the difference synthesis in the H_2O plane and perpendicular to it. The lone pair of the H_2O oxygen atom is found as a peak of 0·25 (4) e Å^{-3} at 0·4 Å from the O nucleus and almost on the bisector of the $Li^+\cdots Ow\cdots Li^+$ angle, in contrast to the room-temperature experiment (Fig. 3*b* of Hermansson & Thomas, 1983) where the lone-pair peak is significantly off the bisector.

The charge distribution of the nitrate ion has also been investigated: uronium nitrate (Harkema, 1971), $NaNO_3$ (Göttlicher & Knöchel, 1980), thiotrithiazyl nitrate (Moss, Guru Row & Coppens, 1980) and $LiNO_3.3H_2O$ (Hermansson, Thomas & Olovsson, 1984). Hermansson *et al.* (1984) showed the sp^2 hybridization of the nitrate O atom clearly. The formate ion has also been studied: $LiHCOO.H_2O$ (Thomas, Tellgren & Almlöf, 1975) and $KH(HCOO)_2$ (Hermansson & Tellgren, 1983). It seems that the terminal O atoms of these planar anions, NO_2^-, NO_3^- and HCO_2^-, take sp^2 hybridization.

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Structure of Homilite, $Ca_{2.00}(Fe_{0.90}Mn_{0.03})B_{2.00}Si_{2.00}O_{9.86}(OH)_{0.14}$

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Abstract. $M_r = 370{\cdot}0$, monoclinic, $P2_1/a$, $a = 9{\cdot}786\,(2)$, $b = 7{\cdot}621\,(2)$, $c = 4{\cdot}776\,(1)$ Å, $\beta = 90{\cdot}61\,(2)°$, $V = 356{\cdot}2\,(1)$ Å^3, $Z = 2$, $D_x = 3{\cdot}451$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 37{\cdot}7$ cm^{-1}, $F(000) = 364{\cdot}58$, room temperature, final $R = 0{\cdot}033$ for 1596 observed reflections. The crystal structure consists of sheets built up from alternating SiO_4 and BO_4 tetrahedra, parallel to (001). The calcium and iron atoms are located between the sheets and form a CaO_8 tetragonal antiprism and an FeO_6 octahedron respectively.

Introduction. Homilite was first described by Paikjull (1876), and later Tennyson (1978) determined the lattice parameters. It has been known that homilite has close structural relationships with gadolinite, $RE_2FeBe_2Si_2O_{10}$, (RE = rare earth), datolite, $CaBSiO_4(OH)$, *etc.* However, the structure analysis of homilite, which enables us to compare the structures of these minerals, has not yet been accomplished. The authors, therefore, have investigated its crystal structure. The homilite specimen used in the present study is from Stokö near Brevig, Langesundfjord, Norway.

Experimental. The electron microprobe analysis of the specimen gave SiO_2, 31·58; FeO, 17·03; MnO, 0·50; CaO, 29·43 wt %, and revealed the presence of trace amounts of rare-earth elements: Y, La, Ce, Pr, Nd and Dy. The ion microprobe mass analyzer indicated that boron is present in major amounts, while

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Table 1. *Atomic coordinates with e.s.d.'s in parentheses and equivalent isotropic temperature factors*

$B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + 2\beta_{13}ac\cos\beta)$.

	x	y	z	B_{eq}(Å^2)
Ca	0·33389 (3)	0·10224 (5)	0·00685 (7)	0·64
Si	0·08380 (5)	0·26757 (6)	0·53051 (10)	0·55
B	0·3382 (2)	0·4081 (2)	0·4596 (4)	0·52
Fe	0	0	0	0·61
O(1)	0·0359 (1)	0·3991 (2)	0·7625 (3)	0·83
O(2)	0·4553 (1)	0·2991 (2)	0·3283 (2)	0·64
O(3)	0·2090 (1)	0·3352 (2)	0·3304 (3)	0·70
O(4)	0·1439 (1)	0·0863 (2)	0·6828 (3)	0·66
O(5)	0·3411 (1)	0·4130 (2)	0·7583 (3)	0·73

beryllium is present only in trace amounts. The empirical formula, normalized to 10 oxygen, is $Ca_{2.00}(Fe_{0.90}Mn_{0.03})B_{2.00}Si_{2.00}O_{9.86}(OH)_{0.14}$, where the mole number of boron was assumed to be equal to that of silicon, and the hydrogen atoms were introduced to compensate the charge due to the vacancy at the Fe site.

