

Clinobarylite, $\text{BaBe}_2\text{Si}_2\text{O}_7$: structure refinement, and revision of symmetry and physical properties

Sergey V. Krivovichev, Kiel, St. Petersburg, Viktor N. Yakovenchuk, Apatity, Thomas Armbruster, Bern, Yulia Mikhailova, Apatity, and Yakov A. Pakhomovsky, Apatity

With 1 figure and 5 tables

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Abstract: The structure of clinobarylite, $\text{BaBe}_2\text{Si}_2\text{O}_7$, a rare mineral from the Khibina alkaline massif, Kola peninsula, has been refined in the orthorhombic space group $Pmn2_1$, $R1 = 0.030$ ($wR2 = 0.082$, $S = 1.167$) for 469 unique observed reflections with $|F_o| \geq 4\sigma_F$. The structure is based upon tetrahedral framework consisting of BeO_4 and SiO_4 tetrahedra. BeO_4 tetrahedra share corners to form chains parallel to the c axis. The chains are interlinked by the Si_2O_7 groups oriented parallel to the a axis. The Ba^{2+} cations are in the framework channels and are coordinated by eleven O atoms. The obtained orthorhombic symmetry of clinobarylite is in contradiction with the monoclinic symmetry recently reported for this mineral by CHUKANOV et al. (2003). Optical properties and powder X-ray diffraction pattern of clinobarylite have been re-studied and no contradictions with orthorhombic symmetry have been observed. Comparison of clinobarylite and its dimorph, barylite, show that tetrahedral frameworks in the structures of these minerals are based upon tetrahedral sheets of the same type. According to this description, barylite and clinobarylite can be considered as 2O- and 1O-polytypes of $\text{BaBe}_2\text{Si}_2\text{O}_7$, respectively.

Key words: clinobarylite, barylite, crystal structure, Khibina massif, Kola peninsula.

Introduction

Clinobarylite, $\text{BaBe}_2\text{Si}_2\text{O}_7$, has been described recently by CHUKANOV et al. (2003) from the Yukspor Mt., Khibina massif, Kola peninsula, Russia. Clinobarylite is a dimorph of barylite, $\text{BaBe}_2\text{Si}_2\text{O}_7$, a mineral known since

1876 (GAINES et al. 1997). Crystal structure determination (RASTSVETAeva & CHUKANOV 2003) demonstrated that clinobarylite is monoclinic, space group $Pm11$. Reported monoclinic symmetry of clinobarylite is responsible for its name as a monoclinic analogue of barylite which is orthorhombic. In this paper, we report results of our re-investigation of clinobarylite. First, we analyze data on this mineral reported by CHUKANOV et al. (2003) and RASTSVETAeva & CHUKANOV (2003), and then we present results of our own studies that unambiguously show that clinobarylite is in fact orthorhombic. Consequently, we provide revised data on its symmetry, structure and optical properties. In conclusions, we discuss in more details structural differences of clinobarylite and barylite.

Previous studies

RASTSVETAeva & CHUKANOV (2003) reported clinobarylite as monoclinic, space group $Pm11$. The unit-cell parameters were given by RASTSVETAeva & CHUKANOV (2003) as $a = 11.618(3)$, $b = 4.904(1)$, $c = 4.655(1)$ Å, $\beta = 89.94(2)^\circ$ (the same parameters are reported by CHUKANOV et al. 2003). However, with the space-group setting $Pm11$, the monoclinic angle should be α and not β as reported by these authors. It is evident from Fig. 1a of the paper by RASTSVETAeva & CHUKANOV (2003) that the monoclinic angle is actually α and not β . Thus, the proper (though non-standard) setting of the structure is $Pm11$, $a = 11.618(3)$, $b = 4.904(1)$, $c = 4.655(1)$ Å, $\alpha = 89.94(2)^\circ$. Note that the α angle is very close to 90° and there is no clear evidence that the structure should really be monoclinic. Analysis of the atomic coordinates reported by RASTSVETAeva & CHUKANOV (2003) has revealed an additional symmetry operator $(0.5 - x, 0.5 - y, 0.5 + z)$ that works especially well if all atoms are shifted by -0.0088 along the b axis. Obviously, the operator $(0.5 - x, 0.5 - y, 0.5 + z)$ corresponds to the twofold screw 2_1 axis parallel to the c axis, centered at $x = 1/4$, $y = 1/4$ in the original unit cell. Addition of this symmetry operation to the space group $Pm11$ produces an orthorhombic space group $Pmn2_1$ with unit-cell parameters $a = 11.618(3)$, $b = 4.904(1)$, $c = 4.655(1)$ Å. The following pairs of atoms given by RASTSVETAeva & CHUKANOV (2003) are symmetry equivalent: Ba(1)–Ba(2), Be(1)–Be(2), Si(1)–Si(2), O(1)–O(4), O(2)–O(3), O(5)–O(7), O(6)–O(8). These atoms are symmetrically equivalent within 0.025 Å for Ba and Si and within 0.06 Å for Be and O. Thus, structural data on clinobarylite suggest that this mineral is most probably orthorhombic, space group

