

Mn-BEARING HELLANDITE-(Y) FROM THE HEFTETJERN PEGMATITE, TØRDAL, NORWAY

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ABSTRACT

Mn-bearing hellandite-(Y) occurs as pinkish yellow granular crystals (up to sub mm) in the Sc-rich granite pegmatite at Heftetjern, Tørdal, Telemark, Norway. Associated minerals are quartz, albite, Sc- and Ce-bearing epidote, hingganite-(Y), and an undetermined Ca-bearing hingganite-related mineral. Electron microprobe analyses give an empirical formula as $\text{Ca}_{1.34}\text{Mn}_{1.07}\text{Y}_{2.75}\text{Ce}_{0.02}\text{Nd}_{0.02}\text{Sm}_{0.01}\text{Gd}_{0.01}\text{Dy}_{0.05}\text{Er}_{0.05}\text{Yb}_{0.15}\text{Al}_{0.94}\text{Fe}_{0.08}\text{Si}_{3.99}\text{B}_{4.33}\text{O}_{22.00}(\text{OH})_{2.00}$ on the basis of $\text{Al}+\text{Fe}+\text{Si} = 5$ and 24 anions per formula unit. The lattice parameters were refined from diffraction data obtained using a Gandolfi camera with an imaging plate and Ni filtered $\text{CuK}\alpha$; a 18.693(17), b 4.651(3), c 10.178(7) Å, $\beta = 111.37(6)^\circ$, V 824.1(10) Å³. The crystal structure was refined from single-crystal XRD data obtained with a CCD-diffractometer and graphite-monochromated $\text{MoK}\alpha$. The refinement with anisotropic atomic displacement parameters converged to $R_1 = 0.0269$ for 1567 reflections [$I > 2\sigma(I)$] and 0.0318 for all 1768 reflections, resulting in the structural formula $^{M3}(\text{Ca}_{0.56}\text{Mn}_{0.44})_2^{M4}(\text{Y}_{0.43}\text{Ca}_{0.23}\text{Ln}_{0.14}\square_{0.20})_2^{M2}(\text{Y}_{0.94}\text{Ln}_{0.06})_2^{M1}(\text{Al}_{0.92}\text{Fe}_{0.08})\text{Si}_4\text{B}_4\text{O}_{21.21}(\text{OH})_{2.79}$. In this hellandite-(Y), Mn^{2+} replaces Ca^{2+} at the $M3$ site and there is a significant vacancy at the $M4$ site. The T site is vacant, and, instead, the O5 position is occupied by (OH)⁻.

Keywords: hellandite-(Y), borosilicate, manganese, Heftetjern, Norway.

INTRODUCTION

Hellandite-(Y) is the premier member of the hellandite group, which consists of borosilicate minerals having the general formula (Oberti *et al.* 2002), $X_4Y_2ZT_2\text{Si}_4\text{B}_4\text{O}_{22}W_2$ or $M3_2M4_2M2_2M1T_2\text{Si}_4\text{B}_4\text{O}_{22}(\text{OH},\text{F},\text{O})_2$, where $X = M3 + M4$; $M3 = \text{Ca}^{2+}, \text{Y}^{3+}$; $M4 = \text{Na}^+, \text{Ca}^{2+}, \text{Y}^{3+}, \text{Ln}^{3+}$ (lanthanoids, La–Lu); $Y = M2 = \text{Y}^{3+}, \text{Ln}^{3+}, \text{Th}^{4+}, \text{U}^{4+}, \text{Ca}^{2+}$; $Z = M1 = \text{Mg}, \text{Al}^{3+}, \text{Fe}^{3+}, \text{Mn}^{3+}, \text{Ti}^{4+}$; $T = \text{Li}^+, \text{Be}^{2+}, \square$ (vacancy), (H). The distinguishing feature of the crystal structure of hellandite-(Y) is $\text{B}_4\text{Si}_4\text{O}_{22}$ chains of five-membered Si- and B-tetrahedra (Mellini & Merlino 1977). The chains are connected by eight-coordinated polyhedra of Ca and rare earth elements (REE: Y + Ln)

(at the X and Y sites) and Al octahedra (the Z site) to form a three-dimensional structure. Mazzi *et al.* (1979) pointed out that filling the vacant site tetrahedrally surrounded by 4 O atoms between the chains would transform the chain structure into a sheet structure related to semenovite-(Ce) and gadolinite. The additional tetrahedrally coordinated site (T) is occupied by Be and/or Li; partially in hellandite-(Y) (Oberti *et al.* 1999) and considerably in mottanaite-(Ce) (Della Ventura *et al.* 2002). A variety of isomorphous substitutions are observed in hellandite-(Y). Cations Th^{4+} , U^{4+} , and Na^+ substitute for the essential constituents of hellandite-(Y), REE^{3+} and Ca^{2+} , at the X and Y sites. The $\text{REE}^{3+}/\text{Ca}^{2+}$ ratio varies from the ideal ratio of hellandite-

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FIG. 1. A photomicrograph of Mn-bearing hellandite-(Y).

(Y) and hellandite-(Ce) (3/3) towards those of tadhikite-(Ce) (2/4) and mottanaite-(Ce) (1/5). Replacements of Al^{3+} by Fe^{3+} , Ti^{4+} , and Mg^{2+} are usually observed in hellandite-(Y). The charge balance for substitutions between heterovalent cations is maintained by coupled substitutions to form solid solutions with complex chemistry. Tadhikite-(Ce) (Chernitsova *et al.* 1982), ciprianiite, and mottanaite-(Ce) (Della Ventura *et al.* 2002) are the other endmembers of solid solution series, and are independent species in the hellandite group. Manganese

has been reported as a minor constituent of hellandite-(Y), *e.g.*, with only 0.41 wt.% MnO in Norwegian material (Ofteidal 1965), 0.58 wt.% in Canadian material (Hogarth *et al.* 1972), and 0.70 wt.% in an Italian sample (Mellini & Merlino 1977). Mellini & Merlino (1977) described the minor Mn (less than 0.1 *apfu*) as the divalent (Mn^{2+}) substituent for Ca^{2+} in hellandite-(Y). On the other hand, Oberti *et al.* (1999) and Della Ventura *et al.* (2002) considered the slight amounts of Mn (not greater than 0.02 *apfu*) to be trivalent (Mn^{3+}) substituting for Al^{3+} at the Z (M1) site in hellandite-(Y) and ciprianiite.

