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The crystal structures of whewellite and weddellite: re-examination and comparison

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Abstract

Whewellite, $CaC_2O_4 \cdot H_2O$, and weddellite, $CaC_2O_4 \cdot (2 + x)\ H_2O$ where $x \leq 0.5$, occur in sediments, in plants, and in urinary stones.

Their crystal structures have been refined to $R = 0.033$ and $R = 0.032$ respectively, using new sets of X-ray diffraction data, collected on a single-crystal diffractometer.

Refined cell parameters are: $P2_1/c$, $a = 6.290(1)$, $b = 14.583(1)$, $c = 10.116(1)$A, $\beta = 109.46(2)°$, $Z = 8$ for whewellite; $I4/m$, $a = 12.371(3)$, $c = 7.357(2)$A, $Z = 8$ for weddellite.

During refinement of whewellite, three out of four H atoms could be located, and split positions, partially occupied, for the two independent water molecules were found. In weddellite refinement, it was possible to locate all the H atoms, and a split position for the "zeolitic" water was found; a maximum water content of 2.5 H_2O was confirmed.

The comparison of the structures explains the relationships existing between some repeats of the two minerals and shows the differences between the Ca coordination polyhedra. A possible correlation between the structural features and the mechanism of formation of the two mineral species is suggested.

The symmetry and planarity of the oxalate groups are discussed.

Introduction

Whewellite and weddellite are hydrated oxalates of calcium, respectively $CaC_2O_4 \cdot H_2O$ and $CaC_2O_4 \cdot (2+x)H_2O$ (with $x \leq 0.5$), found naturally in plant tissues, in sediments as a mineral of organic origin, and in urinary stones. About 70 percent of human urinary stones contain whewellite and/or weddellite, either singly or mixed with other components, mostly phosphates, uric acids, or urates.

The frequency with which the two minerals are found in association in natural sources suggested a re-examination and a comparison of their crystal structure as the first stage of an investigation on their genetic relationships.

Previous structure analyses were carried out by Cocco (1961) and Cocco and Sabelli (1962) for whewellite (two-dimensional photographic data, $R = 0.14$) and by Sterling (1965) for weddellite (three-dimensional photographic data, $R = 0.13$).

Experimental

Single crystals of whewellite and weddellite were obtained from urinary calculi. A Philips PW 1100 single-crystal diffractometer was used to collect the X-ray diffraction data. Table 1 presents the experimental details. On the crystals of both species three standard reflections monitored at three-hour intervals showed less than 3.5 percent intensity variation during the data collection. X-ray data were processed with a locally modified version of a program specifically written for PW 1100 (Hornstra and Stubbe, 1972). Intensities were corrected for absorption following the method of North *et al.* (1968). Programs for structure determination, refinement and drawing of figures by Germain *et al.* (1971), Busing *et al.* (1962), and Johnson (1965) were employed. The scattering curves for neutral atoms given by the *International Tables for X-ray Crystallography* (1974) were used. High-precision methods using an ω scan of four intense reflections from each of several lattice rows provided accurate data for unit-cell parameter computations.

Results of the structure refinements

Whewellite

The orientation and the dimensions of the cell axes (Table 1) are consistent with one of the two alternative orientations proposed by Arnott *et al.* (1965)

Table 1. Crystal and diffraction data

Property	Whewellite	Weddellite
Formula	$CaC_2O_4 \cdot H_2O$	$CaC_2O_4 \cdot (2+x)\ H_2O$ $(x \leqslant 0.5)$
Space group	$P2_1/c$	$I\ 4/m$
a (A)	6.290(1)	12.371(3)
b (A)	14.583(1)	
c (A)	10.116(1)	7.357(2)
β (°)	109.46(2)	
V (A^3)	876.228	1125.927
Z	8	8
Crystal dimensions (mm)	0.36×0.36×0.20	0.30×0.25×0.20
Radiation (A)	Mo $K\alpha$, λ =0.71069	Mo $K\alpha$, λ =0.71069
Monochromator	graphite	graphite
μ (cm^{-1})	13.0	10.5
Scan width (°)	2.50	1.00
Scan speed ($° s^{-1}$)	$0.1° s^{-1}$	$0.025° s^{-1}$
ϑ range (°)	2 – 40	2 – 30
Maximum $(\sin \vartheta)/\lambda$	0.90444	0.70353
Measured reflections	5152	1789 (hkl, $\bar{k}hl$)
Independent reflections	5152	889
Observed reflections (whit $I \geqslant 3\sigma I$)	2864	607

(space group $P2_1/c$). The values given for c and β by Cocco (1961) are not consistent with $P2_1/c$ orientation assumed by this author, but seem to agree better with a $P2_1/n$ orientation (Leavens, 1968).

