

Table 2. *Coordinates of water protons* (Å)Values in parentheses are taken from Galigné *et al.* (1970).

		<i>x</i>	<i>y</i>	<i>z</i>	Distance O _w -proton (Å)
I Vector	Proton 1	2.221	4.650	3.31	1.097
<i>p-p</i> vector of H ₂ O(1)		(2.0324)	(4.7763)	(3.31)	(0.87)
	Proton 2	2.221	6.230	3.31	1.097
		(2.0324)	(6.1037)	(3.31)	(0.87)
II Vector	Proton 1	0	2.629	4.101	0.996
<i>p-p</i> vector of H ₂ O(2)		(0)	(2.5459)	(4.0117)	(0.9)
	Proton 2	0	2.629	2.519	0.996
		(0)	(2.5459)	(2.6083)	(0.9)

Location of protons of water molecules

Assuming a linear H-bond in each case and putting in the value $r=1.581$ and 1.583 Å (as determined by this investigation) in the two cases, the coordinates of the water protons were calculated. The results are given in Table 2.

The assumption of a linear H-bond appears to be more valid for H₂O(2) than for H₂O(1). The latter might be involved in slightly non-linear H-bonds. This point must be borne in mind when one considers the co-ordinates of the protons of H₂O(1). The distances between the protons and their parent oxygen atoms are 1.097 and 0.996 Å respectively. These values appear to be 'normal', contributing to fairly strong H-bonds. An IR spectrum was obtained for the salt and this also suggests such normal H-bonds. On the other hand, using the values of the coordinates of the protons given by Galigné *et al.* (1970), if 2α is calculated from the inter-proton distances, improbable values of the order

of 18 and 15 Oersteds are obtained. Thus, our p.m.r. data lead to a more probable location for the protons.

Conclusion

The p.m.r. data on single crystals of lithium acetate dihydrate confirm the space group *Cmmm* and the presence of the two types of water molecules. The water oxygen atoms are tetra-coordinated, in H₂O(2) by four H, and H₂O(1) by two Li and two H. More probable locations for the water protons have been obtained.

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The Crystal and Molecular Structure of a Lactone Oxime

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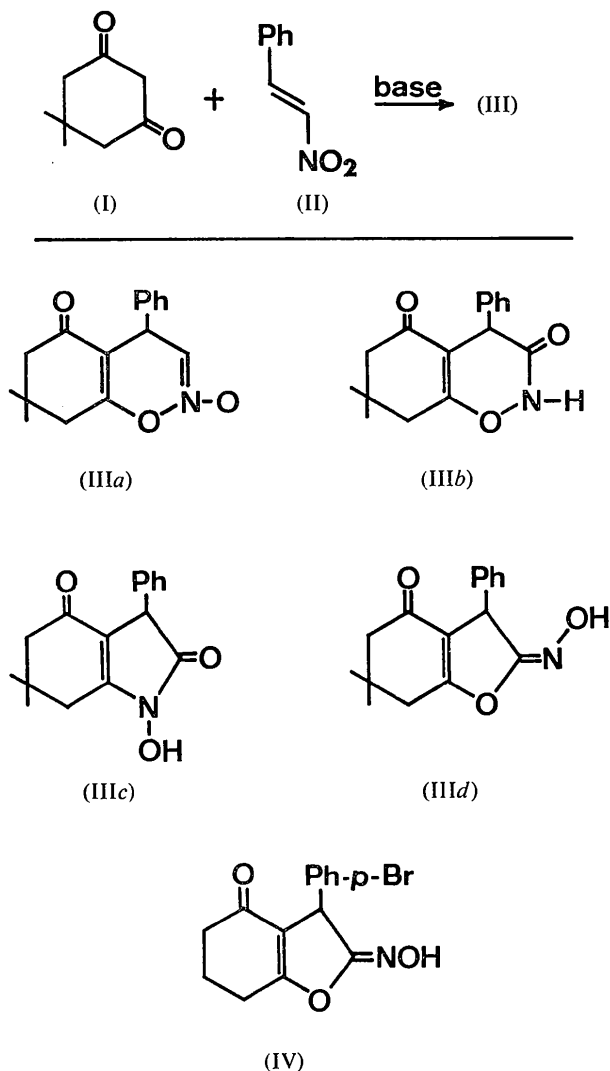
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The base-catalyzed condensation of dimedone with β -nitrostyrene gives an unusual lactone oxime, C₁₆H₁₇NO₃, which crystallizes in the monoclinic space group, *P2₁/n*. The unit-cell parameters are $a=13.158$, $b=8.657$, $c=12.205$ Å, and $\beta=98.84^\circ$. Data were collected on an automated diffractometer; the structure was solved by direct methods and refined to an *R* value of 0.044 by the least-squares method. The oxime is exclusively *anti* with respect to the oxygen of the lactone ring and, in the crystalline state, is hydrogen-bonded to the carbonyl oxygen of an adjacent molecule.

