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ITSIITE, $\text{Ba}_2\text{Ca}(\text{BSi}_2\text{O}_7)_2$, A NEW MINERAL SPECIES FROM YUKON, CANADA: DESCRIPTION AND CRYSTAL STRUCTURE

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ABSTRACT

Itsiite, ideally $\text{Ba}_2\text{Ca}(\text{BSi}_2\text{O}_7)_2$, is a new mineral from the Gun claim, just south of the Itsi Range, Yukon Territory, Canada. The new mineral occurs in low temperature, late-stage veins in direct association with cerchiaraitite-(Fe), diopside, pyrite, quartz, sphalerite, and witherite. Itsiite occurs as colorless and light blue to medium greenish-blue tetragonal plates up to 1 mm across. The plates are flattened on {001} and exhibit the forms {001}, {101}, and {112}. The mineral is transparent, has a vitreous luster, and is non-fluorescent. It has a white streak and Mohs hardness of approximately 5½. It is brittle, with splintery fracture, and one perfect cleavage on {001}. The calculated density based upon the empirical formula and single-crystal unit cell is 3.644 g/cm³. The mineral is optically uniaxial (-), with $\omega = 1.623(1)$ and $\epsilon = 1.619(1)$ (white light). The mineral is nonpleochroic. Electron-microprobe compositions (average of 3) provided: Na₂O 0.06, BaO 46.35, CaO 7.35, FeO 0.15, Al₂O₃ 0.17, TiO₂ 0.06, SiO₂ 34.91, B₂O₃ 10.41 (from structure), total 99.46 wt.%. The empirical formula (based on 14 O *apfu*) is $\text{Ba}_{2.06}(\text{Ca}_{0.89}\text{Al}_{0.02}\text{Na}_{0.01}\text{Fe}_{0.01}\text{Ti}_{0.01})_{\Sigma 0.94}(\text{Si}_{3.96}\text{B}_{2.04})_{\Sigma 6.00}\text{O}_{14}$. Itsiite is tetragonal, $I\bar{4}2m$, a 10.9515(5), c 10.3038(7) Å, V 1235.79(11) Å³, and $Z = 4$. The nine most intense lines in the X-ray powder diffraction pattern are [d_{obs} in Å(I)(hkl)]: 5.50(42)(200); 3.746(100)(202); 3.446(60)(301); 3.100(51)(222); 2.899(96)(321,312); 2.279(44)(323); 2.145(69)(224,501); 1.8257(41)(503,334,305); 1.7584(43)(532,523). The crystal structure ($R_1 = 1.8\%$ for 992 $F_o > 4\sigma F$) is based upon a zeolite-like tetrahedral framework of corner-sharing tetrahedra consisting of four-membered silicate rings alternating with four-membered borate rings. The framework contains channels along each axis that host Ba^{2+} in ninefold coordination and Ca^{2+} in sixfold coordination. The structure is very similar to those of hyalotekite and kapitsaite-(Y).

Keywords: itsiite, new mineral, borosilicate, crystal structure, hyalotekite, kapitsaite-(Y), Gun claim, Yukon Territory, Canada

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INTRODUCTION

Recently, Kampf *et al.* (2013) described the new minerals cerchiarait-(Fe) and cerchiarait-(Al) from the the Cerchiara mine, Liguria, Italy [cerchiarait-(Fe)] and the Esquire #7 and #8 claims along Big Creek, Fresno County, California, USA [cerchiarait-(Fe) and cerchiarait-(Al)]. They noted these phases occurring at several other Ba-rich contact metamorphic deposits, one of which is the Gun claim, Yukon Territory, Canada. Subsequently, retired mineral dealer Forrest Cureton approached one of the authors (ARK) about verifying the presence of cerchiarait-(Fe) and/or cercharait-(Al) on a specimen from the Gun claim that he had acquired about 25 years earlier in an exchange with the Geological Survey of Canada. That specimen did, in fact, prove to contain cerchiarait-(Fe); however, more significantly, a major component of the specimen is the new mineral itsiite, described herein.