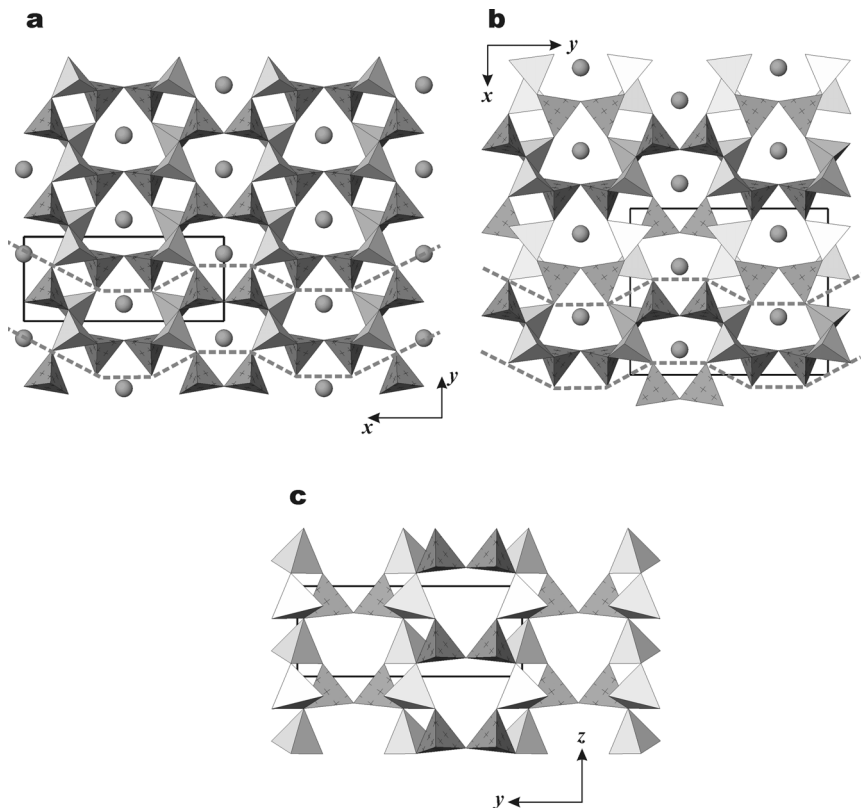


Fig. 1. Crystal structures of clinobarylite (a) and barylite (b), and sheet of chains of corner-sharing BeO₄ tetrahedra and Si₂O₇ groups in the structure of barylite (c). Legend: SiO₄ tetrahedra = cross-hatched and shaded, grey; BeO₄ tetrahedra = shaded; Ba atoms are shown as circles. In (a) and (b), dashed lines show sheets similar to those shown in (c).

*Pmn*2₁. However, if clinobarylite is truly monoclinic, this should also be reflected in its optical properties and (probably) its X-ray powder-diffraction pattern.

The optical constants of clinobarylite were reported by CHUKANOV et al. (2003) as following: biaxial, optically positive, $\alpha = 1.698(3)$, $\beta = 1.700(3)$, $\gamma = 1.705(5)$, $2V_{\text{meas}} = 70(10)^\circ$, $2V_{\text{calc}} = 65^\circ$. The orientation of the optical axes relative to the crystallographic axes was given as $Z = b$, $X \wedge a = 6^\circ$, $Y \wedge c = 5.5^\circ$. This is in obvious contradiction with the space group *Pm*11 reported

by RASTSVETAeva & CHUKANOV (2003). If the a axis is a unique monoclinic axis (as implied from the $Pm11$ setting), then one of the optical axis should be parallel to a and the angle $X^\wedge a$ makes no sense unless optics in clinobarylite has an anomalous character. In addition, in a metrically orthorhombic unit cell, $X^\wedge a$ and $Y^\wedge c$ must be equal, whereas CHUKANOV et al. (2003) reported them to be different by 0.5° which is again unacceptable.

