NIOBOPHYLLITE[1]—THE NIOBIUM ANALOGUE OF ASTROPHYLLITE; A NEW MINERAL FROM SEAL LAKE, LABRADOR

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ABSTRACT

Niobophyllite, the niobium analogue of astrophyllite, is described for the first time. A chemical analysis indicates the following composition: $(K,Na)_{2.95}(Fe,Mn)_{6.85}$ $(Nb,Ti)_{2.02}(Si,Al)_{7.70}(O,OH,F)_{31}$. Niobophyllite is triclinic, with space group $P1$ or $P\bar{1}$ and the following unit-cell dimensions: $a = 5.391$ Å, $b = 11.88$ Å, $c = 11.66$ Å, $\alpha = 113.1°$, $\beta = 94.5°$ and $\gamma = 103.1°$. The specific gravity is 3.42 (meas.) and 3.406 (calc.). Niobophyllite is biaxially negative with $2V = 60°$ and the following refractive indices: $\alpha = 1.724$, $\beta = 1.760$ and $\gamma = 1.772$. Z is normal to the plane of perfect cleavage, in contrast to astrophyllite, where X is normal to the cleavage.

INTRODUCTION

During an investigation of samples from the beryllium-bearing alkaline syenite in the Seal Lake area of Labrador, a brown micaceous mineral with an unusual combination of composition and optical properties was found to occur in some abundance. Further study showed that this mineral could be regarded as a new species, the niobium analogue of astrophyllite (Nickel, 1963). The name proposed for this mineral is niobophyllite—"niobo" for its niobium content and "-phyllite" for its micaceous nature.

The geology of the area has been interpreted as consisting of a series of interbedded volcanic rocks and gneisses intruded by alkaline syenite (Evans & Dujardin, 1961; Brummer & Mann, 1961). The niobophyllite was found within a band of paragneiss consisting chiefly of albite and arfvedsonite. Accessory minerals identified to date include aegirine-augite, barylite, eudidymite, neptunite, schizolite, pyrochlore, joaquinite, sphalerite, and galena.

In general, the mineralogy exhibits some similarity to that of certain bodies of alkaline syenite in other parts of the world, notably Langesund-fjord, Norway (Brögger, 1890), Narsarsuk, Greenland (Flink, 1901) and

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[1]Name approved by the I.M.A. Commission on New Minerals and Mineral Names.

the Kola Peninsula, U.S.S.R. (Vlasov, Kuz'menko & Yes'kova, 1959). The Seal Lake assemblage, however, contains several minerals that appear to make this deposit unique; much more work will be required before its mineralogy is fully known.

GENERAL DESCRIPTION

The niobophyllite occurs in the gneiss as sub-parallel, polycrystalline aggregates (Fig. 1), either intergrown with other dark minerals or as thin, discontinuous seams in the feldspar (Fig. 2). When the rock is broken it tends to split along the seams of niobophyllite, frequently resulting in surfaces coated predominantly with this mineral. These surfaces are chocolate-brown in colour and are somewhat slippery to the touch.