Introduction

Several plausible structures (III*a*–III*c*) can be written for the product C₁₆H₁₇NO₃ of the base-catalyzed con-

densation of dimedone (I) and β -nitrostyrene (II). Berestovitskaya, Sopova & Perekalin (1967) apparently were the first to synthesize this compound, to which they assigned the structure (III*a*).



As we have reported in a preliminary communication (Dominianni, Chaney & Jones, 1970) X-ray diffraction studies show the correct structure to be the unusual lactone oxime (III*d*), which we had not previously considered. The I.U.C. name for the compound is 6,7-dihydro-6,6-dimethyl-3-phenylbenzofuran-2,4(3*H*,5*H*)-dione, 2-oxime. This structure is of particular interest because at the time we submitted the above-mentioned communication we were aware of no other example of a lactone oxime. By coincidence, a preliminary report on the structure of another closely related lactone oxime (IV) was published at the same time as ours by Ansell, Moore & Nielsen (1970). Details of their structure work have recently appeared (Ansell, Moore & Nielsen, 1971). We wish to report here the complete details of our structure analysis on (III*d*).

Experimental

The reaction product (III) crystallized from methanol as colorless, elongated prisms melting in the range

170–173°, with decomposition. Combustion analysis and molecular weight (272.3) determination by mass spectroscopy confirmed the empirical formula, $C_{16}H_{17}NO_3$. Precession and Weissenberg photographs showed the Laue symmetry to be $2/m$ (C_{2h}). Systematic absences in the reflections $0k0$ with k odd and $h0l$ with $h+l$ odd indicated the centrosymmetric, monoclinic space group, $P2_1/n$. The a and c axes were chosen to give space group $P2_1/n$ rather than the more common $P2_1/c$ or $P2_1/a$ in order to obtain the β angle nearest 90°. The unit-cell parameters were accurately measured using a four-circle diffractometer with copper radiation. The density of the crystals was determined by flotation in an aqueous solution of zinc sulfate. The crystal parameters are given in Table 1.

Table 1. *Crystal parameters*

Formula	$C_{16}H_{17}NO_3$
Molecular weight	271.3
Space group	$P2_1/n$
Z	4
a	$13.158 \pm 0.002 \text{ \AA}$
b	8.657 ± 0.002
c	12.205 ± 0.002
β	$98.84 \pm 0.01^\circ$
V	1373.7 \AA^3
ρ_o	1.317 g.cm^{-3}
ρ_c	1.312
Crystal size	$0.2 \times 0.3 \times 0.5 \text{ mm}$

Intensity data were collected on a Picker automated diffractometer, using nickel-filtered copper radiation. The instrument was operated in the θ - 2θ mode with a scanning speed of $1^\circ/\text{min}$ and a variable scan width (2.5° minimum) to compensate for $K\alpha_1$ - $K\alpha_2$ splitting. Backgrounds were counted on each side of the peak for 30 sec. A check reflection (433), which was remeasured after every fifty reflections, showed a loss of intensity of less than 6 percent during the data collection. 2418 reflections, of which 884 were 'less-than' reflections, were observed from the $\pm hkl$ quadrant in the 2θ range from 5 to 134° . Reflections were indicated as 'less-than' if the peak intensity was less than two standard deviations above background. In order to correct for the small apparent crystal deterioration, the raw intensities were scaled using the periodic check reflection. Lorentz and polarization corrections were applied to the intensities and their standard deviations, and the data were placed on an absolute scale using a Wilson plot.

The structure was solved by direct methods, using an iterative multiple solution application of the Sayre equation. The three origin-defining reflections and four reflections of the starting set were as follows.

