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The structure of nekoite, $Ca_3Si_6O_{15}\cdot 7\ H_2O$, a new type of sheet silicate

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Abstract

Nekoite, $Ca_3Si_6O_{15} \cdot 7\ H_2O$, is a new type of sheet silicate. The sample studied, from Caxias do Sul, Rio Grande do Sul, Brazil, is triclinic, space group $P1$, with $a = 7.588$, $b = 9.793$, $c = 7.339$Å, $\alpha = 111.77$, $\beta = 103.50$, $\gamma = 86.53°$; $Z = 1$. The structure was solved through a combination of direct methods and the Patterson synthesis, and refined to a final R factor of 6.8 percent. The crystal structure consists of tetrahedral sheets interlayered with octahedral chains. The tetrahedral Si_6O_{15} sheets are obtained by interconnecting a "Dreierkette" so as to form alternating bands of 5- and 8-membered rings. Ca-octahedral chains, which crosslink the tetrahedral sheets, are formed by a double strand of octahedra, of which three out of four are occupied and the fourth is empty. Water molecules occupy the empty spaces between these chains. The balance of the electrostatic valences suggests that only oxygen atoms occupy the vertices of the tetrahedral sheet, while only water molecules occur outside the tetrahedral sheet.

Introduction

Nekoite is a rare hydrated calcium silicate, first discovered at Crestmore, Riverside County, California by Eakle (1917), who identified it as okenite on the basis of its chemical composition and optical properties. Based on studies of the same mineral from Eakle's holotype, Gard and Taylor (1956) showed it to be a new species, and named it nekoite. Chalmers *et al.* (1962) and Nicol (1971) have reported further results on the chemical composition and dehydration of nekoite from Crestmore.

Vezzalini (1979) recently described a second occurrence of nekoite in the cavities of a basaltic rock from Caxias do Sul, Rio Grande do Sul, Brazil. Nekoite occurs as tufts of well-shaped vitreous prismatic needles, up to 5 mm long, lying on large transparent crystals of heulandite and apophyllite which coat the surface of the cavities. These thin flat prisms of nekoite are only exceptionally good single crystals. Normally they occur as thin laths, up to 0.4 mm wide and 0.003 mm thick, of two or more individuals, which in turn can be twinned.

The usually accepted composition is $Ca_3Si_6O_{15} \cdot 8\ H_2O$. Chemical analyses of nekoite given in the literature are similar except for the water content, which varies from 6 H_2O (Eakle, 1917) to 8 H_2O (Gard and Taylor, 1955; Chalmers *et al.*, 1962).

A structural model for nekoite was proposed by Mamedov and Belov (1958), but has not been verified. Nicol (1971) announced the publication of the structure of nekoite which has not yet been published. The aim of our research is to determine the crystal structure of nekoite and to provide a better knowledge of its crystal chemistry.

Experimental

An untwinned fragment of nekoite (0.28 × 0.07 × 0.02 mm in size) was obtained from Vezzalini's specimen by cutting a twinned lath. Preliminary Weissenberg photographs confirmed the lattice parameters given by Vezzalini, which are reported along with other crystal data and experimental conditions in Table 1. The unit cell orientation has been chosen according to the recommendations of the Commission on Crystallographic Data of the International Union of Crystallography (Kennard *et al.*, 1967). The orientation of the triclinic unit cell assumed by Nicol (1971) can be derived by means of the transformation matrix 010/001/100.

Diffraction intensities were measured in the ω-scan mode with a Philips PW 1100 single-crystal automatic X-ray diffractometer (Centro di Cristallografia Strutturale del CNR, Pavia, Italy) and graphite-monochromatized Cu$K\alpha$ radiation at room temperature. For each reflection, repeated scans were made

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Table 1. Crystal and refinement data