Itsiite is named for the Itsi Mountain Range (Fig. 1), a spectacular, glaciated, multi-peaked massif within the Selwyn Mountains proper. The Gun claim is located in a cirque on a smaller, unnamed mountain separated from the Itsi Range by the Ross River. The Gun claim is only about 5 km from the base of the Itsi Range. "Itsi" means "wind" in the language of the Kaska, a First Nations people of the area.

The new mineral and name have been approved by the Commission on New Minerals, Nomenclature and Classification of the International Mineralogical

Association (IMA 2013-085). The holotype specimen (in several pieces) is deposited in the collections of the Natural History Museum of Los Angeles County, 900 Exposition Boulevard, Los Angeles, CA 90007, USA, catalogue number: 64072.

OCCURRENCE

The mineral occurs at the Gun claim ($62^{\circ}50'50''\text{N}$ $130^{\circ}0'51''\text{W}$) in a cirque 4 km SE of Wilson Lake along the Ross River head waters and immediately south of the Itsi Range, Yukon Territory, Canada. The Gun claim was originally staked for its Zn content in 1957 by the Newmont Mining Corporation (Montgomery 1960). It is the type locality for pellyite (Montgomery *et al.* 1972, Meagher 1976). The holotype specimen of the new mineral was collected by H. Gary Ansell and Ian Jonasson on July 15, 1984, during an expedition for the Geological Survey of Canada (GSC) and was assigned catalogue number 064493 in the Canadian National Mineral Collection. As noted above, the specimen was later acquired by Forrest Cureton, who recently provided it to us for further study. The GSC documentation provided with the specimen indicates that it was collected from Trench 1 located at $62^{\circ}50'45''\text{N}$ $130^{\circ}0'20''\text{W}$.

The Gun claim is a Ba-rich skarn deposit associated with a quartz monzonite (McNeil *et al.* 2013). The mineralization is similar to that in a number of other barium silicate deposits in western North



FIG. 1. Itsi Range looking NNW from the Gun claim.

America (Alfors & Pabst 1984), the most well-known of which are those in eastern Fresno County, California (Walstrom & Leising 2005). The minerals found at the Gun claim include alforsite, barite, cerchiaraita-(Al), cerchiaraita-(Fe) (Kampf *et al.* 2013), diopside, gillespite, pellyite, pyrite, quartz, sanbornite, sphalerite, taramellite, and witherite. The new mineral occurs in low temperature, late stage veins in direct association with cerchiaraita-(Fe), diopside, pyrite, quartz, sphalerite, and witherite. Crystals of the new mineral are commonly heavily included with cerchiaraita-(Fe).

PHYSICAL AND OPTICAL PROPERTIES

Itsiite occurs as intergrowths of colorless and light blue to medium greenish-blue tetragonal plates up to 1 mm across (Fig. 2). The plates are flattened on {001} and exhibit the forms {001}, {101}, and {112} (Fig. 3); the forms {101} and {112} are generally stri-

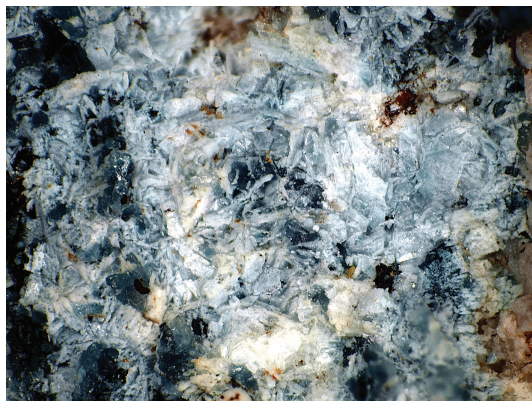


FIG. 2. White to light greenish-blue itsiite plates with darker greenish-blue quartz crystals. The greenish-blue color in both cases is due to inclusions of cerchiaraita-(Fe); FOV = 4 mm.