The crystal structure, re-determined by means of the direct methods, is consistent with that published by Cocco (1961) and by Cocco and Sabelli (1962).

Isotropic temperature factors were used in the first least-squares cycles of the refinement; successively the atoms were treated anisotropically. A pseudo-cell with $b' = b/2$ (the intensities of the reflections with k even are far greater than those of the reflections with k odd) makes the atoms of the pairs O(1)–O(3), O(2)–O(4), Ca(1)–Ca(2), C(3)–C(4), O(6)–O(7), O(5)–O(8), and W(1)–W(2) pseudo-equivalent (see Fig. 1 and 2). In order to avoid correlation effects among the variables during calculations, the coordinates and thermal factors of the first or second atom of a pair were refined separately, as were the scale factor and the coefficient of secondary extinction (Zachariasen, 1963). From a difference Fourier map, three of the four hydrogen atoms were located and two maxima, double the height of that of the hydrogens, were interpreted as split positions of the two water molecules (respectively 0.73 and 0.39A away from them). The occupancy of these positions W(10) and W(20) and, in turn, that of the main positions W(1) and W(2) were refined with other cycles of least squares, together with the coordinates and the anisotropic thermal factors of the non-hydrogen atoms. Fixed isotropic temperature factors were used for the atoms H(11), H(21), H(22), W(10), and W(20). The final atomic parameters are shown in Tables 2 and 3; bond lengths and angles in Table 4. The observed and computed structure factors are compared in Table 5.[1] The final value of the discrepancy index was $R = 0.033$ for the 2864 observed reflections.

Weddellite

The values of the cell parameters (Table 1) agree well with those of Sterling (1965). The atomic coordinates of this author, with the exclusion of those relative to oxygen of the "zeolitic" water, were used to start the refinement. During the first cycles, carried out using isotropic thermal factors, the examination of the difference Fourier map confirmed the presence of zeolitic water, with incomplete occupancy, in the channel running along the four-fold axis, with coordinates similar to those given by Sterling. In the last stages of refinement, difference Fourier maps allowed us to locate the hydrogen atoms of the two molecules of non-zeolitic water and to find a split position W(30) for the oxygen of the zeolitic water, the occupancy of which was refined alternately with that of the main position W(3) (the distance between the two positions was 0.57A). Fixed isotropic temperature factors were used for the atoms H(5), H(6), and W(30). The final atomic parameters are given in Tables 6 and 7, lengths and bond angles in Table 8. The observed and computed structure factors are compared in Table 9.[1] The final value of the discrepancy index was $R = 0.032$ for the 889 observed reflections.

Discussion and comparison of the structures

Whewellite

In whewellite (Fig. 1) the coordination polyhedra of the pseudo-equivalent atoms Ca(1) and Ca(2) are distorted square antiprisms: in each of them seven of the oxygens belong to five oxalic groups and one to a water molecule. Each Ca polyhedron shares three edges with three adjacent Ca polyhedra. In this way

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[1] To receive a copy of Tables 5 and 9, order Document AM-79-116 from the Business Office, Mineralogical Society of America, 2000 Florida Avenue, NW, Washington, DC 20009. Please remit $1.00 in advance for the microfiche.