Here, we describe Mn-bearing (greater than 1 *apfu*) hellandite-(Y) occurring as tiny pinkish yellow granular crystals (Fig. 1) in the Sc-rich granite pegmatite at Heftetjern, Tørdal, Telemark, Norway.

OCCURRENCE AND GEOLOGICAL BACKGROUND

The Heftetjern cleavelandite-amazonite pegmatite (Bergstøl & Juve 1988, Juve & Bergstøl 1990) is situated in a marshy area between Høydaalen and Skarsfjell in Tørdal, Telemark county, southern Norway, at an altitude of ~650 m above sea level, at 59°8.6' N, 8°45.4' E (Fig. 2). A striking geochemical feature of the Heftetjern pegmatite is the high level of Sc. Beryllium, Y, Sn, and Ta are also characteristic elements. The mineralogy and geochemistry of the Heftetjern pegmatite have been reported and

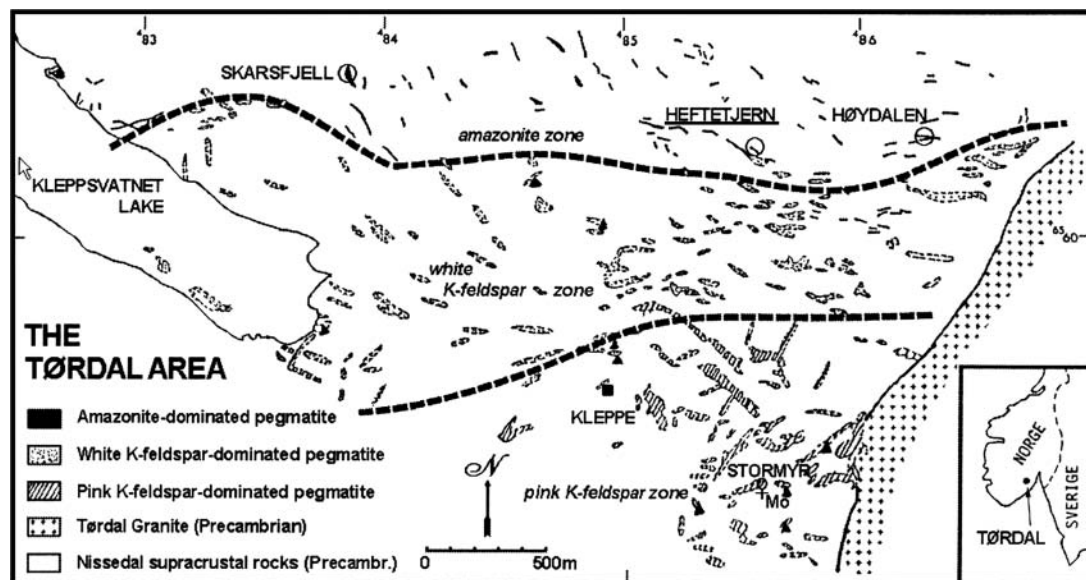


FIG. 2. Regional geological sketch-maps of Heftetjern, Tørdal, Telemark, Norway, reproduced after Segalstad & Raade (2003). Stippled lines separate zones with pegmatites dominated by green amazonite, white K-feldspar, and pink K-feldspar.

TABLE 1. CHEMICAL COMPOSITION OF Mn-BEARING HELLANDITE-(Y)

wt. %	1	2	3	4	5	6	mean	s.d.	Tahara**	Predazzo***	Wakefield****
Al ₂ O ₃	4.78	4.73	4.96	4.92	4.88	4.85	4.85	0.08	3.82	3.04	3.42
Fe ₂ O ₃	0.54	0.62	0.69	0.60	0.70	0.59	0.62	0.06	0.45	3.77	1.70
TiO ₂	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0.85	<0.2	0.117
Mn ₂ O ₃											0.478
CaO	7.62	7.79	7.64	7.66	7.65	7.56	7.65	0.07	16.64	15.22	12.74
SrO	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]				0.059
MnO	7.72	7.65	7.72	7.78	7.61	7.78	7.71	0.06	0.92	0.70	
Y ₂ O ₃	31.85	31.86	30.77	30.94	32.17	32.02	31.60	0.54	21.58	18.07	28.11
La ₂ O ₃	0.08	0 [†]	0.01	0 [†]	0.13	0 [†]	0.04	0.05	0 [†]	16.0 [‡]	0.113
Ce ₂ O ₃	0.44	0.24	0.31	0.31	0.13	0.33	0.29	0.09	1.10		0.133
Pr ₂ O ₃	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.162
Nd ₂ O ₃	0.37	0.36	0.37	0.41	0.34	0.40	0.37	0.02	2.09		1.90
Sm ₂ O ₃	0.16	0.22	0.23	0.31	0.18	0.39	0.25	0.08	1.10		1.89
Eu ₂ O ₃	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.092
Gd ₂ O ₃	0.39	0.08	0.25	0.15	0.24	0.02	0.19	0.12	1.49		3.23
Dy ₂ O ₃	0.81	0.94	0.78	1.29	0.88	0.70	0.90	0.19	1.97		3.56
Ho ₂ O ₃	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0.58		
Er ₂ O ₃	1.00	0.92	0.73	1.14	0.82	0.95	0.93	0.13	1.32		2.42
Yb ₂ O ₃	2.88	3.05	2.74	3.38	2.83	3.06	2.99	0.21	1.50		1.96
ThO ₂	0 [†]	0 [†]	0.15	0.17	0 [†]	0.24	0.09	0.10	0 [†]		0.885
UO ₂	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.130
SiO ₂	24.55	24.51	24.46	24.27	24.05	24.27	24.35	0.17	24.84	25.57	22.25
B ₂ O ₃	15.97	16.12	15.53	14.06	15.81	14.45	15.32	0.78	14.39	14.85	12.88
BeO											0.041
Li ₂ O											0.003
H ₂ O	1.28 ^c	1.14 ^c	2.20 ^c	2.78 ^c	1.25 ^c	2.31 ^c	1.83 ^c		3.62 ^c	4.50 ^c	1.40
F	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]				0.216
O = F											-0.091
Total	100.44	100.23	99.54	100.17	99.67	99.92	99.98		98.26		99.74
<i>apfu</i>	1	2	3	4	5	6	Mean				
Al	0.92	0.91	0.95	0.95	0.95	0.94	0.94		0.73	0.56	0.712
Fe	0.07	0.08	0.08	0.07	0.09	0.07	0.08		0.06	0.45	0.226
Ti	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0.11	<0.03	0.016
Mn											0.021
ΣAlFe	0.99	0.99	1.03	1.02	1.04	1.01	1.01		0.90	1.04	0.975
Ca	1.33	1.37	1.33	1.34	1.35	1.33	1.34		2.88	2.55	2.412
Sr	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]				0.002
Mn	1.07	1.06	1.06	1.08	1.06	1.08	1.07		0.13	0.09	
ΣCaMn	2.40	2.43	2.39	2.42	2.41	2.41	2.41		3.01	2.64	2.414
Y	2.77	2.77	2.66	2.70	2.82	2.80	2.75		1.85	1.51	2.640
La	0.00	0 [†]	0.00	0 [†]	0.01	0 [†]	0.00		0 [†]	0.78 [‡]	0.007
Ce	0.03	0.01	0.02	0.02	0.01	0.02	0.02		0.55		0.009
Pr	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.010
Nd	0.02	0.02	0.02	0.02	0.02	0.02	0.02		1.05		0.120
Sm	0.01	0.01	0.01	0.02	0.01	0.02	0.01		0.06		0.115
Eu	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.006
Gd	0.02	0.00	0.01	0.01	0.01	0.00	0.01		0.08		0.189
Dy	0.04	0.05	0.04	0.07	0.05	0.04	0.05		0.11		0.203
Ho	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0.03		
Er	0.05	0.05	0.04	0.06	0.04	0.05	0.05		0.07		0.135
Yb	0.14	0.15	0.14	0.17	0.14	0.15	0.15		0.08		0.105