ated. The mineral is transparent, has a vitreous luster, and is non-fluorescent. It has a white streak and Mohs hardness of approximately 5½ based on scratch tests. It is brittle, with splintery fracture, and has one perfect cleavage on {001}. The density could not be measured because the crystals are generally heavily included with cerchiaraita. The calculated density based upon the empirical formula and single-crystal cell is 3.644 g/cm³. The mineral is optically uniaxial (-), with the following optical constants measured in white light: $\omega = 1.623(1)$, $\epsilon = 1.619(1)$. The mineral is nonpleochroic. Crystals slowly decompose in concentrated HCl (about 1 h) and are unreactive in concentrated H₂SO₄ (days).

CHEMICAL COMPOSITION

Chemical analyses (3) were obtained at Queen's University using a JEOL JXA-8230 electron microprobe (WDS mode, 15 kV, 10 nA, 5 µm beam diameter). The presence of B was confirmed by EMPA, but accurate analyses of B could not be made. Consequently, B was calculated from the structure determination (Si + B = 6 and O = 14). The structure determination clearly shows the mineral to be free of H₂O and CO₂. Analytical data are given in Table 1.

The empirical formula (based on 14 O *apfu*) is Ba_{2.06}(Ca_{0.89}Al_{0.02}Na_{0.01}Fe_{0.01}Ti_{0.01})_{Σ0.94}(Si_{3.96}B_{2.04})_{Σ6.00}O₁₄. The simplified structural formula is Ba₂Ca(BSi₂O₇)₂, which requires BaO 45.59, CaO 8.34, B₂O₃ 10.35, SiO₂ 35.73, total 100 wt.%.

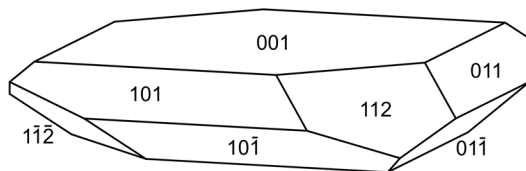


FIG. 3. Crystal drawing of itsiite (clinographic projection in standard orientation).

TABLE 1. ANALYTICAL RESULTS FOR ITSIIITE

Constituent	wt.%	Range	SD	Standard
Na ₂ O	0.06	0.03–0.09	0.03	albite
BaO	46.35	46.09–46.50	0.23	barite
CaO	7.35	7.24–7.49	0.13	anorthite glass
FeO	0.15	0.07–0.20	0.07	syn. fayalite
Al ₂ O ₃	0.17	0.15–0.21	0.03	adularia
TiO ₂	0.06	0.01–0.10	0.05	rutile
SiO ₂	34.91	34.85–35.01	0.09	adularia
B ₂ O ₃	10.41*			
Total	99.46			

*Calculated based upon the structure.

The Gladstone-Dale compatibility index, $1 - (K_P/K_C)$, as defined by Mandarino (1981), provides a measure of the consistency among the average index of refraction, calculated density, and chemical composition. For itsite, the compatibility index is 0.006 (superior) based on the empirical formula.

X-RAY CRYSTALLOGRAPHY AND STRUCTURE DETERMINATION

Both powder and single-crystal X-ray studies were carried out using a Rigaku R-Axis Rapid II curved imaging plate microdiffractometer, with monochromatized $\text{MoK}\alpha$ radiation. For the powder-diffraction study, observed values of d and intensities were derived by profile fitting using the JADE 2010 software (Materials Data, Inc.). The powder data presented in Table 2 show good agreement with the calculated powder data based upon a whole-pattern-fitting Rietveld structure refinement using JADE 2010. Unit-cell parameters refined from the powder data are a 10.936(2), c 10.297(2) Å, and V 1231.4(4) Å³.

