

Identity of “freyalite”, an alleged rare earth-rich variety of thorite, and its pre-metamict composition

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Abstract

This paper presents electron microprobe analyses of the type specimen of “freyalite”, a REE-Th mineral found in a nepheline syenite pegmatite of the Langesundfjord district (S. E. Norway). Freyalite was previously assumed to be related to thorite, but these data indicate that it is more accurately described as a thorian melanocerite (caryocerite) which has been altered with subsequent leaching of Na, Ca and F. The composition of the material may be expressed by the formula $(RE, Ca, etc.)_x(B, Si, etc.)_3(O, OH, F)_{13}$. In an analysis of the least altered part of the specimen $x = 4.017$: $(RE_{1.870}Y_{0.167}Th_{0.413}Zr_{0.104}Ca_{1.129}Fe_{0.095}Na_{0.239})(B_1Si_{1.823}P_{0.159}S_{0.019})(O_{7.067}OH_{3.176}F_{2.757})$ (boron and hydroxyl contents estimates only). This evidence discredits the name “freyalite”. The crystalline precursor of the metamict melanocerite was probably a boron- and fluorine-rich member of the mosandrite–gotzenite or cerite groups.

The Subcommittee on New Minerals and Mineral Names of the International Mineralogical Association has approved the discreditation of “freyalite”.

Introduction

In his classic monograph of the mineralogy of the nepheline syenite pegmatites of the Langesundfjord area, S. E. Norway, Brøgger (1890) described a number of calcium and rare earth bearing minerals which he grouped together with thorite. These include calcliothorite, eucrasite and freyalite. Of these, freyalite has the highest rare earth content and has gained a certain importance in mineralogical literature in regard to the proposed substitution of Ce^{4+} for Th^{4+} in thorite. Frondel (1958) used Brøgger's (1890) results to postulate extensive solid solution between thorite and the hypothetical component $CeSiO_4$, while Semenov (1963) extended the name “freyalite” to cover the $CeSiO_4$ component. This approach is somewhat in conflict with the views of Brøgger (1890), who expressed some doubt concerning the status of freyalite, and suggested that the mineral might be classified with tritomite or melanocerite rather than with thorite. Struntz (1977), on the other hand, classified freyalite as an alteration product of thorite; a view which is clearly not in agreement with Brøgger's (1890) descriptions.

Previous discussion of the chemical composition of freyalite has been based on an analysis by Damour (1878), which was cited by Brøgger (1890). This analysis

(Table 2) was recalculated by Neumann (1985) as: $(Ce_{0.416}RE_{0.016}Th_{0.248}Zr_{0.026}Fe_{0.072}Mn_{0.058}Na_{0.174})(Si_{0.773}Al_{0.227})(O_{2.716}OH_{1.281})$, a formula which corresponds remarkably well with thorite stoichiometry, allowing substitution of hydroxyl for oxygen. However, as the material was analyzed by bulk, wet chemical techniques, determining groups of elements rather than single components and not taking into account any possible heterogeneities in the sample, it was felt that it was a poor foundation for some of the wider conclusions drawn from it. A reexamination of the freyalite type specimen was therefore undertaken.

The type specimen

The type specimen of freyalite was collected by M. Thr. Esmark who also named the mineral; it is in the possession of the Mineralogical-Geological Museum, University of Oslo. To the best of the present authors' knowledge, no other samples of the assumed species exist. The locality where the sample was collected is not accurately known. The original labels of the specimen indicate the island Arø in the western part of the Langesundfjord (S. E. Norway); Brøgger (1890) however suggested that the Barkevik skerries, which are islands a few kilometers further east in the Langesundfjord may be the true locality.

The material described as freyalite is closely similar to thorite from the Langesundfjord district in terms of its

¹ Deceased October 24th, 1983.

physical properties. It is yellowish brown, translucent in thin splinters, and has a vitreous luster. It forms the central part of a small nodule (diameter about 5 cm) and is enclosed by and partly intergrown with, the major minerals of the host pegmatite, which are alkali feldspar, nepheline and green pyroxene or amphibole. No external crystal faces or cleavages are present. Towards the edges, the material is darker, and has a duller luster; the material analyzed by Damour (1878) was taken from the central, vitreous part of the specimen, while no analyses of the outer zones of the nodule exist.