TABLE 1. (CONTINUED)

wt. %	1	2	3	4	5	6	mean	s.d.	Tahara**	Predazzo***	Wakefield****
Th	0 [†]	0 [†]	0.01	0.01	0 [†]	0.01	0.00		0 [†]		0.036
U	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]		0 [†]		0.005
ΣREE	3.08	3.06	2.95	3.08	3.11	3.11	3.06		2.45	2.29	3.580
Si	4.01	4.01	3.97	3.98	3.96	3.99	3.99		4	4	3.933
B	4.51	4.55	4.35	3.98	4.50	4.10	4.33		4	4	3.930
Be											0.017
Li											0.002
H	1.39	1.25	2.38	3.04	1.37	2.54	2.00		3.89	2.34	1.647
F	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]	0 [†]				0.121

*Present study. **Miyawaki *et al.* (1987). ***Mellini & Merlino (1977). ****Oberti *et al.* (2002).

[†]Under the detection limit, <0.1 wt.% for F and <0.01 wt.% for the others. [‡]Total Ln, lanthanoids, La–Lu.

^c Calculated.

discussed on several occasions (Raade *et al.* 2002, Cooper *et al.* 2006, Lussier *et al.* 2009, Kristiansen 2009, Kolitsch *et al.* 2010). The numerous granite pegmatites of the Tørdal area are situated within the Nissedal volcano-sedimentary outlier. Both the outlier (1300–1200 Ma) and the older basement gneiss (1520–1500 Ma) are intruded by the Tørdal granite (960–850 Ma), which is regarded as the source of the pegmatite swarms (Bergstøl & Juve 1988). The Rb–Sr dating of lepidolite and microcline from the Skarsfjell pegmatite gave ages between 950 and 880 Ma (Neumann 1960), but Kulp *et al.* (1963) estimated the age to be closer to 1000 Ma. From trace-element analyses of different rock types it has been suggested that some of the tin and a major part of the scandium in the cleavelandite-amazonite pegmatites of the area probably have their source in the volcanogenic rocks of the Nissedal outlier, which were penetrated by the pegmatitic fluids (Bergstøl & Juve 1988). The Tørdal area in Telemark is geochemically different from the other granite pegmatite areas in south Norway, being characterized by tin, scandium, yttrium, beryllium, and lithium. The cleavelandite-amazonite pegmatites at the nearby Høydalen and at Skarsfjell were described by Oftedal (1942). Here, “zinnwaldite” and

cassiterite are the most common Li- and Sn-minerals, respectively.

The Heftetjern pegmatite is famous for its unique assemblage of Sc-minerals and Sc-bearing minerals, such as the new species kristiansenite (Raade *et al.* 2002), oftedalite (Cooper *et al.* 2006), and heftetjernite (Kolitsch *et al.* 2010), as well as bazzite (Juve & Bergstøl 1990), scandiobabingtonite and cascandite (Raade & Erambert 1999), and thortveitite (Kristiansen 2009); the latter is also intimately intergrown with three of the former (Raade *et al.* 2004). According to the classification of Černý (1992), the Tørdal pegmatites are of mixed LCT-NYF (Li, Cs, Ta - Nb, Y, F) affinity.

