

Hundholmenite-(Y) from Norway – a new mineral species in the vicanite group: descriptive data and crystal structure

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ABSTRACT

Hundholmenite-(Y) occurs as pale brown, subhedral crystals up to 1 mm across, embedded in REE-bearing fluorite, in a granitic pegmatite at Hundholmen, Tysfjord, Nordland County, north Norway. Two other occurrences in the same area are the granitic pegmatites at Stetind and Lagmannsvik. The simplified formula, obtained from wavelength-dispersive EMP analyses and boron determination by ICP-AES, is $(Y, REE, Ca, Na)_{15}(Al, Fe^{3+})Ca_xAs_{1-x}^{3+}(Si, As^{5+})Si_6B_3(O, F)_{48}$ ($x = 0.78$). The mineral is trigonal, $R3m$, with $a = 10.675(6)$, $c = 27.02(2)$ Å, $V = 2667(5)$ Å³, $Z = 3$. The crystal structure was refined to $R1 = 0.037$ for 1720 observed reflections. Hundholmenite-(Y) is isostructural with okanoganite-(Y) and vicanite-(Ce), and the differences in site populations are discussed. The strongest seven reflections of the X-ray powder-diffraction pattern [d_{obs} in Å, (I) (hkl)] are: 4.38 (33) (202), 3.114 (43) (214), 2.972 (100) (027), 2.947 (76) (125), 2.924 (66) (303, 033), 2.681 (36) (220) and 1.978 (37) (235). The mineral is optically uniaxial (–) with ω 1.7578(5) and ϵ 1.7487(5). The Mohs hardness is ~5–6; $D_{calc} = 5.206(9)$ g/cm³.

KEYWORDS: hundholmenite-(Y), new mineral species, vicanite group, REE borosilicate, electron microprobe data, crystal structure, Hundholmen granite pegmatite, Norway.

Introduction

THE new mineral species, hundholmenite-(Y), characterized and described in this paper, is a member of the vicanite group (Strunz and Nickel, 2001), which up to now includes the REE borosilicates okanoganite-(Y) and vicanite-(Ce). The chemical and structural data on the new mineral have significantly added to the understanding of the crystal-chemical properties of this uncommon mineral group.

Okanoganite-(Y) was described as a new species by Boggs (1980) with the chemical formula $(Na, Ca)_3(Y, Ce, Nd, La)_{12}Si_6B_2O_{27}F_{14}$

and cell dimensions $a = 10.72(1)$ and $c = 27.05(8)$ Å. A recent study of its crystal structure (Boiocchi *et al.*, 2004) has resulted in a revision of the formula (see below). It is curious that with a calculated density of 4.37 g/cm³, based on an erroneous formula and a correct unit cell, Boggs (1980) obtained a very good match with his measured density of 4.35 g/cm³. On type-locality material, Boiocchi *et al.* (2004) calculated a density of 4.96 g/cm³ for okanoganite-(Y).

Vicanite-(Ce) was described as a new mineral species by Maras *et al.* (1995), and it was apparent from its powder-diffraction pattern and unit-cell data that the mineral was closely related to okanoganite-(Y). A subsequent crystal-structure determination and refinement (Ballirano *et al.*, 2002) clarified for the first time the layered structure of the vicanite-group minerals (trigonal, space group $R3m$).

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Hundholmenite-(Y) is named for the type locality, the Hundholmen granitic pegmatite in north Norway. The mineral and name were approved by the IMA Commission on New Minerals and Mineral Names (2006-005). The type material is held in the mineral collection of the Natural History Museum, University of Oslo (catalogue number 41590).

Occurrence and mineral parageneses

Hundholmenite-(Y), initially thought to be identical with okanoganite-(Y), was first encountered in 1989 in a granitic pegmatite at Stetind, Tysfjord, Nordland County, north Norway. The mineral was subsequently reported from granitic pegmatites at Lagmannsvik, Hamarøy, Nordland and at Hundholmen, Tysfjord, Nordland. The description of the new mineral is based on material from Hundholmen, which consequently becomes the type locality.

The Hundholmen granitic pegmatite is situated on a small peninsula 7 km NW of Kjølsvik in Tysfjord, Nordland County. This very large pegmatite was mined extensively for K-feldspar (surface area ~1600 m²), quartz (surface area ~700 m²) and fluorite (Foslie, 1941) in the period 1906–1958. The Hundholmen pegmatite is well known for the occurrence of cm-sized crystals of thalénite-(Y) (Vogt, 1922) and non-metamict gadolinite (Nilssen, 1973).

Hundholmenite-(Y) from Hundholmen occurs as anhedral grains or subhedral crystals, equant to tabular in shape, up to 1 mm in size, forming aggregates up to 3 mm across. It is embedded in massive REE-bearing fluorite ('ytrofluorite' of Vogt, 1911). The only closely associated mineral is allanite-(Ce). In the Stetind occurrence, hundholmenite-(Y) forms slightly rounded, tabular mm-sized crystals, rarely up to 5–7 mm across, embedded in REE-bearing fluorite. The crystals may form intergrown aggregates or V-shaped twins. Small interpenetration twins, like the one pictured by Boggs (1980) for okanoganite-(Y), have been observed in vugs. Other accessory minerals in the massive REE-bearing fluorite include bastnäsite-(Ce), britholite-(Y), törnebohmitite-(Ce), rowlandite-(Y), allanite-(Ce), yttrilite-(Y) and thalénite-(Y). As for the Lagmannsvik occurrence, hundholmenite-(Y) occurs as tiny crystals in a REE-bearing fluorite matrix or as small aggregates along joints. The crystallization of REE-bearing fluorite and associated minerals took place at a late stage of the

pegmatite formation; hundholmenite-(Y) is thus a late-stage, primary pegmatite mineral. REE minerals formed by secondary alteration of other minerals (e.g. gadolinite-(Y) and REE-bearing fluorite) include tengerite-(Y), kainosite-(Y), adam-site-(Y), kamphaugite-(Y) and calcioancylite-(Ce).

The pegmatites at Stetind, Lagmannsvik and Hundholmen are, together with numerous other granitic pegmatites of the area, thought to be genetically related to the Tysfjord granite (Foslie, 1941), which has been dated at 1742±46 Ma (Andresen and Tull, 1986). In contrast to the granitic pegmatites of south Norway, some of the pegmatites of this area are characterized by the occurrence of several primary and secondary arsenic minerals (Raade *et al.*, 2006). Hundholmenite-(Y) also contains a small amount of As.