X-ray diffraction pattern of clinobarylite reported by CHUKANOV et al. (2003) has only one diffraction peak that is not indexable in the orthorhombic space group $Pmn2_1$ suggested in this study. This is the 001 peak at $d = 4.671 \text{ \AA}$; its intensity on a 100 %-scale is 3. This reflection is also absent if the diffraction pattern is calculated on the basis of the space group $Pm11$ and atomic coordinates reported by RASTSVETAeva & CHUKANOV (2003). All facts mentioned above indicated that clinobarylite is most probably orthorhombic, which prompted our re-investigation of this mineral using crystals from the type locality, i. e. Yukspor Mt., Khibina massif, Kola peninsula, Russia.

Structure refinement

A crystal of clinobarylite flattened on (010) was selected for single-crystal structure study. The crystal was mounted on a Bruker PLATFORM goniometer equipped with a 1K SMART CCD detector with a crystal-to-detector distance of 5.4 cm. A hemisphere of three-dimensional data was collected using $\text{MoK}\alpha$ X-radiation and frame widths of 0.3° in ω , with 45 s used to acquire each frame. The unit-cell dimensions were refined on the basis of 532 reflections (Table 1). The systematic absences of reflections are consistent with the orthorhombic space-group $Pmn2_1$, which is in agreement with the space group derived from analysis of atomic coordinates given by RASTSVETAeva & CHUKANOV (2003). The data were reduced using the Bruker program SAINT. A semi-empirical absorption-correction based upon the intensities of equivalent reflections was applied modelling the crystal as a (010) plate, and the data were corrected for Lorentz, polarization, and background effects. A total of 1363 intensities was measured; there were 498 unique reflections ($R_{\text{INT}} = 0.033$), with 469 classified as observed ($F_o > 4sF_o$).

Scattering curves for neutral atoms, together with anomalous dispersion corrections, were taken from (IBERS & HAMILTON 1974). The Bruker SHELXTL Version 5.1 system of programs was used for the refinement of

Table 1. Crystallographic data and refinement parameters for clinobarylite.

a (Å)	11.650(10)	Crystal size (mm)	$0.30 \times 0.08 \times 0.007$
b (Å)	4.922(4)	Radiation	MoK α
c (Å)	4.674(4)	Total Ref.	1363
V (Å ³)	268.0(2)	Unique Ref.	498
Space group	$Pmn2_1$	Unique $ F_o \geq 4\sigma_F$	469
F_{000}	296	R_1	0.030
μ (mm ⁻¹)	7.84	wR_2	0.082
Z	2	S	1.167
D_{calc} (g/cm ³)	4.01		

Note: $R_1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$; $wR_2 = \{\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where $P = (F_o^2 + 2F_c^2)/3$; $s = \{\Sigma [w(F_o^2 - F_c^2)] / (n - p)\}^{1/2}$ where n is the number of reflections and p is the number of refined parameters.

the crystal structure on the basis of F^2 . The structure was solved by direct methods and refined to a final agreement index (R_1) of 0.030, calculated for the 469 unique observed reflections ($|F_o| \geq 4\sigma_F$), and a goodness-of-fit (S) of 1.167. The racemic twinning model was included into the refinement and resulted in significant improvement of its quality. The final model included atom positional parameters, and anisotropic displacement parameters for Ba and Si. Attempts to refine Be and O atoms anisotropically resulted in physically unrealistic displacement parameters which is probably a result of the dominance of the Ba scattering power over all other atoms present in the structure and the racemic twinning of the crystal studied. The final atom parameters and bond-valence sums are given in Table 2, and selected interatomic distances are in Table 3. Observed and calculated structure-factors are available from the authors upon request.