In thin-section, the mineral is strongly pleochroic from brownish-yellow (parallel to the flakes) to orange-red (normal to the flakes). Because of its dark colour and relatively high refractive indices, internal details of the niobophyllite bands are difficult to resolve unless strongly convergent light is used for illumination.
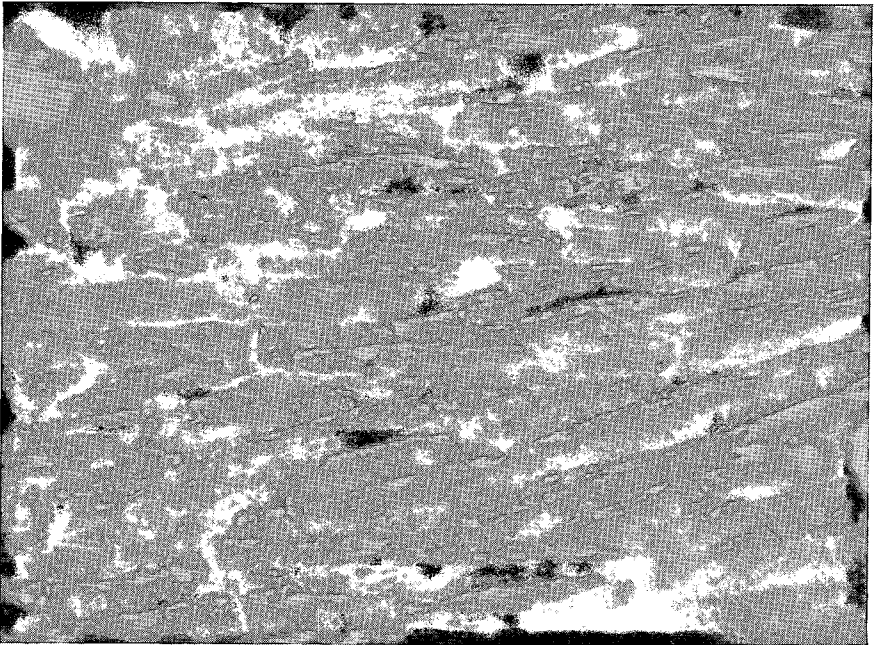

FIG. 1. Thin-section photomicrograph of a polycrystalline aggregate of niobophyllite grains in sub-parallel arrangement. 970✕.
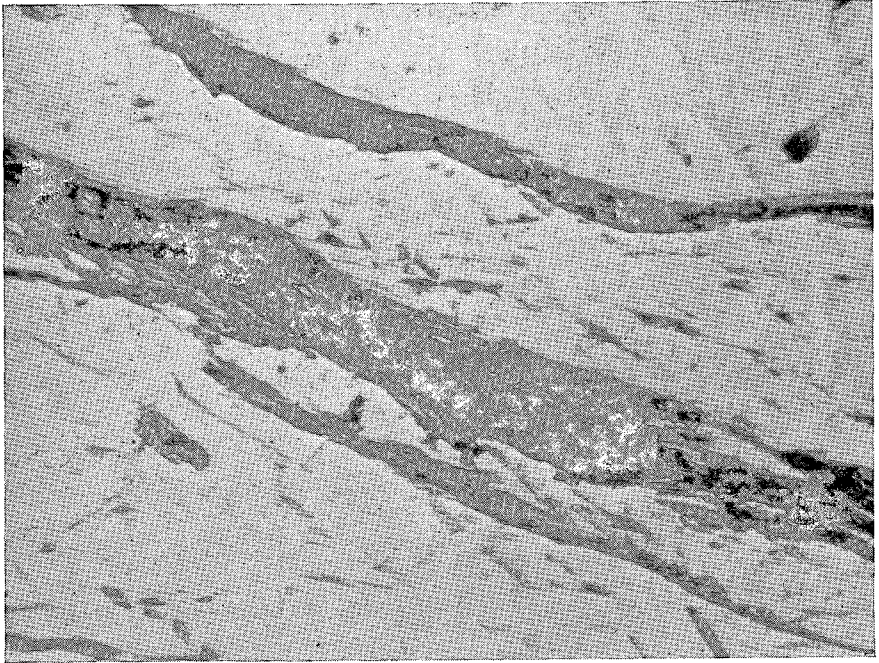
FIG. 2. Thin-section photomicrograph showing sinuous bands of niobophyllite (grey) in feldspar (white). 185×.

OPTICAL AND PHYSICAL PROPERTIES

Because of the fine-grained nature of niobophyllite, accurate determinations of optical and physical properties can be made only with difficulty.

Niobophyllite has one perfect cleavage parallel to two of the crystal axes. This plane, by analogy with the micas, is designated as the (001) plane. A secondary, ill-defined cleavage can also be observed in some (001) cleavage flakes. The trace of this cleavage is parallel to the a-axis, and it can thus be defined as an (0kl) cleavage.