Physical data

The colour of hundholmenite-(Y) varies from pale reddish brown to greyish brown (Hundholmen and Stetind) and from greyish yellow to grey (Lagmannsvik). It has a white streak and is non-fluorescent in short- and long-wave ultraviolet radiation. The translucent crystals have a vitreous to adamantine lustre. Mohs hardness was estimated to be ~5–6. Cleavage and parting are not present; the mineral is brittle with uneven to subconchoidal fracture. The mineral sinks in Clerici solution, indicating a density greater than 4.2 g/cm³. Density measurement with a Berman balance was not attempted owing to inclusions of REE-bearing fluorite. The calculated density, using the empirical formula and single-crystal unit-cell data, is 5.206(9) g/cm³.

The mineral is nonpleochroic and colourless in thin fragments. It is uniaxial negative with refractive indices ω 1.7578(5) and ϵ 1.7487(5), measured at 589 nm, on Hundholmen material, by the spindle-stage method of Medenbach (1985).

Chemical and IR spectroscopic data

Analytical details

Electron microprobe (EMP) analyses were made in three different laboratories on material from Hundholmen and Stetind (Table 1). Wavelength-dispersive analyses were performed on the crystal from Hundholmen that was used for single-crystal study (sample GR1) with a Cameca SX100 electron probe at 20 kV operating voltage and 20 nA beam current, with 10 s counting time (20 s

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TABLE 1. EMP analyses (wt.%) of hundholmenite-(Y) from Hundholmen and Stetind, north Norway.

	— Hundholmen GR1 ¹ —			Hundholmen HH1		Stetind ST1	
	1	2	3	4	5	6	7
Na ₂ O	0.31	0.28–0.34	0.27	0.26	0.39	0.29	0.52
CaO	6.45	6.32–6.56	3.15	5.63	6.59	6.08	7.27
MnO	0.07	0.00–0.16	0.03	n.a.	n.a.	n.a.	n.a.
PbO	0.14	0.06–0.20	0.02	n.a.	n.a.	n.a.	n.a.
B ₂ O ₃	3.92 ²		3.08	n.a.	n.a.	n.a.	n.a.
Al ₂ O ₃	1.23	1.09–1.38	0.66	0.90	0.94	1.47	1.47
Fe ₂ O ₃	0.79 ³	0.67–0.88 ³	0.27	0.36	0.60	0.04	0.00
Y ₂ O ₃	18.07	17.75–18.27	4.39	16.16	16.34	17.19	17.71
La ₂ O ₃	4.84	4.39–5.31	0.81	3.99	4.96	4.09	4.15
Ce ₂ O ₃	12.70	11.91–13.43	2.12	11.01	12.59	11.01	11.42
Pr ₂ O ₃	1.73	1.40–2.00	0.29	1.71	1.83	1.93	1.64
Nd ₂ O ₃	7.87	7.34–8.28	1.28	7.85	8.01	8.66	8.62
Sm ₂ O ₃	2.93	2.52–3.35	0.46	3.14	2.90	2.29	3.00
Eu ₂ O ₃	0.83	0.60–1.06	0.13	0.71	0.65	n.a.	n.a.
Gd ₂ O ₃	4.39	3.56–4.93	0.66	5.14	4.34	3.93	4.16
Tb ₂ O ₃	0.49	0.39–0.64	0.07	0.62	0.51	0.38	0.51
Dy ₂ O ₃	4.20	3.43–4.73	0.62	3.32	4.09	2.27	2.63
Ho ₂ O ₃	0.80	0.57–1.01	0.12	0.53	0.37	n.a.	n.a.
Er ₂ O ₃	3.16	1.96–4.00	0.45	3.47	2.90	2.87	2.80
Tm ₂ O ₃	0.79	0.68–0.93	0.11	0.34	0.20	n.a.	n.a.
Yb ₂ O ₃	3.53	3.14–3.82	0.49	4.11	3.30	3.92	3.59
Lu ₂ O ₃	0.78	0.59–0.96	0.11	0.63	0.49	n.a.	n.a.
SiO ₂	15.05	14.87–15.31	6.87	12.88	14.43	14.62	14.55
ThO ₂	0.09	0.04–0.15	0.01	n.a.	n.a.	n.a.	n.a.
P ₂ O ₅	0.14	0.00–0.71	0.05	n.d.	n.d.	n.a.	n.a.
As ₂ O ₃	0.80 ⁴		0.22				
As ₂ O ₅	1.09 ⁴	(1.93–2.10) ⁵	0.26	1.95	1.76	n.a.	n.a.
F	7.75	7.53–8.27	11.18	6.49	8.40	6.61	8.65
Sum	104.94			(91.20)	(96.59)	(87.65)	(92.69)
–O≡F ₂	3.26			2.73	3.54	2.78	3.64
Total	101.68			(88.47)	(93.05)	(84.87)	(89.05)

1: Mean of 13 analyses

2: Range of 13 analyses

3: Atoms per formula unit, based on (O + F) = 48

4: Mean of 2 analyses, dark areas on BSE image (Fig. 1a)

5: Mean of 14 analyses, light areas on BSE images (Fig. 1a)

6: One analysis, dark area on BSE image (Fig. 1b)

7: Mean of two analyses, light areas on BSE image (Fig. 1b)

¹ Crystal that was used for structure refinement

² Analysed with ICP-AES

³ Value of 0.28 wt.%, which is too small, is replaced by result obtained by Robert Gault (the range refers to three single analyses)

⁴ Recalculated according to structure data

⁵ Range of total As₂O₅; mean value 2.02 wt.%

n.a.: not analysed

n.d.: not detected

for P-*K*β) and a beam diameter of 10 μm. The probe standards and peaks were: omphacite (Na-*K*α), wollastonite (Ca-*K*α and Si-*K*α), pyro-

phanite (Mn-*K*α), galena (Pb-*M*α), Al₂O₃ (Al-*K*α), Fe₂O₃ (Fe-*K*α), REEPO₄ (*L*α for Y, La, Ce, Tb and Yb; *L*β for Pr, Nd, Eu, Gd, Dy, Ho, Er,

Tm and Lu), synthetic oxide with Y, Sm, Tb (Sm- $L\beta$), synthetic glass with 14.97 wt.% ThO₂ (Th- $M\alpha$), Durango apatite (P- $K\beta$), GaAs (As- $L\alpha$), synthetic fluorphlogopite (F- $K\alpha$). The REE standards are described by Jarosewich and Boatner (1991). Ti was sought but not detected. Backscattered electron (BSE) imaging reveals a very slight and irregular zoning in sample GR1. The mean result of 13 individual analyses, obtained from different zones, is presented in Table 1, where also the range of variation in the analytical data and the number of atoms per formula unit (a.f.p.u.), on the basis of (O + F) = 48, are given. The total As content, obtained as As₂O₅ 2.02 wt.%, has been recalculated to As₂O₃ and As₂O₅, according to the structure data.

