Miyahisaite, (Sr,Ca)₂Ba₃(PO₄)₃F, a new mineral of the hedyphane group in the apatite supergroup from the Shimoharai mine, Oita Prefecture, Japan

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Miyahisaite, $(Sr,Ca)_2Ba_3(PO_4)_3F$, a new mineral of the hedyphane group in the apatite supergroup, is found in the Shimoharai mine, Oita Prefecture, Japan. Miyahisaite is colorless and occurs as a pseudomorphic aggregate (up to about 100 µm in size) along with fluorapatite in the quartz matrix in a namansilite-rich layer of the chert. Its hardness is 5 on the Mohs scale, and its calculated density is 4.511 g/cm³. The empirical formula of miyahisaite is $(Sr_{1.366}Ca_{0.717})_{\Sigma 2.083}Ba_{2.911}P_{3.002}O_{12}(F_{0.898}OH_{0.088}Cl_{0.014})_{\Sigma 1.00}$, which is representatively shown as $(Sr,Ca)_2$ Ba₃(PO₄)₃F. Its simplified ideal formula is written as $Sr_2Ba_3(PO_4)_3F$, which requires 23.25 wt% SrO, 51.62 wt% BaO, 23.89 wt% P₂O₅, 2.13 wt% F, and -0.90 wt% F = O, for a total of 100.00 wt%. The mineral is hexagonal with a space group *P6₃/m*, unit cell parameters *a* = 9.921 (2) Å, *c* = 7.469 (3) Å, and *V* = 636.7 (3) Å³, and *Z* = 2. The eight strongest lines in the powder XRD pattern [*d* (Å), (*I*/*I*₀), *hkI*] are 3.427 (16) 102, 3.248 (22) 120, 2.981 (100) 121, 2.865 (21) 300, 1.976 (23) 123, 1.874 (16) 140, 1.870 (15) 004, and 1.864 (17) 402. The mineral was formed by the reaction between fluorapatite and the Ba-bearing fluid that produced the aegirine-rich layer with hydrous Ba-rich minerals during the late-stage activity.

Keywords: Miyahisaite, New mineral, Hedyphane group, Apatite supergroup, Shimoharai mine

INTRODUCTION

Apatite supergroup minerals are found in many geological settings and make up an important class of rock-forming minerals. The standard formula for the apatite supergroup minerals is ${}^{\text{IX}}M1_2 {}^{\text{VII-IX}}M2_3 (TO_4)_3 X$, and they are formally divided into five groups based on their crystallographic and chemical features: the apatite, hedyphane, belovite, britholite, and ellestadite groups. Although the minerals can incorporate various M-type cations such as Ca, Sr, Ba, and Pb, the cation size mismatch causes site splitting and cation ordering (Pasero et al., 2010). *M*-type cations are disordered and ordered in the apatite and hedyphane groups, respectively, since large cations are placed in the M2 site after the M1 site is filled by the smaller cations in the $P6_3/m$ structure. Indeed, Ca and Pb ordering in the structures of the hedyphane group due to the cation size mismatch and Ca-Sr ordering are also expected to potentially produce new mineral species in the hedyphane group. In the present study, we found a new Sr-Ba fluorophosphate, (Sr,Ca)₂Ba₃(PO₄)₃F, from the Shimoharai

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mine, Oita Prefecture, Japan. Considering the allocation of ions to different sites in apatite supergroup minerals, this mineral is understood to be a new member of the hedyphane group in the apatite supergroup. This new mineral is named "miyahisaite" in honor of Professor Michitoshi Miyahisa (1928-1983), who contributed greatly to the advancement of mineralogy, petrology, and economic geology in Japan through his research on many ore deposits on Kyushu. The data and name were approved by the IMA Commission on New Minerals, Nomenclature and Classification (IMA2011-043). The type specimen of miyahisaite has been stored at the National Museum of Nature and Science, Tokyo, Japan (NSM M-41299). Here, we describe the new mineral, miyahisaite.