The specific gravity, determined by the suspension of individual flakes in Clerici solution, was found to be 3.42. The calculated specific gravity is 3.406.

The optical determinations were made on several tiny flakes that appeared to be monocrystalline; some of these, however, on subsequent investigation by single-crystal x-ray diffraction methods, were found to be twinned on (001). The twinning cannot be detected optically because the twin members are related by reflection on the (001) plane which

contains two principal vibration directions and is, therefore, an optic symmetry plane. The resultant optical effect is thus exactly as if there were no twinning, and the optical determinations made on the twinned crystals are equally valid for single crystals.

A single (or twinned) flake of niobophyllite gives a centred, obtuse bisectrix interference figure of negative sign. Consequently, the optical directions X and Y lie in the cleavage plane and the refractive indices α and β can be measured readily on flakes lying flat on a microscope slide. The Z direction is normal to the flake and the refractive index γ is therefore much more difficult to measure. Attempts to measure γ according to the Universal Stage method suggested by Ferguson & Peacock (1943), were unsuccessful because the difference between β and γ is not sufficiently great. Efforts were also made at measuring γ in flakes mounted on edge, but no accurate results could be obtained. The value given in Table 1 was obtained by calculation from the other refractive indices and the value of $2V$.

TABLE 1. OPTICAL AND PHYSICAL PROPERTIES OF NIOBOPHYLLITE,
ASTROPHYLLITE AND KUPLETSKITE

	Niobophyllite (This paper)	Astrophyllite *	Kupletskite (Semenov, 1956)
Refractive indices			
α	1.724	1.676–1.691	1.656 (calc.)
β	1.760	1.703–1.726	1.699
γ	1.772 (calc.)	1.731–1.758	1.731
Optic Angle $2V$	$-60°$	$+70°$ to $+88°$	$-70°$
Optic Orientation	$X \wedge b = +13°$	$X \perp (001)$	
	$Y = a$	$Y \wedge b = +13°$	
	$Z \perp (001)$	$Z = a$	
Elongation	negative	positive	positive
Pleochroism			
X	brownish-yellow	reddish-orange	orange-yellow
Y	brownish-yellow	yellow	
Z	orange-red	lemon-yellow	brown
Specific Gravity	3.42	3.3–3.4	3.20–3.23
Cleavage	(001) perfect	(001) perfect	(001) perfect

*The astrophyllite data are taken from Floor (1961) and Winchell & Winchell (1951), except for the optic orientation, which was determined by the present authors on astrophyllite from El Paso County, Colorado. Winchell & Winchell (1951) give optical data for one astrophyllite with much higher refractive indices than the others; these have not been included here because the reference given is apparently in error, and cannot be substantiated.

Attempts were made to measure the optic angle $2V$ on the universal stage, both on horizontal flakes and on those mounted vertically, but the optic axes could not be located in either position—in horizontal flakes because the obtuse angle $(180°-2V)$ is too large, and in vertical flakes because the extinction positions are masked by optical aberrations pro-

duced by the large vertical surfaces. A value for $2V$ was finally obtained by means of the spindle stage following the procedure suggested by Tocher (1962).

The results of the optical and physical determinations are summarized in Table 1. The corresponding data for astrophyllite and kupletskite, the manganese-rich variety of astrophyllite, are given for comparison.

The refractive indices of niobophyllite are much higher than those of either astrophyllite or kupletskite. The pleochroism exhibited by astrophyllite and niobophyllite is different relative to their optical directions, although similar when referred to the cleavage, i.e. brownish-yellow parallel to the cleavage and orange-red normal to it.