Because of initial problems with the calculation of a chemical formula for hundholmenite-(Y), it was decided to reanalyse sample GR1 in another laboratory. Robert Gault of the Canadian Museum of Natural History kindly performed three single analyses. The two sets of analytical data on GR1 are quite similar, except for Fe₂O₃. The mean concentration obtained from the 13 analyses, 0.28 wt.% Fe₂O₃, is obviously too low and has been replaced by the new result, 0.79 wt.%, in Table 1.

Table 1 also contains the results of some early reconnaissance EMP analyses on hundholmenite-(Y) from Hundholmen (sample HH1) and from Stetind (sample ST1). Although the Stetind analyses are incomplete, lacking data for Eu, Ho, Tm, Lu and As, they are still useful for assessing the chemical variability of the mineral. These wavelength-dispersive analyses were performed with a Cameca Camebax instrument

at 20 kV and 18.5 nA with 10 s counting time, scanning areas of 10 $\mu\text{m} \times 10 \mu\text{m}$. The probe standards employed and peaks are identical to those used on the Cameca SX100 instrument (see above), except that both synthetic fluorphlogopite and fluorite were used as F standards, with similar results. For sample HH1, some elements were analysed separately as trace elements with long counting times: Eu and Ho (30 s), Tm (50 s), Lu and As (20 s). The HH1 and ST1 samples show distinct and complicated chemical zonation on BSE images (Fig. 1); it is differentiated between the analytical results on dark and bright BSE areas in Table 1.

H₂O was not found in a 0.1 mg sample with a LECO RC-412 multiphase CH analyser; the detection limit is said to be ~0.05 wt.% H₂O. In spite of this result, the O9 atom can probably be interpreted as an OH group from the bond-valence sums (see below). CO₂ is not present according to the infrared spectrum. B₂O₃ was measured on a bulk sample weighing 23.65 mg by ICP-AES after dissolution in concentrated HCl. The result, 3.80 wt.%, has been corrected for an estimated contamination with 3% REE-bearing fluorite to 3.92 wt.% B₂O₃.

Infrared spectroscopy

The infrared (IR) spectrum of the mineral, dispersed in a KBr pellet, shows minor and broad absorptions at 3440 and 1645 cm^{-1} , due to water molecules. This is probably absorbed water, as H₂O could not be determined chemically. However, the structure refinement indicates that a minor amount of hydroxyl is present (see below).

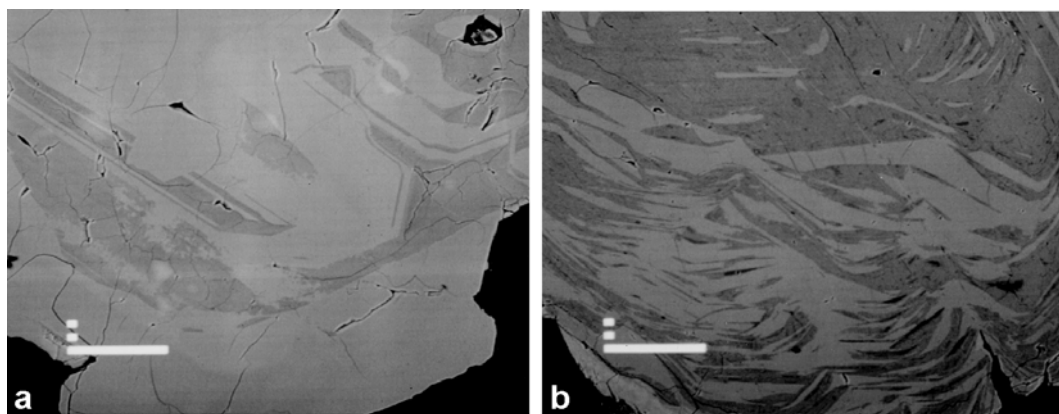


FIG. 1. BSE images of hundholmenite-(Y) from (a) Hundholmen (sample HH1) and (b) from Stetind (sample ST1). Scale bars are 100 μm .

A small absorption at 1440 cm^{-1} is tentatively assigned to the arsenite ion $(\text{AsO}_3)^{3-}$. This is based on a comparison with the IR spectrum of the arsenite mineral magnussonite presented by Graeser *et al.* (1994). The rest of the spectrum is difficult to interpret owing to the chemical and structural complexity of the mineral. A broad absorption ‘trough’ between ~ 1300 and 600 cm^{-1} has small maxima at 1257 , 1130 , 1056 , 989 , 927 , 866 and 783 cm^{-1} . Better defined absorptions occur at 643 , 565 and 521 cm^{-1} .

Analytical results

The main chemical differences between the dark and bright zones (with relatively lighter and heavier elements, respectively) in samples HH1 and ST1 (Fig. 1) are the distinctly lower F and moderately lower Na and Ca contents of the former (Table 1). The Stetind sample is notable in being nearly devoid of Fe.

The analytical data for the Hundholmen samples GR1 and HH1 are not very different. The empirical formula, on the basis of $(\text{O} + \text{F}) = 48$, for the hundholmenite-(Y) crystal that was used for the structure determination (see analytical data for GR1 in column 1 of Table 1), is: $(\text{Y}_{4.39}\text{REE}_{7.73}\text{Ca}_{3.15}\text{Na}_{0.27}\text{Mn}_{0.03}\text{Pb}_{0.02}\text{Th}_{0.01})_{\Sigma 15.60}(\text{Al}_{0.66}\text{Fe}_{0.27})_{\Sigma 0.93}\text{As}_{0.22}(\text{Si}_{0.69}\text{As}_{0.26}\text{P}_{0.05})_{\Sigma 1.00}\text{Si}_{6.18}\text{B}_{3.09}(\text{O}_{36.82}\text{F}_{11.18})_{\Sigma 48}$. Here, trivalent and pentavalent As are partitioned according to the structure data. The simplified formula, taking into account the mutually exclusive As^{3+} and Ca sites

in the structure, is: $(\text{Y},\text{REE},\text{Ca},\text{Na})_{15}(\text{Al},\text{Fe}^{3+})\text{Ca}_x\text{As}_{1-x}(\text{Si},\text{As}^{5+})\text{Si}_6\text{B}_3(\text{O},\text{F})_{48}$ ($x = 0.78$). The Gladstone–Dale relationship gives a compatibility index $1 - (\text{K}_p/\text{K}_c) = 0.045$, rated as ‘good’ (Mandarino, 1981).