OCCURRENCE

Many manganese ore deposits are embedded in the chert of Triassic to Jurassic age distributed in the Chichibu belt of the Saiki area, Oita, Japan (Yoshimura, 1969). A lowgrade metamorphism of prehnite-pumpellyite to actinolite facies has been recognized in this area (Imai et al., 1971). The Shimoharai mine exploits one of the bedded manganese deposits in the chert and is located in the Yayoi Udoki area in Saiki City. Several ore deposits were also found in the same chert block (Yoshimura, 1969), and unique Na-Mn pyroxene and Ba-rich minerals have previously been found in the chert from the Shimoharai mine (Minakawa et al., 1994). The chert from the Shimoharai mine is divided into three layers: black braunite-rich, purple namansilite-rich, and brown aegirine-rich layers (Fig. 1; Color version of Figure 1 is



Figure 1. Photograph of miyahisaite-bearing chert from the Shimoharai mine, Oita Prefecture, Japan. The chert consists mainly of quartz and is divided into three layers: black braunite (Brn)-rich, purple namansilite (Nm)-rich, and brown aegirine (Aeg)-rich layers. Miyahisaite occurs in the namansilite-rich layer. Color version of Figure 1 is available online from http://joi.jlc. jst.go.jp/JST.JSTAGE/jmps/110901. available online from http://joi.jlc.jst.go.jp/JST.JSTAGE/ jmps/110901). The braunite-rich layer is the main manganese ore of the Shimoharai mine, and its constituents are braunite and quartz in a matrix. Namansilite is an uncommon mineral in general in bedded manganese deposits, while it is a principal constituent of the chert layer from the Shimoharai mine and is scattered in the quartz matrix. A small amount of fluorapatite is also found only in this layer. The aegirine-rich layer crosses the other layers as a vein and includes a negligible amount of quartz, while its main constituents are aegirine and cymrite. Rare Ba-rich minerals such as strakhovite, benitoite, and witherite have been also found in this layer. Noélbensonite, a quite rare Ba-rich mineral, also occurs in the aegirinerich layer, and it forms aggregates of up to 5 mm in size at the boundary between the aegirine-rich layer and the other layers (Minakawa, 1995). After the layer formation, a several-millimeter-thick quartz-rich vein crosses the chert, and arfvedsonite with high K and Mn contents also occurs in this quartz-rich vein. The details of such rare minerals will be reported after future studies.

Miyahisaite, a fluorophosphate of Ba and Sr, occurs in the namansilite-rich layer, although the other Ba-rich minerals occur in the aegirine-rich layer. It is scattered in the namansilite-rich layer along its boundary with the aegirine-rich layer. Miyahisaite occurs in anhedral grains with submicron to 10 μ m sizes and forms aggregates up to about 100 μ m in size. It is closely associated with fluorapatite in the quartz matrix of the namansilite-rich layer (Fig. 2). The miyahisaite partially or completely



Figure 2. Backscattered electron images of miyahisaite (Mh). Miyahisaite occurs as aggregates with fluorapatite up to about 100 µm in size in the quartz matrix in the namansilite-rich layer.

replaces the fluorapatite, and relict fluorapatite pieces are often found in the miyahisaite aggregates (Fig. 2). No significant association with other minerals was found in the investigated occurrences of miyahisaite.

APPEARANCE AND PHYSICAL PROPERTIES

Miyahisaite is colorless white in powder, and its luster is

Table 1. Representative chemical composition of miyahisaite

	wt%	Range(4)
CaO	4.69	2.95-4.69
SrO	16.51	16.49-16.67
BaO	52.05	52.05-54.27
P ₂ O ₅	24.85	24.38-24.98
F	1.99	1.61-1.99
Cl	0.06	0.04-0.06
-0≡F + Cl	-0.85	
H_2O^*	0.09	
Total	99.39	
	O = 12	
Ca	0.717	
Sr	1.366	
Ba	2.911	
Р	3.002	
Total	7.997	
F	0.898	
Cl	0.014	
OH	0.088	
Total	1	

 H_2O is calculated as OH + F + Cl = 1.

vitreous and transparent. No fluorescence was observed under long- or short-wave ultraviolet radiation. Its Mohs hardness was measured to be 5 by scratch tests performed on the aggregates using small pieces of fluorite, apatite, and orthoclase feldspar. The density was calculated to be 4.511 g/cm³ using an empirical formula. Other physical and optical properties could not be measured or observed because of the small grain size.

CHEMICAL COMPOSITION

The chemical analyses of miyahisaite were carried out using a JEOL JSM-5400 electron microprobe with an Oxford WDS 400 wavelength-dispersive spectroscopy (WDS; 15 kV, 10 nA, beam diameter 1-2 μ m). The standard materials used were CaSiO₃, SrF₂, BaF₂, GaP, and NaCl for Ca, Sr and F, Ba, P, and Cl, respectively. Energy-dispersive spectroscopy (EDS) analyses (15 kV, 0.4-0.8 nA, beam diameter 1-2 μ m) were also carried out to obtain the Ba-Sr-Ca ratio of miyahisaite.