The most striking difference between niobophyllite and the other two minerals is in optic orientation (Fig. 3). In astrophyllite, X is normal to
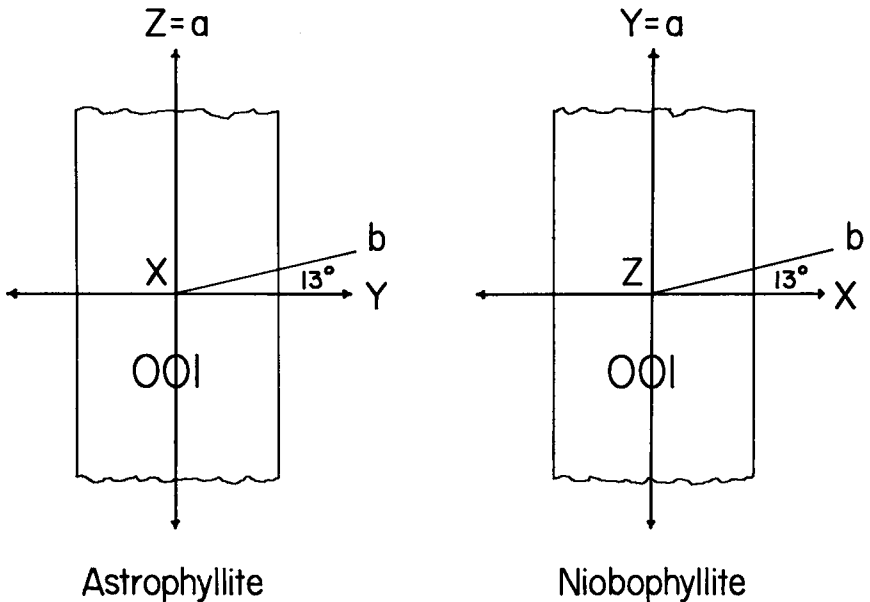

FIG. 3. Optic orientation of astrophyllite and niobophyllite.

the cleavage and the mineral consequently exhibits positive elongation (length-slow). Since kupletskite also has positive elongation, it can be assumed to have the same orientation as astrophyllite. In niobophyllite, however, Z is normal to the cleavage, resulting in negative elongation (length-fast). The niobophyllite orientation is abnormal, in that practically all phyllosilicates have their lowest refractive index in the direction normal to the cleavage. This anomalous orientation has also been noted

in certain chlorites, but no satisfactory structural reason has yet been given for this phenomenon.

CHEMICAL COMPOSITION

Because the niobophyllite is extremely fine-grained and is intergrown with other minerals, a concentrate pure enough for chemical analysis could not be obtained by the usual methods of concentration, including heavy-liquid and magnetic separations. Consequently, sufficient niobophyllite for analysis (600 mg.) had to be laboriously hand-picked under a stereomicroscope.

The results of the chemical analysis are shown in Table 2, with analyses of the related minerals, astrophyllite and kupletskite, included for com-

TABLE 2. CHEMICAL ANALYSES OF NIOBOPHYLLITE,
ASTROPHYLLITE AND KUPLETSKITE

	Niobophyllite, Seal Lake, Canada (1)	Astrophyllite, El Paso Co., Colorado, U.S.A. (2)	Astrophyllite, Langesundfjord, Norway (3)	Kupletskite, Kola Pen., U.S.S.R. (4)
Na_2O	2.49%	2.54%	3.18%	2.14%
K_2O	5.51	5.01	6.12	5.00
CaO	0.72	—	1.16	2.52
BaO	—	—	—	0.16
R.E. Oxides	1.50*	—	—	—
FeO $\rangle$ $Fe_2O_3 \rangle$	23.74	26.10 6.56	16.58 $\rangle$ 4.36 $\rangle$	9.29
MgO	0.16	0.30	0.63	2.30
MnO	9.83	3.48	15.18	25.62
CuO	—	0.42	—	—
TiO_2	2.94	13.58	8.02	11.34
Nb_2O_5	14.76	—	—	1.57
Ta_2O_5	0.52	0.80(?)	—	—
ZrO_2	—	2.20	5.34	0.59
Al_2O_3	0.89	0.70	1.72	1.34
SiO_2	33.40	34.68	33.81	33.07
H_2O^+	3.64	3.54	3.43	3.86
H_2O^-	0.08	—	—	0.94
F	0.46	—	0.70	0.61
Total	100.64	99.91	100.23	100.35
$-O \equiv F$	0.19		0.30	0.26
	100.45		99.93	100.09