As we can conclude from our study, the systematic absences, internal consistency of the dataset, and atomic coordinates are in agreement with the orthorhombic symmetry of clinobarylite. All geometric parameters (bond lengths, angles, etc.) are similar to those obtained by RASTSVETAeva & CHUKANOV (2003) in the space group $Pm11$.

Structure description

A general description of the structure of clinobarylite was given by RASTSVETAeva & CHUKANOV (2003). BeO₄ tetrahedra share corners to form

Table 2. Atomic coordinates, displacement parameters (\AA^2), and bond-valence sums* (BVS, v. u. = valence units) for clinobarylite.

Atom	x	y	z	U_{eq}	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	BVS
Ba $\frac{1}{2}$	0.2027 (12)	0.5079 (1)	0.0127 (2)	0.0127 (2)	0.01354 (33)	0.01192 (34)	0.01256 (37)	-0.0002 (6)	0	0	1.74
Si	0.6282 (56)	0.67564 (16)	-0.0141 (4)	0.0064 (5)	0.0044 (12)	0.00579 (81)	0.00900 (91)	-0.0033 (14)	0.0024 (13)	-0.0010 (15)	4.06
Be	0.75195 (75)	0.1671 (16)	-0.0147 (42)	0.0067 (19)							2.00
O (1)	0.63911 (39)	0.3521 (10)	-0.9436 (13)	0.0044 (11)							1.85
O (2)	0.77733 (44)	0.1340 (12)	-0.3534 (13)	0.0072 (12)							1.99
O (3) $\frac{1}{2}$		0.7741 (17)	-0.0767 (21)	0.0144 (21)							2.35
O (4)	0.63300 (11)	0.7324 (5)	-0.3550 (10)	0.0091 (13)							1.89

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

* Calculated using bond-valence parameters from (BROWN & ALTERMATT 1985).

Table 3. Selected bond lengths (Å) in the structure of clinobarylite.

Si–O(3)	1.598(4)	Ba–O(1)	2.760(6) 2 ×
Si–O(4)	1.618(9)	Ba–O(4)	2.858(6) 2 ×
Si–O(2)	1.629(7)	Ba–O(3)	2.867(6)
Si–O(1)	1.631(6)	Ba–O(4)	3.100(6) 2 ×
<Si–O>	1.62	Ba–O(1)	3.121(6) 2 ×
		Ba–O(2)	3.313(6) 2 ×
Be–O(4)	1.61(1)	<Ba–O>	3.02
Be–O(2)	1.62(2)		
Be–O(1)	1.63(1)		
Be–O(2)	1.70(1)		
<Be–O>	1.64		

chains parallel to the *c* axis. The chains are interlinked by the Si₂O₇ groups oriented parallel to the *a* axis (Fig. 1 a). The Ba²⁺ cations are in the framework channels and are coordinated by eleven O atoms.

X-ray powder diffraction study

The powder X-ray diffraction pattern of clinobarylite (Table 4) was obtained using a DRON-2 diffractometer operated at 20 kV and 30 mA (Bragg-Brentano geometry, CuK α -radiation). Table 4 gives comparison of the obtained powder X-ray diffraction pattern to that given for clinobarylite by CHUKANOV et al. (2003) and one calculated on the basis of atomic coordinates given above. The only reflection, 001, that violates the *Pmn*2₁ space group in the pattern given CHUKANOV et al. (2003) is absent in our data. This reflection has also zero intensity in the theoretical X-ray diffraction pattern calculated from crystal-structure data given by RASTSVETAJEVA & CHUKANOV (2003). Thus the X-ray powder diffraction data are in agreement with the orthorhombic symmetry of clinobarylite suggested here.

Optical properties

As the optical properties given for clinobarylite by CHUKANOV et al. (2003) are rather contradictory (see above), we have re-determined optical constants. The mineral was found to be orthorhombic, with following orientation of main vibration directions: *X* = *b*, *Y* = *c*. Clinobarylite is biaxial, opti-

Table 4. Powder X-ray diffraction data on clinobarylite.