The material is completely metamict; on heating to 1000°C in air for 24 hours, it gives a tritomite-B X-ray diffraction pattern (see Neumann et al., 1957). This is a composite pattern, whose strongest lines originate from a cubic phase isostructural with uraninite.

Analytical methods

A polished, carbon-coated grain mount of the original freyalite specimen was analysed for major and minor elements with the electron microprobe. The instrument used was an ARL-EMX microprobe equipped with a LINK energy dispersive analyser unit. Data were reduced on-line with the program ZAF4/FLS, allowing a total of 14 elements in any single analysis. The acceleration voltage was 15 kV. The synthetic, three element standards of Åmli and Griffin (1975) were used for calibration of the rare earths. Care had to be taken during calibration to choose energy intervals giving the least overlap of peaks. Accuracy and precision were checked against the synthetic REE-silicate glasses of Drake and Weill (1972). Standard chemical data are given in Table 1, together with approximate detection limits for the rare earth oxides estimated from counting statistics. Because of high ZAF-correction factors, the precision of these analyses is poor compared to those involving only the common major elements. It may be noted that for elements other than Yb, accuracy and precision are comparable to results obtained with the more comprehensive (ZAF4 + /FLS) program (for example Styles and Young, 1983). Thorium and the remaining constituents of the analyses present fewer peak overlap problems and were analysed with routine energy-dispersive microprobe techniques. Fluorine cannot be analyzed with sufficient accuracy and precision with an energy dispersive microprobe and was therefore analyzed separately using an automatic wavelength-dispersive Cameca Camebax instrument at the Central Institute for Industrial Research, Oslo. Electron backscatter photomicrographs of the sample were taken with a Jeol Superprobe scanning electron microscope at the Continental Shelf Institute (IKU), Trondheim, Norway. The bulk content of boron was determined semiquantitatively, using a Zeiss-Hilber emission spectrograph at the Central Institute for Industrial Research, Oslo. A separate fragment of the central part of the specimen was used for this analysis. Another fragment of the specimen was used for combined thermogravimetry/DTA analysis, using a Mettler thermobalance at the Chemistry Department, University of Oslo. The sample was heated to 1000°C in a nitrogen atmosphere, with Al₂O₃ as a reference substance. The cold junction of the thermocouple was kept at 25°C.

Results

Heterogeneity

The grain mount made for microprobe analysis contains fragments of both dull (peripheral) and vitreous (central)

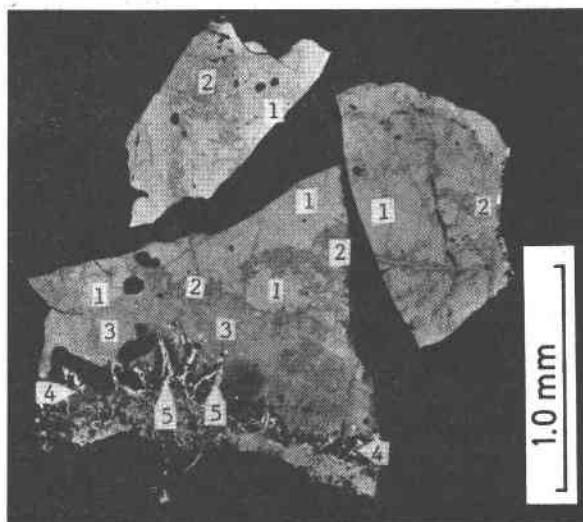


Fig. 1. Low magnification electron backscatter photomicrograph of the microprobe mount made from the freyalite type specimen. Numbers identifying the different phases: 1-3: see Table 2; 4: intergrowth of REE- and Ca-REE-silicates; 5: Thorite or thorumite. Phase 1 (mean atomic number ca. 17) has the second highest mean atomic number of the phases present, superseded only by thorite (mean atomic number 22.67). A cerian thorite (CeTh(SiO₄)₂ with mean atomic number 20) would have a shade between those of phases 1 and 5.