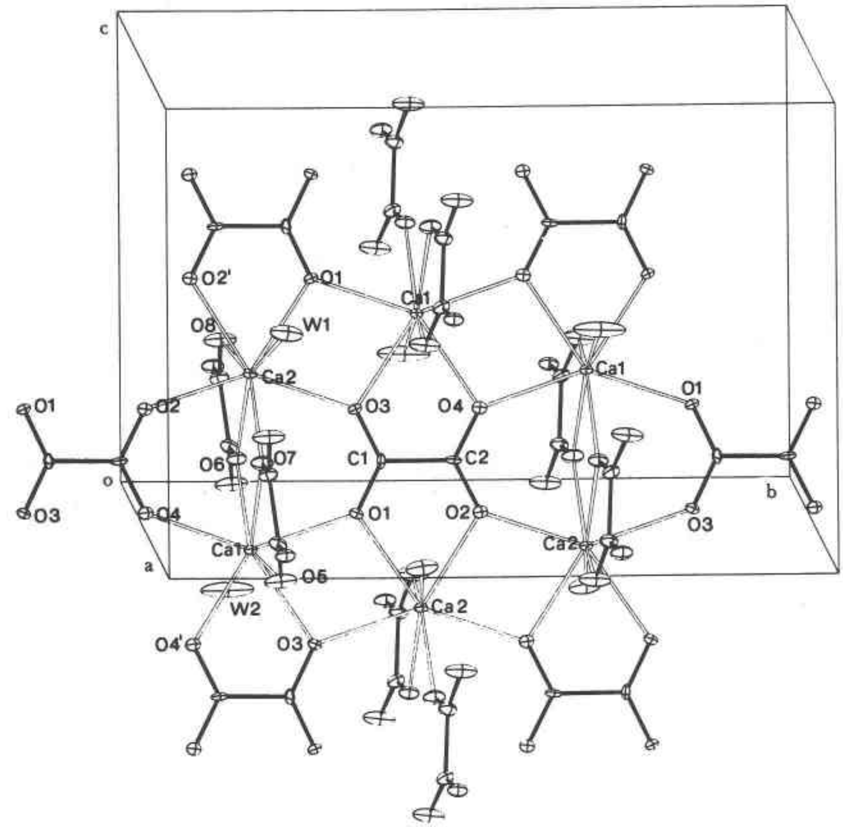


Fig. 1. Whewellite, section almost parallel to (100).

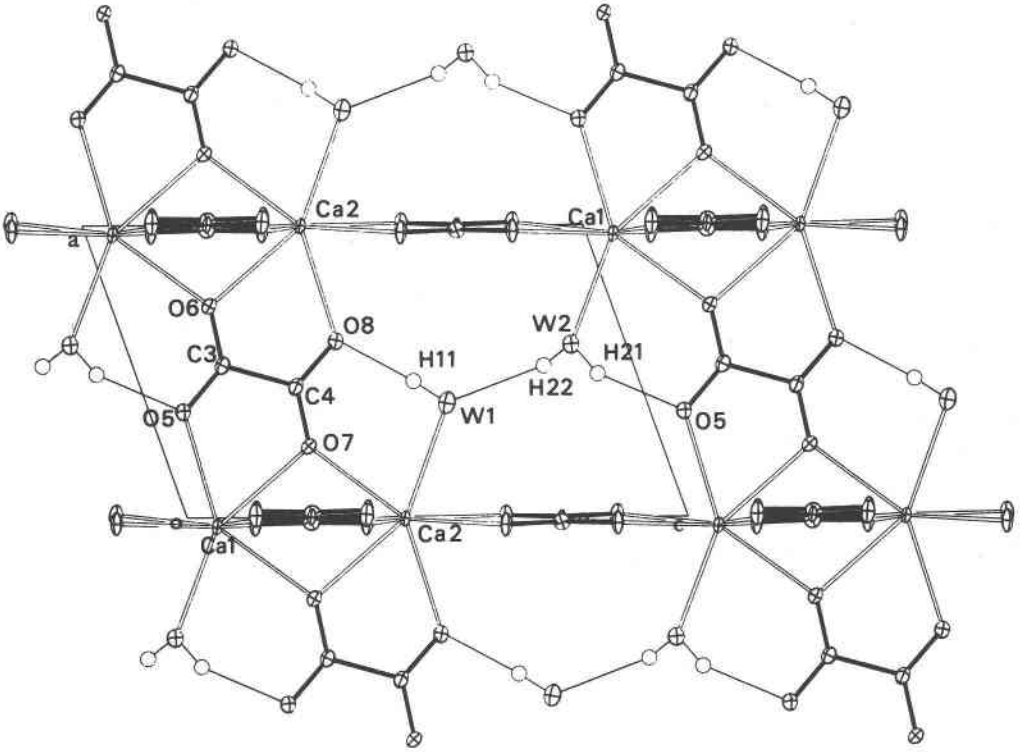


Fig. 2. Whewellite, section parallel to (010).