The representative chemical composition determined by WDS is listed in Table 1. The H₂O content was calculated as OH + F + Cl = 1 per formula unit by analogy with the apatite group minerals. The simplified and ideal formulae are different from each other: the empirical formula is $(Sr_{1.366}Ca_{0.717})_{\Sigma 2.083}Ba_{2.911}P_{3.002}O_{12}(F_{0.898}OH_{0.088}Cl_{0.014})_{\Sigma 1.00}$, leading a simplified formula of $(Sr,Ca)_2$ $Ba_3(PO_4)_3F$, and the ideal formula is written as Sr_2Ba_3 $(PO_4)_3F$, which requires 23.25 wt% SrO, 51.62 wt% BaO, 23.89 wt% P₂O₅, 2.13 wt% F, and -0.90 wt% F = O, for a total of 100.00 wt%. Miyahisaite is a Sr- and Ba-



Figure 3. X-ray diffraction pattern for miyahisaite. Bars below the diffraction pattern indicate the peak positions after the refinement of the unit cell parameters for miyahisaite (top), fluorapatite (middle), and quartz (bottom).

Table 2. Powder XRD data for miyahisaite

h k l	<i>I/I</i> ₀	d _{obs.}	d calc.	$d_{\rm obs}/d_{\rm calc.}$ -1	h k l	<i>[/]</i> ₀	d _{obs.}	d calc.	$d_{\rm obs}/d_{\rm calc}$ -1
100			8.5927		304	4.8	1.5658	1.5642	0.00102
101			5.6371		502	6.8	1.5617	1.5612	0.00032
110	0.3	4.9547	4.9610	-0.00127	233	7.0	1.5459	1.5455	0.00026
200	13.6	4.2979	4.2963	0.00037	150			1.5433	
111	14.0	4.1336	4.1325	0.00027	332	7.8	1.5115	1.5121	-0.00040
002	14.9	3.7359	3.7345	0.00038	151			1.5114	
201			3.7242		143	0.6	1.4978	1.4978	0.00000
102	16.3	3.4273	3.4250	0.00067	224			1.4918	
120	22.3	3.2478	3.2477	0.00003	242	0.8	1.4895	1.4892	0.00020
112			2.9836		105			1.4717	
121	100.0	2.9805	2.9784	0.00071	134	0.7	1.4700	1.4698	0.00014
300	20.8	2.8645	2.8642	0.00010	115			1.4321	
202	3.8	2.8219	2.8186	0.00117	600			1.4304	
301			2.6743		152	0.7	1.4260	1.4263	-0.00021
220			2.4805		503			1.4143	
122			2.4507		340			1.4126	
103			2.3913		205			1.4110	
130	7.9	2.3834	2.3832	0.00007	404	1.3	1.4118	1.4093	0.00177
221	1.1	2.3543	2.3541	0.00008	601	1.6	1.4074	1.4065	0.00064
302			2.2727		341	2.4	1.3875	1.3880	-0.00036
131			2.2704		333			1.3775	
113	7.8	2.2267	2.2252	0.00067	250			1.3759	
203	3.6	2.1572	2.1541	0.00144	243			1.3601	
400	5.6	2.1483	2.1482	0.00005	125	5.0	1.3584	1.3571	0.00096
222	14.9	2.0664	2.0662	0.00011	234			1.3556	
401			2.0645		251	5.9	1.3549	1.3532	0.00126
132	11.1	2.0091	2.0090	0.00006	602	2.2	1.3367	1.3372	-0.00037
123	22.6	1.9764	1.9759	0.00024	305			1.3245	
230			1.9713		144	5.1	1.3229	1.3231	-0.00015
231	10.8	1.9060	1.9060	-0.00003	342			1.3213	
303	1.5.4		1.8790		153	~ 1		1.3117	
140	15.6	1.8741	1.8751	-0.00053	160	3.4	1.3111	1.3104	0.00052
004	15.3	1.8704	1.8673	0.00166	252	7.3	1.2897	1.2911	-0.00108
402	17.3	1.8635	1.8621	0.00075	161			1.2907	
104			1.8247		225			1.2797	
141			1.8186		135	0.0	1 2 4 2	1.2657	0.00004
223			1.7572		504	0.9	1.2042	1.2645	-0.00024
114	0.4	1 7420	1.7470	0.00022	006	0.7	1.2405	1.2448	0.00137
232	0.4	1./429	1.7433	-0.00023	603	2.4	1.2413	1.2414	-0.00008
500			1.7210		440	2.9	1.2400	1.2402	-0.00019
204	1.4	1 7150	1./103	0.00100	334			1.2360	
204	1.4	1./139	1.7123	0.00199	102			1.2303	
142	5.7	1.0/23	1.0737	-0.00204	2 4 3	2.0	1 2295	1.2320	0 00008
220			1.0740		343 350±700	5.9 4 1	1.2203	1.2200	-0.00008
103			1.0337		3307700	4.1	1.2209	1.2275	-0.00049
210			1.0204		4 U J 2 A A	3 /	1 2258	1.2204	0.00041
1240	62	1 6221	1.0237	0.00204	<u> </u>	5.4	1.2230	1.2233	0.00041
124	0.Z 9 1	1.0221	1.0100	0.00204	441 701-251	12	1 2107	1.2233	-0.00050
2 / 1	0.1	1.0101	1.0140	0.00093	116	1.2	1.210/	1.2113	-0.00030
241	1.0	1.300/	1.3008	-0.0004	110			1.20/4	
a = 9.921	(2) Å, $c =$	• 7.469(3) Å	V = 636.8(3)) A'					