Chemical formula	$Ca_3Si_6O_{15} \cdot 7H_2O$
Space group	P1
a	7.558(3) Å
b	9.793(5)
c	7.339(5)
α	111.77(7)°
β	103.50(5)
γ	86.53(3)
Volume	492.3(4) Å^3
Z	1
D_{meas}	2.23±0.01 g·cm^{-3}*
D_{calc}	2.21
λ (CuKα)	1.5418 Å
μ (CuKα)	113.7 cm^{-1}
Crystal size	0.28 x 0.07 x 0.02 mm
Scan mode	ω
Maximum 2θ	100°
ω scan width	1.80°
ω scan speed	0.03°/sec.
Number of independent reflections	889
Observed reflections (I > 2σ(I))	612
Maximum percent variation in standard reflections intensities	5.4 %
Final R (omitting zeros)	0.068
Final R (including zeros)	0.134
Final Rw (omitting zeros)	0.088

E.s.d.'s in parentheses refer to last digit.
* *From Nicol(1971).*

until the preset maximum number (3) or the preset number of counts (400) was reached. Due to the small dimension of the crystal, 277 weak reflections were skipped by the diffractometer program during the data collection, because they fulfilled the relationship, $I_t - 2\sqrt{I_t} < I_b$, where I_t is the intensity in counts/sec measured at the top of the scan, and I_b is the mean intensity in counts/sec of both backgrounds measured during a preliminary run of 5 sec at each end of the scan. The background counting time t′ at each end of the scan was $t' = (I_b - I_t)\,t/2$, where t is the scan time and I_t is the mean intensity in counts/sec during the scan. The "unobserved" reflections were assigned values of half the minimum observed intensity. Three standard reflections, monitored at four-hour intervals, showed no variation in intensity greater than 5.4 percent.

Intensities were corrected for Lorentz-polarization effects and for absorption, the latter correction being carried out by a combination of the semiempirical method proposed by North *et al.* (1968) and the absorption correction for a spherical shape (μ = 113.7 cm^{-1}, r = 0.001 cm), and then converted to $|F_{obs}|$ and $\sigma(|F_{obs}|)$ according to Davies and Gatehouse (1973).

Table 2. Final atomic coordinates and isotropic temperature factors

Atom	x	y	z	B(Å2)
Si(1)	0.1572(18)	0.1374(12)	0.0791(19)	0.9(2)
Si(2)	-0.1287(20)	-0.1229(13)	-0.0428(20)	1.7(2)
Si(3)	-0.1380(17)	-0.1058(12)	0.3978(18)	1.0(2)
Si(4)	0.1602(19)	0.1414(13)	0.6624(20)	1.2(2)
Si(5)	0.1802(18)	0.3783(14)	0.4897(19)	1.2(2)
Si(6)	0.0695(17)	0.7051(13)	0.6358(18)	1.3(2)
O(1)	-0.0218(34)	0.0291(25)	-0.0046(34)	2.5(6)
O(2)	0.1023(34)	0.2796(24)	0.2485(36)	2.2(5)
O(3)	-0.0366(33)	-0.2616(24)	-0.1835(34)	2.1(6)
O(4)	0.3357(34)	0.0605(27)	0.1573(37)	2.6(6)
O(5)	-0.3354(32)	-0.1078(27)	-0.1182(36)	2.1(6)
O(6)	-0.0921(36)	-0.1487(25)	0.1810(36)	2.9(6)
O(7)	0.1717(33)	0.1939(21)	-0.0926(33)	1.7(5)
O(8)	-0.0323(33)	0.0384(25)	0.5439(35)	2.6(6)
O(9)	-0.0679(31)	-0.2401(22)	0.4612(33)	1.5(5)
O(10)	0.1169(31)	0.2931(22)	0.6219(32)	1.4(5)
O(11)	-0.3558(30)	-0.0813(24)	0.3768(33)	1.5(5)
O(12)	0.3313(35)	0.0589(26)	0.5923(38)	2.3(5)
O(13)	0.0751(29)	0.5289(24)	0.5339(33)	1.6(5)
O(14)	0.3995(34)	0.3923(25)	0.5464(36)	2.2(5)
O(15)	0.2543(39)	0.7873(30)	0.7148(43)	3.7(6)
Ca(1)	0.5366(14)	0.1658(9)	0.4731(14)	2.6*
Ca(2)	0.4671	-0.1608	0.5595	1.5*
Ca(3)	0.4493(16)	-0.1628(10)	0.0297(15)	1.5*
W(1)	0.5054(40)	-0.4210(29)	-0.0770(42)	3.9(6)
W(2)	0.5499(45)	-0.4132(31)	0.4586(46)	4.9(7)
W(3)	0.7084(50)	0.2084(36)	0.8207(52)	8.0(10)
W(4)	0.7141(48)	0.2183(36)	0.2877(50)	7.4(10)
W(5)	0.0825(45)	0.5093(36)	0.0357(49)	6.7(9)
W(6)	0.7598(45)	0.4588(31)	0.1661(46)	5.2(7)
W(7)	0.2745(36)	-0.2432(24)	0.2251(36)	2.2(6)