parts of the freyalite specimen. Figure 1 shows a low magnification electron backscatter photomicrograph of the mount, illustrating differences in mean atomic number as shades of grey, the lightest shade representing the highest mean atomic number. At least five compositionally different phases may be recognized in Figure 1, forming domains with apparently sharp contacts. One of these, designated as phase 1, is found only as rounded relics. Along fractures and towards the rims of the specimen phase 1 is mantled by successive zones of phases 2 and 3, both of which have lower mean atomic numbers. The outermost part of the sample is a heterogeneous crust, consisting of intergrowths of phase 4 (black) and phase 5 (white). Phase 5 has the highest mean atomic number in the sample. From the relations illustrated in Figure 1, phase 1 is interpreted to be the best representative of the primary, nonmetamict mineral, while all other phases represent successive stages of replacement; these most probably evolved during or after metamictization.

Chemical composition

New microprobe analyses of phases 1, 2 and 3 found in the type specimen of freyalite are presented in Table 2. Wet chemical analyses of freyalite, melanocerite, tritomite and caryocerite (thorian melanocerite) from the Langesundfjord pegmatites (cited from Brøgger 1890), are included for comparison.

The microprobe analyses differ from the original bulk analysis of freyalite in several ways: Calcium and fluorine

Table 1. Rare earth compositions of the four standard glasses of Drake and Weill (1972).

	Literature-value	This work	Detection limit
La ₂ O ₃	4.28	4.52± 0.28	1.2
Ce ₂ O ₃	4.00	4.18± 0.14	0.4
Pr ₂ O ₃	4.44	4.14± 0.28	1.3
Nd ₂ O ₃	4.26	4.12± 0.32	0.6
Sm ₂ O ₃	4.26	3.91± 0.10	0.8
Eu ₂ O ₃	4.40	4.70± 0.27	1.0
Gd ₂ O ₃	4.46	4.47± 0.51	0.9
Tb ₂ O ₃	4.35	4.35± 0.41	0.6
Dy ₂ O ₃	4.36	4.35± 0.31	0.6
Ho ₂ O ₃	4.41	4.70± 0.38	0.6
Er ₂ O ₃	4.36	4.26± 0.91	0.5
Tm ₂ O ₃	4.35	4.02± 0.60	0.5
Yb ₂ O ₃	4.26	2.74± 1.37	0.7
Lu ₂ O ₃	4.26	4.03± 0.80	0.7
Y ₂ O ₃	4.08	3.95± 0.19	0.3

Data on elements other than La, Ce and Nd are included to indicate approximate upper limits for rare earth elements present in the "freyalite" in amounts below the detection limits. All data are in weight percent oxides. The analytical uncertainty (one standard deviation) and the detection limits are derived from the microprobe's counting statistics. Note the poor accuracy and precision of the ytterbium analyses.

(previously unreported) prove to be important constituents, and silica is lower than previously believed. Aluminum, manganese, and potassium (Damour, 1878) do not exceed the microprobe detection limits (around 0.1 wt.% oxide) in our analyses. Sodium, calcium and fluorine contents, and the sum of the microprobe analyses, decrease progressively from phase 1 to phase 3. Silica, on the other hand, increases.

Qualitative microprobe analyses show that the outer crust of the specimen consists of ordinary thorite or thorumite with low rare earth content (phase 5) intergrown with a number of different thorium-free REE or Ca-REE silicates (grouped together as phase 4).

For phases 1, 2 and 3, La, Ce and Nd define straight, or nearly straight, light rare earth-enriched chondrite normalized distribution patterns. The slopes are nearly identical for all three phases. No distribution anomalies for cerium can be detected (Fig. 2) indicating that little or no Ce was in the 4+ state during the evolution of the material.

