

Gadolinite-(Ce) from Skien, southwestern Oslo region, Norway

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Abstract

Cerium-rich gadolinite containing the larger REE occurs in a syenite pegmatite near Skien, Norway. It shows REE zoning with a rim rich in Ce and poor in Y (25 weight percent Ce_2O_3 ; 2 weight percent Y_2O_3), relative to the core (15 weight percent Ce_2O_3 ; 14 weight percent Y_2O_3). X-ray studies show that the unit cell is slightly larger than for gadolinites-(Y). The chondrite-normalized REE pattern of the gadolinite-(Ce) shows strong enrichment of the larger REE relative to the smaller REE. This is believed to be related to the larvikite-derived pegmatitic liquids; it is not believed to have resulted through leaching of the basaltic country rock.

Introduction

Both homilite and gadolinite are isostructural with datolite, and the inadequately described mineral variety erdmannite (Brøgger, 1890) has an intermediate composition between homilite and gadolinite (Ito and Mori, 1953; Semenov *et al.*, 1963; Ito and Hafner, 1974). The general gadolinite formula can be written as $A_2BC_2Si_2O_{10}$ where $A = REE, Ca, Th, K$ in 8-fold coordination; $B = Fe^{2+}, Fe^{3+}, Mg, Mn$ in 6-fold coordination; $C = Be, B, Al$ in 4-fold coordination. REE is here used to mean the 15 elements La through Lu and yttrium.

Type gadolinite, $REE_2Fe^{2+}Be_2Si_2O_{10}$, can form solid-solution series involving the replacement of Fe^{2+} and REE^{3+} by other divalent and trivalent ions respectively (Ito, 1965; Ito and Hafner, 1974). A partial replacement of REE^{3+} by Ca^{2+} with valence compensation of Fe^{2+} will form calciogadolinite with the general formula $(REE_{2-x}Ca_x)(Fe^{2+}_{1-x}Fe^{3+}_x)Be_2Si_2O_{10}$ (Nakai, 1938; Ito, 1967; Ito and Hafner, 1974). A gadolinite-like mineral having vacant octahedral sites (\square) and stabilized with two OH^- with a gadolinite formula $REE_2\square Be_2Si_2O_8(OH)_2$ has been described as an unnamed mineral from Tuva, USSR (Semenov *et al.*, 1963; Ito and Hafner, 1974). Ito and Hafner (1974) defined gadolinite as belonging to a partial solid solution series in the type-gadolinite-calciogadolinite- $REE_2\square Be_2Si_2O_8(OH)_2$ three component system.

In naturally-occurring gadolinites, Y is usually the dominant element of those in 8-fold coordination. Gadolinites enriched in the larger rare-earth elements

have been reported by Eakins (1886) and Schetelig (1922). Goldschmidt and Thomassen (1924) included gadolinite among the so-called complex minerals showing a wide variation in the relative abundance of rare-earth elements, in contrast to minerals showing REE selection. The existence of cerium-rich gadolinite was questioned by Vainshtein *et al.* (1958), but Vainshtein *et al.* (1960) report cerium-rich gadolinites as accessory minerals in coarse-grained alaskites in the USSR. Only relative REE-distributions are given for these gadolinites, but approximately 16 weight percent Ce_2O_3 would be expected assuming a total of 50 weight percent REE-oxides in the gadolinites.

The terminology *cer-gadolinite* seems to originate from Schetelig's (1922) description of gadolinite from Fyresdal (formerly named Fyrrisdal), Norway. This contained a total of 23.40 weight percent of the larger REE, which was taken as a lower limit for cer-gadolinite by Tröger (1969). Following the nomenclature scheme for the rare-earth minerals proposed by Levinson (1966), only two gadolinite species names should be applied: gadolinite-(Y) and gadolinite-(Ce), each named after the predominant rare-earth element.

Preliminary chemical analyses of gadolinite from syenite pegmatite material near Skien, southwestern Oslo Region, showed it to be extremely cerium-rich (about 24 weight percent Ce_2O_3), and it was therefore subjected to a comprehensive study.

