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Nafertisite, a layer titanosilicate member of a polysomatic series including mica

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Abstract: Nafertisite, $(\text{Na,K})_3(\text{Fe}^{2+}, \text{Fe}^{3+}, \square)_{10}[\text{Ti}_2(\text{Si}, \text{Fe}^{3+}, \text{Al})_{12}\text{O}_{37}](\text{OH}, \text{O})_6$, is an alkaline titanosilicate from the Khibina massif (Kola Peninsula, Russia). It is monoclinic $A2/m$ with $a = 5.353(4)$, $b = 16.176(12)$, $c = 21.95(2)$ Å, $\beta = 94.6(2)^\circ$, $Z = 2$.

The comparison with bafertisite, $\text{Ba}_2(\text{Fe}, \text{Mn})_4[\text{Ti}_2\text{Si}_4\text{O}_{17}](\text{OH}, \text{O})_3$, and astrophyllite, $(\text{K}, \text{Na})_3(\text{Fe}, \text{Mn})_7[\text{Ti}_2\text{Si}_8\text{O}_{27}](\text{OH}, \text{O})_4$, allowed to obtain a structural model for nafertisite which has been tested against X-ray diffraction data obtained from a very poor crystal. On the basis of a bafertisite-like B module $(\text{A}, \square)_2(\text{M}, \square)_4[\text{X}_2\text{T}_4\text{O}_{17}](\text{OH})_2$ and a mica-like M module $(\text{A}, \square)(\text{M}, \square)_3[\text{T}_4\text{O}_{10}](\text{OH})_2$, a polysomatic series BM_n can be defined where bafertisite, astrophyllite, nafertisite and mica are the members with $n = 0, 1, 2, \infty$, respectively. It is called *heterophyllosilicate series* because the crystal structures of the members consist of 2:1 HOH layers where O is an octahedral sheet and H is a tetrahedral-like sheet which differs from the T sheet of the phyllosilicates for the insertion of Ti octahedra; these play a role similar to that of Si tetrahedra. A group of titanosilicates is shown to be derivatives of the bafertisite structure with different interlayer contents.

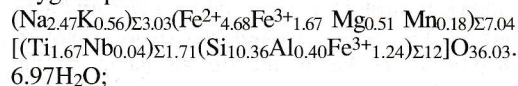
Key-words: nafertisite, crystal structure, polysomatic series, titanosilicates, phyllosilicates.

Introduction

About 500 different minerals occur in the hyperalpine rocks of the Khibina massif (Kola Peninsula, Russia) including more than 100 new species (Khomyakov, 1995). Nafertisite is an alkaline titanosilicate from this massif recently described by Khomyakov *et al.* (1995) (see also Ferraris *et al.*, 1994).

Dark brown-green [100] fibrous aggregates of

nafertisite have been found in a drill core from an alkaline pegmatite of Mount Kukisvumchorr (SW of Khibina massif) with calcite, aegirine and hewaldite. Khomyakov *et al.* (1995) report the following empirical formula on the basis of 43 oxygens pfu



the presence of both Fe^{2+} and Fe^{3+} is proved by

0935-1221/96/0008-0241 \$ 2.25

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Mössbauer spectroscopy and wet chemical analyses. In Khomyakov (1995) nafertisite is mentioned under the code M71.

Experimental

Scanning (SEM) and transmission (TEM) electron microscopy of nafertisite showed that its millimetric macroscopic fibres consist of thin and narrow ($< 20 \mu\text{m}$) $\{001\}$ cleavage laths which are $[100]$ elongated. In TEM images some laths appear rolled around this direction (Fig. 1a). With the beam perpendicular to the plane laths, $hk0$ selected area electron diffraction (SAED) patterns are easily obtained, whereas poor and deformed $h0l$ patterns are obtained from rolled laths with the $[010]$ direction close to the incident beam. Strong and weak streaks are present along the $[001]$ and $[010]$ directions, in the order (Fig. 1b, c). X-ray diffractograms show extended disorder around the $[100]$ fiber axis.

