

## Crystal structure refinement of lawsonite

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### Abstract

Lawsonite,  $\text{Ca}^{[6]}\text{Al}_2^{[6]}\text{Si}_2^{[4]}\text{O}_7(\text{OH})_2 \cdot \text{H}_2\text{O}$ , is found in low-temperature, high-pressure metamorphic rocks. It is a hydrous counterpart of anorthite. A specimen from the type locality, Tiburon Peninsula, Marin County, California, was used for an X-ray study:  $a = 8.795(3)$ ,  $b = 5.847(1)$ ,  $c = 13.142(6)\text{\AA}$ ,  $V = 675.8\text{\AA}^3$ ,  $Z = 4$ ,  $D_{\text{calc}} = 3.088 \text{ g.cm}^{-3}$ , space group *Ccmm*. Refinement of 865  $F_{\text{obs}}$  gave an  $R = 0.0256$ . The structure is based on a three dimensional framework generated by cross-linking ribbons composed of edge sharing single chains of Al coordination octahedra and lateral bridging silicate groups. The openings of the framework accommodate the Ca atoms and the water molecules. The observed mean distances are  $\text{Si}^{[4]} - \text{O} = 1.633$ ,  $\text{Al}^{[6]} - \text{O} = 1.913$  and  $\text{Ca}^{[6]} - \text{O} = 2.421\text{\AA}$ . The hydrogen atoms participate in bent and bifurcated hydrogen bonds. Individual cation-anion distances conform well to the extended electrostatic valence rule.

### Introduction

The crystal structure of lawsonite,  $\text{CaAl}_2\text{Si}_2\text{O}_7(\text{OH})_2 \cdot \text{H}_2\text{O}$ , was determined by Wickman (1947) in space group *C222*<sub>1</sub>. The Si-O bond lengths in the  $\text{Si}_2\text{O}_7$  group as found by Wickman ranged from 1.59 to 1.72 Å. Subsequently, the structure was refined by Rumanova and Skipetrova (1959). They found that it could be described in the centric space group *Ccmm* and that the Si-O distances were lying in a narrow range: between 1.65 and 1.69 Å. Recently Pabst (1977) pointed out that lawsonite is denser than anorthite, its anhydrous equivalent. The volume per oxygen atom in lawsonite is 18.74 Å<sup>3</sup>, while in anorthite it is 20.94 Å<sup>3</sup>. Lawsonite is one of several especially dense hydrous silicates found in the Franciscan Formation, California. It is typical of a low-temperature, high-pressure metamorphic paragenesis.

### Experimental

A clear, almost colorless specimen of lawsonite from the type locality, Tiburon Peninsula, Marin County, California (USNM R3922) was divided into two parts; one was ground into a sphere with a diameter of 0.04 cm, the other was used for a microprobe analysis. Data were collected on an automatic four-circle diffractometer ( $\lambda\text{AgK}\alpha = 0.56083\text{\AA}$ ), using procedures described by Baur and Khan (1970). The

unit cell constants were refined from the setting of 20 reflections measured on a single crystal X-ray diffractometer. The resulting cell constants agree closely with those reported by Davis and Pabst (1960). Reciprocal space was searched in a sphere of radius  $\sin\theta/\lambda = 0.84\text{\AA}^{-1}$ . The total number of measured reflections was 11,268, which were averaged to yield 1,928 unique reflections. Of these 1,063 had an intensity of less than two sigma, and were not used in the refinement. The systematic absences (*hkl* only present with  $h + k = 2n$ ; and *0kl* only with  $l = 2n$ ) are consistent with space group *Ccmm* as noted by Rumanova and Skipetrova. The successful refinement confirms this choice. Lorentz-polarization corrections were applied, but because of the small linear absorption coefficient and the size of the crystal, an absorption correction was not necessary. Scattering factors were taken from the *International Tables for X-ray Crystallography* (1974). The refinement of the 865 observed unique structure factors started with the parameters reported by Rumanova and Skipetrova (1959) and refined in a few cycles to an  $R (= \sum ||F_o| - |F_c|| / \sum |F_o|)$  of 0.027 (with anisotropic temperature factors). A difference synthesis revealed likely positions for the hydrogen atoms. Upon further refinement including the hydrogen atoms an  $R$  of 0.0256 was achieved. The positional (Table 1) and the thermal (Table 2) parameters were used to