dominant fluorophosphate member of the hedyphane group of minerals in the apatite supergroup.

CRYSTALLOGRAPHY

The angle-dispersive X-ray diffraction (XRD) pattern for

miyahisaite was collected using the synchrotron X-ray source on the NE1 beam line of PF-AR, KEK, Japan. This beam line provides a 30-µm-diameter collimated beam of monochromatized X-ray radiation ($\lambda = 0.4111$ Å). The XRD spectra were collected using the Debye-Scherrer method and recorded using an imaging plate detector. The sample-to-detector distance and inclination of the detector were calibrated using a standard material (CeO₂). Two-dimensional XRD images were integrated with respect to 2 θ to obtain conventional one-dimensional diffraction profiles using the IPAnalyzer and PDIndexer software developed by Seto et al. (2010).

The collimated X-rays irradiated the miyahisaite aggregate on the slide glass from the slide side. Although the background intensities were high because of the slide, the diffraction pattern from miyahisaite was clearly observed, along with those of fluorapatite and quartz. The X-ray diffraction pattern after the subtraction of the background is shown in Figure 3, and the XRD data are listed in Table 2. The eight strongest lines in the powder XRD pattern $[d(Å), (I/I_0), hk]$ are 3.427 (16) 102, 3.248 (22) 120, 2.981 (100) 121, 2.865 (21) 300, 1.976 (23) 123, 1.874 (16) 140, 1.870 (15) 004, and 1.864 (17) 402. The XRD peaks obtained for miyahisaite can be indexed using the hexagonal $P6_3/m$ structure of the apatite supergroup, the unit cell parameters are a = 9.921 (2) Å, c = 7.469 (3) Å, and V = 636.7 (3) Å³, and Z = 2. The c/aratio calculated from the unit cell parameters is 0.753.

DISCUSSION

Miyahisaite is one of the fluorophosphate members in the apatite supergroup and is characterized by high amounts of Ba and Sr with a small amount of Ca as $(Sr,Ca)_2Ba_3$ $(PO_4)_3F$. Miyahisaite would be compositionally indistinguishable from the Ba and F analogs of stronadelphite $Sr_5(PO_4)_3F$ and alforsite $Ba_5(PO_4)_3Cl$ of the apatite group (Table 3), respectively. if it was described as $(Ba,Sr,Ca)_5$ $(PO_4)_3F$. However, miyahisaite shows less chemical variation, and its compositions lie on the line indicating

 Table 3. Comparative data on miyahisaite and other related minerals of the apatite supergroup

Name	Miyahisaite	Alforsite	Stronadelphite	
Ideal formula	Sr ₂ Ba ₃ (PO ₄) ₃ F	Ba ₅ (PO ₄) ₃ Cl	Sr ₅ (PO ₄) ₃ F	
Subgroup	Hedyphane	Apatite	Apatite	
a/Å	9.921(2)	10.25	9.845	
c/Å	7.469(3)	7.64	7.383	
Reference	This study	1	2	

1, Newberry et al., (1981); 2, Pekov et al., (2010).