CHEMICAL COMPOSITION

The chemical composition was determined by means of a JEOL 8800M electron microprobe analyzer (WDS mode, 15 kV, 20 nA, and 2 µm beam diameter) employing standard materials as follows; wollastonite for Ca and Si, rhodonite for Mn, sillimanite for Al, fayalite for Fe, danburite for B, REEP₅O₁₄ for each rare earth element (Y and lanthanoids, La–Lu), ThO₂ for Th, and UO₂ for U. The B $\bar{K}\alpha$ analytical line was monochromatized by the LDE2 layered dispersion element on the spectrometer. No other element with atomic number ≥ 5 , except for C from carbon coating and O as a constituent, was detected. The results of chemical analyses with ZAF corrections for six points within the fragment used for the Gandolfi X-ray diffraction investigation are given together with the mean values in Table 1. A few sets of chemical data for Mn-bearing hellandite-(Y) from the literature are also given for comparison. The empirical formula derived from the mean data is Ca_{1.34}Mn_{1.07}Y_{2.75}Ce_{0.02}Nd_{0.02}Sm_{0.01}Gd_{0.01}Dy_{0.05}Er_{0.05}Yb_{0.15}Al_{0.94}Fe_{0.08}Si_{3.99}B_{4.33}O_{22.00}(OH)_{2.00} on the basis of Al + Fe + Si = 5 and 24 anions per formula unit. Oberti *et al.* (2002) pointed out that the hellandite group minerals show an almost constant 4 Si and 4 B

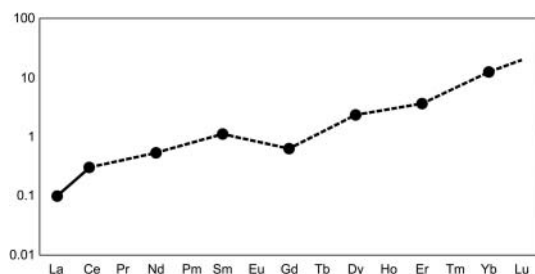


FIG. 3. Chondrite-normalized lanthanoid distribution patterns of Mn-bearing hellandite-(Y).

TABLE 2. POWDER X-RAY DIFFRACTION DATA FOR THE Mn-BEARING HELLANDITE-(Y)

$//h_0$	Hefetjern Present Study		h	k	l	Tahara Miyawaki <i>et al.</i> (1987)		Predazzo Mellini & Merlino (1977)	
	d_{meas}	d_{calc}				l	d'	l_{calc}	d_{calc}
2	9.50	9.44	0	0	1				
2	8.73	8.66	2	0	0	vw	8.60	13	8.84
5	8.03	8.01	$\bar{2}$	0	1				
10	5.48	5.46	2	0	1	w	5.49	9	5.57
10	5.00	4.98	$\bar{2}$	0	2	vw	5.03	1	5.06
60	4.66	4.63	0	1	0	s	4.69	43	4.72
3	4.53	4.56	$\bar{3}$	0	2				
5	4.23	4.20	$\bar{1}$	1	1			1	4.27
11	4.11	4.08	2	1	0	m	4.12	24	4.16
6	4.04	4.01	$\bar{2}$	1	1				
3	3.68	3.67	$\bar{3}$	1	1			3	3.73
3	3.64	3.62	2	0	2			1	3.69
4	3.55	3.53	2	1	1				
21	3.50	3.48	4	0	1	w	3.53	13	3.55
66	3.41	3.39	$\bar{2}$	1	2	s	3.42	74	3.45
7	3.33	3.31	0	1	2			11	3.36
4	3.27	3.25	$\bar{3}$	1	2			2	3.31
35	3.17	3.16	4	1	0	s	3.19	84	3.22
		3.15	$\bar{4}$	0	3			20	3.20
		3.15	0	0	3			12	3.20
		3.10	$\bar{6}$	0	1	w	3.14	13	3.16
63	3.04	3.03	$\bar{4}$	1	2	s	3.08	74	3.08
63	2.87	2.85	2	1	2	s	2.89	100	2.91
87	2.80	2.78	4	1	1	s	2.82	85	2.84
5	2.75	2.73	$\bar{2}$	1	3			1	2.78
10	2.67	2.66	2	0	3			9	2.71
100	2.62	2.61	4	1	3	s	2.64	76	2.65
		2.60	0	1	3			72	2.65
59	2.59	2.57	$\bar{6}$	1	1	s	2.62	80	2.62
10	2.53	2.52	$\bar{2}$	0	4	w	2.56	12	2.56
		2.52	$\bar{6}$	1	2			4	2.57
7	2.46	2.45	6	1	0	vw	2.48	11	2.50
17	2.37	2.36	0	0	4	vw	2.39	16	2.40
33	2.33	2.32	0	2	0	m	2.34	13	2.36
11	2.29	2.31	$\bar{8}$	0	2			20	2.36
14	2.27	2.28	$\bar{6}$	0	4	vw	2.31	13	2.32
14	2.23	2.22	$\bar{3}$	1	4	w	2.25	22	2.26
16	2.19	2.18	7	1	0	w	2.21	23	2.23
43	2.15	2.15	$\bar{7}$	1	3	m	2.17	23	2.19
		2.14	6	0	2			18	2.18
		2.14	3	1	3			25	2.18
15	2.11	2.10	0	1	4	w	2.13	8	2.14
12	2.08	2.07	$\bar{8}$	1	2	vw	2.10	6	2.11
7	2.06	2.06	$\bar{8}$	1	1			4	2.10
		2.05	$\bar{6}$	1	4			4	2.08
8	2.01	2.00	$\bar{4}$	2	2			3	2.04
31	1.936	1.941	6	1	2			10	1.979
		1.928	4	2	1	m	1.950	24	1.964
46	1.871	1.866	$\bar{4}$	2	3	s	1.886	34	1.899
		1.865	0	2	3			30	1.897
		1.854	$\bar{6}$	2	1			36	1.889