This work		CHUKANOV et al. 2003		Calculated*		<i>h</i>	<i>k</i>	<i>l</i>
<i>I</i> _{meas}	<i>d</i> _{meas} (Å)	<i>I</i> _{meas}	<i>d</i> _{meas} (Å)	<i>I</i> _{calc}	<i>d</i> _{calc} (Å)			
7	5.86	13	5.83	52	5.83	2	0	0
3	4.90	5	4.914	2	4.92	0	1	0
		3	4.671					
20	4.529	24	4.529	27	4.53	1	1	0
12	4.336	19	4.332	31	4.338	1	0	1
		3	3.746	1	3.760	2	1	0
56	3.388	84	3.389	85	3.389	0	1	1
17	3.253	45	3.249	26	3.254	1	1	1
26	3.042	40	3.043	74	3.049	3	1	0
28	2.986	36	2.983	85	2.987	3	0	1
58	2.929	55	2.926	100	2.930	2	1	1
				5	2.913	4	0	0
				1	2.554	3	1	1
8	2.500	5	2.501	19	2.507	4	1	0
100	2.458	100	2.458	32	2.461	0	2	0
22	2.404	14	2.404	8	2.408	1	2	0
31	2.336	48	2.335	31	2.337	0	0	2
20	2.265	15	2.265	13	2.267	2	2	0
20	2.206	21	2.206	40	2.209	4	1	1
				7	2.178	0	2	1
20	2.171	20	2.168	17	2.169	2	0	2
16	2.139	11	2.137	13	2.141	1	2	1
7	2.105	4	2.106	15	2.106	5	1	0
				16	2.085	5	0	1
23	2.077	20	2.077	4	2.079	3	2	0
				17	2.077	1	1	2
8	2.038	8	2.036	8	2.040	2	2	1
2	1.983			3	1.985	2	1	2
2	1.941			7	1.942	6	0	0
14	1.898	24	1.897	15	1.899	3	2	1
				2	1.880	4	2	0
14	1.853	34	1.854	20	1.855	3	1	2
2	1.823	4	1.822	3	1.823	4	0	2
7	1.742	12	1.745	8	1.744	4	2	1
4	1.708			5	1.709	4	1	2
12	1.693	18	1.694	9	1.695	0	2	2
7	1.684			13	1.685	6	1	1
8	1.677	17	1.675	6	1.677	1	2	2
2	1.639			0.1	1.641	0	3	0
9	1.625	14	1.625	8	1.627	2	2	2
				1	1.625	1	3	0

Table 4. Continued.

This work		CHUKANOV et al. 2003		Calculated*		<i>h</i>	<i>k</i>	<i>l</i>	
<i>I</i> _{meas}	<i>d</i> _{meas} (Å)	<i>I</i> _{meas}	<i>d</i> _{meas} (Å)	<i>I</i> _{calc}	<i>d</i> _{calc} (Å)				
6	1.590	6	1.590	7	1.591	5	2	1	
6	1.576	7	1.578	}	2	1.579	2	3	0
7	1.564	6	1.567		2	1.577	7	1	0
					2	1.568	7	0	1
					11	1.565	5	1	2
7	1.546	4	1.548		2	1.553	3	2	2
				2	1.548	0	3	1	
				2	1.544	1	0	3	
21	1.532	35	1.533	11	1.534	1	3	1	
4	1.524	5	1.523	4	1.524	6	2	0	
5	1.510	4	1.509	2	1.511	3	3	0	
12	1.494	26	1.495	5	1.496	2	3	1	
				3	1.494	7	1	1	
				5	1.494	6	0	2	
				6	1.485	0	1	3	
				3	1.449	6	2	1	
8	1.486			7	1.446	3	0	3	
10	1.446	6	1.446	9	1.439	2	1	3	
				9	1.430	4	3	0	
				2	1.378	7	2	0	
				1	1.371	5	2	2	
13	1.428	27	1.428	}	2	1.367	4	3	1
2	1.376			}	1	1.323	4	1	3
3	1.365	4	1.366		2	1.322	7	2	1
5	1.337	15	1.337		3	1.309	2	3	2
3	1.322	5	1.322	1	1.308	1	2	3	
7	1.307	14	1.308	3	1.307	7	1	2	
				5	1.295	5	0	3	
				3	1.284	2	2	3	
3	1.276			3	1.277	6	2	2	
4	1.268			2	1.269	3	3	2	
3	1.252			4	1.253	8	2	0	
6	1.245	10	1.248	1	1.248	9	0	1	
				5	1.247	3	2	3	
2	1.236			4	1.236	8	0	2	
12	1.222			2	1.224	1	4	0	
13	1.218	30	1.216	6	1.220	4	3	2	