(1) This paper; D. J. Charette, analyst. Alkali determinations by Miss E. M. Penner.
(2) König (1877).
(3) Gossner & Reindl (1934); average of two analyses.
(4) Semenov (1956); average of two analyzed samples.
*A qualitative spectrographic analysis indicates that cerium and lanthanum oxides are the predominant rare earths present.

parison. The analyses of the three minerals are similar with respect to alkalis, alumina, silica and water. However, there are major differences

in the amounts of iron, titanium, manganese and niobium oxides present, niobophyllite being relatively high in iron and niobium, the El Paso astrophyllite in iron and titanium, and kupletskite in manganese and titanium. The Langesundfjord astrophyllite, by virtue of its Fe:Mn ratio, appears to occupy an intermediate position between the El Paso astrophyllite and the kupletskite.

X-RAY CRYSTALLOGRAPHY

The crystals of niobophyllite that were examined by x-ray diffraction consisted of small cleavage fragments chosen from the hand-picked concentrate used for the chemical analysis. From optical examinations, these fragments appeared to be single crystals, and where the x-ray diffraction work confirmed this to be so, the same crystal was used to obtain both optical and crystallographic information. In many fragments, however, twinning was present, although in a manner that did not affect the extinction positions. In others, the x-ray diffraction work indicated the presence of parallel growth, and these fragments were discarded.

The single-crystal cleavage fragments were examined using a Buerger precession camera with unfiltered CuK or MoK radiations. Films were made with the crystals mounted on the goniometer head in various orientations, and the diffractions obtained were interpreted as representing a lattice with triclinic symmetry. The reported unit-cell dimensions represent composite values obtained both by direct measurement of films for some of the crystal orientations, and also by calculation from information provided by films for other orientations.

Since the single-crystal x-ray diffraction work indicates that niobophyllite is triclinic, any one of several possible unit cells could be used to define the lattice. However, a primitive cell with three obtuse angles appears to be the logical choice. The axes are named so that the plane of perfect cleavage is (001), thereby following the convention employed for other micaceous silicates. The space group is indeterminate, either of the triclinic space groups, $P1 = C_1^1$ and $P\bar{1} = C_i^1$, being possible. The dimensions of the unit cell, in direct and reciprocal space are as follows:

Direct		Reciprocal	
$a = 5.391\text{Å},$	$\alpha = 113.1°,$	$a^* = 0.19383,$	$\alpha^* = 65.0°,$
$b = 11.88\text{Å},$	$\beta = 94.5°,$	$b^* = 0.09534,$	$\beta^* = 79.2°,$
$c = 11.66\text{Å},$	$\gamma = 103.1°.$	$c^* = 0.09492,$	$\gamma^* = 73.7°.$

The axial ratios of the direct cell are $a:b:c = 0.454:1:0.981$, and the cell volume is 657.3 Å³. The twinning present in many of the fragments conforms to the law: twin plane = (001).

Previous workers investigating the crystallography of astrophyllite chose face-centred unit cells (Gossner & Reindl, 1934; Woodrow, 1963). Although a centred lattice is superfluous in the triclinic system since it can always be reduced to a primitive lattice, the primitive cell was transformed to an A-face-centred cell for purposes of comparing the niobophyllite unit-cell dimensions with those of astrophyllite. The index transformation is $h_A = h_P$, $k_A = -h_P-k_P$ and $l_A = h_P + k_P + 2l_P$ (where the subscripts A and P refer to the face-centred and primitive cells respectively). The new cell dimensions are shown in Table 3. A more detailed account of the crystallographic investigation of the El Paso astrophyllite will be given in another paper.