Optical spectroscopic analysis indicates the presence of boron at a bulk concentration between 0.3 and 3.2 wt.% B₂O₃. Thermogravimetric analysis indicates a total loss on ignition of 9.1 wt.% in the interval 100 to 1000°C (Fig. 3). The dehydration curve shows that the sample lost water in a stepwise fashion on heating. Loss of mass below ca.

480°C is probably due to liberation of molecular (adsorbed) water. Loss of hydroxyl groups is probably the cause of the steep step on the curve at about 500°C; the smaller loss of weight at high temperatures is probably a result of liberation of fluorine or of volatile, fluorine-bearing components produced on heating (e.g., SiF₄). Only the low-temperature step of weight-loss corresponds to a well-defined endothermic peak on the DTA-curve (Fig. 3, lower curve). At higher temperatures the DTA-curve shows a broad endothermic continuum.

Discussion of results

Identity of "freyalite"

As can be seen from Table 2, the calcium content of phase 1 is much too high, and the silica too low, to justify the earlier assumption of its equivalence with thorite. The analyses cannot be constrained to conform with thorite stoichiometry, unless very extensive OH-substitution is

Table 2. Chemical compositions of the freyalite type specimen and related materials.

	Phase ¹ 1	Phase ¹ 2	Phase ¹ 3	FREY ²	MELAN ³	TRITO ³	CARYO ³
	Weight percent oxides						
SiO ₂	14.23	16.57	18.25	20.02	13.07	13.54	12.97
Al ₂ O ₃					0.83	1.18	0.87
B ₂ O ₃					3.19	7.31	4.70
Fe ₂ O ₃	0.98	1.01	0.64	2.47	2.09	1.67	1.36
Mn ₂ O ₃				1.78	1.22	0.67	0.66
MgO					0.14		0.17
CaO	8.23	6.94	2.70		8.62	7.04	7.37
Na ₂ O	0.96	0.48	0.11	2.33 ⁴	1.45	1.40	1.42
La ₂ O ₃	15.21	15.08	14.93	2.47 ⁵	12.94	16.31	14.34
Ce ₂ O ₃	20.84	20.16	20.94	28.80	24.16	22.07	20.18
Nd ₂ O ₃	3.89	3.75	3.73		7.67 ⁸	5.57 ⁸	6.75 ⁸
Y ₂ O ₃	2.44	1.64	2.04		9.17	2.97	2.21
ZrO ₂	1.70	1.13	1.31	6.31 ⁶	0.46	1.09	0.47
ThO ₂	14.18	13.51	16.06	28.39	1.66	9.51	13.64
Ta ₂ O ₅					3.65	1.15	3.11
P ₂ O ₅	1.47	1.35	1.14		1.29		0.86
CO ₂				0.82 ⁷	1.75		0.35
F	6.81	2.02	0.85		5.78	4.29	5.63
H ₂ O				7.40	3.01	6.40	3.11
SO	0.15	0.08	0.10				
-O=F	2.87	0.85	0.36		2.42	1.81	2.37
Sum	88.22	82.87	82.44	100.79	99.83	100.36	99.46

1: Bulk B₂O₃ content between 0.3 and 3.2 wt%, loss on ignition 9.1 wt%. 2: Original analysis of freyalite, Damour (1878). 3: Melanocerite (MELAN), tritomite (TRITO) and caryocerite (CARYO) from Brøgger (1890). 4: Includes K₂O. 5: Includes Nd₂O₃. 6: Includes Al₂O₃. 7: Includes unspecified volatiles. 8: Includes Sm₂O₃ ("Di₂O₃").

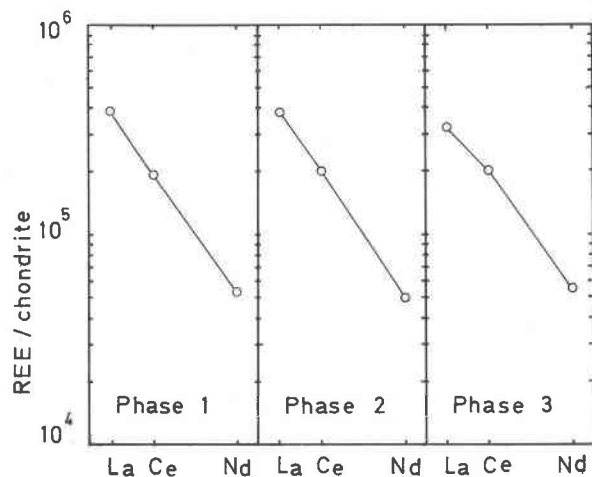


Fig. 2. Chondrite normalized rare earth distribution patterns of phases 1 to 3 in the freyalite type specimen. Normalized to the 9-chondrite values of Haskin et al. (1968).