			E	
Origin	9	1	6	+4.13
	$\bar{3}$	0	1	+2.76
	$\bar{9}$	1	7	+3.31
Starting set	$\bar{6}$	1	4	2.65
	$\bar{3}$	0	3	2.78
	10	1	4	3.25
	2	5	8	3.64

Each of these reflections had a high E value and a large number of Σ_2 interactions. 319 reflections, down to an E value of 1.56, were included in the phase-determining set. Of the 16 solutions obtained from permutation of the four starting set phases, one solution clearly stood out above the others, with a consistency index of 0.82. This solution, with starting set phases $--+-$, also showed the most rapid convergence. Comparison of the phases from this solution with those obtained from the final least-squares refinement shows that all 319 phases were correct. An E map calculated from this set clearly showed all twenty non-hydrogen atoms and gave the first indication that the structure was the unexpected lactone oxime, (III*d*), above.

Refinement

The structure was refined by the least-squares procedure, minimizing the function $\sum w(k|F_o| - |F_c|)^2$. Weights were chosen equal to the reciprocals of the variances calculated from counting statistics. The atomic scattering factors used were those given by Hanson, Herman, Lea & Skillman (1964). An isotropic temperature factor of 3.61, the value calculated from the Wilson plot, was used initially for all nonhydrogen atoms. Three cycles of block-diagonal matrix least-squares brought the usual R index to 0.11. The temperature factors were converted to anisotropic, and an additional three cycles brought R down to 0.09.

At this point, a difference Fourier map was calculated, from which the positions of all 17 hydrogen atoms were readily apparent. The lowest hydrogen atom had a density of $0.7 \text{ e.}\text{\AA}^{-3}$, and there was no extraneous peak greater than that value. All hydrogen atoms were included in the least-squares refinement, with isotropic temperature factors equal to the temperature factors of atoms to which they are attached. Eight cycles of

block-diagonal least-squares refinement, during which the hydrogen atom positions were refined, brought the R index to a final value of 0.044. At this point, all non-hydrogen atom parameter shifts were less than one-third the error in the parameter.

Of the 1571 reflections included in the final least-squares cycle, 37 were 'less-than' reflections which calculated larger than the threshold value. The estimated standard deviations for the positional parameters for heavy atoms are in the range 0.002 to 0.005 Å; for hydrogen atoms, the standard deviations are 0.02 to 0.05 Å. The final heavy atom parameters and their e.s.d.'s are listed in Table 2, and the corresponding values for the hydrogen atoms are listed in Table 3. The final observed and calculated structure factors are given in Table 4.

Table 3. *Hydrogen atom parameters and their standard deviations (in parentheses)*

Values for the coordinates have been multiplied by 10^3 . The temperature factors are in the form $\exp(-B \sin^2 \theta/\lambda^2)$.

	Bonded to	x				B
		x	y	z	B	
H(21)	C(1)	487 (3)	366 (4)	426 (3)	2.1 (0.9)	
H(22)	C(5)	321 (3)	479 (4)	21 (3)	2.7 (0.9)	
H(23)	C(5)	239 (3)	485 (5)	114 (3)	2.7 (1.0)	
H(24)	C(12)	234 (3)	880 (5)	44 (3)	3.8 (1.1)	
H(25)	C(12)	163 (3)	731 (5)	68 (3)	3.5 (1.0)	
H(26)	C(12)	227 (3)	726 (5)	-49 (3)	3.5 (1.0)	
H(27)	C(13)	429 (3)	847 (5)	51 (4)	4.2 (1.2)	
H(28)	C(13)	415 (3)	684 (5)	-31 (3)	3.6 (1.1)	
H(29)	C(13)	485 (3)	673 (5)	82 (3)	3.6 (1.1)	
H(30)	C(7)	355 (3)	860 (5)	230 (3)	2.9 (1.0)	
H(31)	C(7)	278 (3)	704 (5)	260 (3)	2.9 (1.0)	
H(32)	C(16)	600 (3)	537 (5)	189 (3)	2.9 (1.0)	
H(33)	C(17)	787 (3)	549 (5)	185 (4)	4.8 (1.2)	
H(34)	C(18)	911 (3)	438 (5)	333 (3)	3.3 (1.0)	
H(35)	C(19)	844 (3)	313 (5)	482 (3)	2.8 (1.0)	
H(36)	C(20)	669 (3)	289 (4)	486 (3)	2.3 (0.9)	
H(37)	O(11)	522 (3)	-60 (5)	399 (3)	3.8 (1.1)	