Table 1. Lawsonite, positional parameters\*

Atom		$\bar{x}$	$\bar{y}$	$\bar{z}$
Ca	4 mm	0.33305(5)	0	$\frac{1}{4}$
Al	8 $\bar{1}$	$\frac{1}{4}$	$\frac{1}{4}$	0
Si	8 m	0.98040(5)	0	0.13298(3)
O(1)	4 mm	0.0495(2)	0	$\frac{1}{4}$
O(2)	16 $\bar{1}$	0.3788(1)	0.2726(1)	0.11690(6)
O(3)	8 m	0.1378(1)	0	0.06505(8)
O(h4)	8 m	0.6391(1)	0	0.0479(1)
O(w5)	4 mm	0.6097(2)	0	$\frac{1}{4}$
H(w)	8 m	0.639(4)	0	0.194(3)
H(oh)	8 m	0.587(6)	0	0.056(4)
H(w) <sub>geom.</sub>	8 m	0.6731	0	0.1905
H(oh) <sub>el.</sub>	8 m	0.5329	0	0.0704

\* Estimated standard deviations are listed in parentheses in units of the least significant digit. The numbers of equivalent positions and their point symmetries are given for every atom. For atoms H(w)<sub>geom</sub> and H(oh)<sub>el</sub> see text.

calculate a final set of structure factors (Table 3<sup>1</sup>). The root-mean-square thermal displacements and their orientations are recorded in Table 4.

### Crystal data

Cell constants  $a = 8.795(3)$ ,  $b = 5.847(1)$ ,  $c = 13.142(6)$  Å;  $V = 675.8$  Å<sup>3</sup>; space group  $Ccmm$ ;  $D_{ca1c} = 3.088$  g · cm<sup>-3</sup>;  $Z = 4$ ;  $\mu(\text{AgK}\alpha) = 8.05$  cm<sup>-1</sup>;  $\mu R = 0.161$ . A microprobe analysis yielded (weight percent): 31.09 Al<sub>2</sub>O<sub>3</sub>, 37.75 SiO<sub>2</sub>, 0.90 Fe<sub>2</sub>O<sub>3</sub>, 17.18 CaO,

<sup>1</sup> To obtain a copy of this table order Document AM78-067 from the Business Office, Mineralogical Society of America, 1909 K Street, N.W., Washington, D.C. 20006. Please remit \$1.00 in advance for the microfiche.

0.33 TiO<sub>2</sub>, traces of MnO and K<sub>2</sub>O. The sum including 2H<sub>2</sub>O is 98.54 percent. This corresponds to Ca<sub>0.981</sub>(Al<sub>1.953</sub>Fe<sub>0.036</sub>Ti<sub>0.013</sub>)Si<sub>2.013</sub>O<sub>7</sub>(OH)<sub>2</sub> · H<sub>2</sub>O and shows that the crystal is close to the ideal composition CaAl<sub>2</sub>Si<sub>2</sub>O<sub>7</sub>(OH)<sub>2</sub> · H<sub>2</sub>O. This analysis corroborates observations made on lawsonites from other localities (see Davis and Pabst, 1960; Ernst *et al.*, 1970).

### Description of structure

The individual non-hydrogen atom positions determined in this refinement differ on the average by 0.05 Å from the positions found by Rumanova and Skipetrova (1959). The single largest discrepancy was observed for O(h4) which has shifted in the course of the refinement by 0.13 Å. The average change in the individual Ca-O, Al-O, Si-O, O(h4)-O and O(w5)-O distances is 0.05 Å.