Occurrence

The Bjørkedalen valley (Fig. 1) is situated 9 km SE of Skien in the southwestern part of the Oslo Region.

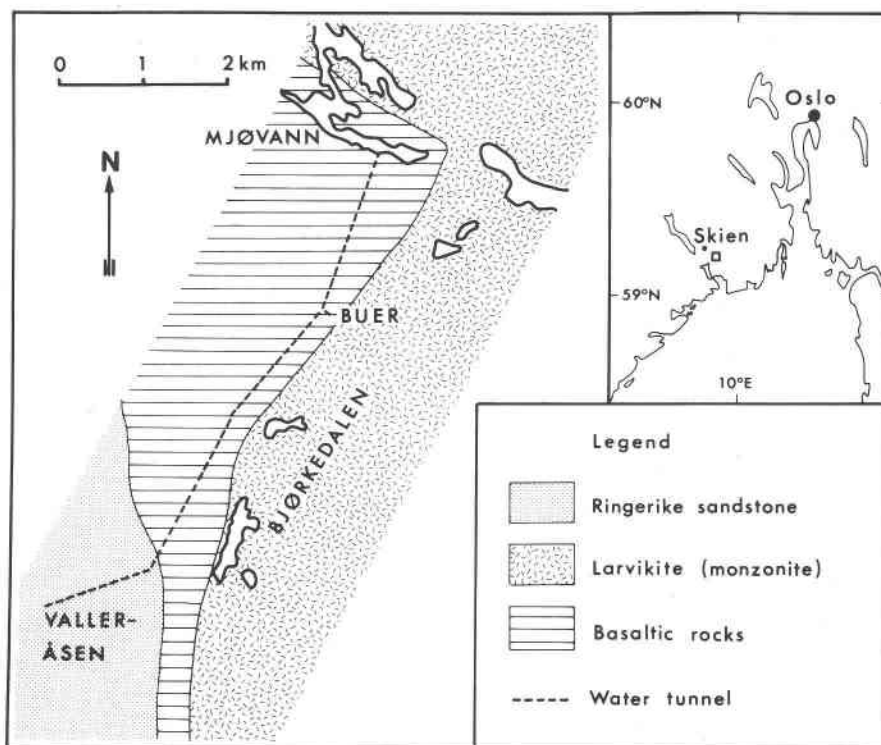


Fig. 1. Geological sketch map of the Bjørkedalen area, 9 km SE of Skien town.

The rare mineral occurrences in the syenite pegmatites along the Permian larvikite (monzonite) contact have become famous through the work by Brøgger (1890), who very briefly mentioned the pegmatites in "Birkedalen" (older name for Bjørkedalen). Bjørkedalen pegmatites lie along the same contact between basaltic rocks and larvikite as the famous Låven, Arø, and Stokkø island deposits in the Langesundsfjord, some 20 km to the south.

Syenite pegmatites occur in Bjørkedalen in a 200 m broad zone along the basaltic rocks/larvikite contact. Three types of pegmatites are observed: (1) pegmatites with predominant microcline and magnetite; (2) pegmatites with predominant microcline and nepheline; (3) pegmatites with predominant microcline and aegirine. In general, type 1 and type 2 appear as "dikes" oriented parallel to the contact, while type 3 only appears as vein fillings in thin (5–20 cm) cracks in the basaltic rocks and perpendicular to the contact between basaltic rocks and larvikite. Both homilite and erdmannite have earlier been described from the syenite pegmatites in the Langesundsfjord area (Brøgger, 1890). These have not been found in Bjørkedalen, but gadolinite occurs in the type 3 pegmatite.