Table 2. Atomic coordinates, occupancies, and equivalent isotropic temperature factors of whewellite*

Atom	Occ.	x/a	y/b	z/c	$\beta_H(A^2)$
C(1)	1	0.9832(2)	0.3201(1)	0.2452(2)	0.80
C(2)	1	1.0009(2)	0.4270(1)	0.2492(1)	0.77
C(3)	1	0.5189(2)	0.1266(1)	0.1812(1)	1.07
C(4)	1	0.4505(2)	0.1173(1)	0.3131(1)	1.08
O(1)	1	0.9756(2)	0.2826(1)	0.1322(1)	1.16
O(2)	1	1.0066(2)	0.4659(1)	0.1395(1)	1.14
O(3)	1	0.9799(2)	0.2819(1)	0.3550(1)	1.17
O(4)	1	1.0073(2)	0.4658(1)	0.3614(1)	1.21
O(5)	1	0.3614(2)	0.1418(1)	0.0690(1)	2.20
O(6)	1	0.7245(2)	0.1227(1)	0.1974(1)	1.16
O(7)	1	0.2438(2)	0.1229(1)	0.2957(1)	1.31
O(8)	1	0.6073(2)	0.1068(1)	0.4264(1)	2.24
Ca(1)	1	0.9676(1)	0.1243(1)	0.0546(1)	0.67
Ca(2)	1	0.9968(1)	0.1236(1)	0.4357(1)	0.66
W(1)	0.85	0.3932(3)	0.3459(1)	0.1022(2)	1.81
W(2)	0.86	0.5913(3)	0.3829(3)	0.3908(2)	4.48
H(11)	1	0.487	0.372	0.051	(5.00)
H(21)	1	0.510	0.364	0.426	(5.00)
H(22)	1	0.530	0.367	0.320	(5.00)
W(10)	0.15	0.388	0.396	0.099	(1.80)
W(20)	0.14	0.584	0.409	0.392	(4.50)

* Standard deviations in parentheses; equivalent isotropic temperature factors after Hamilton (1959).

polyhedral layers are formed parallel to (100), the good cleavage plane. Within the layers, Ca atoms occur at the vertices of hexagons which have at their center one of the two independent oxalic groups. These oxalic groups lie in the (100) plane with the C(1)–C(2) bonds nearly parallel to the *b* axis. The layers are connected to one another through the second series of oxalic groups and the water molecules (Fig. 2): oxalic groups alternate with the water molecules and form ribbons lying in the (010) plane and running along *c*. Bonding between the pseudo-equivalent water molecules W(1) and W(2) and the pseudo-equivalent oxygens O(8) and O(5) is through the pseudo-equivalent hydrogen atoms H(11) and H(21). The bond between the two water molecules takes place through hydrogen H(22). The hydrogen atom H(12), which probably does not take part in hydrogen bonds and which, in any case, cannot respect the pseudo-symmetry with H(22) for steric reasons, was not identified.

Weddellite

In weddellite the calcium coordination polyhedron is a distorted square antiprism but, of the eight oxy-

Table 3. Analysis of the anisotropic thermal parameters in whewellite*

Atom	r.m.s.	U_ia	U_ib	U_ic
C(1)	0.062	96	172	84
	0.102	118	82	11
	0.126	151	86	99
C(2)	0.070	65	98	170
	0.103	86	8	98
	0.117	154	89	96
C(3)	0.096	22	89	132
	0.111	68	88	42
	0.138	88	178	89
C(4)	0.088	37	89	146
	0.112	53	86	56
	0.143	87	176	89
O(1)	0.080	72	115	155
	0.102	78	25	115
	0.165	158	86	92
O(2)	0.081	68	61	150
	0.100	100	29	61
	0.163	155	88	95
O(3)	0.082	70	69	159
	0.098	93	21	69
	0.168	160	85	91
O(4)	0.082	74	124	146
	0.105	72	35	124
	0.168	156	84	94
O(5)	0.105	107	82	143
	0.110	17	89	126
	0.246	91	172	97
O(6)	0.088	27	91	136
	0.114	63	97	47
	0.154	94	173	94
O(7)	0.085	170	88	60
	0.117	80	86	30
	0.170	91	176	86
O(8)	0.104	32	90	141
	0.109	58	95	52
	0.250	93	175	93
Ca(1)	0.074	59	90	168
	0.092	102	14	93
	0.107	146	104	101
Ca(2)	0.072	58	90	167
	0.095	89	1	90
	0.105	148	89	102
W(1)	0.112	63	75	162
	0.141	28	101	84
	0.191	93	161	107
W(2)	0.109	56	92	165
	0.123	34	88	76
	0.379	90	177	88