Examination by an Italstructure 4-circle single-crystal diffractometer (MoK α radiation; graphite monochromator; 55 mA, 50 kV) of a very small crystal ($0.72 \times 0.02 \times 0.03 \text{ mm}$) allowed to collect diffraction intensities up to $\theta = 20^\circ$ ($4401 \pm h \pm k \pm l$ reflections in the P lattice; $\theta/2\theta$ scan; θ range 1.4° ; background to scan time ratio = 0.5; scan speed variable with intensity between 1 and $12^\circ \text{ min}^{-1}$; 1 standard reflection measured every 100 reflections). The quality of the intensities was poor because of both the small dimensions of the crystal, which caused low signal to background ratio, and some disorder of the fibres around $[100]$ and of the structure (see below), which caused considerable broadening of the peaks. Altogether only 491 independent reflections ($R_{\text{merge}} = 0.15$) have $|F_o| > 2\sigma(|F_o|)$ in the space group $A2/m$ which was finally adopted for nafertisite (see below), in spite of the presence of 22 weak reflections with $k + l = 2n + 1$ and $I_o > 3\sigma(I_o)$ (A instead of C lattice has been chosen for an easier comparison with related structures).

The following lattice parameters were obtained by least-squares refinement of 33 reflections from an X-ray powder diffraction pattern (Table 1): $a = 5.353(4)$, $b = 16.176(12)$, $c = 21.95(2) \text{ \AA}$, $\beta = 94.6(2)^\circ$. The discrepancy between the calculated density (2.83 Mg/cm^3), based on the given empirical formula ($Z = 2$), and the measured density [$2.7(1) \text{ Mg/m}^3$, pycnometer (Khomyakov *et al.*, 1995)] is due to the fibrous nature of the material.

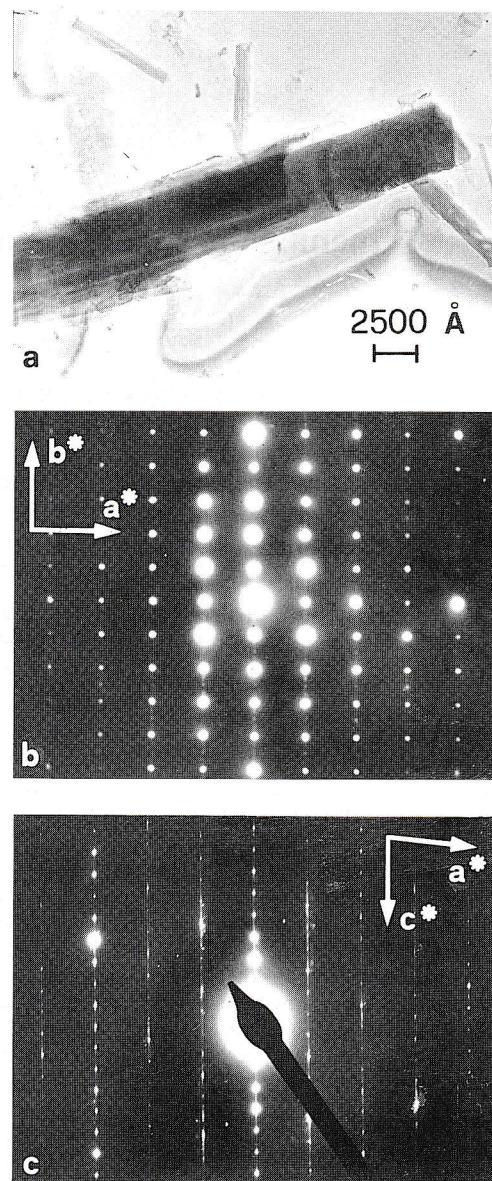


Fig. 1. TEM data of nafertisite: (a) a $\{001\}$ lath rolled around $[100]$; (b) $hk0$ pattern overexposed to show some weak $k = 2n + 1$ reflections; (c) $h0l$ pattern from a rolled lath.

The crystal structure

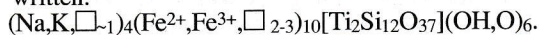
Related structures

Because of the mentioned poorness of the diffraction data and space group uncertainty, a so-

Table 1. X-ray powder pattern of nafertisite; Guinier-Lenné camera, CuK α graphite monochromatized radiation; quartz as internal standard. Indexing has been done according to the pattern calculated (I_c) from the crystal structure. Starred indexes have not been included in the cell refinement. I_m visually estimated intensities on a relative scale. Broad reflections are marked with b.