REE pattern

Chondrite-normalized REE patterns of hundholmenite-(Y) (Fig. 2) (CI chondrite values from McDonough and Sun, 1995) are smooth, especially for the LREEs; larger variations occur in HREEs with the lowest concentrations (e.g. Ho and Tm) and can be ascribed to analytical inaccuracy. There is no systematic difference in REE concentrations between hundholmenite-(Y) from Hundholmen and Stetind, nor is there any pronounced difference between bright and dark areas on the BSE images. The patterns are remarkably flat with no relative enrichment in LREEs or HREEs. ‘Yttrifluorite’ from Hundholmen, in which hundholmenite-(Y) is embedded, has been shown by Sverdrup (1968) not to be enriched in Y as the name would imply but rather in LREEs, with a likely variation in REE composition from place to place within the pegmatite body.

X-ray diffraction (XRD)

Powder XRD

The powder XRD pattern (data in Table 2) was recorded with a Siemens D5005 diffractometer,

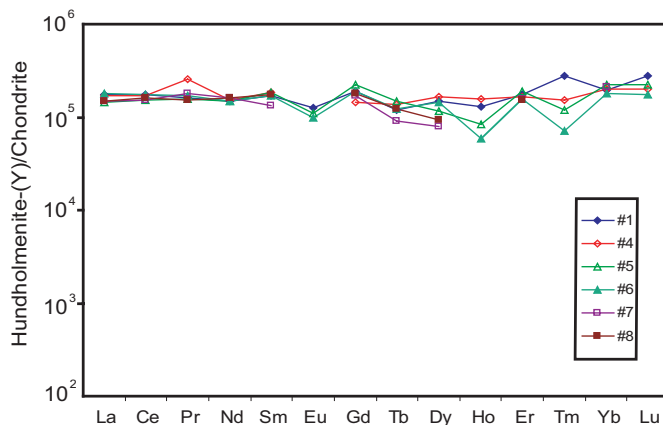


FIG. 2. Chondrite-normalized REE patterns for hundholmenite-(Y) (analyses in Table 1). CI chondrite values from McDonough and Sun (1995). 1: Hundholmen, Sample GR1 (Table 1), 4: Hundholmen, sample GR1 (analysed by R. Gault, not in Table 1). Patterns 5, 6, 7 and 8 correspond to analyses 4, 5, 6 and 7 in Table 1.

run at 40 kV and 40 mA and equipped with a curved graphite secondary monochromator and scintillation detector, using Cu- $K\alpha$ radiation and Bragg-Brentano geometry. The powdered sample was dispersed with ethyl alcohol on a (510) silicon plate which was rotated during exposure. No internal standard was included; the instrument was calibrated with a quartz sample. The range of

2θ was 5–75°, although only reflections below 53° are reported here. The step size was 0.05° and the step time 2 s. A step size of 0.01° did not give well resolved reflections, probably owing to chemical zoning in the sample. Background subtraction was applied; the intensities of the diffraction lines were measured as peak heights above background and normalized to 100 for the strongest reflection. Cell parameters, refined from the powder data with the program Celref Version 3 (Laugier and Bochu, 2002), are $a = 10.704(4)$, $c = 27.07(1)$ Å, $V = 2686(1)$ Å³.

TABLE 2. X-ray powder-diffraction data for hundholmenite-(Y).

hkl	d_{meas} (Å)	d_{calc} (Å)	hkl
4	8.98	9.02	003
9	7.62	7.65	012
5	5.47	5.47	104
8	5.36	5.35	110
7	4.67	4.68	015
6	4.57	4.57	021
33	4.38	4.39	202
8	3.82	3.82	024
17	3.567	3.569	107
8	3.517	3.521	205
6	3.484	3.475	211
6	3.470*	3.449	116
12	3.399	3.392	122
43	3.114	3.111	214
29	3.095	3.090	300
22	3.011	3.007	009
100	2.972	2.969	027
76	2.947	2.941	125
66	2.924	2.923	303 033
14	2.736	2.733	208
36	2.681	2.676	220
23	2.594	2.596	217
6	2.576*	2.566	223
5	2.548	2.549	036 306
12	2.520	2.526	312
18	2.159	2.155	309 039
26	2.144	2.142	2.1.10
19	2.132*	2.141	137
10	2.098	2.101	232
21	2.030	2.029	324
22	2.000	1.999	229
37	1.978	1.979	235
27	1.974	1.974	413 143
8	1.897	1.899	0.2.13
9	1.862	1.863	327
25	1.821	1.822	0.3.12 3.0.12
31	1.788	1.788	054
22	1.783	1.784	330 2.0.14
12	1.754	1.754	505

Diffraction, Cu- $K\alpha_1$ radiation ($\lambda = 1.5406$ Å)
 Refined cell parameters: $a = 10.704(4)$, $c = 27.07(1)$ Å
 * not used for calculation of cell parameters

Single-crystal XRD

A suitably sized single crystal of hundholmenite-(Y) was used for the collection of XRD data. Data were collected on a Bruker AXS diffractometer with a four-circle goniometer and a SMART 1000 CCD detector operated at 40 kV and 37 mA with graphite-monochromated Mo- $K\alpha$ radiation. The SMART program (Bruker AXS, 1998) was used for the determination of the cell orientation and for data collection, SAINT for the integration of intensities, and XPREP to perform empirical absorption corrections and for preparing SHELX files; all are Bruker AXS programs. The structure was solved by direct methods in SHELXS-97 (Sheldrick, 1997). The final refinements were done in SHELXL-93 (Sheldrick, 1993), applying the atomic position scheme of the vicinite-(Ce) structure. Information relevant to the data collection and structure determination is given in Table 3. The final atomic coordinates, anisotropic displacement parameters and equivalent isotropic displacement factors for the hundholmenite-(Y) structure are shown in Table 4; bond distances and selected angles are presented in Table 5. A table of structure factors is deposited with the Principal Editor and can be accessed at www.minersoc.org/pages/e_journals/deposited/Raade_structure_factor_data.sft

Crystal structure

The structures of the vicinite-group minerals have three distinct layers parallel to (001):

(1) Layer A contains an octahedron with Al, Fe³⁺ and Ti as the central atoms, sharing corners with six SiO₄ tetrahedra in the form of a pinwheel (Fig. 3). BO₄ tetrahedra form threefold rings, and each BO₄ tetrahedron of the rings shares a corner with one SiO₄ tetrahedron, forming an (Si₃B₃O₁₈)¹⁵⁻ polyanion.

TABLE 3. Crystal data, data-collection information and refinement details for hundholmenite-(Y).