E.s.d.'s in parentheses refer to last digit.
**Equivalent isotropic temperature factors after Hamilton(1959).*

Solution and refinement of the structure

The E-statistics showed an intermediate result between a centrosymmetric and a noncentrosymmetric situation (Karle *et al.*, 1965); therefore any sub-

Table 3. Anisotropic temperature factors (× 10^4) and magnitudes (Å) of the principal axes of thermal ellipsoids of Ca atoms

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ca(1)	136(21)	10(10)	75(23)	2(11)	13(17)	6(12)
Ca(2)	133(21)	12(11)	68(23)	8(12)	24(17)	8(12)
Ca(3)	138(20)	2(10)	72(21)	11(7)	16(18)	12(8)

	Axis 1	Axis 2	Axis 3
Ca(1)	0.06(3)	0.13(2)	0.20(1)
Ca(2)	0.07(3)	0.13(2)	0.19(1)
Ca(3)	0.02(9)	0.13(2)	0.20(1)

E.s.d.'s in parentheses refer to last digit.
The anisotropic temperature factor has the form:
$\exp(-h^2\beta_{11}-k^2\beta_{22}-l^2\beta_{33}-2hk\beta_{12}-2hl\beta_{13}-2kl\beta_{23})$

Table 4. Interatomic distances (Å) and angles (°)

Atoms	Distance	Atoms	Distance	O-Si-O angle
Si(1)-tetrahedron				
Si-O(1)	1.63(3)	O(1)-O(2)	2.54(3)	103.9(13)
-O(2)	1.60(2)	-O(4)	2.68(3)	112.3(13)
-O(4)	1.60(3)	-O(7)	2.58(3)	107.4(13)
-O(7)	1.58(2)	O(2)-O(4)	2.69(3)	114.3(13)
mean	1.60	-O(7)	2.50(3)	103.9(12)
		O(4)-O(7)	2.67(3)	114.2(13)
Si(3)-tetrahedron				
Si-O(6)	1.60(3)	O(6)-O(8)	2.56(3)	108.7(13)
-O(8)	1.55(2)	-O(9)	2.50(3)	103.4(13)
-O(9)	1.58(2)	-O(11)	2.64(3)	109.3(13)
-O(11)	1.63(2)	O(8)-O(9)	2.58(3)	111.4(13)
mean	1.59	-O(11)	2.60(3)	109.7(12)
		O(9)-O(11)	2.69(3)	114.0(12)
Si(5)-tetrahedron				
Si-O(2)	1.65(2)	O(2)-O(10)	2.67(3)	107.7(12)
-O(10)	1.65(2)	-O(13)	2.60(3)	106.2(13)
-O(13)	1.59(2)	-O(14)	2.70(3)	111.2(13)
-O(14)	1.62(2)	O(10)-O(13)	2.60(3)	106.5(12)
mean	1.63	-O(14)	2.66(3)	108.5(12)
		O(13)-O(14)	2.73(3)	116.2(12)