TABLE 2. (CONTINUED)

hkl_0	Heftetjern Present Study		h	k	l	Tahara Miyawaki <i>et al.</i> (1987)		Predazzo Mellini & Merlino (1977)	
	d_{meas}	d_{calc}				l	d'	l_{calc}	d_{calc}
12	1.829	1.826	$\overline{10}$	0	1			4	1.864
11	1.825	1.825	$\overline{10}$	0	3			4	1.861
9	1.790	1.782	$\overline{8}$	0	5	w	1.816	12	1.813
18	1.739	1.741	8	0	2	w	1.766	16	1.776
		1.732	10	0	0			12	1.768
		1.731	$\overline{10}$	0	4			11	1.763
6	1.713	1.718	2	0	5			11	1.747
20	1.695	1.691	$\overline{4}$	0	6	w	1.714	16	1.717
		1.687	4	1	4			14	1.718
10	1.669	1.663	$\overline{8}$	1	5			6	1.692
10	1.661	1.653	0	2	4			13	1.681
		1.637	$\overline{8}$	2	2			18	1.668
		1.622	10	1	0	m	1.645	21	1.656
21	1.594	1.588	$\overline{4}$	1	6	w	1.613	31	1.613
19	1.577	1.571	6	2	2	w	1.590	22	1.601
7	1.552	1.552	2	2	4			2	1.579
		1.544	0	3	0			7	1.572
3	1.533	1.539	8	0	3				
3	1.517	1.512	$\overline{12}$	0	1			3	1.543
3	1.505	1.499	$\overline{12}$	0	4			2	1.529
6	1.464	1.464	$\overline{12}$	1	3				
		1.440	$\overline{4}$	3	2	vw	1.454	4	1.466
		1.437	$\overline{12}$	1	1			4	1.467
6	1.433	1.427	$\overline{12}$	1	4			8	1.454
7	1.418	1.412	$\overline{8}$	2	5	vw	1.429	2	1.437
2	1.407	1.386	$\overline{4}$	3	3	w	1.402	9	1.411
17	1.392	1.386	0	3	3			8	1.410

apfu. The excess B in the present analysis possibly comes from errors in the ZAF corrections, especially an overestimation for the absorption (A) correction, in addition to the relatively large standard deviation (0.78 for B_2O_3 in Table 1). A *proforma* calculation based on $B = 4$, $Al + Fe + Si = 5$ and 24 anions per formula unit gave lower B_2O_3 , 14.16 wt.%, and increased H_2O , 2.73 wt.%, with 99.73 total %, leading to the formula $Ca_{1.34}Mn_{1.07}Y_{2.75}Ce_{0.02}Nd_{0.02}Sm_{0.01}Gd_{0.01}Dy_{0.05}Er_{0.05}Yb_{0.15}Al_{0.94}Fe_{0.08}Si_{3.99}B_4O_{21.02}(OH)_{2.98}$.

The chondrite-normalized lanthanoid distribution patterns of Mn-bearing hellandite-(Y) show an increasing trend with enrichment of heavy REEs having smaller ionic radii comparable to Y (Fig. 3). This trend can typically be observed in many of the Y-predominant REE minerals. Nevertheless, the trend is not a simple slope, but shows the second peak at Pr, one of the light REEs. The lanthanoids content, which was not determined with the electron microprobe, was estimated by means of interpolation with those of the determined lanthanoids having neighboring atomic numbers; 0.03Lu, 0.02Tm, 0.01Ho,

0.05Tb, 0.04Eu, and 0.03Pr *apfu*. The chemical formula including the infinitesimal lanthanoids with charge compensating H is $Ca_{1.34}Mn_{1.07}Y_{2.75}Ln_{0.38}Al_{0.94}Fe_{0.08}Si_{3.99}B_4O_{21.23}(OH)_{2.77}$, which was used for the occupancy parameters in the crystal structure refinement.

Hellandite-(Y) from the Heftetjern pegmatite, Tørdal, Norway, shows a notable chemical feature in the enrichment of Mn with deficiency of Ca (Table 1). As the amounts of Al and Fe are sufficient to fill the octahedral *M1* (or *Z*) site, Mn probably occupies the *M2*, *M3*, and/or *M4* (*X*, *Y*) sites together with Ca, Y, and Ln (lanthanoids; La–Lu). The distribution of the cations among the *M* sites was examined in the crystallographic investigation as described below. The valence of Mn was estimated as divalent after a bond-valence calculation based on the crystal chemistry, *i.e.*, the isomorphous substitution between Ca and Mn. Our microprobe analyses show a deficiency of the cations at the *M2*, *M3*, and *M4* sites, $(Ca_{1.34}Mn_{1.07}Y_{2.75}Ln_{0.38})_{\Sigma 5.54}$, relative to the ideal 6 *apfu*.

TABLE 3. CRYSTAL DATA, DATA COLLECTION INFORMATION, AND REFINEMENT DETAILS

Chemical formula	$\text{Ca}_{1.34}\text{Mn}_{1.07}\text{Y}_{2.75}\text{Yb}_{0.38}\text{Al}_{0.92}\text{Fe}_{0.08}\text{Si}_4\text{B}_4\text{O}_{21.21}(\text{OH})_{2.79}$
Temperature	296 K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/a$ (# 13)
Unit cell parameters	$a = 18.609(8)$ Å $b = 4.6307(19)$ Å $c = 10.143(4)$ Å $\beta = 111.475(4)^\circ$ $V = 813.4(6)$ Å ³ $Z = 2$
Density (calculated)	4.06 Mg/m ³
Absorption coefficient	13.70 mm ⁻¹
$R(000)$	944
Crystal size	0.12 × 0.07 × 0.05 mm ³
Oscillation width	0.5° (in ω)
Exposure time per frame	5 s
Theta range for data collection (θ)	2.35 to 27.18°
Index ranges	$\bar{1}7 \leq h \leq 23$, $5 \leq k \leq 4$, $\bar{1}2 \leq l \leq 12$
Reflections collected	4089
Independent reflections	1768
Reflections [$I > 2\sigma(I)$]	1567
R_{int}	0.0208
Refinement method	Full-matrix least-squares on F^2
restraints / parameters	6 / 191
Goodness-of-fit on F^2	0.784
Final R indices [$I > 2\sigma(I)$]	$R1 = 0.0269$, $wR2 = 0.0879$
R indices (all data)	$R1 = 0.0318$, $wR2 = 0.0934$
Largest diff. peak and hole	1.082 and -1.013 e/Å ³

X-RAY CRYSTALLOGRAPHY

X-ray diffraction

The powder X-ray diffraction pattern was obtained from Debye rings recorded on an imaging plate in a Gandolfi camera with a diameter of 114.6 mm using Ni filtered $\text{CuK}\alpha$ radiation. The data were processed with a Fuji BAS-2500 bio-image analyzer using a computer program written by Nakamuta (1999). The lattice parameters were refined with diffraction angle data calibrated with a Si-standard reference material (NBS #640b) and using a computer program by Toraya (1993). Table 2 shows the X-ray powder diffraction data for Mn-bearing hellandite-(Y). The lattice parameters were refined in the monoclinic system from the diffraction data: a 18.693(17), b 4.651(3), c 10.178(7) Å, β 111.37(6)°, V 824.1(10) Å³, which are very close to those of hellandite-(Y) in the published literature (e.g., Mellini & Merlino 1977, Oberti *et al.* 1999, Oberti *et al.* 2002).