Wavelength	0.71073 Å
Crystal system: space group	Trigonal: $R\bar{3}m$
Unit-cell dimensions	$a = 10.675(6)$ Å, $c = 27.02(2)$ Å
Volume	$2667(5)$ Å ³
Z	3
$F(000)$	3287
Absorption coefficient	18.33 mm ⁻¹
Crystal size	$0.07 \times 0.11 \times 0.18$ mm
θ range for data collection	2.26 to 29.92°
Index ranges	$-12 \leq h \leq 0$, $0 \leq k \leq 14$, $-37 \leq l \leq 36$
Reflections collected	1817
Unique reflections	938 ($R1 = 0.032$)
Refinement method	Full-matrix least-squares on F^2
Obs. refls. / restraints / parameters	1720 / 0 / 167
Goodness-of-fit on F^2	1.118
Final R indices [$F_o > 4\sigma(F_o)$]	$R1 = 0.037$, $wR2 = 0.089$
R indices (all data)	$R1 = 0.039$, $wR2 = 0.089$
Largest difference peak and hole	1.75 and -1.45 eÅ ⁻³

(2) Layer B contains a tetrahedron ($Si3/As1$) which may incorporate variable amounts of Si, As⁵⁺ and P. This tetrahedron occurs in two orientations with the same probability of occurrence. In vicanite-(Ce), a BO₃ triangle is also present in this layer.

(3) Layer C contains two very close atom sites: the $As2$ site with As³⁺ in three-fold pyramidal coordination and the Ca/Na site with Ca or Na in ten-coordination, which are mutually exclusive (Fig. 4).

A three-dimensional network of five different polyhedra with coordinations from eight to ten

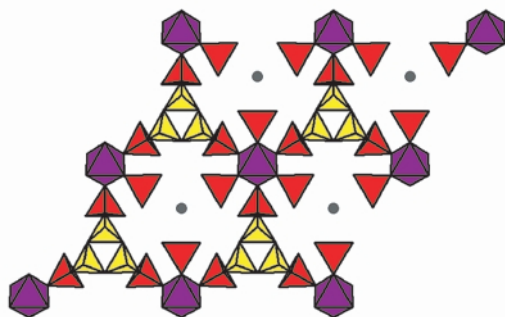


FIG. 3. Projection of layer A on (001) with the Al/Fe octahedron (blue) at the origin. Six SiO₄ tetrahedra (red) form a pinwheel around the octahedron by corner-sharing, and three BO₄ tetrahedra (yellow) form a ring by corner-sharing. The polyanion (Si₃B₃O₁₈)¹⁵⁻ is composed of three Si and three B tetrahedra. The $Si3/As1$ site in layer B is shown as a circle. The corresponding tetrahedron has two possible orientations.

($M1$ to $M5$) contains Y, REE, Ca and Th and provides connections between the three layers.

Table 6 shows site-scattering factors and site populations in minerals of the vicanite group. A bond-valence calculation for hundholmenite-(Y) is shown in Table 7, based on the assigned site populations in Table 6, with bond-valence parameters taken from Brese and O'Keeffe (1991).

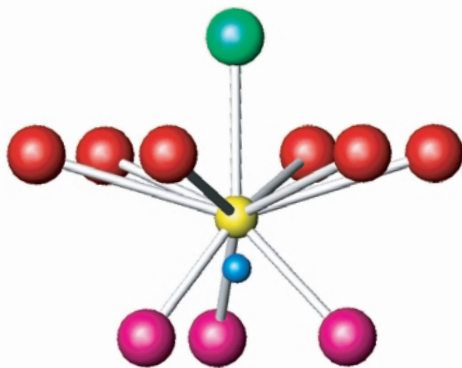


FIG. 4. The two close sites of Ca (yellow) and As³⁺ (blue) in the disordered part of layer C (the c axis is vertical). Having a distance of $0.70(2)$ Å, the two positions are mutually exclusive. Ca is ten-coordinated to one fluorine atom (site $F13$) (green), six oxygen atoms (site $O6$) (red) at a long distance of $2.881(4)$ Å, and three mixed fluorine/oxygen atoms (site $OF12$) (magenta). The Ca-F bond lies along the c axis. As³⁺ is bonded to the three $OF12$ atoms in a pyramidal configuration.

TABLE 4. Fractional coordinates and displacement parameters (\AA^2) for hunderholmenite-(Y). M = site multiplicity.