postulated, both by the mechanism $4(\text{OH}) = \text{SiO}_4$ and by substitution of OH for O bonded to Si. The material analyzed by Damour (1878) must have been strongly contaminated with the major phases of the pegmatite dyke, in order to account for the high aluminum, silica, potassium and manganese. On the other hand, the compositional similarity of the least altered part of the freyalite specimen with thorian melanocerite ("caryocerite") is striking. The composition of phase 1 differs from the old analysis of thorian melanocerite mainly in its higher SiO_2 content.

The melanocerite group is an imperfectly understood group of boron-bearing, metamict minerals, whose composition is commonly given as $(\text{Ce}, \text{Ca})_5(\text{Si}, \text{B})_3\text{O}_{12}(\text{OH}, \text{F}) \cdot n\text{H}_2\text{O}$ (Fleischer, 1983). According to the usage of Clark (1984), only melanocerite (including the thorian variety "caryocerite") and tritomite may be recognized as valid species. Spencite, earlier assumed to be a distinct mineral (Jaffe and Molinski, 1962), should be classified as tritomite-(Y). The members of the melanocerite group are distinguished from each other by minor chemical differences.

The nature of the crystalline precursor

No crystalline species with composition corresponding to the melanocerite formula are known. From the compositional similarity with britholite, the minerals have commonly been assumed to belong structurally to the silicate apatites (Kupriyanova and Sidorenko, 1963; Ewing and Chakoumakos, 1982). The X-ray diffraction evidence used to support this assumption is ambiguous. Small differences in chemical composition of the metamict material and in the physical conditions during heating may stabilize different crystalline phases (Neumann et al., 1957; Hogarth et al., 1973). Borneman-Starynkevich (1968) suggested that the melanocerite-group minerals give a mixture of three different phases on heating: A britholite (incorporating tri-

valent rare earths and thorium), a ceric oxide and a glass (containing fluorine, boron, aluminum, ferric iron and excess rare earths). In the present case, the high proportions of Th and Ce in the freyalite sample may be assumed to influence strongly the recrystallization process, stabilizing a cubic $(\text{Th}, \text{Ce})\text{O}_2$ phase which is responsible for the tritomite-B pattern obtained. In the freyalite type specimen, both mean atomic numbers and the sums of the microprobe analyses decrease from phase 1 to phase 3. Simultaneously, the SiO_2 -content increases. Although phase 1 is least affected by alteration processes, there is no reason to believe that its composition is identical to that of its crystalline precursor. Similar observations in other occurrences have led to suggestions that the crystalline precursors may have had compositions significantly different from that indicated by the idealized formula. Borneman-Starynkevich (1968) suggested that the melanocerite-group minerals could derive from minerals with datolite-type stoichiometry, while Hogarth et al. (1973) suggested hellandite as a precursor for the tritomite-(Y) phase from Bancroft, Ontario (Canada). Although calculation of structural formulas for metamict and altered material may not be a valid pro-