* Root mean square thermal vibrations along the ellipsoid axes (A) and angles (°) between the crystallographic axes and the principal axes (U_i) of the vibration ellipsoid. E.s.d. of r.m.s. range from 0.001 to 0.003

Table 4. Interatomic distances (A) and bond angles (°) in whewellite (standard deviations in parentheses)

Atoms	Distances	Atoms	Distances
Ca(1)-O(1)	2.434(2)	Ca(2)-O(1)	2.452(2)
-O(3)	2.461(2)	-O(2)	2.420(2)
-O(4)	2.449(2)	-O(2')	2.425(2)
-O(4')	2.435(2)	-O(3)	2.438(2)
-O(5)	2.446(1)	-O(6)	2.450(1)
-O(6)	2.427(1)	-O(7)	2.425(1)
-O(7)	2.481(1)	-O(8)	2.432(1)
-W(2)	2.399(2)	-W(1)	2.542(2)
Average	2.442	Average	2.448
C(1)-C(2)	1.563(2)	C(3) -C(4)	1.538(2)
C(1)-O(1)	1.254(2)	C(3) -O(5)	1.254(2)
-O(3)	1.249(2)	-O(6)	1.250(2)
C(2)-O(2)	1.257(2)	C(4) -O(7)	1.255(2)
-O(4)	1.257(2)	-O(8)	1.249(2)
Average	1.255	Average	1.252
W(1)-O(8)	2.654(2)	H(21)-O(5)	1.96
-W(2)	2.820(2)	-W(2)	0.77
W(2)-O(5)	2.683(2)		
		H(22)-W(1)	2.11
H(11)-O(8)	1.70	-W(2)	0.73
-W(1)	0.98		

Atoms	Angles	Atoms	Angles
O(1)-C(1)-O(3)	127.55(17)	O(5)-C(3)-O(6)	126.75(16)
O(1)-C(1)-C(2)	116.07(14)	O(5)-C(3)-C(4)	115.91(13)
O(3)-C(1)-C(2)	116.38(14)	O(6)-C(3)-C(4)	117.25(13)
O(2)-C(2)-O(4)	126.33(17)	O(7)-C(4)-O(8)	127.07(17)
O(2)-C(2)-C(1)	116.74(14)	O(7)-C(4)-C(3)	116.59(14)
O(4)-C(2)-C(1)	116.92(14)	O(8)-C(4)-C(3)	116.32(14)
H(21)-W(2)-H(22)	97.2		

Table 6. Atomic coordinates, occupancies, and equivalent isotropic temperature factors of weddellite*

Atom	Occ.	x/a	y/b	z/c	β_H(A^2)
C	1	0.4464(2)	0.2415(2)	0.1053(3)	1.23
O(1)	1	0.3564(1)	0.2458(1)	0.1829(2)	1.46
O(2)	1	0.2355(2)	0.4634(1)	0.1799(2)	2.79
Ca	1	0.1993(1)	0.3011(1)	0.0	0.97
W(1)	1	0.1490(3)	0.1145(2)	0.0	2.75
W(2)	1	0.0192(2)	0.3841(3)	0.0	2.97
W(3)	0.58	0.0	0.0	0.6830(19)	7.20
H(1)	1	0.166	0.071	0.105	(5.00)
H(2)	1	0.350	0.018	0.115	(5.00)
W(30)	0.17	0.0	0.0	0.240	(7.00)

* Standard deviations in parentheses; equivalent isotropic temperature factors after Hamilton (1959).

gens, six belong to four oxalic groups and two to water molecules: with respect to whewellite, calcium is coordinated to one less oxalic group and one more water molecule. If we assume that in solution the formation of calcium oxalates and their precipitation take place by substitution, in the Ca^{2+} hydration sphere, of the water molecules with oxalate ions, weddellite should be the first stage of this process.