The structure can be conveniently described as composed of edge sharing chains of Al coordination octahedra (with Al at  $y = 0.25$  and  $0.75$ ) arranged parallel to the [010] direction (Fig. 1). The octahedra in these chains share the edge O(3)-O(h4). The O(2) vertices (which are not in the shared edges) of neighboring octahedra are laterally bridged by silicate groups which alternate at heights  $y = 0$  and  $0.5$ . The symmetry of this ribbon of octahedra and tetrahedra is 2<sub>1</sub> and its composition is Al<sub>2</sub>SiO<sub>10</sub>(OH)<sub>2</sub>. Similar ribbons have been found in vauxite (Baur and Rama Rao, 1968) where their composition is AlFePO<sub>10</sub>(OH)<sub>2</sub>. Gottardi (1968) has described analogous ribbons in ilvaite, epidote, pumpellyite and chevkinite. He makes the point that these octahedral chains with lateral bridging tetrahedra can be consid-

Table 2. Lawsonite, anisotropic thermal parameters\* for non-hydrogen atoms, isotropic thermal parameters for hydrogen atoms

Atom	$\beta_{11}$ or B	$\beta_{22}$	$\beta_{33}$	$\beta_{12}$	$\beta_{13}$	$\beta_{23}$
Ca	0.00300(5)	0.00530(10)	0.00133(2)	0	0	0
Al	0.00150(5)	0.00309(11)	0.00106(2)	-0.00012(5)	-0.00007(2)	0.00003(4)
Si	0.00140(4)	0.00343(10)	0.00099(2)	0	0.00002(2)	0
O(1)	0.0023(2)	0.0081(4)	0.0009(1)	0	0	0
O(2)	0.0024(1)	0.0047(2)	0.0014(1)	-0.0008(1)	-0.0003(1)	0.0004(1)
O(3)	0.0017(1)	0.0042(2)	0.0013(1)	0	0.0003(1)	0
O(h4)	0.0019(1)	0.0048(2)	0.0018(1)	0	0.0004(1)	0
O(w5)	0.0040(2)	0.0226(7)	0.0014(1)	0	0	0
H(w)	2.6(0.9)					
H(oh)	7.0(1.6)					

\* The anisotropic thermal parameters are defined by  $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$

Table 4. Lawsonite, thermal ellipsoids\*

Atom	Axis	Displacement	Angle with axis		
			[100]	[010]	[001]
Ca	1	0.096(1)	90	0	90
	2	0.108(1)	90	90	0
	3	0.109(1)	0	90	90
Al	1	0.072(1)	64(10)	26(10)	89(2)
	2	0.077(1)	154(9)	65(10)	97(2)
	3	0.097(1)	96(2)	88(2)	7(2)
Si	1	0.074(1)	2(2)	90	92(2)
	2	0.077(1)	90	0	90
	3	0.093(1)	88(2)	90	2(2)
O(1)	1	0.091(3)	90	90	0
	2	0.095(3)	0	90	90
	3	0.118(3)	90	0	90
O(2)	1	0.081(2)	53(4)	37(4)	89(4)
	2	0.095(2)	127(4)	62(5)	130(3)
	3	0.119(1)	121(2)	67(2)	40(3)
O(3)	1	0.078(2)	16(3)	90	106(3)
	2	0.085(2)	90	0	90
	3	0.108(2)	74(3)	90	16(4)
O(h4)	1	0.083(2)	15(2)	90	105(2)
	2	0.091(2)	90	0	90
	3	0.128(2)	75(2)	90	15(2)
O(w5)	1	0.111(4)	90	90	0
	2	0.125(3)	0	90	90
	3	0.198(3)	90	0	90

\* Root-mean-square thermal displacements along principal axes and their orientations relative to a, b and c.

ered as the building blocks of structures such as lawsonite.