(Sr + Ca):Ba = 2:3 (Fig. 4). Since the standard formula for the apatite supergroup minerals is ${}^{IX}M1_2^{VII-IX}M2_3$ $(TO_4)_{2}X$, the compositional ratio of miyahisaite is in harmony with the supergroup's cation sites, as Sr + Ca and Ba occupy the M1 and M2 sites, respectively. The disorder and the order of the M-type cations are individually distinguished to separate groups such as the apatite (disordered) and the hedyphane (ordered) group. even in the same symmetry (Pasero et al., 2010). The cation size mismatch causes the ordering, because the M1 site is small and the M2 site is large, and small Ca and large Pb cations are ordered in the structures such as those of the hedyphane group. Ca-Sr ordering is also expected to produce potentially new mineral species in the hedyphane group due to this cation size mismatch (Pasero et al., 2010). Figure 5 shows the ionic size ratios among Ca, Sr, Ba, and Pb for different coordination numbers based on results obtained by Shannon and Prewitt (1969).



Figure 4. Chemical variation of miyahisaite in the Ca-Sr-Ba system. The dotted line indicates (Sr + Ca):Ba = 2:3. Miyahisaite shows little chemical variation, and the compositions follow the dotted line.



Figure 5. Plot of ionic ratios for different coordination numbers based on the effective ionic radii obtained by Shannon and Prewitt (1969).

The *M* sites in the apatite structure have seven to nine coordinated sites, and the Ba/Sr ratios are similar to the Sr/Ca ratios for these coordination numbers. This suggests that Ba and Sr may also prefer to be ordered in the apatite structure. On the other hand, the Sr/Ca ratio is considerably low in nine-coordinated sites such as the M1 site. This also suggests that a small amount of Ca can be easily accommodated in the M1 site even if Sr is dominant in the M1 site. According to the allocation of ions to sites in the apatite supergroup minerals, where the large cation is placed in the M2 site after the M1 site is filled by small cations (Pasero et al., 2010), miyahisaite, (Sr.Ca)₂Ba₃ $(PO_4)_3F$, is considered to be a member of the hedyphane group with an ordered structure rather than the apatite group with a disordered structure, since Sr + Ca and Ba may occupy the M1 and the M2 sites, respectively.

Miyahisaite partially or completely replaces fluorapatite and occurs only in the boundary area between the namansilite- and aegirine-rich layers, while fluorapatite is scattered throughout the whole namansilite-rich layer and was formed from radiolarians during the chert formation. The aegirine-rich layer crosses the namansilite-rich layer and includes hydrous Ba-rich minerals as the main constituent. Miyahisaite may have been formed by the reactions between fluorapatite and the fluid that caused the formation of the aegirine-rich layer during the latestage activity. Although the intrusion of Ba-rich fluid is quite unique even in manganese deposits, which often show a wide variety, miyahisaite might be found in a similar geological setting. A similar deposit to this one at the Shimoharai mine has been found at the Shiromaru mine, Tokyo, Japan. The Shiromaru mine deposit is located in the Chichibu belt of Jurassic age, consists of bedded manganese chert in association with Ba-rich minerals, and has experienced low-grade metamorphism (e.g., Kato et al., 1987; Matsubara et al., 2000; 2004), just like the deposits from the Shimoharai mine. Further surveys searching for minerals from the Shimoharai and Shiromaru mines are highly desirable to help us understand the peculiar activity of Ba-rich fluid on the bedded manganese chert deposit in the Chichibu belt.

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SUPPLEMENTARY MATERIAL

Color version of Figure 1 is available online from http:// joi.jlc.jst.go.jp/JST.JSTAGE/jmps/110901.

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Errata

The following are errata for the original article entitled "Miyahisaite, (Sr,Ca)₂Ba₃(PO₄)₃F, a new mineral of the hedyphane group in the apatite supergroup from the Shimoharai mine, Oita Prefecture, Japan" by the Daisuke NISHIO-HAMANE, Yukikazu OGOSHI and Tetsuo MINAKAWA (Vol. 107, no. 3, 121-126, 2012). These are authors' mistakes.

Page 121, Abstract line 7; 100.00 wt% should be read as 99.99 wt%

Page 123, CHMICAL COMPOSITION line 20; 100.00 wt% should be read as 99.99 wt%.

Page 123, Table 1, Total 7.997 should be read as 7.996.

Page 124, Table 2 in hkl list, line12, 115 and line 13, 600 should be read as 600 and 115, respectively.