Each polyhedron of calcium in weddellite makes a link to two adjacent polyhedra (Fig. 3), changing the layers found in whewellite to chains running along *c* (axis 4). The link between these chains is made by

Table 7. Analysis of the anisotropic thermal parameters in weddellite*

Atom	r.m.s.	U_ia	U_ib	U_ic	Atom	r.m.s.	U_ia	U_ib	U_ic
C	0.109	80	95	169	W(1)	0.143	95	174	90
	0.118	15	100	79		0.152	90	90	0
	0.144	101	168	86		0.247	174	84	90
O(1)	0.106	130	87	40	W(2)	0.153	153	63	90
	0.123	42	102	50		0.183	90	90	0
	0.171	102	167	95		0.236	116	153	90
O(2)	0.109	89	139	49	W(3)	0.287	87	3	90
	0.131	83	49	41		0.287	103	13	90
	0.277	173	86	85		0.330	90	90	180
Ca	0.104	90	90	180					
	0.109	116	26	90					
	(0.119)	153	116	90					

* Root mean square thermal vibrations along the ellipsoid axes (A) and angles (°) between the crystallographic axes and the principal axes (U_i) of the vibration ellipsoid. E.s.d. of r.m.s. range from 0.001 to 0.004

Table 8. Interatomic distances (A) and bond angles (°) in weddellite (standard deviations in parentheses)

Atoms	Distances	Atoms	Distances
Ca–W(1)	2.391(3)	O(2)–W(1)	2.918(3)
–W(2)	2.453(3)	–W(2)	2.866(3)
–O(2) × 2	2.446(2)		
–O(1) × 2	2.461(2)	H(1)–O(2)	2.04
–O(1^{1}) × 2	2.501(2)	–W(1)	0.96
Average	2.458	H(2)–O(2)	1.86
		–W(2)	1.05
C–C	1.549(4)	W(3)–W(3)	2.69 (3)
		–O(2) × 4	3.303(2)
C–O(1) × 2	1.252(3)	–W(1) × 4	3.293(9)
–O(2) × 2	1.246(3)	W(30)–W(30)	3.53
Average	1.249	–O(2) × 4	3.35
		–W(1) × 4	2.92

Atoms	Angles	Atoms	Angles
O(2)–C–O(1)	126.67(18)	H(1)–W(1)–H(1)	106.54(27)
O(2)–C–C	116.15(12)	H(2)–W(2)–H(2)	107.11(29)
O(1)–C–C	117.17(11)		

means of (· · · oxalate–water–oxalate· · ·) ribbons lying in planes parallel to (100). In these ribbons the water molecules W(1) and W(2) and the oxygens O(2) are bonded through hydrogens H(5) and H(6).

The repetition around the four-fold axis of the Ca chains and the oxalate-water ribbons generate a channel (Fig. 4) in which zeolitic water occurs in two alternative and very close positions, W(3) and W(30). The distance between two W(3) water molecules related by a symmetry center corresponds to that of a strong hydrogen bond; the closest contacts of water W(30) occur with four non-zeolitic W(1) waters. The sum of the occupancies of the two alternative positions W(3) and W(30) turns out to be *Occ* = 0.58 + 0.17 = 0.75, which, taking multiplicity into account, brings the contribution of zeolitic water to 0.37; thus the formula of the weddellite examined is $CaC_2O_4 \cdot 2.37H_2O$.

Sterling (1965) observed that the multiplicity of the position of the zeolitic water limits the maximum water content of weddellite to $2.5H_2O$; however, Gérard *et al.* (1968) later postulated the existence of weddellite with three molecules of water, on the basis of thermal analyses of samples kept in water-vapor-controlled atmosphere. In order to check this hypothesis, a systematic research of possible cavities existing in the structure was carried out; two "holes" (with coordinates *0, 0.50, 0.25* and *0, 0, 0*) were identified, but they appear too small to hold water molecules, which in this case would have too short contacts (less than 2.35A) with other water molecules. Therefore a