Atoms	Distance	Atoms	Distance	O-Si-O angle
Si(2)-tetrahedron				
Si-O(1)	1.64(2)	O(1)-O(3)	2.65(3)	109.7(13)
-O(3)	1.61(2)	-O(5)	2.61(3)	109.4(13)
-O(5)	1.55(2)	-O(6)	2.72(3)	108.5(12)
-O(6)	1.71(3)	O(3)-O(5)	2.68(3)	116.1(13)
mean	1.63	-O(6)	2.61(3)	104.0(12)
		O(5)-O(6)	2.66(3)	108.9(14)
Si(4)-tetrahedron				
Si-O(7^{V})	1.66(2)	O(7^{V})-O(8)	2.68(3)	106.4(12)
-O(8)	1.69(3)	-O(10)	2.56(3)	102.7(11)
-O(10)	1.62(2)	-O(12)	2.72(3)	114.7(13)
-O(12)	1.57(3)	O(8)-O(10)	2.61(3)	111.4(13)
mean	1.63	-O(12)	2.71(3)	109.7(12)
		O(10)-O(12)	2.70(3)	114.0(12)
Si(6)-tetrahedron				
Si-O(3^{VI})	1.63(2)	O(3^{VI})-O(9^{VII})	2.65(3)	106.1(12)
-O(9^{VII})	1.68(2)	-O(13)	2.58(3)	105.9(12)
-O(13)	1.61(2)	-O(15)	2.61(4)	111.1(14)
-O(15)	1.54(3)	O(9^{VII})-O(13)	2.63(3)	105.9(11)
mean	1.61	-O(15)	2.66(4)	111.5(14)
		O(13)-O(15)	2.66(3)	115.7(14)

Ca(1)-octahedron		Ca(2)-octahedron		Ca(3)-octahedron	
Ca-O(4)	2.35(2)	Ca-O(5'')	2.37(2)	Ca-O(4)	2.24(3)
-O(11')	2.41(2)	-O(11')	2.44(2)	-O(5')	2.35(2)
-O(12)	2.41(2)	-O(12)	2.28(2)	-O(11')	2.49(2)
-O(14)	2.32(3)	-O(15^{III})	2.36(3)	-O(15^{IV})	2.33(3)
-W(3)	2.47(4)	-W(2)	2.39(3)	-W(1)	2.40(3)
-W(4)	2.32(3)	-W(7)	2.41(2)	-W(7)	2.51(2)
mean	2.38	mean	2.38	mean	2.39

Superscripts refer to the following positions

None	x	y	z	V	x	y	z + 1
I	x + 1	y	z	VI	x	y + 1	z + 1
II	x + 1	y	z + 1	VII	x	y + 1	z
III	x	y - 1	z	VIII	x	y	z - 1
IV	x	y - 1	z - 1	IX	x + 1	y + 1	z

Water-oxygen and water-water distances < 3.30Å

W(1)-O(14^{IV})	2.65(3)	W(4)-O(8')	3.27(4)
-O(15^{IV})	3.26(4)	-W(6)	2.87(4)
-W(6^{III})	2.82(4)	W(5)-O(2)	3.16(4)
-W(7)	3.10(4)	-O(3^{VII})	3.19(4)
W(2)-O(14^{III})	2.62(3)	-O(7)	2.96(4)
-W(6^{III})	2.84(4)	-O(10^{VIII})	3.05(4)
-W(7)	3.18(4)	-W(6)	2.96(5)
W(3)-O(1'')	2.99(4)	-W(7^{VII})	2.63(4)
-O(8')	3.12(4)	W(6)-O(2')	3.12(4)
-O(12)	3.10(4)	-O(9^{IX})	3.08(3)
-W(6^{V})	2.76(4)	-O(13')	3.05(4)
W(4)-O(1')	3.27(4)	W(7)-O(4)	3.27(3)
-O(2')	3.14(4)	-O(6)	2.86(3)
-O(4)	3.12(4)	-O(11')	3.06(3)
		-O(12)	3.15(3)

E.s.d.'s in parentheses refer to last digit.

sequent calculation was performed in space group $P1$, which was later confirmed.