TABLE 3. UNIT-CELL DIMENSIONS FOR NIOBOPHYLLITE AND
ASTROPHYLLITE, REFERRED TO FACE-CENTRED CELLS

	Niobophyllite, Seal Lake, Labrador (this paper)	Astrophyllite, El Paso Co., Colorado (this paper)	(Woodrow, 1963)	Astrophyllite, Langesundfjord, Norway (Gossner & Reindl, 1934)	
a	5.391 Å	5.368 Å	5.36 Å	5.41 Å	(b)
b	11.88 Å	11.86 Å	11.76 Å	11.72 Å	(a)
c	21.16 Å	21.15 Å	21.08 Å	21.14 Å	(c)
α	95.2°	95.1°	94.9°	$\sim$94°	(β)
β	87.7°	87.6°	90.0°	$\sim$90°	(α)
γ	103.2°	103.2°	103.2°	$\sim$90°	(γ)

The powder diffraction pattern that was obtained using a 114.6 mm diameter Debye-Scherrer camera with filtered $CoK\alpha$ radiation, was readily indexed from the single-crystal data. The calculated spacing for each (hkl) was obtained from the reciprocal lattice dimensions using the following formula:

$$1/d^2 = h^2A + k^2B + l^2C + klD + lhE + hkF, \text{ where}$$
$$A = a^{*2} = 0.03758, \qquad D = 2b^*c^* \cos \alpha^* = 0.00765,$$
$$B = b^{*2} = 0.00909, \qquad E = 2c^*a^* \cos \beta^* = 0.00687,$$
$$C = c^{*2} = 0.00901, \qquad F = 2a^*b^* \cos \gamma^* = 0.01037.$$

Since the triclinic cell is primitive, there are no extinction rules, and all (hkl) spacings were calculated for the range of values to be indexed. The powder diffraction data are given in Table 4. Only those (hkl) values required to index the observed diffractions are listed, and they correspond in all cases to the more intense diffractions of the single-crystal films.

The molecular weight of niobophyllite calculated from the measured specific gravity of 3.42 and the unit-cell volume of 657.3 Å^3 is 1354.0. Calculation of the atomic proportions, using this value and the chemical analysis shown in Table 2, yields a cell containing a total of about 31

atoms of oxygen, hydroxyl and fluorine. This agrees with the formula given for astrophyllite by Woodrow (1963). Recalculating the atomic

TABLE 4. NIOBOPHYLLITE: X-RAY POWDER DIFFRACTION PATTERN

Int. (est.)	d (meas.) (Å)	d (calc.) (Å)	(hkl)
9	10.52	10.54	001
		10.49	$01\bar{0}$
3	9.68	9.78	$0\bar{1}1$
$\frac{1}{2}$	6.15	6.23	$01\bar{1}$
2	5.76	5.77	$0\bar{2}1$
1	5.26	5.249	$\bar{1}10$
		5.244	020
$\frac{1}{2}$	5.15	5.159	100
1	4.696	4.739	$1\bar{1}1$
		4.658	$\bar{1}11$
3 br	4.42	4.405	$11\bar{1}$
	4.363	4.336	$\bar{1}20$
		4.325	101
		4.312	$12\bar{1}$
$\frac{1}{2}$	4.20	4.188	110
3 br	4.059	4.087	$\bar{1}02$
		4.060	021
$\frac{1}{2}$	3.87	3.857	$0\bar{1}3$
4 br	3.741	3.762	$\bar{1}21$
		3.756	$11\bar{2}$
		3.738	$0\bar{2}3$
10 br	3.506	3.523	111
		3.512	003
		3.509	$13\bar{1}$
		3.502	$121\bar{}$
		3.496	030
$\frac{1}{2}$	3.40	3.402	$12\bar{2}$
		3.383	102