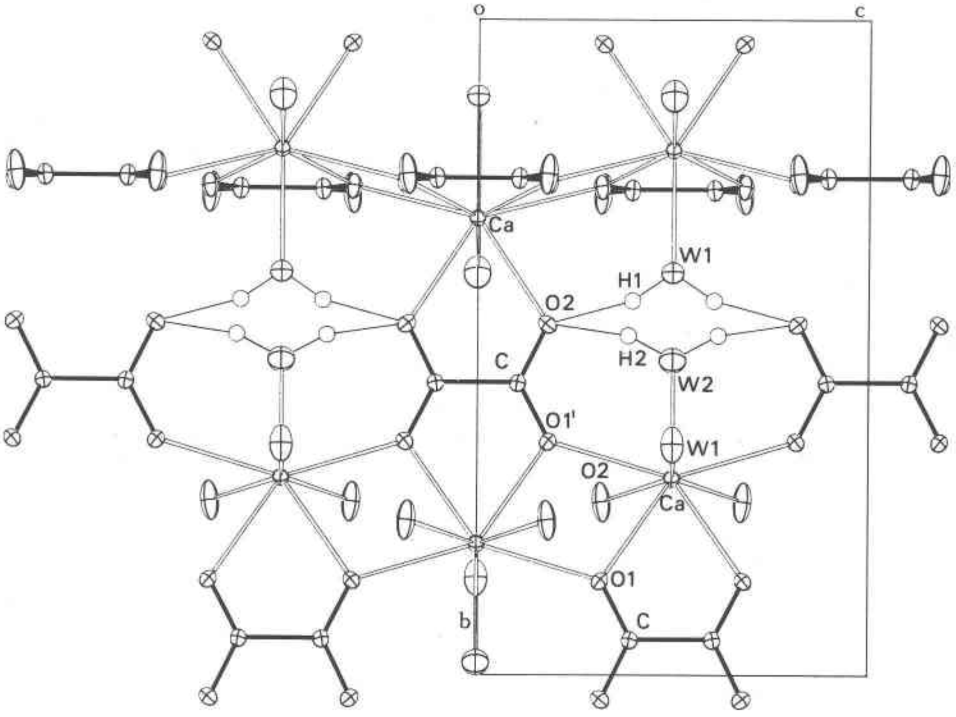


Fig. 3. Weddellite, section parallel to (100).

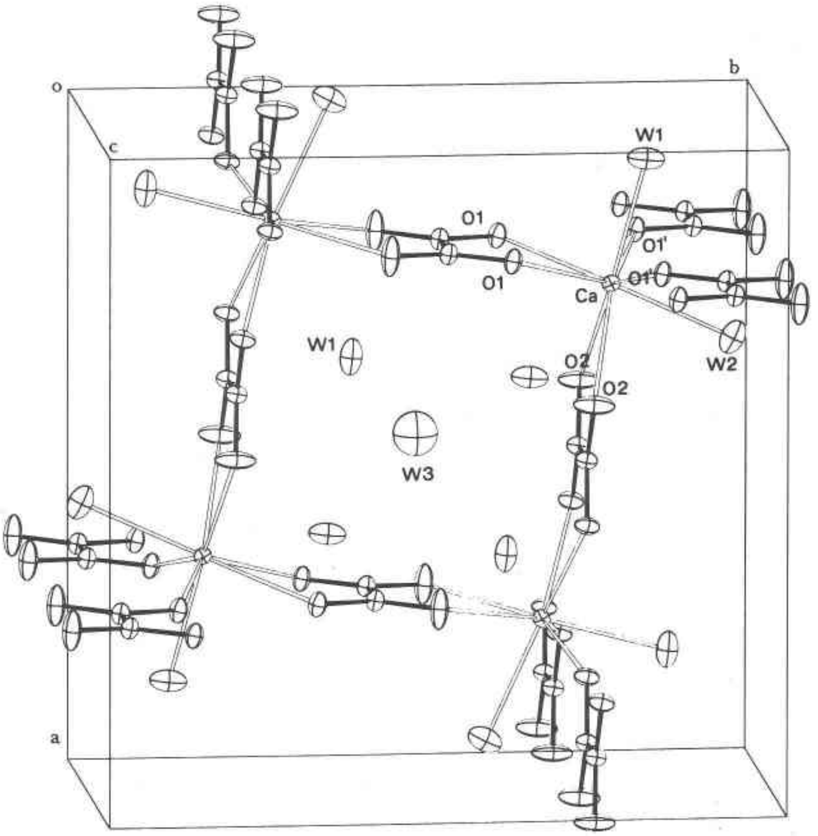


Fig. 4. Weddellite, section almost parallel to (001).

greater degree of hydration implies another crystalline phase, which could be that of the trihydrate hypothesized by some authors (Walter-Levy and Laniepiece, 1964; Gérard *et al.*, 1968; Doremus *et al.*, 1976).