The gadolinite-bearing syenite pegmatite material was found on the dumps near the outlet of a water

tunnel at Buer (see Fig. 1). The samples contain pegmatite-filled cracks which are characterized by long (up to 10 cm) aegirine crystals oriented perpendicular to the wall-rock. The order of crystallization has been determined by studies of the mineral paragenesis in hand specimens and in thin and polished sections. The primary sequence of crystallization in this pegmatite material is as follows [(1) means earliest, (5) last; minerals dominant listed in italics; minerals in subordinate amounts listed in parentheses]: (1) pyrochlore, *aegirine*, zircon, (apatite), (titanite); (2) pyrophanite, (magnetite); (3) loparite, chevkinite, gadolinite-(Ce), zircon; (4) biotite; (5) *microcline*. The later hydrothermal sequence of crystallization is: (1) (helvite), (galena), (molybdenite), *albite*; (2) (apophyllite), (quartz), (calcite).

The gadolinite is black, vitreous, and shows conchoidal fracture. It occurs as irregular masses with a maximum diameter of 20 mm. In thin section it is olive green, non-pleochroic, and isotropic (totally amorphous, metamict) due to a trace amount of Th, with $n = 1.78$. Specific gravity, determined using pycnometers filled with toluene at 20°C, is 4.20 g/cm³. The mineral is highly fractured and is surrounded by an orange to yellow thin (up to 0.5 mm) alteration rim.

Table 1. Chemical composition (weight percent oxides) and structural formulas (no. of atoms) of gadolinite-(Ce) from Bjørkedalen, Skien. Structural formula based on 10 oxygens.

	2 (rim)			3 (int.)			4 (core)	
SiO ₂	22.99	2.052		23.17	2.035		23.68	2.075
BeO	8.83*	1.893] 1.986	8.83	1.863] 1.951	8.83*	1.858
B ₂ O ₃	0.55*	0.085		0.55	0.083		0.55*	0.083
Al ₂ O ₃	0.07	0.008		0.05	0.005		0.04	0.004
TiO ₂	0.13	0.009] 0.839	0.14	0.009] 0.889	0.14	0.009
FeO	9.41	0.702		10.03	0.737		10.11	0.741
MnO	1.20	0.091		1.33	0.099		1.28	0.095
MgO	0.28	0.037] 2.152	0.33	0.044] 2.160	0.32	0.042
CaO	2.74	0.262		2.67	0.251		3.13	0.294
Na ₂ O	0.11	0.019		0.10	0.017		0.11	0.019
K ₂ O	0.53	0.060		0.53	0.060		0.54	0.060
Y ₂ O ₃	2.95	0.140		6.78	0.317		10.23	0.477
La ₂ O ₃	16.14	0.531		14.00	0.454		11.91	0.385
Ce ₂ O ₃	24.54	0.802		21.25	0.683		18.02	0.578
Pr ₂ O ₃	3.27	0.106		2.95	0.094		2.33	0.074
Nd ₂ O ₃	5.05	0.161		4.36	0.149		4.35	0.136
Sm ₂ O ₃	0.15	0.005		0.39	0.012		0.52	0.016
Eu ₂ O ₃	nd			0.07†	0.002		nd	
Gd ₂ O ₃	nd			0.92†	0.027		nd	
Tb ₂ O ₃	0.24	0.007		0.29	0.008		0.27	0.008
Dy ₂ O ₃	0.64	0.018		1.01	0.029		0.35	0.010
Ho ₂ O ₃	0.28	0.008		0.39	0.011		0.44	0.012
Er ₂ O ₃	0.44	0.013		0.76	0.021		1.06	0.029
Tm ₂ O ₃	0.08	0.002		0.13	0.004		0.17	0.005
Yb ₂ O ₃	0.61	0.016		0.65	0.017		0.91	0.024
Lu ₂ O ₃	0.08	0.002		0.13	0.004		0.05	0.001
Sum	101.31	7.029		102.18	7.035		100.34	7.035

Traces: Th, U, Zr, Sr, Ba. Not detected: Sc.

* Expected value assuming no zoning. † Value from mass spectrometer (see text). nd = not detected.