Table 2. *Heavy-atom parameters and their standard deviations (in parentheses)*

All values have been multiplied by 10^4 . The anisotropic temperature factor is expressed in the form $\exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)]$.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
C(1)	5060 (2)	3766 (4)	3386 (2)	42 (2)	76 (4)	44 (2)	-2 (2)	2 (2)	0 (3)
C(2)	4634 (2)	2359 (4)	2726 (3)	43 (2)	80 (4)	51 (2)	-2 (3)	9 (2)	-4 (3)
O(3)	4018 (2)	2812 (2)	1737 (2)	63 (2)	78 (3)	56 (2)	0 (2)	-8 (1)	-7 (2)
C(4)	3846 (2)	4361 (4)	1838 (3)	42 (2)	81 (4)	55 (2)	0 (3)	6 (2)	3 (3)
C(5)	3111 (3)	5121 (4)	955 (3)	51 (2)	102 (5)	62 (3)	-4 (3)	-5 (2)	7 (3)
C(6)	3264 (3)	6871 (4)	995 (3)	42 (2)	103 (5)	64 (3)	5 (3)	2 (2)	15 (3)
C(7)	3413 (3)	7420 (4)	2215 (3)	57 (2)	100 (5)	69 (3)	18 (3)	1 (2)	-1 (3)
C(8)	4273 (2)	6613 (4)	2975 (3)	46 (2)	90 (5)	51 (2)	-2 (3)	10 (2)	-2 (3)
C(9)	4382 (2)	4999 (4)	2749 (2)	37 (2)	80 (4)	49 (2)	-3 (2)	7 (2)	1 (3)
N(10)	4659 (2)	921 (3)	2880 (2)	55 (2)	94 (4)	60 (2)	-5 (2)	5 (2)	-1 (3)
O(11)	5256 (2)	530 (3)	3896 (2)	74 (2)	99 (4)	63 (2)	0 (2)	-1 (2)	10 (2)
C(12)	2291 (3)	7638 (5)	350 (3)	49 (2)	159 (7)	88 (4)	16 (3)	-3 (2)	27 (4)
C(13)	4194 (3)	7327 (5)	451 (3)	56 (3)	184 (7)	83 (3)	-7 (4)	12 (2)	27 (4)
O(14)	4809 (2)	7280 (3)	3743 (2)	74 (2)	96 (3)	60 (2)	2 (2)	-4 (2)	-12 (2)
C(15)	6210 (2)	3997 (4)	3375 (2)	41 (2)	87 (4)	44 (2)	0 (3)	6 (2)	1 (3)
C(16)	6563 (3)	4763 (5)	2516 (3)	43 (2)	173 (7)	58 (3)	4 (3)	7 (2)	27 (4)
C(17)	7616 (3)	4937 (5)	2506 (3)	49 (2)	198 (8)	76 (3)	-6 (4)	12 (2)	27 (4)
C(18)	8317 (3)	4316 (5)	3349 (3)	48 (2)	152 (6)	75 (3)	3 (4)	6 (2)	18 (4)
C(19)	7971 (3)	3561 (4)	4209 (3)	43 (2)	139 (6)	61 (3)	8 (3)	-3 (2)	14 (3)
C(20)	6919 (2)	3375 (4)	4225 (3)	46 (2)	99 (5)	48 (2)	3 (3)	2 (2)	9 (3)

Table 4. Observed and calculated structure factors

In each group, the three columns from left to right are the values of h, F_o, and F_c. Entries marked with an asterisk are 'less-than' reflections.

Table with multiple columns containing numerical data for structure factors. The table is organized into groups, with each group containing three columns of values (h, F_o, F_c). Asterisks indicate 'less-than' reflections. The data is dense and spans the entire page.