Several attempts to solve the structure by direct methods (program MULTAN, Germain *et al.*, 1971) were unsuccessful. Space group $P\bar{1}$ was also considered during the trials. On the other hand there was no simple interpretation of the three-dimensional Patterson function, nor was there any evidence in favor of the structural model proposed by Mamedov and Belov (1958). The actual solution of the structure was found by combining the information derived from electron density maps obtained from some of the solutions of MULTAN and from the Patterson synthesis. This procedure gave the positions of a few atoms; the remaining were found in successive stages of Fourier refinement. The refinement was carried out by alternating calculation of electron density maps and least-squares cycles; the latter were calculated with a modified version of the ORFLS program (Busing *et al.*, 1963). The final residual indices were $R = 0.068$ and $Rw = 0.088$ respectively for the observed reflections. In the final cycles of the refinement anisotropic temperature factors were used for the Ca atoms. Atomic scattering factors (Cromer and Mann, 1968) for fully-ionized atoms were used throughout. Final atomic coordinates and isotropic thermal parameters are listed in Table 2. The origin of the cell was chosen in a pseudocenter of symmetry

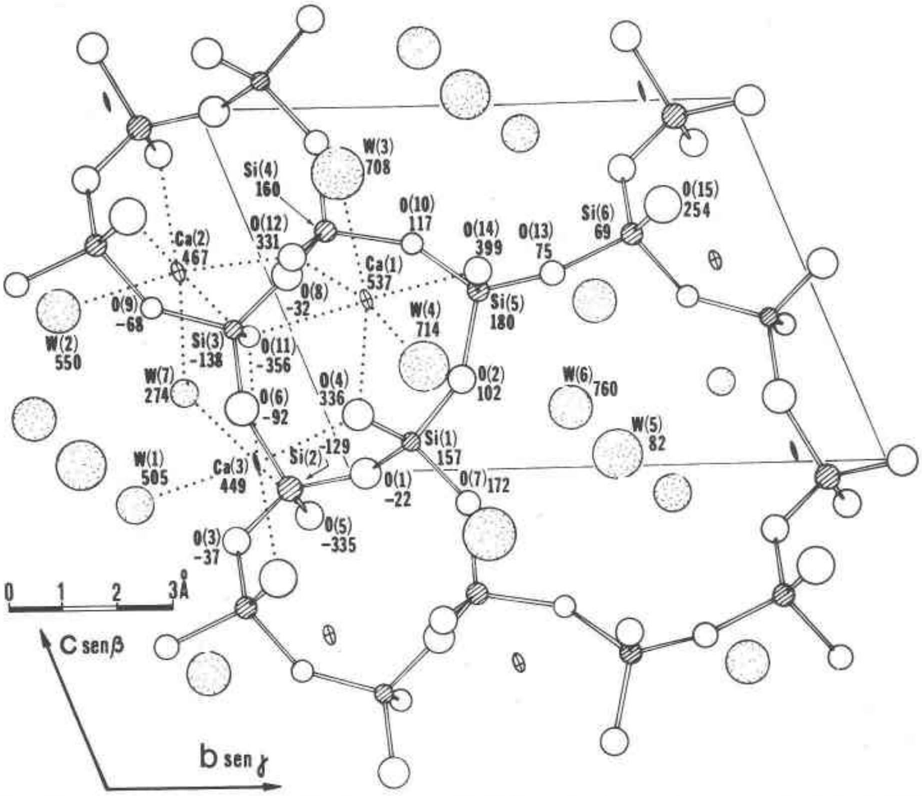

Fig. 1. Projection of the structure along [100]. Numbers give the heights of the atoms in thousands of the cell edge. Only atoms listed in Table 2 are coded.

to emphasize the deviation from centricity of the structure. Table 3 gives the anisotropic temperature factors of the Ca atoms, along with the dimensions of thermal ellipsoids. Bond distances and angles are listed in Table 4. The F_{obs} and F_{calc} table has been deposited.[1] The final model was checked by a difference Fourier synthesis, which showed no significant residual electron densities. Figure 1 is a projection of the structure along [100].

Description and discussion of the structure

Nekoite is a sheet silicate, which is formed by interconnecting tetrahedra sheets and octahedral chains (Fig. 2).