A transparent crystal fragment was ground into elliptic tablet shape with diameter 0·14 mm and thickness 0·07 mm. Rigaku AFC-5 four-circle automated diffractometer equipped with Rotaflex RU-200 X-ray generator (operating conditions: 50 kV 160 mA). Lattice parameters refined by least-squares fitting of 25 2θ values, $15 < 2\theta < 30°$.

Intensities measured up to $\sin\theta/\lambda = 1{\cdot}36$ Å^{-1} within $-17 \leq h \leq 17$, $0 \leq k \leq 14$, $0 \leq l \leq 9$, Mo $K\alpha$ radiation, graphite monochromator, 2θ–ω scan technique, scan rate 2° min^{-1}, three standard reflections after every 50 reflections (max. intensity variation 3%); Lorentz and polarization correction, no absorption correction; 1872 unique reflections, 1596 with $|F_o| \geq 3\sigma(|F_o|)$ used for subsequent crystal-structure analysis. Since homilite has been thought to have a gadolinite-type structure, the atomic parameters of gadolinite (Miyawaki, Nakai & Nagashima, 1984) were used for the initial parameters. Refinement by full-matrix least-squares program *RFINE*2 (Finger, 1969); atomic scattering factors from Cromer & Mann (1968), anomalous-dispersion factors from *International Tables for X-ray Crystallography* (1974). The occupancies of iron and manganese at the Fe site were fixed at 90% and 3% respectively after the chemical composition.

Final R and R_w 0·033 and 0·027 respectively, $w = 1/\sigma^2(|F_o|)$, Δ/σ for all parameters $< 0{\cdot}01$. Final $\Delta\rho$ map excursions -1–2 e Å^{-3}.

Discussion. The final coordinates and equivalent isotropic temperature factors are listed in Table 1.*

The crystal structure of homilite is characterized by the sheets parallel to (001) (Fig. 1*a*). The sheet consists of alternating SiO_4 and BO_4 tetrahedra linked so as to form four- and eight-membered rings. The calcium and iron (manganese) atoms are located above the center of the respective rings, between the alternating SiO_4 and BO_4 sheets (Fig. 1*b*). The linkage of the tetrahedra is topologically identical with that found in datolite (Foit, Phillips & Gibbs, 1973), gadolinite (Miyawaki *et al.*, 1984) and hingganite, $REBeSiO_4(OH)$ (Yakubovich, Matvienko, Voloshin & Simonov, 1983).

The interatomic distances and bond angles were calculated using *UMBADTEA* (Finger, 1968), and are listed in Tables 2 and 3 respectively. Table 2 also shows the interatomic distances reported for gadolinite, datolite and hingganite for comparison.

The silicon atom forms a distorted tetrahedron with four oxygen atoms: O(2), O(3) and O(4) are bridging with neighboring boron atoms, while O(1) is non-bridging. The difference between the non-bridging and the bridging Si—O distances of homilite, 0·086 Å, is as large as that of datolite, 0·083 Å, and is larger than that of gadolinite, 0·028 Å, and of hingganite, 0·023 Å. This may be due to the effect of the neighboring cations to the SiO_4 tetrahedra. *e.g.* Ca^{2+} for homilite and datolite, RE^{3+} for gadolinite and hingganite.

The BO_4 tetrahedron also has a non-bridging B—O(5) bond which is shorter than the bridging B—O bonds. This tendency is observed in the BeO_4 tetrahedron of gadolinite, but not in the $BO_3(OH)$ tetrahedron of datolite nor in $BeO_3(OH)$ of hingganite where the O(5) positions are occupied by OH^-.

The calcium atom forms a tetragonal antiprism with eight oxygen atoms. The absence of cleavage in homilite, despite its sheet structure, accounts for the considerable strength of the Ca—O bonds.