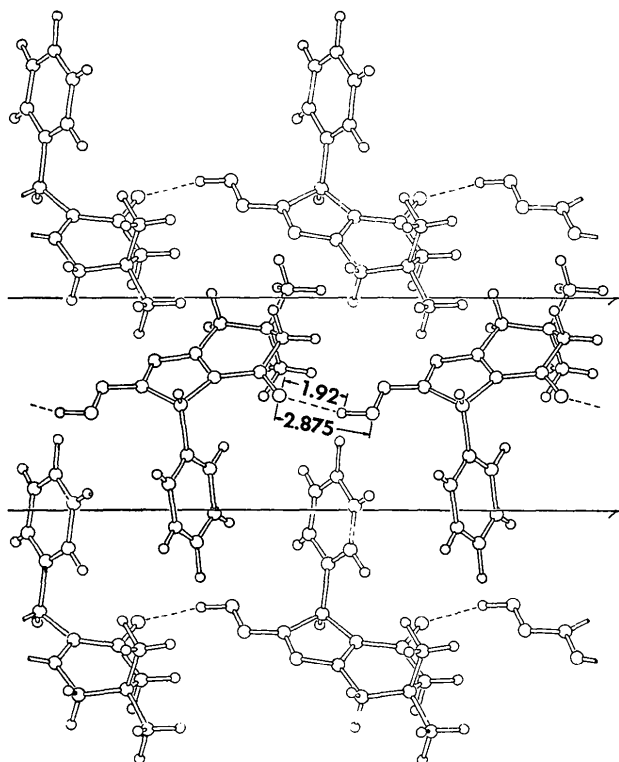


Fig. 1. Packing diagram of the structure as viewed down the c^* axis.

The structure

Fig. 1 shows the crystal structure of the lactone oxime as viewed down the c^* axis. The molecules form chains held together by hydrogen bonding of the oxime hy-

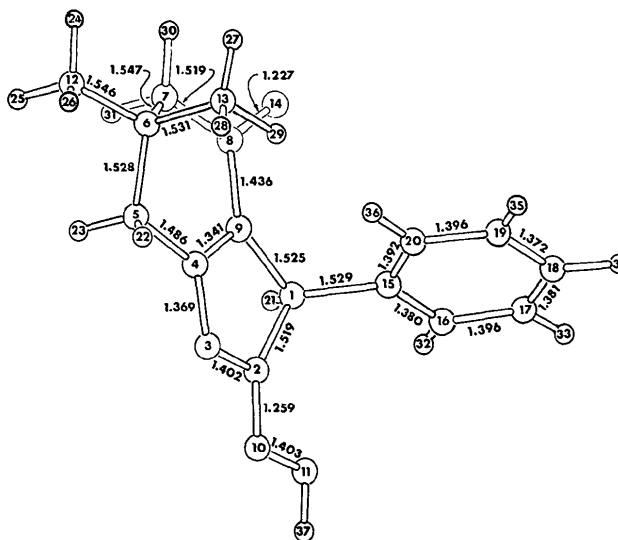


Fig. 2. Bond lengths for atoms other than hydrogen. The average standard deviation is 0.005 Å.

Table 5. Bond distances and angles involving hydrogen atoms

Average standard deviation for bonds is 0.04 Å, for angles of the type H-X-X, 2°, and for angles of the type H-X-H, 3°.

1	Atom 2	3	Distance (Å) or angle (°)	1	Atom 2	3	Distance (Å) or angle (°)
H(21)	C(1)		1.14	H(29)	C(13)		1.05
		C(2)	108			C(6)	110
		C(9)	110	H(30)	C(7)		1.04
		C(15)	112			C(6)	114
H(22)	C(5)		0.98			C(8)	107
		C(4)	112	H(31)	C(7)		1.06
		C(6)	107			H(31)	113
		H(23)	113			C(6)	110
H(23)	C(5)		1.03	H(32)	C(16)		98
		C(4)	105			C(8)	1.11
		C(6)	110			C(15)	119
H(24)	C(12)		1.01			C(17)	120
		C(6)	110	H(33)	C(17)		1.03
		H(25)	105			C(16)	120
		H(26)	114			C(18)	120
H(25)	C(12)		1.06	H(34)	C(18)		1.04
		C(6)	111			C(17)	121
		H(26)	112			C(19)	120
H(26)	C(12)		1.07	H(35)	C(19)		0.97
		C(6)	105			C(18)	122
H(27)	C(13)		1.00			C(20)	117
		C(6)	109	H(36)	C(20)		0.97
		H(28)	108			C(15)	121
		H(29)	112			C(19)	119
H(28)	C(13)		1.02	H(37)	O(11)		0.99
		C(6)	110			N(10)	108
		H(29)	97				

drogen, H(37), of one molecule to the carbonyl oxygen, O(14), of another molecule displaced one unit cell along the b axis. The distance between the two hydrogen-bonded oxygen atoms, O(14) and O(11), is 2.875 Å, and the OH...O angle is 161°. As is often observed in crystal structures, the aromatic rings from adjacent molecules overlap to form a stack which is nearly coaxial with a 2_1 screw axis. Each aromatic ring is inclined at an angle of 30.8° to the screw axis.