The tetrahedral silicate sheet

The tetrahedral sheets are formed by two chains with a period of three tetrahedra each ("Dreierkette"), which are linked together to form double chains with rings of 5 tetrahedra. The double chains are joined laterally with symmetrically equivalent double chains to form rings of 8 tetrahedra. Mamedov and Belov (1958) proposed that nekoite contained tetrahedral sheets formed by 5- and 8-membered rings of tetrahedra. The actual tetrahedral sheet of nekoite differs from that proposed by Mamedov and Belov in the orientation of one tetrahedron. In nekoite one of the chains has two free vertices pointing down and one pointing up, the other chain has all three free vertices pointing up, whereas in the Mamedov and Belov model one of the free vertices is pointing down in the latter chain. If one applies the symbols used for the space groups to the tetrahedral sheet, the symmetry of the single sheet (referred to a monoclinic cell with $a \sim 7.6$, $b \sim 18.7$, $c \sim 7.3$Å and $\beta \sim 103°$) is *A11m*, and not *A112/m* as proposed by Mamedov and Belov.

The calcium octahedral chain

The second important feature of the structure of nekoite is the Ca-octahedral chain. This chain can be described as a double chain formed by two strands of

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[1] To obtain a copy of this table, order Document AM-80-137 from the Business Office, Mineralogical Society of America, 2000 Florida Ave., N.W., Washington, D.C. 20009. Please remit $1.00 in advance for the microfiche.

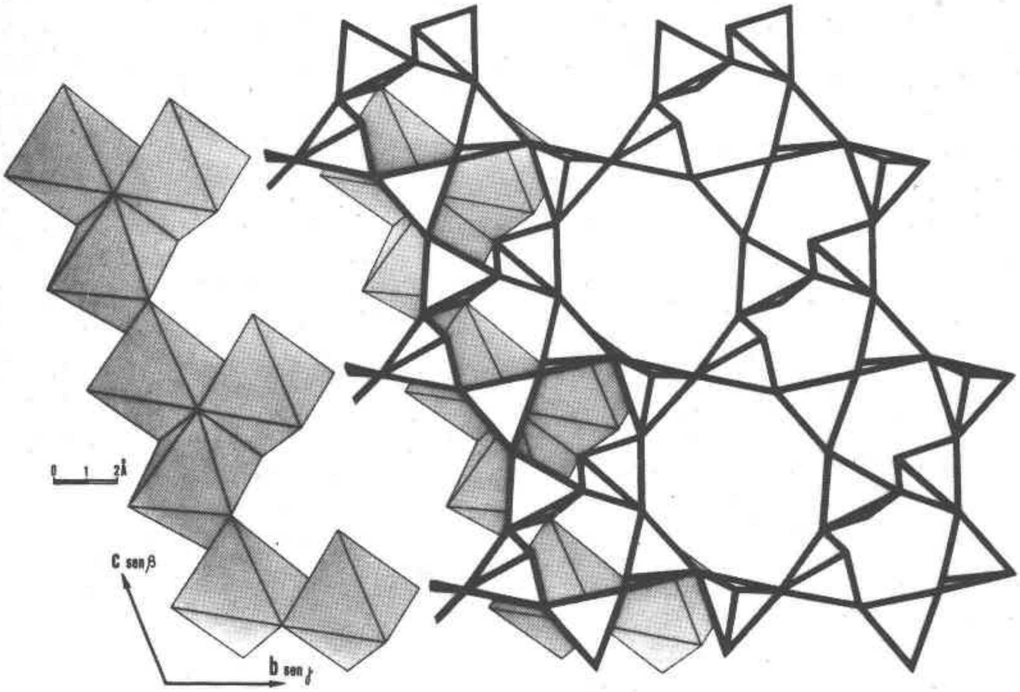

Fig. 2. Tetrahedral sheet and Ca-octahedral chain viewed along [100]. The left side shows only the chain and the right side only the sheet. The octahedral chain is quite regular, whereas the tetrahedral sheet is strongly deformed. The free vertices of the tetrahedral sheet are linked to the octahedral chains which are located above and below the sheet and are correlated by the *a* translation.