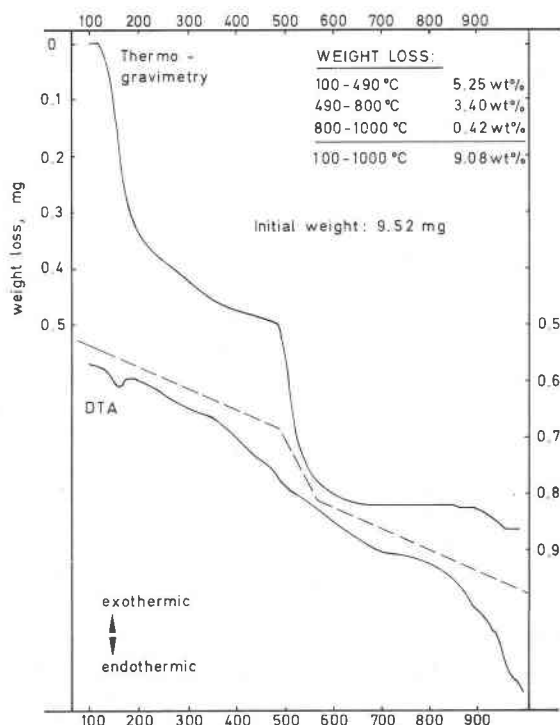


Fig. 3. Combined thermogravimetry and DTA curves for a sample of the freyalite type specimen. The thermogravimetry curve (upper curve) shows the weight loss of the solid sample as a function of temperature. The scale on the figure refers to this curve only. The DTA curve (lower curve, no scale) shows endothermic and exothermic events as indicated on the figure. The inserted table gives loss on ignition (wt.%) in different temperature intervals, as derived from the thermogravimetry curve.

cedure in terms of crystal chemistry, such recalculation of the analyses may be a useful guide to identification of the crystalline precursor. A general formula for a mineral giving rise to the present material on metamictization may be written as: $M_xZ_z(O, OH, F)_y$, where Z represents tetrahedral cations (Si^{4+} and those which may replace it, mainly P^{5+} , S^{6+} , Al^{3+} and B^{3+}) and M represents larger cations contained in non-tetrahedral position(s) (Fron del, 1958, Ito, 1968). Our data indicate that hydration and selective leaching of some components (sodium, calcium and fluorine) has taken place during or after the metamictization of the freyalite type specimen. The relative increase in silica on alteration indicates that the tetrahedral cations were the least mobile during alteration; therefore a fixed value for z in the general formula is the best basis for calculation of cation distributions. Due to the lack of reliable boron data on the freyalite type specimen the formulas given in Table 3 for phases 1, 2 and 3 are based on fixed sums of Si + P + S + Al.

As can be seen from the formulas in Table 3, the old analysis of thorian melanocerite has a cation distribution in good agreement with the general formula for the melanocerite group, but with F in excess of one atom per formula unit. The microprobe analyses cannot be recalculated to a similar formula on a boron-free basis. If a boron content corresponding to one atom per three tetrahedral sites is assumed (in agreement with Brøgger's (1890) data on thorian melanocerite, and roughly corresponding to the extent of B^{3+} substitution which may be calculated from the maximum bulk boron content indicated by the spectrographic analysis); the analysis of phase 1 can be recalculated to a formula with $x = 4.017$: $(RE_{1.870}Y_{0.167}Th_{0.413}Zr_{0.104}Ca_{1.129}Fe_{0.095}Na_{0.239})(B_1Si_{1.823}P_{0.159}S_{0.019})(O_{7.067}OH_{3.176}F_{2.757})$. Taking the selective leaching of some non-tetrahedral cations into account, there is no reason to believe that this formula represents the true composition of the crystalline precursor. It seems logical, however to postulate that the precursor probably had a formula of the type: $(RE, Y, Th, Zr, Ca, Fe, Na)_x(Si, B, P, Al, S)_3(O, OH, F)_{13}$, where $x > 4$, and to compare this with the formulas of known silicate minerals rich in rare earths and thorium in the search for the primary material (see compilations by Ewing and Chakoumakos, 1982; Fleischer, 1983; Clark, 1984). Crystalline species with suitable cation distributions include boron-bearing analogs of britholite ((Ce, Ca)₅(SiO₄, PO₄)₃(OH, F)) and hypothetical boron-rich members of the cerite ((Ce, Ca)₉(Mg, Fe)Si₇(O, OH, F)₂₈) and mosandrite-gotzenite ((Na, Ca, Ce)₃Ti(Si₂O₇)(O, OH, F)₂-(Ca, Na)₇(Ti, Al)₂Si₅O₁₅(F, OH)₃) groups. Other suggested minerals such as hellandite and datolite are less probable as precursors for the present material, due to the higher B/Si ratios of these borosilicates. The high fluorine and (by inference) hydroxyl contents of phase 1 indicate that these components must have substituted in excess of one atom per three tetrahedral cations in the precursor. This renders species which allow little or no fluorine substitution (such as allanite), or which only contain fluorine or hydroxyl in a restricted number of sites (such as britholite)