Refinement of the crystal structure

The X-ray diffraction data for crystal structure analysis were collected from a single crystal of the Mn-bearing hellandite, which was picked from the thin section used for the electron microprobe analyses. The measurements were obtained with a Bruker AXS SMART APEX 2 diffractometer equipped with a CCD area-detector using ω -scans (0.5°/min) and utilizing graphite-monochromated $\text{MoK}\alpha$ radiation ($\lambda = 0.71073$ Å) with 7 seconds per frame exposure time. The reduction and correction for the Lorentz-polarization factors and background effects were carried out as well as an empirical correction for absorption (SAINTPLUS and SADABS). The lattice parameters and diffraction experimental conditions are summarized in Table 3.

The structure was solved using direct methods and referencing the results of the crystal structure analysis by Mellini & Merlino (1977) and was refined with the SHELXTL program (Sheldrick 2008). A full-matrix least-squares refinement with anisotropic atomic

TABLE 4. OCCUPANCY PARAMETERS, ATOMIC FRACTIONAL COORDINATES, EQUIVALENT ISOTROPIC AND ANISOTROPIC ATOMIC DISPLACEMENT PARAMETERS (Å²)

	Occupancy	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}	
Al1	0.92Al 0.08Fe	0	0	0	0.0138(3)	
Y2	0.943(2)Y 0.0567(17)Yb	0.042313(17)	0.02021(7)	0.35947(3)	0.00774(16)	
Ca3	0.560(8)Ca 0.440(8)Mn	0.24672(4)	0.00215(12)	0.65778(7)	0.0104(2)	
Y4	0.433(3)Y 0.231(8)Ca 0.1365(15)Yb 0.000(8)Mn	0.15507(3)	-0.04275(10)	0.92895(5)	0.01532(18)	
Si1	Si	0.09970(5)	0.48676(18)	0.64705(9)	0.0092(3)	
Si2	Si	0.11117(6)	0.49617(18)	0.16171(11)	0.0106(2)	
B1	B	0.1710(2)	0.5301(8)	0.4522(5)	0.0108(8)	
B2	B	0.2544(2)	0.4559(8)	0.1348(4)	0.0102(8)	
O1	O	0.03942(13)	0.2433(5)	0.5624(2)	0.0116(5)	
O2	O	0.17507(13)	0.3119(5)	0.7570(3)	0.0156(5)	
O3	O	0.06910(13)	-0.2935(5)	0.7363(2)	0.0139(5)	
O4	O	0.13177(14)	-0.3257(5)	0.5423(3)	0.0148(5)	
O5	O	0.03821(13)	0.1900(6)	0.8705(2)	0.0127(5)	
O6	O	0.24417(13)	-0.2388(5)	0.8603(3)	0.0149(5)	
O7	O	0.16602(13)	0.2257(5)	0.4484(2)	0.0133(5)	
O8	O	0.12932(15)	0.6732(5)	0.3096(3)	0.0191(6)	
O9	O	0.18864(14)	0.3301(6)	0.1646(3)	0.0252(7)	
O10	O	0.08544(13)	0.7371(5)	0.0408(3)	0.0129(5)	
O11	O	0.05237(13)	0.2411(5)	0.1547(2)	0.0127(5)	
O12	O	0.25	0.3245(8)	0	0.0266(9)	
O13	O	0.25	0.6503(8)	0.5	0.0184(8)	
H5	H	0.036(5)	0.427(19)	0.882(9)	0.14(4) <i>U</i> _{iso}	
	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₂₃	<i>U</i> ₁₃	<i>U</i> ₁₂
Al1	0.0153(8)	0.0146(7)	0.0125(8)	-0.0006(5)	0.0064(6)	-0.0006(5)
Y2	0.0059(2)	0.0096(2)	0.0084(2)	-0.00031(9)	0.00347(16)	-0.00046(9)
Ca3	0.0111(4)	0.0096(3)	0.0099(4)	-0.00089(19)	0.0033(3)	-0.0001(2)
Y4	0.0147(3)	0.0167(3)	0.0185(3)	0.00353(15)	0.0107(2)	0.00422(15)
Si1	0.0076(6)	0.0106(5)	0.0099(6)	0.0000(3)	0.0040(5)	-0.0002(3)
Si2	0.0081(5)	0.0119(5)	0.0113(5)	-0.0002(3)	0.0031(4)	0.0001(3)
B1	0.0102(19)	0.0083(18)	0.015(2)	-0.0018(13)	0.0055(16)	0.0000(13)
B2	0.0086(19)	0.0108(19)	0.0117(19)	0.0001(14)	0.0041(15)	-0.0013(13)
O1	0.0111(11)	0.0108(12)	0.0127(12)	-0.0004(9)	0.0041(9)	-0.0012(9)
O2	0.0087(12)	0.0128(12)	0.0217(13)	0.0007(10)	0.0014(10)	0.0013(9)
O3	0.0110(12)	0.0158(12)	0.0156(12)	0.0000(10)	0.0058(10)	0.0028(9)
O4	0.0145(12)	0.0124(12)	0.0196(13)	0.0017(10)	0.0089(10)	0.0011(9)
O5	0.0113(12)	0.0142(13)	0.0146(12)	-0.0018(9)	0.0069(10)	-0.0008(9)
O6	0.0126(12)	0.0124(13)	0.0199(13)	0.0014(10)	0.0063(10)	0.0018(9)
O7	0.0100(11)	0.0124(13)	0.0162(13)	0.0012(9)	0.0033(10)	-0.0021(9)
O8	0.0250(14)	0.0131(13)	0.0140(12)	-0.0014(10)	0.0009(11)	0.0036(11)
O9	0.0121(13)	0.0203(14)	0.0480(18)	0.0152(13)	0.0169(12)	0.0056(11)
O10	0.0079(11)	0.0155(12)	0.0144(12)	0.0016(9)	0.0029(9)	0.0017(9)
O11	0.0099(11)	0.0165(12)	0.0113(12)	-0.0006(9)	0.0032(9)	-0.0036(9)
O12	0.054(3)	0.0130(19)	0.0133(18)	0	0.0128(18)	0
O13	0.0126(17)	0.0144(18)	0.030(2)	0	0.0094(15)	0