The basaltic wall-rock shows a thin (2–3 cm) re-crystallized border zone against the gadolinite-bearing pegmatite. This zone consists mainly of secondary anhedral aegirine and biotite, with quartz, microcline, plagioclase, and calcite in the groundmass. The Permian Skien basaltic rocks are silica-undersaturated ankaramites, nephelinites, and basanites (Segalstad, 1977). The basaltic rock at Buer has euhedral to subhedral strongly-zoned clinopyroxene phenocrysts up to 5 mm long, with plagioclase, biotite, magnetite, and ilmenite in the groundmass. At Buer the later Permian larvikite (monzonite) intrusive rock is characterized by coarse-grained, anhe-

dral, vein-perthitic ternary feldspar (mean diameter 5–15 mm), olivine pseudomorphs, subhedral aegirine-augite, anhedral reddish brown pleochroic hornblende, biotite, nepheline, apatite, magnetite, and ilmenite.

Chemical analysis

Handpicked unaltered gadolinite material was analyzed for beryllium by three methods. Optical spectrography by H. Bollingberg, Institut for Petrologi, Copenhagen, gave 9.7 weight percent BeO. Wet-chemical analysis by B. Bruun, Mineralogisk-Geologisk Museum, Oslo, gave 8.67 weight percent BeO.

Spectrophotometric analyses with chromazurol S by A. Åsheim, Porsgrunn Fabrikker, Porsgrunn, by a procedure described by Koch and Koch-Dedig (1974), gave 8.83 weight percent BeO. The last value has been adopted. The same gadolinite material was also analyzed for boron by two methods: Optical spectrography by H. Bollingberg gave 0.55 weight percent B₂O₃, which was also the mean of two colorimetric analyses with carmin by B. Bruun. Electron microprobe analyses on polished thin sections were made on an ARL-EMX microprobe at the Central Institute of Industrial Research, Oslo, using a series of natural and synthetic standards. Matrix corrections were made according to Bence and Albee (1968), using correction factors from Albee and Ray (1970) and Åmli and Griffin (1975). The rare-earth elements were analyzed by the procedure described by Åmli and Griffin (1975), using the same standards with the same accuracy as in their work. Each analysis represents the mean of four analyzed points. The analytical results are presented in Tables 1 and 2. In addition wet-chemical titration gave 9.8 weight percent FeO.

Owing to interference from the lightest REE, Eu and Gd could not be analyzed with the electron microprobe. The determination of the heavier REE with the microprobe is also subject to interference, firstly because of the high concentration of lighter REE (Åmli and Griffin, 1975), and secondly because of interference from the relatively high concentration of Mn. The mineral was therefore also analyzed by spark-source mass spectrometry by O. H. J. Christie, Massespektrometrisk Laboratorium, Universitetet i Oslo. The results obtained from different runs showed a considerable spread, due to the inhomogeneity of the REE distribution of the gadolinite (see discussion on zoning below). The determination of the heavier REE by mass spectrometer is also subject to interference from the high concentration of the lighter REE. The mass-spectrometer values listed in Table 1, column 2, have been calculated from their relative sensitivity factors using the known La₂O₃ concentration of point 3 (obtained by the electron microprobe analysis). This point has been assumed to represent the mean composition of the larger gadolinite grain. The mass-spectrometer values represent the mean of 6 runs.

Three different grains of gadolinite were analyzed on the electron microprobe with analyses 2, 3, and 4 (Table 1) representing the rim, intermediate zone, and core, respectively, of one large grain 16 mm long (Fig. 2). Analyses 13 and 16 (rims), and 14 and 17

Table 2. Chemical composition (weight percent oxides) of gadolinites-(Ce) from Bjørkedalen, Skien. Sums of the corresponding elements for analyses no. 2, 3, and 4 (Table 1) are 90.47, 89.76, and 88.89, respectively.