I_m	I_c	d_m (Å)	d_c (Å)	hkl
30	42	13.00	13.01	011
100	100	10.94	10.92	002
<5	2	8.06	8.09	020*
5	2	6.54	6.50	022*
5	2	5.50	5.46	004*
<5	4	5.23	5.24	031
15	9	4.44	4.45	120
<5	1	4.33	4.34	4.32 033 $\bar{1}$ 13*
5	6	4.03	4.04	4.03 040 122*
			4.02	3.98 113* $\bar{1}$ 04
5	1	3.80	3.774	$\bar{1}$ 31*
10	11	3.638b	3.646	006
10	6	3.571	3.574	$\bar{1}$ 24
10	6	3.330	3.344	124
5	4	3.245	3.252	3.223 044 140
5	6	3.040	3.069	017*
5	2	2.790	2.787	135
25	17	2.728	2.735	2.730 008 151
			2.719	144*
20	12	2.641	2.642	$\bar{2}$ 02
15	18	2.547	2.551	153
15	10	2.480	2.483	2.478 $\bar{1}$ 37* $\bar{2}$ 04
			2.476	$\bar{1}$ 46*
5	6	2.385	2.384	$\bar{1}$ 55
<5	2	2.332	2.332	2.324 162* 204
5	5	2.291	2.295	155
5	3	2.242	2.242	$\bar{2}$ 06
5	2	2.193	2.227	2.188 240* 00.10
5	4	2.116b	2.116	2.098 $\bar{1}$ 57 $\bar{2}$ 17
10	7	2.045b	2.074	2.043 206* $\bar{1}$ 66
			2.036	2.030 148 157*
5	5	1.7839	1.7890	1.7745 159 168*
			1.7702	14.10*
5	4	1.7479	1.7633	1.7485 $\bar{2}$ 0.10* $\bar{2}$ 71
<5	2	1.7342	1.7369	1.7338 $\bar{3}$ 22 271*
			1.7210	13.11*
10	4	1.6184b	1.6213	1.6176 $\bar{1}$ 4.12* 0.10.0
<5	4	1.6000b	1.6002	1.5900 0.10.2 16.10*
10	4	1.5629b	1.5624	$\bar{3}$ 51
5	4	1.5462	1.5468	1.5467 351 $\bar{3}$ 53

lution of the crystal structure of nafertisite could only be obtained *via* a structural model deduced from the related titanosilicates bafertisite and astrophyllite. For an easier comparison with these minerals, the formula of nafertisite (nfr) can be written:



Two polytypes have been described for bafertisite (bft), $\text{Ba}_2(\text{Fe,Mn})_4[\text{Ti}_2\text{Si}_4\text{O}_{17}](\text{OH},\text{O})_3$, with cells $a = 5.36$, $b = 6.80$, $c = 10.98$ Å, $\beta = 94^\circ$ ($Z = 1$, s.g. $P2_1/m$; Pen & Shen, 1963) and $a = 10.60$, $b = 13.64$, $c = 11.73$ Å, $\beta = 112.4^\circ$ [reduced cell from Guan *et al.* (1963), $Z = 2$, s.g. Cm]. Two polytypes have also been described for astrophyllite (ast), $(\text{K,Na})_3(\text{Fe,Mn})_7[\text{Ti}_2\text{Si}_8\text{O}_{27}]$

$(\text{OH},\text{O})_4$, with cells $a = 5.36$, $b = 11.63$, $c = 11.76$ Å, $\alpha = 112.1$, $\beta = 103.1$, $\gamma = 94.6^\circ$ [$Z = 1$, s.g. $P\bar{1}$; reduced cell from Woodrow (1967) who used $A\bar{1}$ space group] and $a = 5.35$, $b = 23.00$, $c = 10.43$ Å, $\beta = 102^\circ$ ($Z = 2$, s.g. $C2/m$; Pen & Ma, 1963).