Figs. 2 and 3 show the heavy-atom bond distances and angles from the final least-squares refinement; Table 5 lists the distances and angles for bonds involving hydrogen. Table 6 lists intermolecular distances less than 3.5 Å for nonhydrogen atoms. All the molecular parameters involving heavy atoms appear quite reasonable, especially in the aromatic ring where the consistency is excellent. The oxime C=N and N-O bond lengths are 1.259 and 1.403 Å, respectively; these values fall within the range of bond lengths 1.22 to 1.33 Å for C=N and 1.32 to 1.41 Å for N-O found in other oxime structures (see references given by Ansell, Moore & Nielsen, 1971).

Table 6. Intermolecular distances less than 3.5 Å involving atoms other than hydrogen

Atom 1 is in molecule at x, y, z .

Atom 1 to Atom 2	In molecule at	Distance (Å)
O(14) O(11)	$x, 1+y, z$	2.875*
O(11) O(11)	$1-x, -y, 1-z$	3.020
O(14) N(10)	$x, 1+y, z$	3.320
O(3) C(19)	$\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$	3.395
O(11) O(14)	$1-x, 1-y, 1-z$	3.462
C(7) N(10)	$x, 1+y, z$	3.481

* Hydrogen bond.

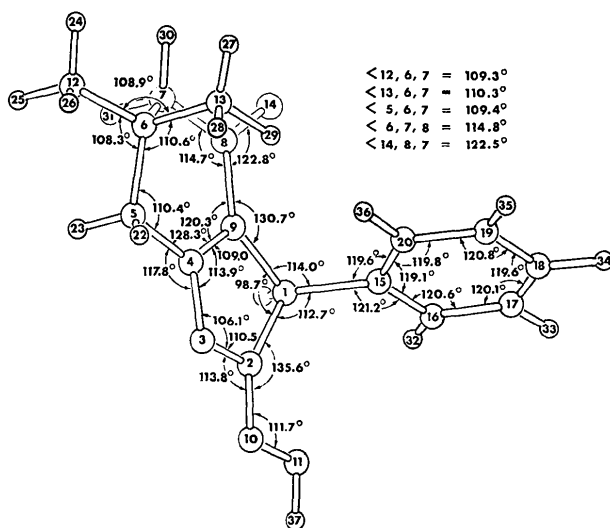


Fig. 3. Bond angles for atoms other than hydrogen. The average standard deviation is 0.3°.

Table 7 gives the results of least-squares plane calculations involving each of the three rings in the molecule. The aromatic ring is quite planar and is turned at an angle of 74.2° to the plane of the lactone ring, which is also nearly planar. The cyclohexenone ring assumes a half-chair conformation and shows a near perfect staggering of the hydrogen substituents on C(5) and C(7), which are adjacent to the *gem*-dimethyl group at C(6).

Table 7. Equations of certain least-squares planes, deviations of atoms from these planes, and angles between planes

Equations of planes given for an orthogonal axial system, where X and Y coincide with the crystal axes a and b , respectively. Atoms marked by an asterisk were included in the least-squares calculation. Angles given are between plane normals.

Plane 1: $-0.084X + 0.859Y + 0.505Z = 4.389$			
C(15)*	0.003 Å	C(1)	-0.033 Å
C(16)*	-0.004	H(32)	0.123
C(17)*	0.006	H(33)	-0.031
C(18)*	-0.008	H(34)	-0.037
C(19)*	0.008	H(35)	0.015
C(20)*	-0.006	H(36)	0.055

Plane 2: $0.885X + 0.043Y - 0.464Z = 3.413$			
C(1)*	0.164 Å	C(5)	-0.293 Å
C(2)*	0.092	C(8)	-0.350
O(3)*	0.109	H(21)	-0.682
C(4)*	-0.104	C(15)	1.519
C(9)*	-0.118	H(37)	-0.246
N(10)*	-0.044		
O(11)*	-0.099		

Angle between planes 1 and 2 is 74.2°.