	13 (rim)	14 (core)	16 (rim)	17 (core)
SiO ₂	22.72	22.99	21.93	22.91
BeO	8.83*	8.83*	8.83*	8.83*
B ₂ O ₃	0.55*	0.55*	0.55*	0.55*
Al ₂ O ₃	0.03	0.02	0.04	0.04
TiO ₂	0.26	0.25	0.16	0.15
FeO	10.68	10.65	10.58	10.00
MnO	1.71	1.89	1.92	1.96
MgO	0.36	0.33	0.26	0.29
CaO	2.80	2.15	1.91	2.26
Na ₂ O	0.10	0.11	0.10	0.12
K ₂ O	0.51	0.52	0.57	0.54
Y ₂ O ₃	11.31	14.82	9.73	8.71
La ₂ O ₃	10.09	9.07	3.01	15.05
Ce ₂ O ₃	18.24	15.55	20.26	19.43
Sum	88.19	87.73	79.85	90.84

* Assumed value

(cores) (Table 2) are of two smaller grains, each 5 mm long. The complete REE distribution has been determined at the points 2, 3, and 4 in the larger gadolinite grain (Fig. 2). The structural formulas based on 10 oxygens (Table 1) have been calculated assuming no zoning in the BeO and B₂O₃ contents (Table 1, 2), and at the intermediate zone analysis (assumed average composition) using the Eu₂O₃ and Gd₂O₃ values from the mass-spectrometric analysis.

The analyses show that the abundances of all elements, except for the REE, are remarkably constant from grain to grain and within each grain. The REE, however, show a considerable inverse variation in the yttrium and the larger REE composition from core to rim. Y₂O₃ is higher in the cores than in the rims, whereas La₂O₃ and Ce₂O₃ are lower in the cores than in the rims. Eakins (1886) reported a maximum of 11.10 weight percent Ce₂O₃ and a minimum of 9.50 weight percent Y₂O₃ in one of the gadolinites from Colorado. The larger grain analyzed here has generally lower Y₂O₃ and higher La₂O₃+Ce₂O₃, respectively, than any gadolinite previously reported, as far as the authors are aware.

A step scan across the larger gadolinite grain with the microprobe (Fig. 2) shows that the core contains 14 weight percent Y₂O₃ and 15 weight percent Ce₂O₃, varying continuously down to 2 weight percent Y₂O₃

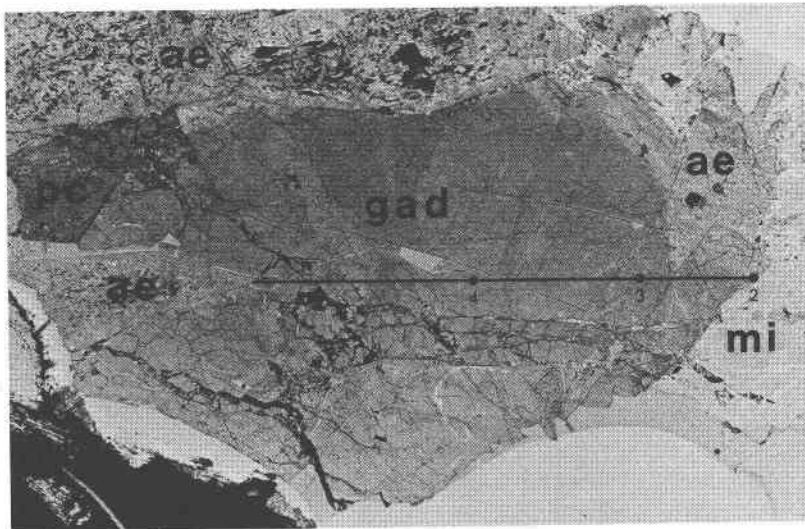
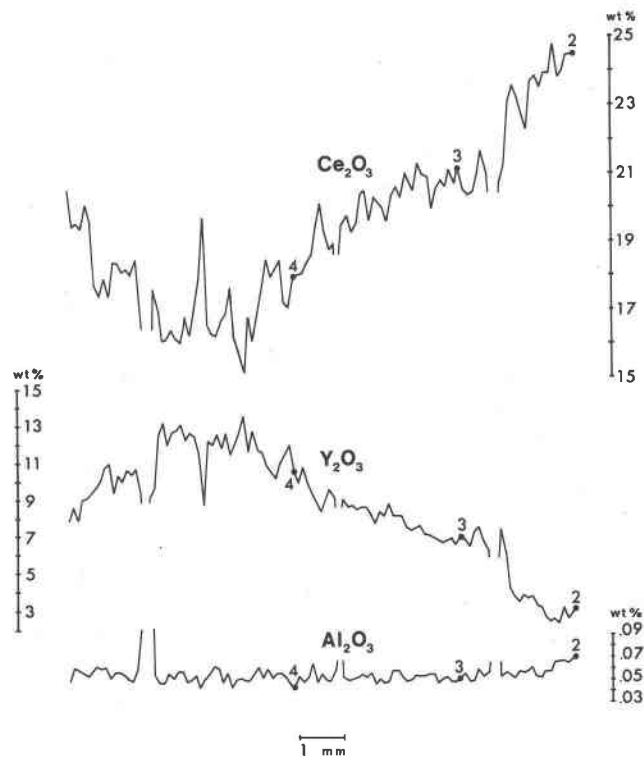