A comparison of bafertisite, astrophyllite and nafertisite shows that:

- these minerals have a common value for a of about 5.4 Å which corresponds to the a value of mica;
- $(b_{\text{ast}} - b_{\text{bft}}) \approx 1/2(b_{\text{nfr}} - b_{\text{bft}}) \approx 4.7$ Å; this value corresponds to $b/2$ in mica;
- d_{001} (or d_{002}) ≈ 11 Å corresponds to the thickness of one structural layer;

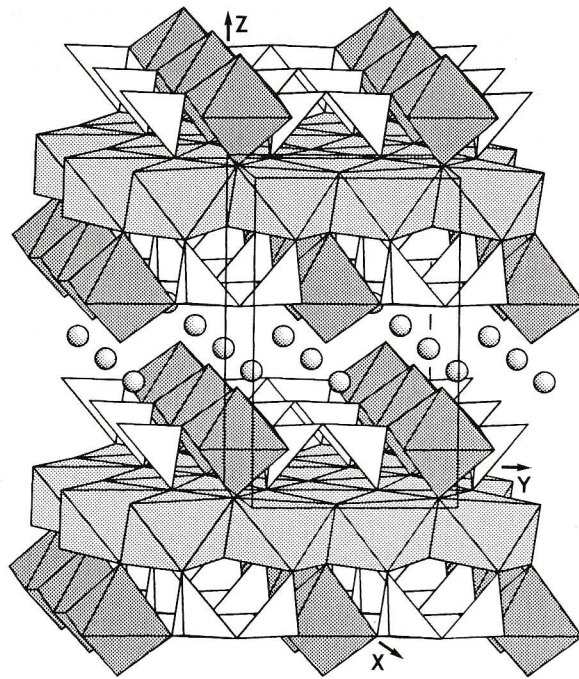


Fig. 2. Perspective view of the crystal structure of bafertisite in the space group $P2_1/m$. Coordinates from Pen & Shen (1963) where the z coordinate of FeII must be read 0.012. Ti octahedra are dark and circles represent Ba.

- the difference between the compositions of astrophyllite and bafertisite and of nafertisite and astrophyllite is about $(A, \square)(M, \square)_3[\text{Si}_4\text{O}_{10}](\text{OH})_2$ (A = interlayer cation; M = octahedral cation); that corresponds to half the difference between the compositions of nafertisite and bafertisite and to the composition of a mica.

Bafertisite and astrophyllite (Fig. 2, 3) are based on phyllosilicate-like structures where:

- a heterogeneous (001) sheet (H) is built by [100] rows of Ti octahedra alternated with strips of Si tetrahedra; the H sheet plays the same role as the tetrahedral sheet (T) in phyllosilicates;
- two H sheets sandwich octahedral M cations (O sheet) and form a 2:1 HOH layer;
- two HOH layers sandwich large A cations.

Ignoring details (like the attachment to the O sheet and the corner sharing of Ti octahedra) and looking along [011], *i.e.* along the shortest Ti-Ti distance across the HOH layer, it turns out that astrophyllite differs from bafertisite only in having an additional mica-like module (M) between two strips of Ti/Si polyhedra (bafertisite-like module, B).

Polytypism and structure

On this basis, a structural model for nafertisite is obtained by inserting two M modules in the bafertisite structure (Fig. 4).

In order to establish details of this model it is necessary to investigate the relationships between the H and O sheets (polytypism). In fact, given the configuration on the HO interface on one side of the O sheet, ten different sites per cell are available on the other side for attaching the corner of a Ti octahedron (Fig. 4). Relative to the position of the Ti octahedron on the reference HO interface, the ten sites can be described by stagger vectors with components $n/6a$ and $m/10b$. The experimental d_{001} value requires a two-layer polytype; the monoclinic cell with $\beta \approx 95^\circ$ constrains the resultant components of the two stagger vectors (one per layer) to be 0 along b and $-1/3 = (c \cos\beta)/a$ along a .

The relative positions of two layers should be considered: *e.g.*, there are single and paired Ti octahedra in bafertisite and astrophyllite, respectively (Fig. 2, 3).