The Rigaku CrystalClear software package was used for processing the diffraction data, including the application of an empirical multi-scan absorption correction using ABSCOR (Higashi 2001). The structure was

solved by direct methods using SIR2004 (Burla *et al.* 2005). SHELXL-2013 (Sheldrick 2008) was used for the refinement of the structure. All cation sites (Ba, Ca, Si, and B) refined to full occupancy with their dominant constituents. In particular, it should be noted that there is no indication of any significant B occupancy at the Si site or any significant Si (or Be) occupancy at the B site. Bond valence summations and bond distances provide further corroboration for these site assignments.

The details of the data collection and the final structure refinement are provided in Table 3. The final coordinates and displacement parameters of the atoms are provided in Table 4. Selected interatomic distances are listed in Table 5 and a bond valence analysis in Table 6. Tables of observed and calculated structure-factors are available from the Depository of Unpublished data on the Mineralogical Association of Canada website [document Itsite CM52_401].

ATOMIC ARRANGEMENT

The crystal structure (Fig. 4) is based upon a zeolite-like tetrahedral framework of corner-sharing tetrahedra consisting of four-membered silicate rings alternating with four-membered borate rings. The framework contains channels along each axis that host Ba^{2+} in

TABLE 2. POWDER X-RAY DATA ($\text{MoK}\alpha$) FOR ITSITE*

I_{obs}	$d_{\text{obs}}(\text{Å})$	$d_{\text{calc}}(\text{Å})$	I_{calc}	hkl	I_{obs}	$d_{\text{obs}}(\text{Å})$	$d_{\text{calc}}(\text{Å})$	I_{calc}	hkl
17	7.73	7.7439	8	1 1 0	5	1.9358	1.9360	5	4 4 0
		7.5044	4	1 0 1	12	1.8809	1.8782	7	5 3 0
42	5.50	5.4758	33	2 0 0			1.8761	4	4 0 4
4	5.16	5.1519	4	0 0 2			1.8467	9	5 0 3
18	4.411	4.4234	9	2 1 1	41	1.8257	1.8234	15	3 3 4
5	4.288	4.2894	8	1 1 2			1.7946	9	3 0 5
100	3.764	3.7522	100	2 0 2			1.7748	3	4 2 4
60	3.446	3.4409	46	3 0 1	43	1.7584	1.7646	13	5 3 2
51	3.100	3.0952	45	2 2 2			1.7499	20	5 2 3
		2.9135	40	3 2 1	14	1.7006	1.7053	8	3 2 5
96	2.899	2.8742	33	3 1 2			1.6873	5	5 4 1
		2.8121	9	2 1 3	9	1.6403	1.6386	9	2 0 6
17	2.748	2.7379	15	4 0 0	3	1.5694	1.5698	5	2 2 6
		2.5813	9	3 3 0			1.5488	3	5 5 0
30	2.583	2.5760	17	0 0 4	13	1.5467	1.5468	5	7 0 1
14	2.504	2.5015	14	3 0 3			1.5385	4	3 1 6
4	2.448	2.4488	4	4 2 0			1.5176	8	5 3 4
		2.3309	10	2 0 4	22	1.5138	1.5009	8	5 0 5
12	2.327	2.3078	4	3 3 2			1.4885	6	7 2 1
44	2.279	2.2753	36	3 2 3	29	1.4860	1.4832	15	5 5 2
7	2.214	2.2117	8	4 2 2			1.4475	5	5 2 5
		2.1478	3	5 1 0	20	1.4400	1.4380	6	7 3 0
		2.1447	14	2 2 4			1.4238	5	7 0 3
69	2.145	2.1424	37	5 0 1	6	1.4127	1.4097	3	2 1 7
		2.0252	3	1 0 5			1.4060	3	4 2 6
29	1.9978	1.9952	21	5 2 1	10	1.3780	1.3779	9	7 2 3

*Only calculated lines with intensities of 3 or greater are listed.