Int. (est.)	d (meas.) (Å)	d (calc.) (Å)	(hkl)
5	3.258	3.261	$0\bar{3}3$
$\frac{1}{2}$	3.12	3.116	$02\bar{2}$
1	3.071	3.070	$\bar{1}22$
6	3.019	3.029	$12\bar{3}$
		3.025	$12\bar{3}$
1	2.977	2.973	$01\bar{3}$
		2.970	$\bar{1}31$
		2.965	031
3 br	2.859	2.883	$0\bar{4}2$
		2.873	$13\bar{3}$
		2.862	112
		2.855	$0\bar{1}4$
		2.842	041
$\frac{1}{2}$	2.82	2.819	121
		2.808	$14\bar{1}$
8	2.778	2.777	$13\bar{1}$
		2.772	$14\bar{2}$
$\frac{1}{2}$	2.72	2.730	$03\bar{4}$
		2.724	$04\bar{3}$
		2.686	$\bar{2}10$
1	2.662	2.658	$\bar{1}40$
		2.658	$\bar{2}11$
3	2.636	2.643	$11\bar{4}$
		2.634	004
		2.626	$\bar{2}20$
7	2.574	2.579	200
		2.578	130
		2.570	$14\bar{3}$
4	2.475	2.480	$\bar{2}12$
		2.474	023

proportions to give exactly 31 anions, assuming all the iron to be divalent and calculating all the rare-earth oxides as Ce_2O_3, results in the proportions shown in Table 5. The atomic proportions of astrophyllite and kupletskite, also calculated on the basis of 31 anions, are included for comparison. The calculated molecular weight for this niobophyllite unit cell is 1348.6, and the calculated specific gravity is 3.406. The chemical composition of the unit cell can be expressed by the following approximate formula:
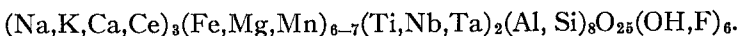
$$(Na,K,Ca,Ce)_3(Fe,Mg,Mn)_{6-7}(Ti,Nb,Ta)_2(Al, Si)_8O_{25}(OH,F)_6.$$

TABLE 5. UNIT-CELL CONTENTS OF NIOBOPHYLLITE, ASTROPHYLLITE AND KUPLETSKITE, BASED ON 31 (O, OH, F) ATOMS PER UNIT CELL

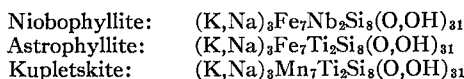| Group	Element	Niobophyllite, Seal Lake, Canada		Astrophyllite, El Paso, Col., U.S.A.		Kupletskite, Kola Pen., U.S.S.R.	
A	Na	1.079		1.059		0.880	
	K	1.572		1.375		1.353	
	Ca	0.173	2.947	—	2.434	0.572	2.818
	Ba	—		—		0.013	
	R.E.	0.123		—		—	
B	Fe″ Fe‴	4.439		4.695 1.062		1.647	
	Mg	0.053	6.354	0.096	6.555	0.727	6.976
	Mn	1.862		0.634		4.602	
	Cu	—		0.068		—	
C	Ti	0.494		2.197		1.808	
	Nb	1.492	2.018	—	2.475	0.150	2.019
	Ta	0.032		0.047		—	
	Zr	—		0.231		0.061	
D	Al	0.235	7.702	0.177	7.636	0.335	7.347
	Si	7.467		7.459		7.012	
X	OH	5.429		5.079		5.460	
	F	0.325	31.000	—	31.000	0.409	31.000
	O	25.246		25.921		25.131	
Mol. Wt.		1348.6		1291.4		1275.2	
Calc. S.G.		3.406		3.376		3.244*	
Meas. S.G.		3.42		3.375		3.215	

*Calculated from the unit-cell parameters of astrophyllite.