Fig. 2. Photomicrograph showing gadolinite-(Ce) from Bjørkedalen, Skien. The curves show the REE zoning in the gadolinite along the horizontal traverse, obtained by electron microprobe analysis in steps of 100 μm . ae = aegirine; gad = gadolinite-(Ce); mi = microcline feldspar; pc = pyrochlore.

and up to 25 weight percent Ce_2O_3 in the rim to the right. The large irregularities in the curves are due to cracks in the mineral.

X-ray crystallography

Unheated gadolinite-(Ce) gave no X-ray diffraction pattern, due to its metamict state. Heated in air,

some or all of the Ce ions are oxidized and expelled from the structure as CeO_2 , which can be identified on X-ray diffraction films. This is the γ -phase reported by Lima de Faria (1964) and Gibson and Ehlmann (1970). To achieve X-ray data for gadolinite-(Ce) the mineral was recrystallized in two ways: in nitrogen at 1000°C for 1 hour; and in a hydro-

thermal bomb at 700°C, 2 kbar, for 48 hours. d -values and cell dimensions have been calculated from X-ray powder diffraction films taken with a Guinier quadruple focussing camera using Fe-radiation, a quartz monochromator, and lead nitrate as internal standard. The diffraction films obtained were identical except for better resolution and sharper lines from the hydrothermally recrystallized sample. The cell dimensions are $a = 4.82 \pm 0.02$, $b = 7.58 \pm 0.02$, $c = 10.01 \pm 0.03$ Å, $\beta = 90^\circ 28' \pm 16'$, $V = 365.9$ Å³; calculated density 4.90 g/cm³.

Data for both the absolute REE abundances and the cell constants have been published for only a few gadolinites. The gadolinites reported from Siberia, USSR (Petrova *et al.*, 1963), Texas, USA (Gibson and Ehlmann, 1970), and Hundholmen, north Norway (Nilssen, 1973) (all after being heated to 1000–1100°C), as well as the synthetic gadolinites investigated by Ito (1965, 1967) and Ito and Hafner (1974) all display approximately the same cell constants: $a \approx 4.74$, $b \approx 7.49$, $c \approx 9.92$ Å, $\beta \approx 90^\circ (\pm 0.5^\circ$ depending on degree of heating), volume ≈ 352 Å³. The Skien gadolinite-(Ce), however, has higher a , b , c , β , and V values than these gadolinites. This must be due to the extraordinarily high contents of the larger REE, as was predicted by Ito (1965) from his experimental work.

Results of DTA

Differential thermal analysis was done on the metamict gadolinite-(Ce) using a Dupont 900 DTA analyzer at a heating rate of 10°C/min in air. The DTA diagram showed a broad and indistinct exotherm at about 250–350°C and a large exotherm at 790°C grading into a smaller exotherm at 810°C. This is in good agreement with the DTA data obtained by Gibson and Ehlmann (1970) on the gadolinite from Texas. Their reported endotherm at 760°C does not occur in our material.