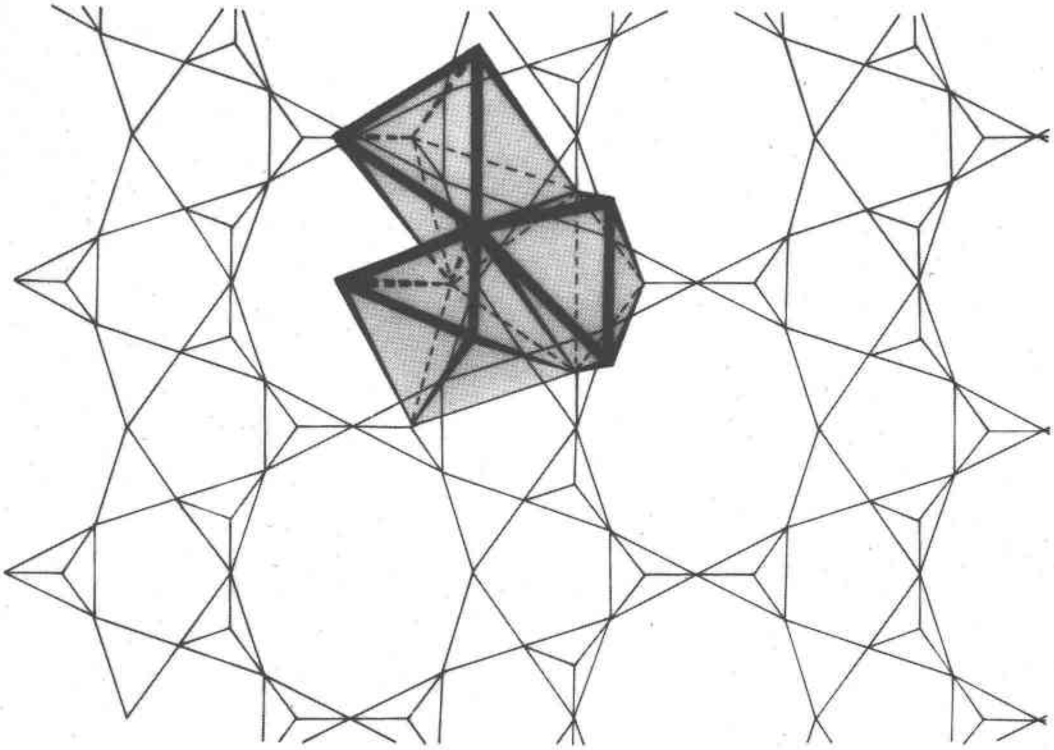

Fig. 3. The tetrahedral sheet in its most regular configuration viewed along [100]; its symmetry is *A*1*lm*. The three independent Ca-octahedra are also shown; part of the vertices of these octahedra are the oxygens of the tetrahedral sheet, and part are water molecules, the positions of which are chosen to provide regularity for the octahedra.

octahedra, one of which is fully occupied, the other being formed by alternate full and empty octahedral positions (Fig. 2). Water molecules occupy the spaces between these chains. This arrangement of octahedra strongly differs from that of Mamedov and Belov, who proposed a Ca-octahedral sheet where one quarter of the octahedra were empty. Each octahedron shares four vertices with oxygens of the tetrahedral sheet, the last two vertices of the octahedron being water molecules. As a consequence of the sharing of these oxygens, the mirror plane disappears and the unit cell becomes triclinic in space group *P*1 with parameters $a = 7.558$, $b = 9.793$, $c = 7.339$Å, $\alpha = 111.77$, $\beta = 103.50$, and $\gamma = 86.53°$. This change of symmetry is shown in Figure 3, where the tetrahedral sheet is in its most regular configuration and the three independent Ca-octahedra are shown with the position of the water molecules chosen so as to obtain the most regular Ca-polyhedra. These polyhedra are very distorted when compared to a regular octahedron; therefore, in the absence of symmetry elements, the structure can be rearranged to obtain more regular Ca-polyhedra and less irregular tetrahedral sheets. The Ca–O distances (Table 4) vary within the ranges of 2.32–2.47, 2.28–2.44 and 2.24–2.51Å for the Ca(1), Ca(2) and Ca(3) octahedra, respectively.