DISCUSSION

The structure of astrophyllite has recently been described by Woodrow (1963). According to Woodrow, the basic unit of the astrophyllite structure consists of a continuous sheet of $(Fe,Mn)O_6$ octahedra sandwiched between two sheets composed of chains of SiO_4 tetrahedra joined by TiO_6 octahedra. These multiple sheets are separated by alkali ions. The unit-cell formula is $K_3Fe_7Ti_2Si_8(O,OH)_{31}$. Because of the similarity of the unit-cell parameters of niobophyllite and astrophyllite, the structures of the two minerals are assumed to be similar, with niobium in niobophyllite replacing the titanium in astrophyllite. The increased charge resulting from the substitution of Nb^{+5} for Ti^{+4} may be balanced, in part, by coupled substitutions (e.g. Al^{+3} for Si^{+4}) but, judging from the major deviations from stoichiometry, is more likely to be balanced by cation vacancies.

The unit-cell formulae shown in Table 5 all exhibit appreciable deviations from the ideal $A_3B_7C_2D_8X_{31}$ formula, with deficiencies in the A, B and D groups and an excess in the C group of atoms. Some improvement is achieved if the excess of C atoms over 2 is allotted in the B group. Even with this expedient, an appreciable deficiency remains in the A group for astrophyllite and kupletskite, in the B group for niobophyllite, and in the D group for all three minerals. A further improvement might be achieved if some of the analyzed water, instead of being allocated entirely to $(OH)^-$, is calculated as the oxonium ion $(H_3O)^+$, and allocated to the A group. However, non-stoichiometry is common in some other phyllosilicates, such as biotite, and may be a characteristic feature of minerals in the astrophyllite group, as well.

Disregarding deviations from the stoichiometric formulae, the generalized compositions of the three mineral end-members can be given as follows:

Niobophyllite: $(K,Na)_3Fe_7Nb_2Si_8(O,OH)_{31}$
Astrophyllite: $(K,Na)_3Fe_7Ti_2Si_8(O,OH)_{31}$
Kupletskite: $(K,Na)_3Mn_7Ti_2Si_8(O,OH)_{31}$

A compilation of the $Mn:(Mn + Fe)$ and $Nb:(Nb + Ti)$ percentage ratios in minerals of the astrophyllite type is given in Table 6.

TABLE 6. PERCENTAGE RATIOS OF $Mn:(Mn + Fe)$ AND $Nb:(Nb + Ti)$ IN MINERALS OF THE ASTROPHYLLITE TYPE

Name and Locality	$\dfrac{Mn \times 100}{Mn + Fe}$	$\dfrac{Nb \times 100}{Nb + Ti}$	References
Astrophyllite, Keivy, U.S.S.R.	3.9	—	Semenov, 1956
Astrophyllite, Naujakasik, Greenland	7.7	—	Kunitz, 1936
Astrophyllite, El Paso Co., Colorado, U.S.A.	9.8	—	König, 1877
Astrophyllite, Khibiny, U.S.S.R.	18.5	—	Semenov, 1956
Astrophyllite, Kola Pen., U.S.S.R.	22.3	—	Kunitz, 1936
Niobophyllite, Seal Lake, Canada	29.2	85.4	This paper
Astrophyllite, Brevik, Norway	29.4	—	Kunitz, 1936
Astrophyllite, Eikaholm, Norway	33.1	—	Brögger, 1890
Astrophyllite, Iles de Los, Guinea	35.3	—	Kunitz, 1936
Astrophyllite, Langesundfjord, Norway	42.4	—	Gossner & Reindl, 1934
Kupletskite, Nepkha Mtn., Lovozero, U.S.S.R.	65.5	21.4	Semenov, 1956
Kupletskite, Kuyvchorr Mtn., Lovozero, U.S.S.R.	83.5	6.0	Semenov, 1956

The values shown in Table 6 suggest that there is probably a complete solid-solution series between astrophyllite and kupletskite, since there appears to be a continuous gradation in the $Mn:(Mn + Fe)$ percentage ratios. The evidence for complete solid solution between niobophyllite and the other two minerals is less clear, since only two niobium values have been reported previously. Consequently, more mineral occurrences that have been suitably analyzed are required before any conclusions as to the isomorphism of niobophyllite and astrophyllite can safely be drawn.

ACKNOWLEDGMENTS

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