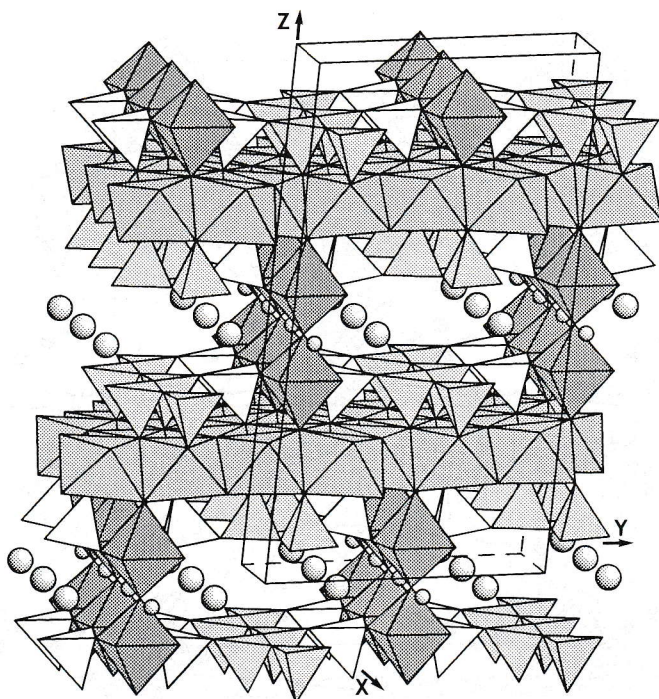


Fig. 3. Perspective view of the crystal structure of astrophyllite in the space group $A1$; coordinates from Woodrow (1967). Ti octahedra are dark; large and small circles represent K and Na respectively. Dotted tetrahedra belong to the (011) undulated slabs of the mica-like modules.

The choice of the latter situation is supported by the final reasonable results.

Five polytypes [symmetry $A2/m$ (1), $P2/c$ (2) and $P2/n$ (2)], compatible with the above constraints, have been derived and tested against the experimental diffraction data, starting from optimized coordinates of the models. Only the $A2/m$ model (stagger vector $\mathbf{a}/6 + \mathbf{b}/2$ for both layers) could be isotropically refined by least-squares (Table 2) obtaining an R value of 0.26 for 491 independent reflections with $|F_o| > 2\sigma |F_o|$, 78 parameters and $1/\sigma^2$ weights¹.

The unsatisfactory refinement and features of the diffraction patterns are due to: (i) stacking faults connected with polytypism ([001] streaks, Fig. 1b); (ii) chain-multiplicity faults connected with polysomatism ([010] streaks, Fig. 1c); (iii) disorder (X-ray diffractograms) and some rol-

ling (Fig. 1a) of the fibres around [100]. In particular, stacking faults producing the mentioned polytypes with P lattice should be responsible for the space group violations.

Crystal chemistry

Even if the poorness of the refinement does not allow a full discussion of crystallochemical aspects (bond lengths and angles not reported), some reasonable results are worth mentioning.

- Difference Fourier maps show an interlayer atom disordered on two sites Na2 and Na2' (Table 2) which are only 2.0 Å apart, and, therefore, are alternatively occupied.

- In agreement with the average chemical analysis, the Fe atoms in the O layer show 0.8 occupancy and the tetrahedral site next to the Ti octahedron has scattering power higher than that of Si, requiring the presence of some Fe^{3+} .

- As astrophyllite (Fig. 3), also nafertisite (Fig. 4) is characterized by a slight waving of the HOH layer.

¹ A list of structure factors may be obtained on request from the authors and is deposited at the Editorial office of the Journal.