Within one of its repeat units each of the octahedral-tetrahedral ribbons is linked through four O(3) atoms to neighboring ribbons by connecting the silicate tetrahedra directly with neighboring octahedra and through two O(1) atoms which connect two each of the tetrahedra into an  $\text{Si}_2\text{O}_7$  group. Therefore the fusion of the ribbons results in a three dimensional framework of composition  $\text{Al}_2\text{Si}_2\text{O}_7(\text{OH})_2$ . This framework is sufficiently open to accommodate one calcium atom and one water molecule per formula unit thus resulting in the overall composition  $\text{CaAl}_2\text{Si}_2\text{O}_7(\text{OH})_2 \cdot \text{H}_2\text{O}$ .

The Ca atom has six near neighbors at 2.400 to 2.494 Å in the form of a distorted octahedron (Table 5). Two additional O(3) atoms at a distance of 2.976 Å are too far removed to be counted into the coordination sphere. Consequently the formula of lawsonite should be written as  $\text{Ca}^{[6]}\text{Al}_2^{[6]}\text{Si}_2^{[4]}\text{O}_7(\text{OH})_2 \cdot \text{H}_2\text{O}$ .

Strunz (1937) has pointed out that the cell constants of lawsonite and ilvaite are very similar. This is true when we compare the cell parameters deter-

mined here, with those reported by Haga and Takeuchi (1976) for ilvaite (in parentheses): 8.795(8.818), 5.847(5.853) and 13.142(13.005) Å. Strunz concluded that lawsonite and ilvaite are isostructural despite the fact that their spacegroups are different (*Pcmm* for ilvaite versus *Ccmm* for lawsonite). The similarity in the b cell constant is due to the length of the repeat unit of the octahedral-tetrahedral ribbons. It corresponds to the height of two octahedra sharing an edge. Similar repeat units ranging in length from 5.6 to 5.9 Å can be observed in epidote, pumpellyite, perrierite and chevkinite. The similarity of the other two cell constants, however, is fortuitous because the arrangement of the ribbons relative to each other is clearly different in lawsonite and ilvaite as has been shown by Gottardi (1968).

The relatively high density of lawsonite as compared to its anhydrous counterpart anorthite (Pabst, 1977) is related to the fact that Al is octahedrally coordinated in lawsonite but only four coordinated in anorthite. Analogous changes in density accompanying changes in coordination number are well known from many pairs of low-pressure high-pressure phases (for instance quartz and stishovite).

### Hydrogen atom positions

The distance O(h4)-H(oh) based on the refined coordinates of H(oh) is only 0.47(5) Å. The corre-

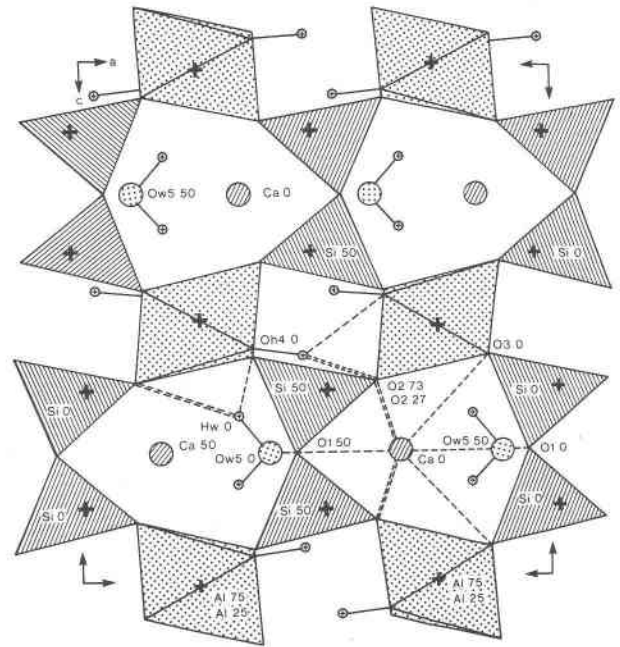


Fig. 1. Lawsonite, view parallel [010]. Next neighbors are indicated by broken lines for one each of the Ca, H(w) and H(oh) atoms. The heights of some atoms are indicated in 100y.