TABLE 3. DATA COLLECTION AND STRUCTURE REFINEMENT DETAILS FOR ITSITE

Diffractometer	Rigaku R-Axis Rapid II
X-ray radiation/power	MoK α ($\lambda = 0.71075 \text{ \AA}$)/50 kV, 40 mA
Temperature	298(2) K
Structural Formula	Ba ₂ Ca(BSi ₂ O ₇) ₂
Space group	<i>I</i> 42 <i>m</i>
Unit cell dimensions	<i>a</i> = 10.9515(5) \AA <i>c</i> = 10.3038(7) \AA
<i>V</i>	1235.79(14) \AA^3
<i>Z</i>	4
Density (for above formula)	3.616 g cm ⁻³
Absorption coefficient	7.217 mm ⁻¹
<i>F</i> (000)	1240
Crystal size	150 x 100 x 25 μm
θ range	3.96 to 30.46°
Index ranges	$-12 \leq h \leq 13, -15 \leq k \leq 12, -14 \leq l \leq 14$
Refls collected / unique	4224 / 1008; <i>R</i> _{int} = 0.023
Reflections with <i>F</i> _o > 4 σ (<i>F</i>)	992
Completeness to $\theta = 30.46^\circ$	98.0%
Max. and min. transmission	0.840 and 0.411
Refinement method	Full-matrix least-squares on <i>F</i> ²
Parameters refined	58
GoF	1.152
Final <i>R</i> indices [<i>F</i> _o > 4 σ (<i>F</i>)]	<i>R</i> ₁ = 0.0177, <i>wR</i> ₂ = 0.0381
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0182, <i>wR</i> ₂ = 0.0384
Absolute structure parameter	-0.009(11)
Largest diff. peak / hole	+1.82 / -0.57 e/ \AA^3

**R*_{int} = $\sum |F_o^2 - F_o^2(\text{mean})| / \sum [F_o^2]$. GoF = $S = \{ \sum [w(F_o^2 - F_c^2)^2] / (n-p) \}^{1/2}$. *R*₁ = $\sum ||F_o| - |F_c|| / \sum |F_o|$.
*wR*₂ = $\{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$; $w = 1 / [\sigma^2(F_o^2) + (aP)^2 + bP]$ where *a* is 0.0158, *b* is 1.238 and *P* is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$.

TABLE 4. ATOM COORDINATES AND DISPLACEMENT PARAMETERS (\AA^2) FOR ITSITE

	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	<i>U</i> _{eq}	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₂₃	<i>U</i> ₁₃	<i>U</i> ₁₂
Ba	0.20510(2)	0.20510(2)	0.99881(3)	0.00998(9)	0.00937(10)	0.00937(10)	0.01120(12)	0.00087(11)	0.00087(11)	-0.00020(9)
Ca	0	0	0.24310(11)	0.0092(2)	0.0077(3)	0.0077(3)	0.0121(5)	0.000	0.000	-0.0012(7)
Si	0.51470(9)	0.20071(8)	0.23644(8)	0.00622(17)	0.0052(5)	0.0047(4)	0.0088(3)	0.0003(3)	0.0004(3)	-0.0004(4)
B	0.5	0.3320(4)	0	0.0044(7)	0.0022(19)	0.0053(19)	0.0055(16)	0.000	0.001(3)	0.000
O1	0.4056(2)	0.4056(2)	0.0665(3)	0.0075(7)	0.0069(12)	0.0069(12)	0.0087(18)	0.0006(9)	0.0006(9)	0.0018(12)
O2	0.5605(2)	0.2525(3)	0.0980(2)	0.0100(5)	0.0081(12)	0.0103(12)	0.0114(11)	0.0036(10)	0.0015(9)	0.0014(11)
O3	0.4973(3)	0.3022(2)	0.3454(2)	0.0112(4)	0.0128(11)	0.0093(11)	0.0117(9)	-0.0014(8)	-0.0019(13)	0.0031(18)
O4	0.6233(3)	0.1106(3)	0.2880(2)	0.0140(5)	0.0146(14)	0.0119(13)	0.0154(12)	0.0010(11)	-0.0030(11)	0.0059(10)