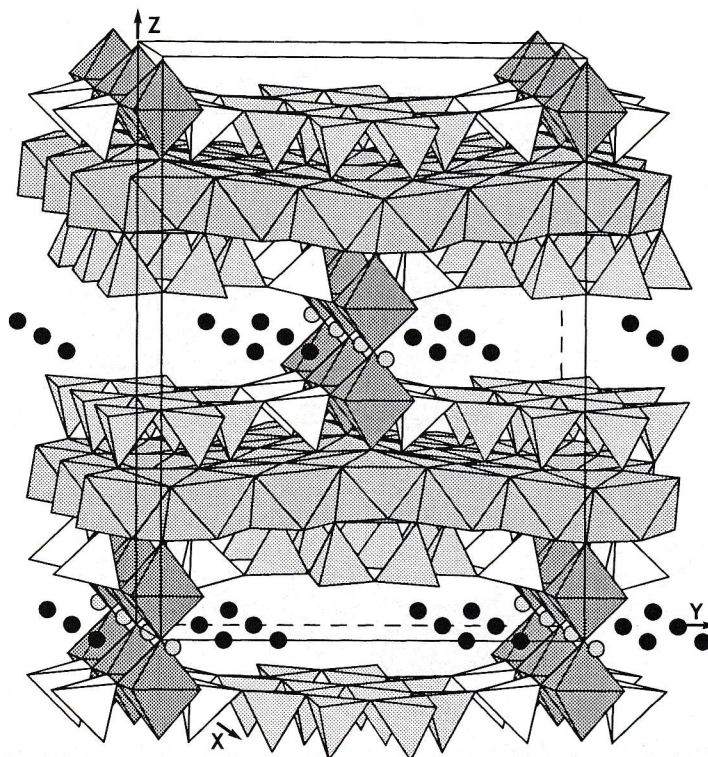


Fig. 4. Perspective view of the crystal structure of nafertisite (this work). Black and dotted circles represent partially and fully occupied alkali sites, respectively. Tetrahedra and octahedra as in Fig. 3.

Since O2 and O5, which account for 6 oxygens pfu, are expected to be OH groups, because they are only coordinated by cations of the *O* sheet, the refined structural model leads to the following ideal crystallochemical formula for nafertisite: $\text{Na}_3\text{Fe}^{2+}_9\text{Fe}^{3+}[\text{Ti}_2\text{Si}_{12}\text{O}_{37}](\text{OH})_6$. In our sample, the octahedral sheet is about 20 % empty and some Fe^{3+} and Al substitute Si; charge balance can be attained by changing the $\text{OH}/\text{O}_{\text{tot}}$ ratio and the content and type of cations, as further discussed below.

Following Liebau (1985), the anion $[\text{Si}_{12}\text{O}_{34}]^{20-}$ forming the silicate strip can be classified as an open branched zweier double chain. On the whole, the *H* sheet represents a new type of complex titanosilicate anion: $[\text{Ti}_2\text{Si}_{12}\text{O}_{37}]^{18-}$.

The polysomatic series

Disregarding details, bafertisite, astrophyllite and nafertisite are members of a polysomatic ser-

ies BM_n built with the following two modules which form (011) undulated slabs running along the shortest Ti-Ti direction across the *HOH* layers (Fig. 5):

- *B* (bafertisite-like module) = $(\text{A}, \square)_2(\text{M}, \square)_4[\text{X}_2\text{T}_4\text{O}_{17}](\text{OH})_2$ (hypothetic bafertisite with corner-sharing Ti octahedra as in astrophyllite; the relative position of the Ti octahedra is not the same in the discussed structures);

- *M* (mica-like module) = $(\text{A}, \square)(\text{M}, \square)_3[\text{T}_4\text{O}_{10}](\text{OH})_2$.

Symbols stand for: A (interlayer cations) = Na, K, Ba, Sr,...; M (*O* sheet cations) = Fe^{2+} , Fe^{3+} , Mn, Mg, Ca,...; X (octahedral cations of the *H* sheet) = Ti (mainly), Nb, Zr,...; T (tetrahedral cations of the *H* sheet) = Si (mainly), Fe^{3+} , Al,...; \square = vacancy.

On the basis of the analogy with the phyllosilicates, the described series is called *heterophyllosilicate polysomatic series*. The prefix *hetero* indicates that, with respect to the phyllosilicates, the members of the new series show *hetero*-

Table 2. Atomic positions and isotropic displacement factors U (\AA^2) for nafertisite. The occupancy of the Fe sites is 0.8; Na2 and Na2' are alternative disordered positions.

