has been analysed with the electron microprobe. The results are in brief as follows:

1. The rare earth minerals from the Pyörönmaa pegmatite, except yttrian spessartine and xenotime, can be regarded as complex minerals, in which both the cerium and yttrium groups are well represented. Y-spessartine and xenotime belong to the yttrium-selective minerals, in which the yttrium group is dominant.

2. The crystallization order of the primary rare earth minerals in the Pyörönmaa pegmatite can be at least partly traced by following the lanthanoid distribution of these minerals.

3. The distribution of lanthanoids of the secondary phases differ greatly from that of the primary phases. The secondary reactions affecting a single mineral may lead to the crystallization of two new phases with different REE contents, e.g., britholite-(Y) = > rowlandite I and rowlandite II. The supergene carbonates are all rich in heavy lanthanoids.

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