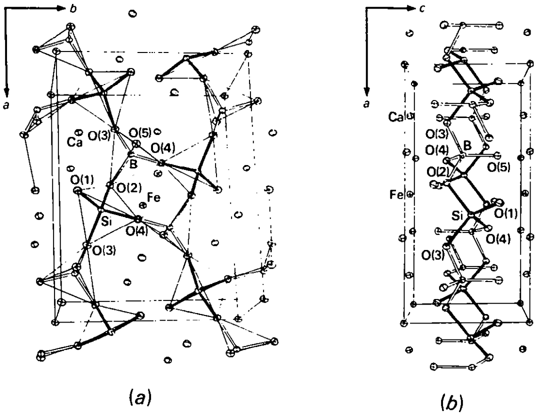

Fig. 1. *ORTEP* (Johnson, 1976) drawings of homilite. (*a*) A view along **c*** showing the linkage of SiO_4 and BO_4 tetrahedra. The tetrahedra are indicated by the single lines. (*b*) A view along **b*** showing the sheet structure made up of the SiO_4 and BO_4 tetrahedra. The iron and calcium atoms are located at $z \simeq 0$, and connect the two sheets.

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* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39767 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Interatomic distances* (Å) *with e.s.d.'s in parentheses*

Homilite[a]		Gadolinite[b]		Datolite[c]		Hingganite[d]	
Ca–O(1)	2·306 (1)	RE–O(1)	2·329 (2)	Ca–O(1)	2·276 (2)	RE–O(1)	2·291 (8)
–O(1′)	2·281 (1)	–O(1′)	2·335 (2)	–O(1′)	2·280 (2)	–O(1′)	2·284 (8)
–O(2)	2·446 (1)	–O(2)	2·392 (2)	–O(2)	2·453 (2)	–O(2)	2·344 (8)
–O(3)	2·626 (2)	–O(3)	2·473 (2)	–O(3)	2·607 (2)	–O(3)	2·686 (8)
–O(3′)	2·660 (2)	–O(3′)	2·691 (2)	–O(3′)	2·658 (2)	–O(3′)	2·460 (8)
–O(4)	2·409 (1)	–O(4)	2·381 (2)	–O(4)	2·420 (2)	–O(4)	2·339 (8)
–O(5)	2·650 (2)	–O(5)	2·497 (2)	–O(5)	2·672 (2)	–O(5)	2·519 (8)
–O(5′)	2·512 (1)	–O(5′)	2·397 (2)	–O(5′)	2·518 (2)	–O(5′)	2·432 (8)
Si–O(1)	1·569 (2)	Si–O(1)	1·612 (2)	Si–O(1)	1·570 (2)	Si–O(1)	1·618 (8)
–O(2)	1·657 (1)	–O(2)	1·641 (2)	–O(2)	1·648 (2)	–O(2)	1·633 (9)
–O(3)	1·645 (1)	–O(3)	1·632 (2)	–O(3)	1·651 (2)	–O(3)	1·641 (9)
–O(4)	1·666 (2)	–O(4)	1·648 (2)	–O(4)	1·661 (2)	–O(4)	1·648 (9)
B–O(2)	1·553 (2)	Be–O(2)	1·651 (3)	B–O(2)	1·475 (3)	Be–O(2)	1·61 (2)
–O(3)	1·507 (2)	–O(3)	1·660 (3)	–O(3)	1·486 (3)	–O(3)	1·63 (2)
–O(4)	1·530 (2)	–O(4)	1·634 (4)	–O(4)	1·464 (3)	–O(4)	1·63 (2)
–O(5)	1·427 (2)	–O(5)	1·592 (3)	–O(5)	1·496 (3)	–O(5)	1·60 (1)
Fe–O(2)	2·238 (1)×2	Fe–O(2)	2·288 (2)×2	Org*–O(2)	2·188×2	Fe–O(2)	2·308 (8)×2
–O(4)	2·181 (1)×2	–O(4)	2·224 (2)×2	–O(4)	2·178×2	–O(4)	2·253 (8)×2
–O(5)	2·038 (1)×2	–O(5)	2·035 (2)×2	–O(5)	2·117×2	–O(5)	2·063 (8)×2