Table 5. Lawsonite, interatomic distances (Å) and angles (°)\*

1. Si		3. Ca	
2 Si-O(2)	1.616(2)	4 Ca-O(2)	2.400(1)
1 Si-O(3)	1.647(1)	1 Ca-O(w5)	2.433(2)
1 Si-O(1)	1.654(1)	1 Ca-O(1)	2.494(2)
mean	1.633	2 Ca-O(3)	2.976(1)
	0-Si-O	mean of 6	2.421
1 O(2)-O(2)	2.659(1)	110.7(1)	mean of 8
2 O(2)-O(3)	2.724(1)	113.2(1)	2.560
2 O(2)-O(1)	2.661(1)	109.0(1)	
1 O(3)-O(1)	2.552(1)	101.3(1)	
means	2.664	109.4	
1 Si-O(1)-Si		136.9(1)	
	2. Al		
2 Al-O(h4)	1.867(1)		
2 Al-O(2)	1.913(1)		
2 Al-O(3)	1.960(1)		
mean	1.913		
	0-Al-O		
2 O(h4)-O(2)	2.652(1)	89.1(1)	
2 O(h4)-O(2)	2.694(1)	90.9(1)	
2 O(h4)-O(3)	2.460(2)	80.0(1)	
2 O(h4)-O(3)	2.932(1)	100.0(1)	
2 O(2)-O(3)	2.740(1)	90.0(1)	
2 O(2)-O(3)	2.738(1)	90.0(1)	
means	2.703	90.0	
		4. H	
		1 H(oh)-O(h4)	[0.980]
		1 H(oh)-O(h4)	2.169
		1 O(h4)-O(h4)	2.752(2)
		1 O(h4)-H(oh)-O(h4)	116.7
		2 H(oh)-O(2)	2.180
		2 O(h4)-O(2)	2.933(1)
		2 O(h4)-H(oh)-O(2)	132.6
		1 H(oh)-H(oh)	1.939
		1 H(oh)-H(w)	2.003
		1 H(w)-O(w5)	[0.960]
		1 H(w)-O(w5)-H(w)	[109.0]
		1 H(w)-H(w)	[1.564]
		1 H(w)-O(h4)	1.898
		1 O(w5)-O(h4)	2.669(1)
		1 O(w5)-H(w)-O(h4)	135.4
		2 H(w)-O(2)	2.445
		2 O(w5)-O(2)	3.229(2)
		2 O(w5)-H(w)-O(2)	138.8

\* The values in square brackets have been preset by assuming a known geometry for the OH and H<sub>2</sub>O groups. The numbers before the atom designations are multiplicities per polyhedron. All distances and angles involving hydrogen atoms are based on H(w)<sub>geom.</sub> and

H(oh)<sub>el.</sub> (Table 1).

sponding O(w5)-H(w) distance measures 0.78(4)Å, and the angle H(w)-O(w5)-H(w) is 141(4)°. While there is no doubt that the hydrogen atoms are in the general vicinity of these positions, it is also clear that these refined positions are not as accurate as their precision might imply.

The point symmetry of the site of the oxygen atom of the water molecule is *mm*. Assuming that the hydrogen atom positions of the water molecule are not disordered there are only four different orientations possible for the molecule. In two of these the hydrogen atoms would be close to the Ca atom to which O(w5) is bonded. This is an unlikely possibility (Baur, 1972). Of the other two orientations one would bring H(w) within 2.56Å of a Ca atom removed by ½*b* from O(w5) while the remaining one is the most likely choice (Fig. 1). In this position, called H(w)<sub>geom.</sub> in Table 1, H(w) is located 1.898Å from O(h4). This distance could correspond to a bent hydrogen bond, since O(w5)-O(h4) measures 2.669Å and the angle O(w5)-H(w)-O(h4) is 135.4° (see Baur, 1965).

The hydrogen atom of the OH group was located by calculating its position of least electrostatic energy employing the program MANIOC (Baur, 1965). This

position, called H(oh)<sub>el</sub> in Table 1, places H(oh) almost equidistantly between H(w) and a centrosymmetrically related H(oh). It also places H(oh) within 2.18Å of two O(2) atoms thus creating a bifurcated hydrogen bond (see Baur, 1965). Actually H(oh) is even closer (2.169Å) to an O(h4) atom, but since the angle O(h4)-H(oh)-O(h4) is 116.7° it is assumed that this contact does not represent a hydrogen bond.