Table 3. Structural formulas of phases 1, 2 and 3 found in the freyalite-type specimen, and of melanocerite-group minerals from the Langesundfjord district.

	Phase 1	Phase 2	Phase 3	MELAN	TRITO	CARYO	TRITH
Si	2.734	2.795	2.849	1.895	1.475	1.704	2.292
P	0.239	0.193	0.151	0.159		0.095	
S	0.028	0.012					
B				0.799	1.374	1.066	0.549
Al				0.143	0.151	0.134	0.159
Ca	1.694	1.254	0.452	1.341	0.822	1.037	1.285
Mg				0.030		0.033	0.142
Fe	0.142	0.128	0.075	0.228	0.137	0.067	0.205
Mn				0.134	0.056	0.134	0.036
Na	0.358	0.157	0.033	0.408	0.296	0.362	
La	1.076	0.938	0.860	0.694	0.655	0.695	0.609
Ce	1.464	1.245	1.196	1.284	0.880	0.971	1.062
Nd	0.266	0.225	0.209	0.397	0.216	0.317	0.307 ¹
Y	0.250	0.147	0.170	0.709	0.172	0.154	0.598 ²
Zr	0.156	0.093	0.100	0.032	0.058	0.030	0.180
Th	0.620	0.518	0.571	0.055	0.236	0.409	0.543
Ta				0.145	0.035	0.111	
F	4.136	1.077	0.420	2.655	1.478	2.340	2.014

All structural formulas are calculated to a basis of 3,000 tetrahedral cations. The compositions of phases 1, 2 and 3 are recalculated on a boron-free basis from microprobe analyses given in Table 2. MELAN, TRITO and CARYO refer to minerals analysed by Brøgger (1890), the analyses are reproduced in Table 2. TRITH is a tritomite from an unknown locality in the Langesundfjord district analysed by Hogarth et al. (1973).

1: Includes Sm. 2: Includes Gd.

less probable as crystalline precursors for the material which was described as freyalite.

Nothing is known about the homogeneity of the melanocerite group minerals analyzed by Brøgger (1890). The compositions indicated by the wet-chemical analyses are similar enough to the present phase 1 to suggest a similar mode of formation. This can only be checked by a restudy of the old materials with techniques like those used in the present study.

Conclusions

From the new analytical data presented, it is concluded that the type material of freyalite is heterogeneous, consisting of thorian melanocerite (caryocerite) and alteration products. "Freyalite" should therefore be deleted from mineralogical terminology. It is not a variety of thorite (Brøgger, 1890; Fron del, 1958), or a Ce^4SiO_4 -component (Semenov, 1963), or an alteration product of thorite (Strunz, 1977). The Commission on New Minerals and Mineral Names of the International Mineralogical Association has approved the discreditation of "freyalite".

It may further be concluded, that speculations in the literature about substitution mechanisms of rare earths in thorite, and the possible existence of a $ThSiO_4$ - $CeSiO_4$ solid solution series, based on the old analysis (Fron del, 1958; Semenov, 1963) are not supported by the new data on the type material of "freyalite". This should be reinvestigated, using other, preferably nonmetamict material.

The crystalline precursor of the present material is not known with certainty, but may have been a boron-bearing member of the mosandrite-gotzenite or cerite groups. Boron-bearing britholite is less likely as a precursor. By inference, the other analyzed melanocerite-group minerals from the Langesundfjord district may have a similar origin.

The other, chemically distinct varieties of thorite described by Brøgger (1890) (eucasite and calciorthorite), should be reinvestigated in order to determine their true status.

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