* Calculated using crystal-structure data determined from single-crystal structure analysis using ATOMS 5.1 (DOWTY 2000).

cally negative, almost neutral, $\alpha = 1.695(5)$, $\beta = 1.702(5)$, $\gamma = 1.708(8)$, $2V_{\text{calc}} = 85(5)^\circ$ (measurements performed by YU. MIKHAILOVA). The negative sign determined for clinobarylite is in contradiction with the positive sign determined for this mineral by CHUKANOV et al. (2003). This may be explained by difficulties associated with determination of optical character of nearly optically neutral crystals.

Discussion

Table 5 gives comparison of the crystallographic characteristics obtained for clinobarylite by CHUKANOV et al. (2003) and in this work, with those determined by barylite by ROBINSON & FANG (1980). It is obvious that clinobarylite is a dimorph of barylite and both these minerals have orthorhombic symmetry. As it was noted by RASTSVETAeva & CHUKANOV (2003) that the structures of barylite and clinobarylite are closely related. In both structures, BeO_4 tetrahedra share corners to form chains elongated along the c axis in both structures. These beryllate groups are interlinked via Si_2O_7 groups to form three-dimensional framework (Fig. 1). The difference between the two structures is in the orientation of the chains of BeO_4 tetrahedra. In barylite, these chains are running up and down (BeO_4 tetrahedra are looking in both up and down directions), whereas, in clinobarylite, they are oriented only up (beryllate tetrahedra are looking in the same direction).

Relationships between the structures of barylite and clinobarylite can also be explained on the basis of sheets of BeO_4 tetrahedra and Si_2O_7 groups parallel to (010) in clinobarylite and to (100) in barylite (the sheets are marked by dashed lines in Figs. 1a and b). These sheets consist of beryl-

Table 5. Crystallographic data on clinobarylite and barylite.

Parameter	Clinobarylite CHUKANOV et al. 2003	This work	Barylite ROBINSON & FANG 1977
a (Å)	11.618	11.650	9.82
b (Å)	4.904	4.922	11.67
c (Å)	4.655	4.674	4.69
α (°)	89.94	—	—
V (Å ³)	265.2	268.0	537.5
Space group	$Pm11$	$Pmn2_1$	$Pnma$

late chains interlinked via Si_2O_7 groups (Fig. 1 c). As beryllate chains are strictly directional (all BeO_4 tetrahedra within the sheet have the same orientation), the sheets are also strictly directional. Beryllsilicate tetrahedral frameworks in barylite and clinobarylite may be considered as based on sheets of the same type. However, in barylite, two adjacent sheets have the opposite orientation, whereas, in clinobarylite, all sheets have the same orientation. As a consequence, the a parameter of clinobarylite is halved compared the corresponding b parameter of barylite (Table 5). It should be noted that, according to this description, barylite and clinobarylite must be considered as 2*O*- and 1*O*-polytypes of $\text{BaBe}_2\text{Si}_2\text{O}_7$, respectively.

It is worthy to note that the name “clinobarylite” was assigned to this mineral because of its (initially thought) monoclinic symmetry, in contrast to barylite, which has an orthorhombic symmetry (CHUKANOV et al. 2003). Here we demonstrated that clinobarylite is in fact orthorhombic, which raises the question concerning validity of its name.

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Authors' addresses:

Dr. S. V. KRIVOVICHEV, Institut für Geowissenschaften – Mineralogie/Kristallographie, Christian-Albrechts-Universität zu Kiel, Olshausenstrasse 40, 24118 Kiel, Germany.

Permanent address: Department of Crystallography, Faculty of Geology, St. Petersburg State University, University Emb. 7/9, 199034 St. Petersburg, Russia.

Corresponding author: E-mail: skrivovi@mail.ru

Dr. VIKTOR N. YAKOVENCHUK, Dr. YAKOV A. PAKHOMOVSKY, YULIA MIKHAILOVA, Geological Institute, Kola Science Center, Russian Academy of Sciences, Apatity 184200 Russia.

Prof. Dr. THOMAS ARMBRUSTER, Laboratorium für chemische and mineralogische Kristallographie, Universität Bern, Freiestrasse 3, CH-3102 Bern, Switzerland.