	x	y	z	U (\AA^2)
Fe1	0.242 (2)	0.3994 (8)	-0.2649 (6)	0.010 (5)
Fe2	0.257 (3)	0.8001 (8)	-0.2453 (6)	0.016 (5)
Fe3	0.258 (3)	0	-0.2319 (9)	0.012 (6)
Si1	0.444 (4)	-0.122 (1)	-0.117 (1)	0.073 (8)
Si2	0.434 (5)	0.312 (2)	-0.133 (1)	0.042 (9)
Si3	-0.064 (5)	0.408 (1)	-0.136 (1)	0.030 (8)
Na1	1/2	0	0	0.05 (2)
Na2	0	-0.154 (4)	0	0.02 (2)
Na2'	0	-0.279 (6)	0	0.02 (3)
Ti	-0.036 (4)	0	-0.0980 (8)	0.005 (6)
O1	0.387 (8)	0.301 (3)	-0.206 (2)	0.02 (2)
O2	0.390 (13)	1/2	-0.223 (3)	0.03 (2)
O3	0.411 (8)	-0.104 (2)	-0.186 (2)	0.01 (1)
O4	-0.109 (10)	0.404 (3)	-0.207 (3)	0.05 (2)
O5	0.104 (9)	0.295 (3)	-0.305 (2)	0.02 (2)
O6	0.075 (13)	1/2	-0.328 (3)	0.02 (2)
O7	0.215 (8)	0.362 (2)	-0.109 (2)	0.02 (1)
O8	0.725 (9)	-0.089 (3)	-0.081 (2)	0.01 (1)
O9	0.247 (12)	0.082 (4)	-0.079 (3)	0.06 (2)
O10	0.455 (8)	0.222 (3)	-0.100 (2)	0.01 (1)
O11	0.752 (10)	0.360 (4)	-0.105 (3)	0.05 (2)
O12	-0.073 (13)	1/2	-0.105 (3)	0.03 (2)
O13	0	0	0	0.00 (2)

geneous H sheets with Ti (mainly) octahedra playing a role similar to that of tetrahedra.

Crystal chemistry

The formula of the general BM_n member of the heterophyllosilicate polysomatic series, which reminds the biopyriboles in sharing M modules, is $\{(A, \square)_2(M, \square)_4[X_2T_4O_{17}](OH)_2\} \{(A, \square)(M, \square)_3[T_4O_{10}(OH)_2]\}_n$ or $(A, \square)_{2+n}(M, \square)_{4+3n}[X_2T_{4+4n}O_{17+10n}](OH)_{2+2n}$.

The following members (polysomes) are known: $n = 0$ bafertisite; $n = 1$ astrophyllite; $n = 2$ nafertisite and $n = \infty$ mica.

An ideal member of the series with a finite value of n is based on $19 + 12n$ oxygens pfu:

- $11 + 6n$ oxygens belong to the H sheet only (plus one, if pairs of Ti octahedra do not share a corner, as in bafertisite);

- $6 + 4n$ oxygens are shared between O and H sheets;

- $2 + 2n$ oxygens belong to the O sheet only and are expected to be OH groups. However, the OH/O_{tot} ratio depends on the content and type of cations.

Crystallography

All the members of the heterophyllosilicate series have the same value of $a \approx 5.4 \text{ \AA} \approx a_{\text{mica}}$. The b value increases about 4.7 \AA for each insertion of a mica module, i.e. $b \approx (6.8 + n4.7) \text{ \AA}$. For an m -layer polytype, $d_{001} \approx m11.0 \text{ \AA}$.

When $n > 0$ the T_2O_7 groups of the H sheet are chess-board arranged; consequently, the ideal bidimensional lattice for the H sheet is primitive or C centered for n even or odd respectively. In the latter case the b axis is doubled (cf. the $C2/m$ polytype of astrophyllite).

Related minerals

The complex Ti/Si anion (H sheet) which occurs in nafertisite has only been found in this mineral so far. The heterogeneous H sheet of astrophyllite, instead, is known for a group of minerals which, however, are just "astrophyllites" with isomorphous substitutions (astrophyllite group): cesiumkupletskite, hydroastrophyllite, kupletskite, magnesiumastrophyllite, niobophyllite, zircophyllite (Fleisher & Mandarino, 1995).

The bafertisite H sheet is definitely widespread. It occurs in several titanosilicates with different interlayer contents, as summarized by Egorov-Tismenko & Sokolova (1990). These authors, emphasizing (001) layers, have connected the bafertisite-like structures to a series which spans from seidozerite $\text{Na}_2\text{MnTiF}_2[\text{Na}(\text{Zr}, \text{Ti})\text{O}(\text{Si}_2\text{O}_7)]_2$, where the Ti octahedra of adjacent HOH layers share one edge and no interlayer space is left, to polyphite, $\text{Na}_{17}\text{Ca}_3\text{Mg}(\text{Ti}, \text{Mn})_4[\text{Si}_2\text{O}_7]_2[\text{PO}_4]_6\text{O}_2\text{F}_6$, where the interlayer consists of $\text{Na}_2\text{Ca}[\text{PO}_4]\text{F}$ (nakaphite). Actu-