Discussion

Plots of the chondrite-normalized lanthanide contents *vs.* ionic radius of Nilssen's (1973) gadolinite from Hundholmen, north Norway, shows a linear pattern from low La ratio ($\sim 1.5 \cdot 10^3$) to high Lu ratio ($\sim 3 \cdot 10^6$) with a distinct negative Eu-anomaly. The chondrite-normalized REE pattern of Gibson and Ehlmann's (1970) gadolinite from Rode Ranch, Texas, is also characterized by a negative Eu-anomaly, but has higher ratios for the larger REE and lower ratios for the smaller REE, displaying a maximum ratio around Tb at 1.12 Å ionic radius. The

Hundholmen gadolinite-(Y) shows a lanthanide distribution pattern with steadily increasing enrichment of the smaller lanthanides relative to chondritic abundances, whereas the Skien gadolinite-(Ce) shows the opposite pattern (Fig. 3), though also having a negative Eu anomaly from the apparent linear trend.

The problem of the origin of the rare minerals of the Norwegian pegmatites has been dealt with by earlier workers. Brøgger (1890) claimed that the mineralogy of the Langesundsfjord pegmatites was strongly dependent on resorption of the basic constituents of the "Augitporphyrit" (= the basaltic rocks) during the injection of the syenite-pegmatite magma. A resorption of wall-rock constituents has evidently taken place in some cases. In the Langesundsfjord area the nepheline-syenite pegmatites penetrating the basic basaltic rocks change to quartz-bearing granite pegmatites when penetrating the Ringerike sandstone (Brøgger, 1890; Dons, 1969). By the same token Goldschmidt (1934) maintained that Sc did not belong to the Evje-Iveland (southern Norway) pegmatite magma, but had been leached out of the surrounding gabbroic rocks, and later participated in forming the Sc-silicate thortveitite. Oftedahl (1960), however, considered the Langesundsfjord pegmatite dikes of magmatic origin, in the sense that they were formed by magmatic gases through leaching of the wall-rock and addition of some soda and rare heavy elements. Abundant aegirine and biotite in the basaltic rocks' border zone adjacent to the gadolinite-bearing pegmatites near Skien indicate that the wall-rocks have received alkalis from the pegmatite liquids. Calcium, however, seems to have been subjected to leaching and transport away from the basaltic border zone.

The 2 cm thick basaltic border zone was cut into slices oriented parallel to the border, crushed and pressed into pellets for X-ray fluorescence analysis, with international basaltic rock standards. This preliminary study shows a regular zoning in the basaltic border zone (from near the pegmatite and into the basaltic rock): 485 → 255 ppm La; 107 → 76 ppm Ce; 164 → 117 ppm Nd. A sample of a similar but unaltered basaltic rock from the same locality showed 45 ppm La, 66 ppm Ce, 84 ppm Nd. We therefore conclude that the movement of the larger REE during crystallization of the pegmatite has been from the dike into the basaltic wall-rock. This seems to indicate that the unusual REE contents of the Skien gadolinite-(Ce) has not originated through leaching of the basaltic country rock, but reflects, in some complex fashion, the REE distribution of the larvi-