Site	M	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}	U_{eq}
<i>M1</i>	9	-0.12915(5)	0.12915(5)	-0.3189(2)	0.0109(4)	0.0109(4)	0.0145(4)	-0.0008(2)	0.0008(2)	0.0039(4)	0.0128(2)
<i>M2</i>	9	-0.13642(6)	0.13642(6)	0.0995(2)	0.0174(4)	0.0174(4)	0.0145(5)	0.0011(2)	-0.0011(2)	0.0065(4)	0.0174(2)
<i>M3</i>	9	0.1153(1)	-0.1153(1)	-0.0989(2)	0.0187(8)	0.0187(8)	0.024(1)	-0.0038(4)	0.0038(4)	0.0125(8)	0.0191(4)
<i>M4</i>	9	0.12303(8)	-0.12303(8)	0.4661(2)	0.0142(5)	0.0142(5)	0.0152(6)	-0.0012(2)	0.0012(2)	0.0032(5)	0.0163(3)
<i>M5</i>	9	0.13867(6)	-0.13867(6)	0.2191(2)	0.0106(4)	0.0106(4)	0.0122(5)	-0.0001(2)	0.0001(2)	0.0052(4)	0.0112(2)
<i>Al/Fe</i>	3	0	0	0	0.020(2)	0.020(2)	0.009(3)	0	0	0.010(1)	0.016(1)
<i>Si1</i>	9	0.1688(2)	-0.1688(2)	0.0426(3)	0.011(1)	0.011(1)	0.020(2)	-0.0006(8)	0.0006(8)	0.006(2)	0.014(1)
<i>Si2</i>	9	-0.1700(3)	0.1700(3)	-0.0402(3)	0.009(1)	0.009(1)	0.014(2)	0.0005(7)	-0.0005(7)	0.004(1)	0.011(1)
<i>B1</i>	9	-0.0794(9)	0.0794(9)	0.2920(5)	0.008(5)	0.008(5)	0.003(5)	0.003(3)	-0.003(3)	0.0001(54)	0.008(3)
<i>Si3/As1</i>	3	0	0	-0.2119(3)	0.014(3)	0.014(3)	0.023(4)	0	0	0.007(1)	0.017(2)
<i>As2</i>	3	0	0	-0.454(1)	0.005(5)	0.005(5)	0.002(11)	0	0	0.002(2)	0.004(4)
<i>Ca/Na</i>	3	0	0	-0.428(1)	0.038(4)	0.038(4)	0.05(2)	0	0	0.019(2)	0.042(5)
<i>O1</i>	9	0.0806(5)	-0.0806(5)	0.0456(4)	0.007(3)	0.007(3)	0.015(5)	0.0005(19)	-0.0005(19)	0.003(4)	0.010(2)
<i>O2</i>	9	0.1853(5)	-0.1853(5)	-0.0151(5)	0.013(4)	0.013(4)	0.022(5)	0.002(2)	-0.002(2)	0.010(4)	0.014(2)
<i>O3</i>	18	0.3246(8)	-0.0682(8)	0.0695(3)	0.010(3)	0.021(4)	0.017(3)	0.001(3)	0.002(3)	0.011(3)	0.015(2)
<i>O4</i>	9	-0.0837(6)	0.0837(6)	-0.0468(5)	0.017(4)	0.017(4)	0.020(5)	-0.005(2)	0.005(2)	0.012(4)	0.016(2)
<i>O5</i>	9	-0.1917(6)	0.1917(6)	0.0180(5)	0.025(5)	0.025(5)	0.015(5)	-0.0007(20)	0.0007(20)	0.020(5)	0.018(3)
<i>O6</i>	18	-0.3290(9)	0.0777(9)	-0.0631(4)	0.012(4)	0.024(4)	0.020(4)	-0.003(3)	-0.006(3)	0.002(3)	0.022(2)
<i>O7</i>	9	0.0773(5)	-0.845(1)	0.3052(4)	0.004(3)	0.005(4)	0.015(4)	-0.001(3)	-0.0004(17)	0.002(2)	0.008(2)
<i>O8</i>	9	-0.0949(5)	0.0949(5)	0.2373(4)	0.010(3)	0.010(3)	0.015(5)	-0.002(2)	0.002(2)	0.006(4)	0.011(2)
<i>O9</i>	9	0.0853(7)	-0.0853(7)	0.1440(4)	0.026(4)	0.026(4)	0.007(4)	0.004(2)	-0.004(2)	0.010(5)	0.021(2)
<i>O10</i>	3	0	0	-0.1501(9)	0.019(6)	0.019(6)	0.04(1)	0	0	0.009(3)	0.026(2)
<i>O11</i>	18	0.164(2)	0.056(2)	-0.2345(6)	0.017(7)	0.03(2)	0.025(7)	0.002(6)	-0.005(5)	0.013(7)	0.023(3)
<i>OF12</i>	9	-0.0869(6)	0.0869(6)	-0.4891(4)	0.020(4)	0.020(4)	0.020(5)	-0.0008(21)	0.0008(21)	0.004(4)	0.023(2)
<i>F13</i>	3	0	0	-0.3375(7)	0.021(5)	0.021(5)	0.030(9)	0	0	0.010(2)	0.024(3)
<i>F14</i>	18	0.0150(8)	0.3029(8)	-0.1614(3)	0.025(4)	0.025(4)	0.029(4)	-0.002(3)	-0.006(3)	0.015(3)	0.025(2)
<i>W</i>	3	0	0	0.4333(7)	0.010(5)*						

$U_{\text{eq}} = 1/3[U_{33} + 4/3(U_{11} + U_{22} - U_{12})]$ (Fischer and Tillmanns, 1988)

* refined isotropically

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TABLE 5. Bond distances (Å) and selected bond angles (°) in hundholmenite-(Y).

<i>M1</i> –F13		2.440(4)	Si1–O2		1.590(12)
–O11		2.458(15)	–O1		1.632(11)
–O3	× 2	2.499(8)	–O3	× 2	1.632(8)
–O5	× 2	2.513(6)	Mean		1.622
–O6	× 2	2.527(9)			
–O7		2.614(10)	O1–Si1–O2		103.9(5)
Mean		2.510	O1–Si1–O3	× 2	106.9(3)
			O2–Si1–O3	× 2	112.2(3)
<i>M2</i> –O5		2.428(11)	O3–Si1–O3		114.0(6)
–O9	× 2	2.513(5)			
–F14	× 2	2.581(7)	Si2–O6	× 2	1.601(8)
–O3	× 2	2.635(8)	–O4		1.605(12)
–O1	× 2	2.636(6)	–O5		1.622(12)
–O11		2.895(18)	Mean		1.607
Mean		2.605			
			O4–Si2–O5		110.7(6)
<i>M3</i> –O8		2.278(10)	O4–Si2–O6	× 2	109.9(4)
–F14	× 2	2.328(7)	O5–Si2–O6	× 2	106.3(4)
–O4	× 2	2.371(7)	O6–Si2–O6		113.8(7)
–O10		2.540(14)			
–O2		2.607(11)	B1–O2		1.454(19)
–O6	× 2	2.906(9)	–O7	× 2	1.491(9)
Mean		2.515	–O8		1.508(17)
			Mean		1.486
<i>M4</i> –O3	× 2	2.285(8)			
–F14	× 2	2.347(7)	O2–B1–O7	× 2	109.0(8)
–OF12	× 2	2.360(6)	O2–B1–O8		108.1(1.1)
–W		2.441(7)	O7–B1–O7		112.1(1.2)
–O11		2.558(14)	O7–B1–O8	× 2	109.3(8)
Mean		2.373			
			Si3/As1–O11	× 3	1.656(15)
<i>M5</i> –O9		2.257(10)	–O10		1.671(26)
–OF12		2.287(11)	Mean		1.660
–O8	× 2	2.323(3)			
–O6	× 2	2.329(8)	O10–Si3/As1–O11	× 3	111.6(5)
–F14	× 2	2.557(7)	O11–Si3/As1–O11	× 3	107.2(6)
–O7		2.588(10)			
Mean		2.394	As2–OF12	× 3	1.866(18)
Al/Fe–O1	× 3	1.933(10)	Ca/Na–OF12	× 3	2.305(29)
–O4	× 3	1.998(12)	–F13		2.444(42)
Mean		1.966	–O6	× 6	2.881(14)
			Mean		2.665
O1–Al/Fe–O1	× 3	83.8(5)			
O1–Al/Fe–O4	× 6	96.0(3)			
O4–Al/Fe–O4	× 3	84.3(5)			

As for the anions, the *O11* site has 4.24 electrons per site (e.p.s.), which could be interpreted as a mixed site with 0.26 O and 0.24 F. However, *O11* is bonded to the *Si3/As1* site, and an Si–F bond is considered less likely. The *O11* site is peculiar in that it is situated in a general position and has an O11–O11 distance of 0.55(4) Å. The *O11* site can therefore only be half occupied. This

is the reason for the two possible orientations of the *Si3/As1* tetrahedron, with the same probability of occurrence. The *OF12* site is filled with 0.64 F and 0.36 O, similar to the situation in vicanite-(Ce). We have tentatively assigned 0.80 F to the *W* site, where okanoganite-(Y) has 1 OH and vicanite-(Ce) has 0.21 H₂O. The total number of F atoms in the formula of hundholmenite-(Y) is

TABLE 6. Site-scattering factors and site populations in minerals of the vicanite group.