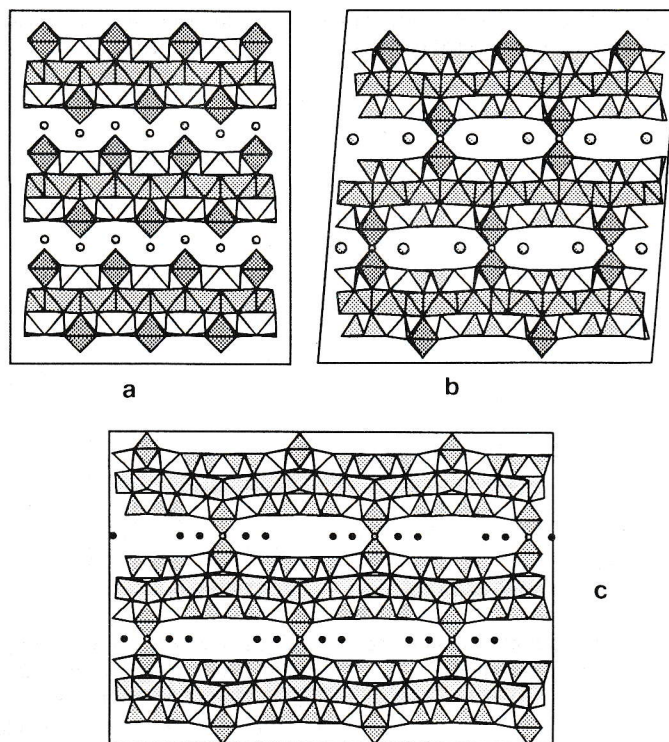


Fig. 5. Projection along [100] of the crystal structures of bafertsite (a), astrophyllite (b) and nafertisite (c). The slope of the (011) undulated slabs of the *B* bafertsite-like (white tetrahedra) and *M* mica-like modules (dotted tetrahedra) increases from (a) to (c).

ally the members of this series can be considered derivatives of bafertsite, *i.e.* the BM_0 member of the heterophyllosilicate series, where the inter-layer contents can be:

- absent (seidozerite, götzenite, rosenbuschite);
- alkalis and/or alkaline earths plus/or (i) H_2O (murmanite, epistolite), (ii) (SO_4) (innelite) and (iii) (PO_4) (lomonosovite, betalomonosovite, vuonnemite, sobolevite, quadruphite and polyphite).

References and characteristics of the quoted minerals can be found in Egorov-Tismenko & Sokolova (1990) and, for quadruphite and polyphite, in Khomyakov *et al.* (1992).

Conclusions

Most of the minerals related to the heterophyllosilicate polysomatic series, including the derivatives of the BM_0 polysome (bafertsite),

occur in the hyperagpaitic rocks of the Kola Peninsula and are genetically related (Khomyakov, 1995). Their structural layers can be built by bafertsite-like (*B*) and mica-like (*M*) modules; the actual composition of these modules and of the interlayer has a large variability, particularly in the derivatives of the BM_0 member. The complex planar anion forming the *H* sheet, $[Ti_2Si_{4+4n}O_{17+10n}]^{(10+4n)-}$, turns out to be particularly well-adapted to the hyperalkaline chemistry of the Khibina massif; it is the last one of a long series of peculiar anions (Khomyakov, 1995) and, *e.g.*, adds to $[AlSi_4O_{13}]^{7-}$ recently described for altisite (Ferraris *et al.*, 1995).

Once more (Ferraris *et al.*, 1986; Veblen, 1991), the concept of polysomatic series is able to correlate minerals which are connected by paragenesis and/or solid state transformations. From a crystallographic point of view, a correlation between properties of a new phase and the features of a polysomatic series can allow an easier characterization.

Acknowledgements: This work was supported by ISF (No. MBE00) and MURST grants. The visit of S.V.S. to Italy was supported by grants of MAE (Roma) and Università degli Studi di Torino. The final version of the paper benefited by helpful comments and suggestions of the referees B. Grobóty and M. Mellini.

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Received 8 March 1995

Modified version received 8 September 1995

Accepted 27 November 1995