displacement parameters was carried out. Scattering factors for neutral atoms were taken from the *International Tables for X-ray Crystallography Volume C* (Wilson 1992). The scattering factor curve for Yb, the predominant lanthanoid in the crystal, was adopted as the representative curve for all of the

lanthanoids. Site occupancy factors were fixed or restrained on the basis of the chemical composition and the cation ordering for hellandite described by Oberti *et al.* (2002). The occupancy of the Al1 (*M*₁) site was fixed at 0.92Al + 0.08Fe. The scattering factors of Fe and Mn are similar, and gave no

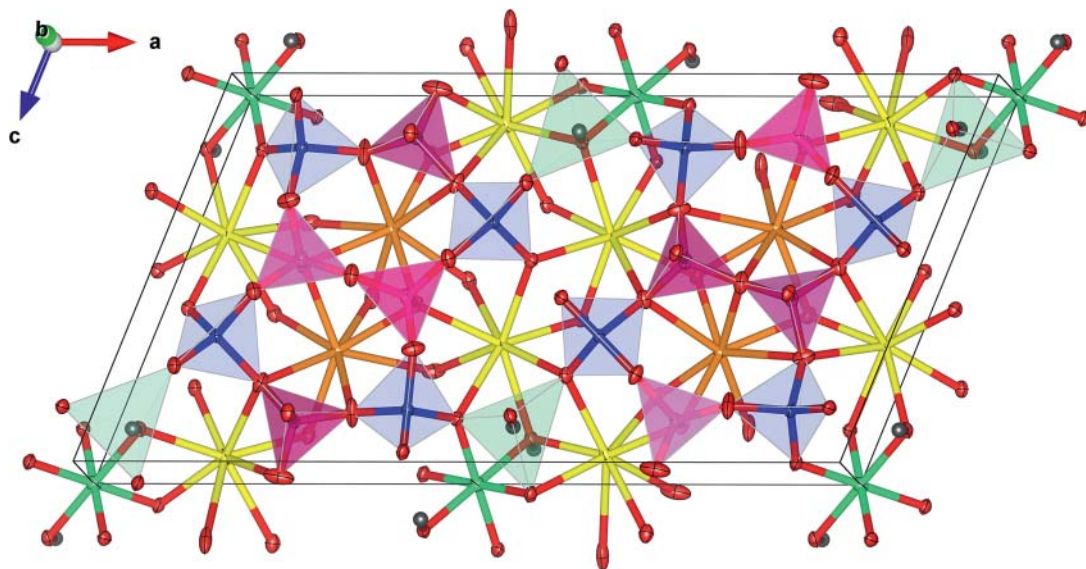


FIG. 4. VESTA (Momma & Izumi 2011) illustration of the crystal structure of Mn-bearing hellandite-(Y). Sites are indicated by colors as follows: green for Al1, orange for Ca3, yellow for Y2 and Y4, blue for Si, pink for B, red for O, and dark gray for H. The Si, B, and Z (vacant in this case) tetrahedra are illustrated in shades of blue, pink, and pale green, respectively.

TABLE 5. INTERATOMIC DISTANCES (Å)

Al1	—O11	1.881(2)	Y2	—O1	2.305(2)
	—O11	1.881(2)		—O3	2.317(2)
	—O5	1.921(2)		—O1	2.322(3)
	—O5	1.921(2)		—O7	2.345(2)
	—O10	1.923(2)		—O11	2.383(2)
	—O10	1.923(2)		—O8	2.462(3)
	mean	1.908		—O5	2.464(2)
H5	—O5	1.11(9)	Y4	—O4	2.553(2)
	—O13	2.300(3)		mean	2.394
	—O9	2.327(3)		—O6	2.214(3)
	—O7	2.346(2)		—O10	2.254(2)
	—O6	2.353(3)		—O5	2.302(2)
	—O2	2.414(3)		—O3	2.332(2)
	—O7	2.480(3)		—O12	2.367(3)
Ca3	—O4	2.534(3)	Si2	—O6	2.444(3)
	—O8	2.682(3)		—O2	2.521(3)
	mean	2.430		—O9	2.827(3)
	—O3	1.599(3)		mean	2.408
	—O1	1.600(2)		—O11	1.594(2)
	—O4	1.644(3)		—O10	1.596(3)
	—O2	1.650(2)		—O9	1.625(3)
Si1	mean	1.623	B2	—O8	1.633(3)
	—O7	1.412(4)		mean	1.612
	—O13	1.479(4)		—O6	1.413(4)
	—O4	1.517(5)		—O12	1.470(4)
	—O8	1.520(5)		—O9	1.484(5)
	mean	1.482		—O2	1.522(5)
				mean	1.473

TABLE 6. BOND-VALENCE TABLE FOR Mn-BEARING HELLANDITE-(Y)