TABLE 5. SELECTED BOND DISTANCES (\AA) FOR ITSITE

Ba–O3 (×2)	2.724(3)	Ca–O3 (×4)	2.350(2)	Si–O3	1.592(2)	B–O1 (×2)	1.479(3)
Ba–O3 (×2)	2.786(3)	Ca–O1 (×2)	2.446(4)	Si–O2	1.615(3)	B–O2 (×2)	1.489(4)
Ba–O2 (×2)	2.802(3)	<Ca–O>	2.382	Si–O4	1.633(3)	<B–O>	1.484
Ba–O4 (×2)	3.114(3)			Si–O4	1.634(3)		
Ba–O1	3.183(4)			<Si–O>	1.619		
<Ba–O>	2.893						

TABLE 6. BOND-VALENCE ANALYSIS FOR ITSITE*

	O1	O2	O3	O4	Σ_c
Ba	0.09	0.25 $\times 2 \rightarrow$	0.31 $\times 2 \rightarrow$ 0.26 $\times 2 \rightarrow$	0.11 $\times 2 \rightarrow$	1.95
Ca	0.27 $\times 2 \rightarrow$		0.36 $\times 4 \rightarrow$		1.98
Si		1.02	1.09	0.98 0.97	4.06
B	0.75 $\times 2 \downarrow \rightarrow$	0.73 $\times 2 \rightarrow$			2.96
Σ_a	1.86	2.00	2.02	2.06	

*Values are expressed in valence units.

Multiplicities indicated by $\times \downarrow \rightarrow$; $Ba^{2+}-O$, $Ca^{2+}-O$, and $B^{3+}-O$ bond strengths from Brown & Altermatt (1985); $Si^{4+}-O$ bond strengths from Brese & O'Keeffe (1991).