(*a*) $Ca_{2.00}(Fe_{0.90}Mn_{0.03})B_{2.00}Si_{2.00}O_{9.86}(OH)_{0.14}$ (present study). (*b*) $RE_{2.00}Fe_{0.86}Be_{2.00}Si_{2.00}O_{9.72}(OH)_{0.28}$ (Miyawaki *et al.*, 1984). (*c*) $CaBSiO_4(OH)$ (Foit *et al.*, 1973). (*d*) $(Y_{0.51}Ln_{0.36}Ca_{0.13})Fe_{0.065}BeSiO_4(OH)$ (Yakubovich *et al.*, 1983).

* Origin.

Table 3. *Bond angles* (°) *with e.s.d.'s in parentheses*

O(1)–Ca–O(1′)	76·35 (6)	O(1)–Si–O(2)	112·13 (6)
O(1)–Ca–O(2)	84·67 (5)	O(1)–Si–O(3)	116·10 (8)
O(1)–Ca–O(3)	79·54 (5)	O(1)–Si–O(4)	109·16 (8)
O(1)–Ca–O(3′)	134·31 (5)	O(2)–Si–O(3)	108·83 (7)
O(1)–Ca–O(4)	109·54 (5)	O(2)–Si–O(4)	105·21 (7)
O(1)–Ca–O(5)	75·54 (5)	O(3)–Si–O(4)	104·59 (7)
O(1)–Ca–O(5′)	144·63 (5)		
		O(2)–B–O(3)	104·9 (1)
O(1′)–Ca–O(2)	81·26 (5)	O(2)–B–O(4)	101·9 (1)
O(1′)–Ca–O(3)	81·67 (5)	O(2)–B–O(5)	114·3 (1)
O(1′)–Ca–O(3′)	115·48 (5)	O(3)–B–O(4)	104·2 (1)
O(1′)–Ca–O(4)	134·36 (5)	O(3)–B–O(5)	115·3 (1)
O(1′)–Ca–O(5)	143·64 (4)	O(4)–B–O(5)	114·8 (1)
O(1′)–Ca–O(5′)	76·98 (4)		
		O(2)–Fe–O(4)	65·62 (5)
O(2)–Ca–O(3)	159·03 (4)	O(2)–Fe–O(4′)	114·38 (5)
O(2)–Ca–O(3′)	56·56 (4)	O(2)–Fe–O(5)	88·82 (5)
O(2)–Ca–O(4)	143·19 (5)	O(2)–Fe–O(5′)	91·18 (5)
O(2)–Ca–O(5)	73·62 (5)	O(4)–Fe–O(5)	89·84 (5)
O(2)–Ca–O(5′)	113·63 (5)	O(4)–Fe–O(5′)	90·16 (5)
O(3)–Ca–O(3′)	143·34 (2)	Si–O(2)–B	119·2 (1)
O(3)–Ca–O(4)	56·62 (4)	Si–O(3)–B	120·3 (1)
O(3)–Ca–O(5)	115·00 (5)	Si–O(4)–B	125·7 (1)
O(3)–Ca–O(5′)	73·96 (5)		
O(3′)–Ca–O(4)	92·90 (4)		
O(3′)–Ca–O(5)	71·24 (5)		
O(3′)–Ca–O(5′)	78·71 (5)		
O(4)–Ca–O(5)	77·37 (5)		
O(4)–Ca–O(5′)	74·51 (5)		
O(5)–Ca–O(5′)	137·26 (3)		

The iron atom in homilite is located at the origin, and forms a distorted octahedron with six oxygen atoms. On the other hand, the Fe site is completely vacant in datolite. However, the volume of the FeO_6 octahedron in homilite is close to that of the corresponding vacant octahedron in datolite. This may indicate that the role of the iron atom in the construction of the structure is not significant.

The framework of the homilite structure is similar to that of datolite, though the Fe site is completely vacant in the structure of datolite. The crystal structure of homilite can be described as being derived from that of datolite by placing the iron atom in the octahedral vacancy, and by charge-compensational replacement of OH^- with O^{2-}. Furthermore, homilite is isomorphous with gadolinite and hingganite.

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