Planarity and symmetry of the oxalic groups

The deviations of the atoms from the least-squares plane (Table 10) and the difference among the bond lengths and angles in each group were submitted to a significance test (Stout and Jensen, 1968, p. 419–421). It was found that:

(a) in whewellite the oxalic group C(1), C(2) . . . appears to be planar, with a symmetry not higher than *2mm*, and the oxalic group C(3), C(4). . . appears to be significantly non-planar, with a symmetry *1*;

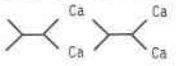
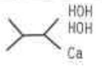
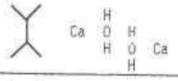
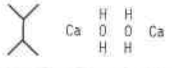
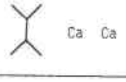
(b) in weddellite the oxalic group appears to be

Table 10. Deviations from planarity in oxalic groups

	Whewellite – Plane I		Whewellite – Plane II		Weddellite	
Equation of the least–squares plane *	5.9322x – 0.9465y – 0.0684z – –5.5155 = 0		0.2622x + 14.4202y + 1.2241z – –2.2035 = 0		0.7045x + 12.3509y + 0.0000z – –3.2891 = 0	
Direction cosines	0.9431 – 0.0649 – 0.0068		0.0417 0.9888 0.1210		0.0569 0.9984 0.0000	
Displacements (A) of atoms from the plane	C(1)	– 0.003(1)	C(3)	– 0.020(1)	C(1)	0.009(2)
	C(2)	0.001(1)	C(4)	– 0.011(1)	C(1')	0.009(2)
	O(1)	– 0.005(1)	O(5)	0.021(1)	O(1)	– 0.002(2)
	O(2)	0.005(1)	O(6)	– 0.003(1)	O(1')	– 0.002(2)
	O(3)	0.006(1)	O(7)	– 0.005(1)	O(2)	– 0.002(2)
	O(4)	– 0.006(1)	O(8)	0.012(1)	O(2')	– 0.002(2)

* Referred to the crystal axes; x, y, z are fractional coordinates

Table 11. Significant repeats in whewellite and weddellite

Sequence	Repeat	Vector
	14.6 A	$[010]_{\text{whew}}$ (see Fig. 1)
	7.3 A	$[001]_{\text{wedd}}$ (see Fig. 3)
	13.3 A	$[201]_{\text{whew}}$ (see Fig. 2)
	12.4 A	$[100]_{\text{wedd}}$ (see Fig. 3)
	10.1 A	$[001]_{\text{whew}}$ (see Fig. 1)

planar, with a symmetry not higher than *2mm* and not lower than *m* (crystallographic symmetry); *mmm* symmetry as proposed by Sterling (1965) must be excluded, consistent with the remarkable difference between the environments and bonds of O(1) and O(2).

Conclusions

The comparison between the crystal structures of weddellite and whewellite reveals marked similarities, which are in agreement with the frequent association of the two minerals and with the easy transformation of the former into the latter.

A simple scheme of linear dimensions (Table 11) shows and explains, with the help of the figures, the relationships existing between some significant repeats of the two compounds (in particular the ratio $c_{\text{wedd}}/b_{\text{whew}} = 1/2$) and displays the distortion of the structure resulting from the dehydration of weddellite to whewellite, which appears clearly by observing, along the repeats $[201]_{\text{whew}}$ and $[100]_{\text{wedd}}$, the change of the hydrogen bond system.

All the structural details given here could be of some interest for the study of the growth morphology of whewellite and weddellite and the possible epitaxial relations between the two minerals hypothesized by Lonsdale (1968) in urinary stones; in addition the structural relations will aid in the understanding of the kinetics of formation and transformation of these compounds on which investigations are in progress in this laboratory.

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