Al1			Y2		Ca3		Y4		Si1		Si2		B1		B2		
O1			0.458 x1	x1					1.067 x1	x1							1.962
			0.437 x1	x1													
O2					0.249 x1	x1	0.194 x1	x1	0.932 x1	x1					0.665 x1	x1	2.039
O3			0.443 x1	x1			0.323 x1	x1	1.070 x1	x1							1.836
O4			0.234 x1	x1	0.180 x1	x1			0.947 x1	x1			0.674 x1	x1			2.035
O5	0.457 x2	x1	0.298 x1	x1			0.350 x1	x1									1.105
O6					0.293 x1	x1	0.444 x1	x1							0.888 x1	x1	1.863
							0.238 x1	x1									
O7			0.411 x1	x1	0.299 x1	x1							0.895 x1	x1			1.813
					0.208 x1	x1											
O8			0.300 x1	x1	0.121 x1	x1					0.976 x1	x1	0.669 x1	x1			2.065
O9					0.315 x1	x1	0.085 x1	x1			0.997 x1	x1			0.737 x1	x1	2.134
O10	0.459 x2	x1					0.398 x1	x1			1.079 x1	x1					1.936
O11	0.512 x2	x1	0.371 x1	x1							1.084 x1	x1					1.967
O12							0.293 x1	x2							0.765 x1	x2	2.117
O13					0.339 x1	x2							0.747 x1	x2			2.171
	2.857		2.952		2.003		2.324		4.016		4.136		2.984		3.055		

significant difference in the parameters correlating the occupancies in the refinement. Ferric iron was adopted as the substituent for Al^{3+} in preference to Mn^{3+} at the Al1 ($M1$) site. However, Mn was considered as Mn^{2+} replacing Ca^{2+} at the Ca3 ($M3$) and Y4 ($M4$) sites in the refinement. The occupancy factors of Y + Yb at the Y2 ($M2$), Ca + Mn at the Ca3 ($M3$), and Y + Ca + Yb + Mn at the Y4 ($M4$) sites were refined but were constrained on the basis of chemical composition. The refinement converged to $R_1 = 0.0286$ for 1567 reflections [$I > 2\sigma(I)$] and 0.0334 for all 1768 reflections.

The final positional and displacement parameters are listed in Table 4. Interatomic distances in the coordination polyhedra are summarized in Table 5. Figure 4 shows an illustration of the crystal structure created using *VESTA* (Momma & Izumi 2011). The bond valences were calculated from the interatomic distances following the procedure of Brown & Altermatt (1985) and using the parameters of Brese & O'Keeffe (1991). The values (Table 6) are weighted averages according to the occupancy parameters in the final refinement.

DISCUSSION

Oberti *et al.* (2002) suggested that Li and/or Be often occupy the T site in hellandite-(Y), instead of H bonding to O at the O5 site, one of the four corners of the T tetrahedron. The difference-Fourier maps of the present crystal structure analysis indicated an electron density (0.9 e) with a distance of 0.98 Å to the O5 site. The final cycle of calculation, refining the fractional coordinates and isotropic displacement parameters of H at this position, converged with the lowest R factors and resulted in crystallographically reasonable interatomic distances. Several comparative calculations with electron density at the T site gave no indication of Li or Be but might represent negligible small occupancies or drift from the H position neighboring the O5 site. The average T -O distance, *ca.* 1.80 Å, corresponds to 3.5 electrons at the T site based on estimation of the (Li + Be) content at the T site from the T -O distance (Oberti *et al.* 2002). The distance between the T site and H neighboring O5 is only 0.7 Å, indicating no simultaneous occupations of H (and Li and/or Be) within the T tetrahedron. Oberti *et al.* (1999) pointed out that the presence of (Be, Li) at the T site is coupled with the substitution of F and/or O for OH at the O5 position. Fluorine was not detected in the electron microprobe analyses. The bond valence sum for the O5 site, 1.07, demonstrates OH⁻ to be predominant there. Consequently, the concentration of Li and/or Be can be insubstantial in the Mn-bearing hellandite-(Y).

The occupancies of the Y2 ($M2$), Ca3 ($M3$), and Y4 ($M4$) sites were refined as 0.943(2)Y + 0.0567(17)Yb, 0.560(8)Ca + 0.440(8)Mn, and 0.433(3)Y + 0.231(8)Ca + 0.1365(15)Yb + 0.000(8)Mn, respectively. The

refinement indicates that there is little Mn at the Y4 ($M4$) site. It is noteworthy that the deficiency of the cations was confirmed as a 0.2 vacancy at the Y4 ($M4$) site. Oberti *et al.* (1999) concluded that no vacancies occur at the M sites in hellandite-(Y) from Latium (Italy), after their complete chemical and structural analyses. On the contrary, the Mn-bearing hellandite-(Y) appears to have a vacancy at the Y4 ($M4$) site.

The cation chemistry of Mn-bearing hellandite-(Y) requires 2.77 (OH) for the 24 anions, namely 24 O in the F-free hellandite-(Y), per formula unit. Approximately 2 (OH) can be at the O5 site. The bond-valence sum (Table 6) suggests that the residual 0.77 (OH) may distribute among O3, O6, and O7, having lower values 1.836, 1.863, and 1.813, respectively. Although the O7 shows the lowest value, the other two, O3 and O7, bond to Y4 ($M4$) and are speculated to be occupied by (OH).

The Mn ion in the Mn-bearing hellandite-(Y) from Hefsetjern, Tørdal, Telemark, Norway is divalent and replaces Ca at the eight-coordinated Ca3 ($M3$) site in the crystal structure. Many Mn-minerals have been recognized in the Hefsetjern pegmatite; helvine [$\text{Mn}^{2+}_4\text{Be}_3(\text{SiO}_4)_3\text{S}$], spessartine [$\text{Mn}^{2+}_3\text{Al}_2(\text{SiO}_4)_3$], scandiobabingtonite [$\text{Ca}_2(\text{Fe}^{2+}, \text{Mn})\text{ScSi}_5\text{O}_{14}(\text{OH})$], and oftedalite [$(\text{Sc}, \text{Ca}, \text{Mn}^{2+})_2\text{K}(\text{Be}, \text{Al})_3\text{Si}_{12}\text{O}_{30}$] (Cooper *et al.* 2006), of which the latter two show the substitution of Mn for Ca. The Mn-bearing hellandite-(Y) suggests a potential occurrence of the Mn-analogue of hellandite-(Y), in which Mn^{2+} dominates over Ca^{2+} , ideally $\text{Mn}_2(\text{YCa})\text{Y}_2\text{AlSi}_4\text{B}_4\text{O}_{22}(\text{OH}, \text{F}, \text{O})_2$, which means the $M3$ site is occupied by only Mn^{2+} .

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