Hydrogen bonds

The balance of the electrostatic valences (Table 5) was computed using the method proposed by Donnay and Allmann (1970). Among the oxygens of the tetrahedral sheet only O(14) receives a sum of electrostatic bond strengths (namely 1.35), which differ greatly from 2.0; if we suppose that this atom is the acceptor of two hydrogen bonds from W(1) and W(2), its value becomes quite normal (1.83).

The IR spectrum of nekoite (Vezzalini, 1979) does not contradict this hypothesis, as it shows no bands in the range 2500–2800 cm^{-1}, this being definitive evidence of the presence of strong Si–OH bonds (Lazarev, 1972).

Among water molecules, only W(7) shows an in-

Table 5. Balance of estimated bond valences

	Ca(1)	Ca(2)	Ca(3)	Si(1)	Si(2)	Si(3)	Si(4)	Si(5)	Si(6)	ΣC^{v}	ΣC^{v*}
O(1)				0.97	0.95					1.92	
O(2)				1.03				0.93		1.96	
O(3)					1.01				0.97	1.98	
O(4)	0.35		0.39	1.03						1.77	
O(5)		0.34	0.35		1.14					1.83	
O(6)					0.82	1.03				1.85	2.00
O(7)				1.07			0.91			1.98	
O(8)						1.14	0.86			2.00	
O(9)						1.07			0.88	1.95	
O(10)							0.99	0.93		1.92	
O(11)	0.32	0.31	0.29			0.97				1.89	
O(12)	0.32	0.38					1.10			1.80	
O(13)								1.05	1.01	2.06	
O(14)	0.36							0.99		1.35	1.83
O(15)		0.34	0.35						1.16	1.85	
W(1)			0.33							0.33	0.10
W(2)		0.33								0.33	0.08
W(3)	0.30									0.30	
W(4)	0.36									0.36	
W(7)		0.32	0.28							0.60	0.21
ΣA^{v}	2.01	2.02	1.99	4.10	3.92	4.21	3.86	3.90	4.02		

ΣA^{V} = *Valences of bonds emanating from cation summed over the bonded anions.*
ΣA^{V} = *Valences of bonds reaching anion.*
* *Values corrected for H bonds.*

termediate value between that of an hydroxyl and that of a water molecule. If W(7) is a donor to O(6) and to W(5), its value decreases to 0.22. In conclusion, all the vertices of the tetrahedral sheet are occupied only by oxygen atoms, while only water molecules are present outside the tetrahedral sheet.

The water content derived from the structure refinement (7 water molecules) does not exactly correspond with the values proposed for the chemical formula: Nicol (1971): 8; Vezzalini (1979): 7.8; Chalmers (in Nicol, 1971): 7.5; Eakle (1917): 6. The atomic arrangement does not allow for other water molecules, and the difference synthesis obtained at the end of the refinement shows no significant residuals. Only two water molecules, W(5) and W(6), are not coordinated to Ca atoms and therefore are probably lost without relevant changes in the structure when the mineral is heated. The loss of the other water molecules, which are strongly bonded to Ca-cations, seems to be impossible without a relevant rearrangement of the structure. The TG and DTG curves reported by Vezzalini (1979) for nekoite from Caxias do Sul show that 2.6 water molecules are lost up to 180°C, whereas the remaining 5.2 are lost at higher temperatures, in sharp step between 200 and 350°C. Probably 2 out of 2.6 water molecules lost below 180°C have a definite site in the structure, the remaining 0.6 molecules being due to absorbed humidity. The above observations correspond with our conclusion, namely the presence of two non-bonded and five bonded water molecules in the structure. These results do not correspond with the thermal dehydration data given by Nicol (1971) for nekoite from Crestmore. In our opinion only a high-temperature X-ray diffraction study of nekoite can give a satisfactory answer to the problem of the thermal behavior of this mineral.

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