Site*	<i>M</i>	Hundholmenite-(Y)			Okanoganite-(Y)			Vicanite-(Ce)		
		sfac	sof	ss e.p.s. X-ray	Site population assigned**	e.p.s. EMPA	ss	Site population	ss	Site population
<i>M1</i>	9	Sm	0.433(4)	53.7	0.64 Sm + 0.36 Y	53.7	52.6	0.72 Ce + 0.28 Y	48.5	0.75 Ce + 0.25 Ca
<i>M2</i>	9	Sm	0.416(4)	51.6	0.75 Sm + 0.25 Ca	51.5	52.3	0.75 Ce + 0.25 Y	47.3	0.74 La + 0.26 Ca
<i>M3</i>	9	Y	0.406(5)	31.7	0.44 Ca + 0.28 Sm + 0.10 Y + 0.09 Na + 0.01 Mn	31.3	27.7	0.59 Ca + 0.41 Y	20.3	0.99 Ca + 0.01 Ce
<i>M4</i>	9	Y	0.571(6)	44.5	0.56 Y + 0.34 Sm + 0.10 Ca	44.9	33.6	0.71 Y + 0.29 Ca	20.7	0.98 Ca + 0.02 Ce
<i>M5</i>	9	Y	0.666(7)	51.9	0.56 Sm + 0.44 Y	51.9	45.4	0.87 Y + 0.13 Th	77.1	0.82 Th + 0.18 Ca
<i>Al/Fe</i>	3	Al	0.242(7)	18.9	0.66 Al + 0.27 Fe	15.6	25.1	0.77 Fe + 0.23 Ti	23.7	0.82 Fe + 0.18 Al
<i>Si3/As1</i>	3	Si	0.180(6)	15.1	0.69 Si + 0.26 As ⁵⁺ + 0.05 P	19.0	14.6	0.60 P + 0.40 Si	28.9	0.77 As ⁵⁺ + 0.23 P
<i>As2</i>	3	As	0.037(6)	7.33	0.22 As ³⁺	7.26	—	empty	13.9	0.42 As ³⁺
<i>Ca/Na</i>	3	Ca	0.13(1)	15.6	0.78 Ca	15.6	11	1 Na	6.38	0.58 Na
<i>O11</i>	18	O	0.53(2)	4.24	0.26 O + 0.24 F (see text)	4.24	4	0.5 O	4	0.5 O
<i>OF12</i>	9	F	0.48(2)	8.64	0.64 F + 0.36 O	8.64	9	1 F	8.6	0.58 F + 0.42 O
<i>W</i>	3	O	0.15(1)	7.20	0.80 F	7.20	8	1 OH	1.7	0.21 H ₂ O

* Sites with full occupancy of a single element are omitted.

** Based on the averaged results of EMP analyses from column 3 in Table 1.

M – multiplicity

sfac – scattering factor

sof – site occupancy factor

ss – site scattering

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TABLE 7. Bond-valence table for hundholmenite-(Y).

Site Coord. Atoms	M1 9 Sm Y	M2 10 Sm Ca	M3 9 Ca Sm Y Na Mn	M4 8 Y Sm Ca	M5 9 Sm Y	Al/Fe 6 Al Fe	Si1 4 Si	Si2 4 Si	B1 4 B	Si3/As1 4 Si As P	As2 3 As ³⁺	Ca/Na 10 Ca	Sum
Charge	3.00	2.75	2.13	2.90	3.00	3.00	4.00	4.00	3.00	4.31	0.66	1.56	
Site ↓													
O1	0.21 2 × ↓ 2 × →					0.53 3 × ↓	0.98						1.93
O2			0.18				1.10		0.80				2.08
O3	0.31 2 × ↓	0.21 2 × ↓		0.51 2 × ↓			0.98 2 × ↓						2.01
O4			0.34 2 × ↓ 2 × →			0.45 3 × ↓		1.05					2.18
O5	0.30 2 × ↓ 2 × →	0.37					1.00						1.97
O6	0.29 2 × ↓		0.08 2 × ↓		0.48 2 × ↓		1.06 2 × ↓					0.08 6 × ↓	1.99
O7	0.23				0.24				0.72 2 × ↓ 2 × →				1.91
O8			0.43		0.49 2 × ↓ 2 × →				0.69				2.10
O9		0.30 2 × ↓ 2 × →			0.58								1.18
O10			0.21 3 × →							0.99			1.62
O11	0.34	0.10		0.25						1.03 3 × ↓			1.72
OF12				0.35 2 × ↓ 2 × →	0.44						0.16 3 × ↓	0.26 3 × ↓	1.56
F13	0.27 3 × →											0.19	1.00
F14		0.19 2 × ↓	0.28 2 × ↓	0.32 2 × ↓	0.19 2 × ↓								0.98
W				0.24 3 × →									0.72
Sum	2.64	2.29	2.22	2.85	3.58	2.94	4.04	4.17	2.93	4.08	0.48	1.45	

9.72 from the structure data (excluding *O11*), as compared to 11.18 from the EMP analysis. This is a satisfactory result considering that F is a difficult element to analyse by EMPA. However, 0.24 F at the *O11* site (see above) incidentally corresponds to 1.44 F a.p.f.u., very close to the ‘missing’ number of F atoms ($11.18 - 9.72 = 1.46$).

The *Ca/Na* site in hundholmenite-(Y) has 15.6 e.p.s., which corresponds to a partially filled site with 0.78 Ca a.p.f.u. The *As2* site is only 0.70(2) Å apart from the *Ca/Na* site (Fig. 4). Consequently, 0.22 As^{3+} is assigned to this site. In vicanite-(Ce), these two sites have 0.58 Na and 0.42 As^{3+} , respectively, whereas in okanoganite-(Y), the *Ca/Na* site is filled with Na, and the *As2* site is empty.

The chemical contents of the *Al/Fe* and *Si3/As1* sites are not in perfect match with the refined scattering values. The Fe content is underestimated, whereas the As content is overestimated. Clearly, in such a complicated EMP analysis, slight inaccuracies in key elements with low concentrations (<2 wt.%) are not easily avoided.