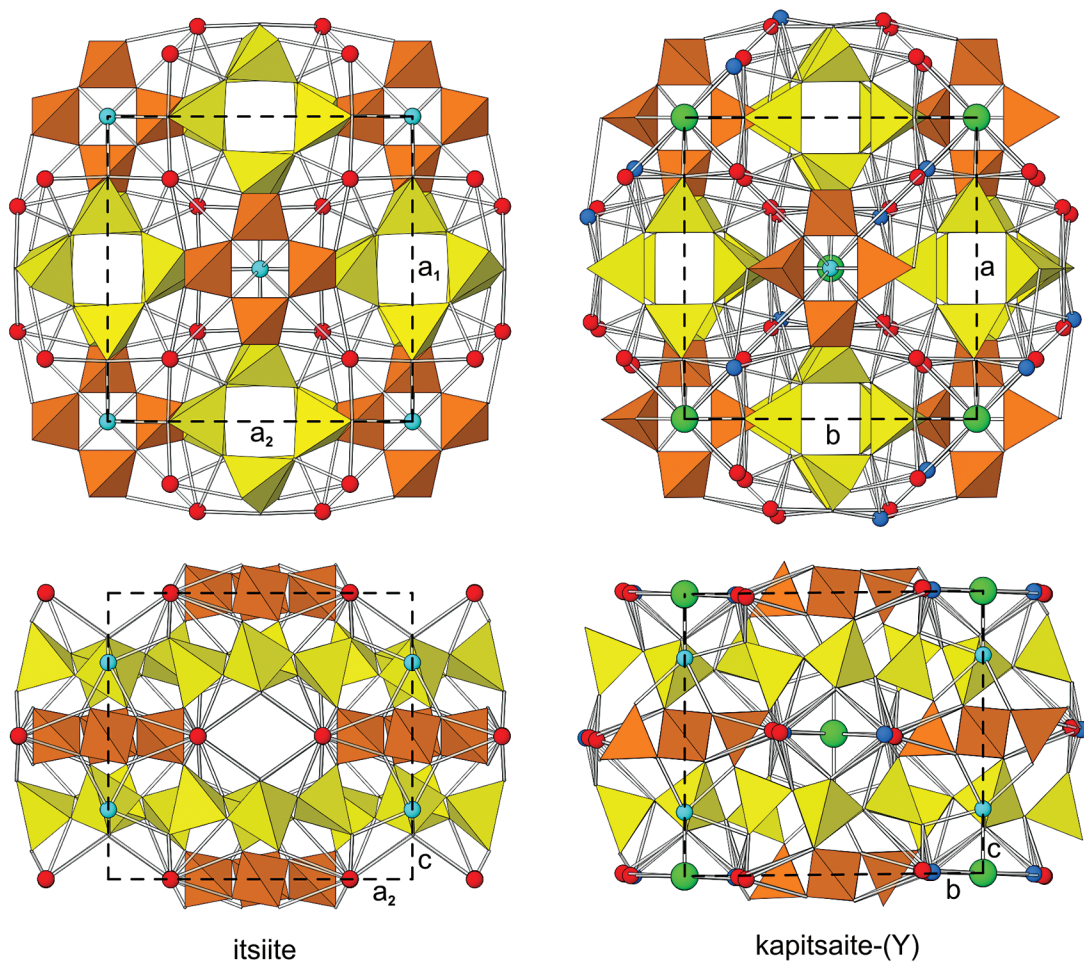


FIG. 4. Structures of itsiite and kapitsaite-(Y). SiO_4 tetrahedra are yellow, BO_4 tetrahedra are orange, Ca atoms in itsiite and Y atoms in kapitsaite-(Y) are light blue, Ba atoms are red, Pb atoms in kapitsaite-(Y) are dark blue and F atoms kapitsaite-(Y) are green.

ninefold coordination and Ca^{2+} in sixfold coordination. The structure is very similar to those of hyalotekite, $(\text{Ba}, \text{Pb}^{2+}, \text{K})_4(\text{Ca}, \text{Y})_2[\text{Si}_8(\text{B}, \text{Be})_2(\text{Si}, \text{B})_2\text{O}_{28}\text{F}]$ (Moore *et al.* 1982, Christy *et al.* 1998), and kapitsaite-(Y), $(\text{Ba}, \text{K}, \text{Pb}^{2+}, \text{Na})_4(\text{Y}, \text{Ca}, \text{REE})_2[\text{Si}_8\text{B}_2(\text{B}, \text{Si})_2\text{O}_{28}\text{F}]$ (Sokolova *et al.* 2000) (Fig. 4).

The structures of hyalotekite and kapitsaite-(Y) differ from that of itsiite in four principal ways. The structure of itsiite is tetragonal ($I42m$), while those of hyalotekite and kapitsaite-(Y) are triclinic ($\bar{1}\bar{1}$). The B site in the itsiite structure is split into two separate sites in the hyalotekite and kapitsaite-(Y) structures, with these sites occupied by differing amounts of B and Si (as well as Be in hyalotekite). The Ba site in itsiite is split into two separate sites in the hyalotekite and kapitsaite-(Y) structures, with one site containing Ba and the other Pb. Finally, an additional channel site at the origin (0,0,0) in the hyalotekite and kapitsaite-(Y) structures is fully occupied by F. Note that we specifically sought F in our EMPA and found none. The largest electron density residual ($+1.82 \text{ e } \text{\AA}^{-3}$) in our structure refinement is located at (0,0,0), 2.50 \AA from the Ca site, but when F is assigned to this site its occupancy refines to 0.070(15), and when O is assigned to this site its occupancy refines to 0.084(18). While it is possible that a small amount of O (as OH or H_2O) or F is hosted at this site in the itsiite structure, the site is clearly predominantly vacant. Furthermore, the presence of a significant amount of either O or F at (0,0,0) in the itsiite structure is also inconsistent with the bond-valence analysis, which provides a BVS of 1.98 *vu* for the Ca site; however, it is worth noting that some substitution by a trivalent cation, *e.g.*, Y^{3+} , at the Ca site, could apparently be accommodated by adding some OH or F at (0,0,0).

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