The hydrogen atom positions as located by X-ray diffraction differ by 0.3Å [distance from H(w) to H(w)<sub>geom.</sub>] and by 0.5Å [from H(oh) to H(oh)<sub>el</sub>] from the calculated positions. The true hydrogen atom positions as they could be determined by neutron diffraction are expected to be closer to the calculated values than to the experimental X-ray values. The hydrogen bonds formed in lawsonite are long, bent, and bifurcated, therefore the thermal motion of the hydrogen bonds must be large, thus making their localization by X-ray diffraction difficult.

The hydrogen positions derived here by geometric and electrostatic arguments are consistent with the infrared measurements and their interpretation reported by Labotka and Rossman (1974) on lawsonite. These authors concluded from the pleochroism of absorption spectra that all oxygen-hydrogen bonds in lawsonite are located in planes parallel to (010).

#### Bond distances and balance of valences

The mean distance Si-O of 1.633Å (Table 5) is close to the value of 1.630Å calculated from the regression equation (Si-O)<sub>mean</sub> = 1.621 + 0.0037CNM - 0.0046NC, where CNM is the mean coordination number of all oxygen atoms in the silicate tetrahedron and NC is the number of bridging oxygen atoms per tetrahedron (Baur, 1976). The individual Si-O bond lengths conform reasonably well to the extended electrostatic valence rule (Baur, 1970) as can be seen from the calculation shown in Table 6. It is apparent that the variations in Si-O bond lengths reflect the variations in bond strengths caused by different coordinations of the individual oxygen atoms. Analogous calculations of the other bond lengths yield for Al-O(h4) 1.880, Al-O(2) 1.900, Al-O(3) 1.960, Ca-O(2) 2.393, Ca-O(w5) 2.421 and Ca-O(1) 2.531Å. The largest discrepancy occurs for Ca-O(1) (0.037 Å), which is not surprising since predicted bond lengths for atoms with a low formal charge usually give less accurate results (Baur, 1970).

The shared edge O(3)-O(h4) between the two Al coordination octahedra is much shorter (2.460Å) than the average of the octahedral edges around Al.

Table 6. Lawsonite (a) Pauling bond strengths,  $p(O)$ , received by the oxygen atoms. (b) Observed and calculated bond lengths Si-O\*

a)						
	Ca	Al	Si	H(d)	H(a)	$\Sigma p(O)$
O(1)	2/6		2x4/4			2.333v.u.
O(2)	2/6	3/6	4/4		1/2x1/6	1.916
O(3)		2x3/6	4/4		1/6	2.167
O(h4)		2x3/6		5/6		1.833
O(w5)	2/6			2x5/6		2.000

b)					
	$p(O)$	$\Delta p(O)$	$d_{calc}$	$d_{obs}$	$ \Delta d $
Si-O(1)	2.333v.u.	0.250v.u.	1.653Å	1.654Å	0.001Å
Si-O(2)	1.916	-0.167	1.615	1.616	0.001
Si-O(2)	1.916	-0.167	1.615	1.616	0.001
Si-O(3)	2.167	0.083	1.638	1.647	0.009
mean	2.083	0	1.630	1.633	0.003

\* H(d) refers to hydrogen bond donors, H(a) to hydrogen bond acceptors. The calculation of bond lengths is based on  $d_{calc} = d_{mean} + 0.091\Delta p(O)$ , Baur (1970).<sup>7</sup>

Valence units: v.u.; observed Si-O distance:  $d_{obs}$ ;  $|\Delta d| = |d_{obs} - d_{calc}|$ ;  $\Delta p(O)$  is the deviation of the individual  $p(O)$  from the mean  $p(O)$  for the polyhedron.

Other shared edges do not occur in this structure unless one considers O(3) to be coordinated to the Ca atom.

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