The correct distribution of Y, REE and Ca between the five *M* sites is problematic. We do not know if Ca is present at all the sites or only at some, or if the REEs are distributed in the same ratio at the different sites. We have used Sm as close to a weighted average of the REEs exclusive of Y. The question of a correct distribution of elements at the *M* sites is virtually impossible to solve. It is clear that Ca should preferably be assigned to the sites with low electron densities (*M3* and *M4*), and Y could be concentrated in polyhedra with the lowest mean bond distances, *M4* and *M5* (Table 5). Site populations based on these assumptions, assigned according to the averaged results of the EMP analyses, are shown in Table 6. However, Table 7 shows that the bond-valence sums are not satisfactory for *M1*, *M2* and *M5*. On the other hand, the sum of the site charges for *M1* to *M5*, 13.78 e, compares well with the sum of the bond valences, 13.58 v.u. The bond-valence sum for *M5* is for some reason as high as 3.58 v.u., although the Th content in our mineral is very small, as opposed to the situation in vicanite-(Ce) and okanoganite-(Y) (Table 6). The *M5* site has higher bond-valence sums than

TABLE 8. Comparative data for minerals of the vicanite group.

	Hundholmenite-(Y) ¹	Okanoganite-(Y) ²	Vicanite-(Ce) ³
Chemical formula	(Y, REE, Ca, Na) ₁₅ (Al, Fe ³⁺) Ca _x As _{1-x} ³⁺ (Si, As ⁵⁺) Si ₆ B ₃ (O, F) ₄₈ $x = 0.78$	(Y, REE, Ca, Na, Th) ₁₅ (Fe ³⁺ , Ti) Na(Si, P) Si ₆ B ₃ (O, F, OH) ₄₈	(Ca, REE, Th) ₁₅ Fe ³⁺ Na _x As _{1-x} ³⁺ (As ⁵⁺ , P) Si ₆ B ₄ (O, F) ₄₇ $x = 0.58$
Space group	Trigonal <i>R3m</i>	Trigonal <i>R3m</i>	Trigonal <i>R3m</i>
Unit-cell parameters (Å)	<i>a</i> 10.675(6) <i>c</i> 27.02(2)	<i>a</i> 10.7108(5) <i>c</i> 27.0398(11)	<i>a</i> 10.8112(2) <i>c</i> 27.3296(12)
Strongest reflections in powder pattern	4.38 (33) (202) 3.114 (43) (214) 3.095 (29) (300) 2.972 (100) (027)	4.38 (41) (202) 3.11 (48) (300) 2.970 (100) (027) 2.939 (95) (125)	7.70 (50) (012) 4.42 (50) (202) 3.13 (50) (214) 2.993 (100) (027)
<i>d</i> (Å) <i>hkl</i>	2.947 (76) (125) 2.924 (66) (303, 033) 2.681 (36) (220) 1.978 (37) (235) 1.788 (31) (054)	2.926 (50) (303) 2.676 (32) (220) 1.978 (35) (325, 413) 1.822 (32) (3.0.12) 1.784 (43) (330, 2.0.14)	2.950 (70) (303) 2.698 (50) (220) 1.839 (50) (3.0.12) 1.802 (50) (2.0.14)
Optical data	Uniaxial (–) ω 1.7578(5) ε 1.7487(5)	Uniaxial (–) ω 1.753(2) ε 1.740(2)	Uniaxial (–) ω 1.757(2) ε 1.722(2)
Density (calc.) g/cm ³	5.206	4.96	4.73

¹ this work; ² Boggs (1980), Boiocchi *et al.* (2004); ³ Maras *et al.* (1995), Ballirano *et al.* (2002)

expected in both okanoganite-(Y) and vicanite-(Ce). The bond-valence sums for all the other cation sites in hundholmenite-(Y) are satisfactory (Table 7). For the *Al/Fe* site, we have used the site population 0.55 Al + 0.45 Fe, derived from the site-scattering value 18.9 e.p.s.

The bond-valence sums for the anions indicate that *O9* is possibly an OH group, which is the case for okanoganite-(Y); *O9* in vicanite-(Ce) hosts an oxygen atom bonded to the three-coordinated B2 atom, which is missing in the two other minerals. However, at the approximate site of the B2 atom in hundholmenite-(Y) [coordinates 0, 0, 0.166(3)], the structure refinement yields a minor site-occupancy factor of 0.06(1), based on the scattering factor for B, corresponding to 1.80 e.p.s. or 0.36 B a.p.f.u. The significance of this small electron density at the B2 site is not easily explained. Hundholmenite-(Y) has an apparent surplus of only 0.09 B atoms in the empirical formula and, more important, the B2–O9 bond length is 1.69(3) Å, far too long for a normal B–O bond [which is 1.365(4) Å for B2–O9 in vicanite-(Ce)]. The only reasonable candidate would seem to be a minor amount of As³⁺. The *O9* site refines to 8 e.p.s. and cannot therefore host F atoms. Its multiplicity gives a maximum of 3 OH groups p.f.u., corresponding to about 1 wt.% H₂O. This amount of water, if actually present, could not be detected in the mineral (see above). Whatever atomic species is situated at B2 in hundholmenite-(Y), it will give a positive contribution to the bond-valence sum for *O9* and decrease the amount of OH in the mineral.

The *O10* and *O11* atoms are slightly but distinctly underbonded, summing to 1.62 and 1.72 v.u., respectively (Table 7). Both atoms are too far (>5 Å) from the *O9* atom to form hydrogen bonds with *O9* as the acceptor atom and *O10* and/or *O11* as the donor atoms. The same two oxygen atoms also have low bond-valence sums in okanoganite-(Y) and vicanite-(Ce). For the remaining cation sites, the bond-valence sums are satisfactory.

Discussion

A comparison of essential data for the three vicanite-group minerals, including simplified formulae, is given in Table 8. Compared to the other two minerals, hundholmenite-(Y) has Al > Fe³⁺ at the *Al/Fe* site in layer A, has Si > As at the *Si3/As1* site in layer B, and Ca > 0.5 at the *Ca/Na* site in layer C.

Including the three-coordinated B2 site in vicanite-(Ce), the vicanite-group minerals have 13 possible cation sites, and might comprise a large number of mineral species. The contents of the four sites *Al/Fe*, *Si3/As1*, *As2* and *Ca/Na* can conveniently be used to discriminate between species of the group, as well as the presence or absence of the B2 site. Also the populations of the mixed (O,F) anion sites *O11* and *OF12*, and (F,OH,H₂O) at the *W* site should be considered. However, the correct distribution of Y, REE, Ca and Th (four variables) between the five *M* sites is not easily made on the basis of site-scattering values. It might therefore be wise to treat *M1* to *M5* as an entity for nomenclatural purposes.

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