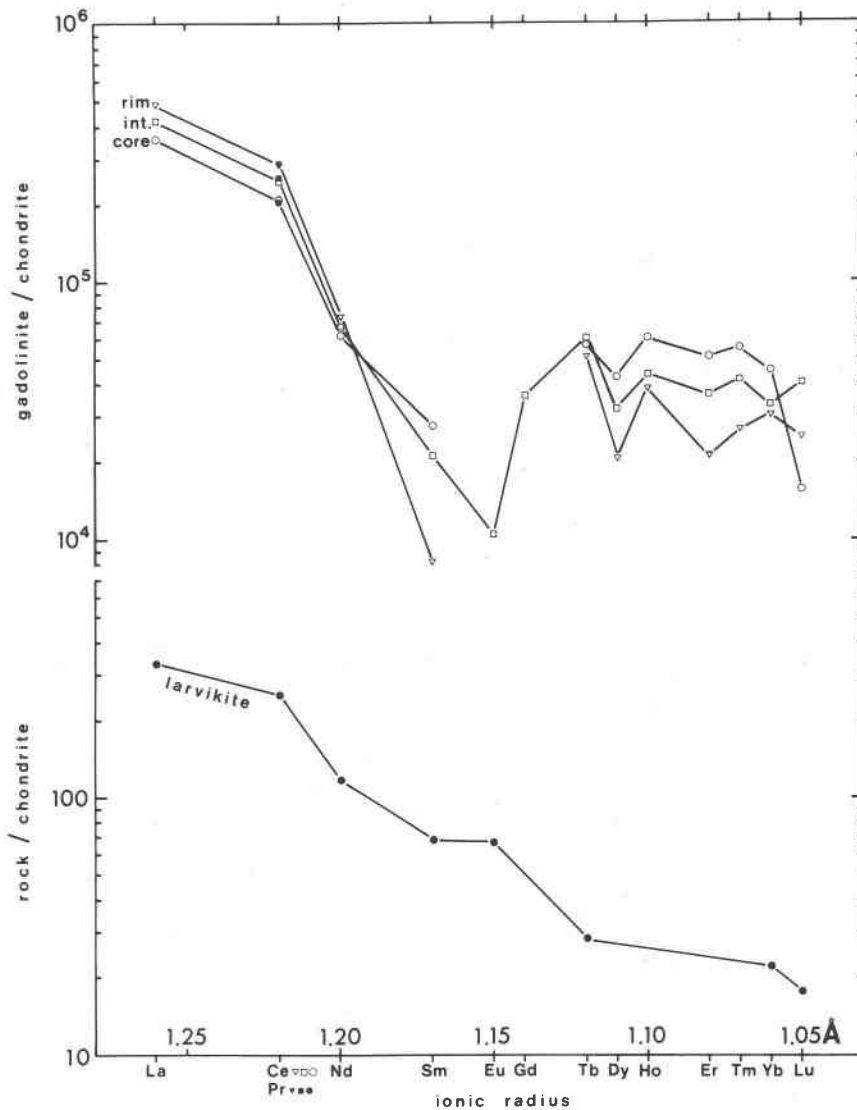


Fig. 3. Gadolinite-(Ce) and larvikite analyses from the Skien district relative to chondrite abundances (Haskin *et al.*, 1971) vs. ionic radius for trivalent ions in 8-fold coordination (Whittaker and Muntus, 1970). The larvikite analysis is from sample 358 of Neumann *et al.* (1977).

kite-derived pegmatitic liquids at its time of crystallization.

Keeping in mind that REE fractionation is likely to occur at several points between the larvikite magma stage and the late-stage crystallization of gadolinite in the pegmatite, the REE pattern of the larvikite sample no. 358 (Neumann *et al.*, 1977) collected 5 km SE of Bjørkedalen is in general (except for the medium-size REE) similar to the Skien gadolinite REE pattern (Fig. 3), when plotting chondrite-normalized REE concentrations vs. ionic radius.

Vainshtein *et al.* (1960) showed that the character

of the REE distribution in gadolinites strongly depends on the environment of their formation. We conclude that the Skien gadolinite-(Ce) has formed magmatically in pegmatites whose origins are closely related to the larvikite magma. The pronounced negative Eu anomaly can be explained by fractional crystallization of feldspar (in which Eu^{2+} substitutes for Ca^{2+} in plagioclase) in the larvikite magma before the pegmatite-forming stage. The observed REE zoning in the gadolinite must be due to changes in the REE of the environment brought about by (1) contemporaneous crystallization of another mineral showing

REE selectivity; (2) marked selectivity of gadolinite for the REE; or (3) selective loss of REE from the open pegmatite system during crystallization.

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