Plane 3: $0.881X + 0.199Y - 0.430Z = 3.986$			
C(4)*	-0.032 Å	C(1)	0.214 Å
C(5)*	-0.151	O(3)	-0.034
C(6)*	0.300	H(22)	0.397
C(7)*	-0.262	H(23)	-1.126
C(8)*	0.070	C(12)	-0.253
C(9)*	0.075	C(13)	1.825
		H(30)	-0.018
		H(31)	-1.319
		O(14)	0.284

Angle between planes 1 and 3 is 83.1°.

Angle between planes 2 and 3 is 9.2°.

The conformation of the oxime in this structure is exclusively *anti* with respect to the oxygen atom of the lactone ring. By contrast, Ansell, Moore & Nielsen (1971) have found in the crystal structure of compound (IV) a roughly $\frac{2}{3}$ to $\frac{1}{3}$ mixture of the *syn* and *anti* oximes. It may be that the packing of the *gem*-dimethyls in the present structure causes crystallization of only the *anti* form from the *syn-anti* mixture.

We wish to express our appreciation to Dr S. J. Dominiani of these laboratories for providing the crystals used in this study and to Mr D. W. Smith for computer assistance. Thanks is also due Professor James M.

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Crystal Structures of $\text{Ca}_2\text{Fe}_{1.43}\text{Al}_{0.57}\text{O}_5$ and $\text{Ca}_2\text{Fe}_{1.28}\text{Al}_{0.72}\text{O}_5^*$

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The crystal structures of $\text{Ca}_2\text{Fe}_{1.43}\text{Al}_{0.57}\text{O}_5$ and $\text{Ca}_2\text{Fe}_{1.28}\text{Al}_{0.72}\text{O}_5$ have been determined from single-crystal data obtained with a Buerger-Supper-Pace-Picker automatic diffractometer. Both are isostructural with brownmillerite, belonging to space group $Ibm2$ (C_{2v}^{22}). Lattice constants are $a = 5.588$ (5), $b = 14.61$ (2), $c = 5.380$ (5) Å and $a = 5.583$ (5), $b = 14.58$ (2), $c = 5.374$ (5) Å respectively. The distribution of the Al^{3+} ions in these crystals and in brownmillerite ($\text{Ca}_2\text{FeAlO}_5$), all synthesized under very similar conditions, is such that 75–76% of the Al^{3+} ions are in tetrahedral sites. The results give new information on the magnetic phase relations of the $\text{Ca}_2\text{Fe}_{2-x}\text{Al}_x\text{O}_5$ system.

Introduction

Crystals of $\text{Ca}_2\text{Fe}_2\text{O}_5$ have their Fe^{3+} ion moments directed along the crystallographic c axis (Gonser, Grant, Wiedersich & Geller, 1966; Corliss, Hastings, Kunmann & Banks, 1966; Friedman, Shakad & Shtrikman, 1967; Takeda, Yamaguchi, Tomiyoshi, Sukase, Sugimoto & Watanabe, 1968); the most probable magnetic space group of $\text{Ca}_2\text{Fe}_2\text{O}_5$ is $Pcm'n'$. In brownmillerite, $\text{Ca}_2\text{FeAlO}_5$, the Fe^{3+} ion moments are aligned along the a axis (Grant, Geller, Wiedersich, Gonser & Fullmer, 1968); the most probable magnetic space group was thought to be $Ic'm'm'$ because although the crystal structure had not been worked out in detail, $Icmm$ was reported to be the most probable space group (Büssem, 1937, 1938; Smith, 1962).

A refinement of the structure of $\text{Ca}_2\text{Fe}_2\text{O}_5$ (Bertaut, Blum & Sagnières, 1959) was carried out (Colville, 1970) to verify the results of a Mössbauer spectroscopic study (Grant, 1969) which showed that the oxygen octahedra and tetrahedra about the Fe^{3+} ions must be considerably distorted. A determination of the crystal structure of brownmillerite (Colville & Geller, 1971) showed that its most probable space group is $Ibm2$.

(In fact, $Icmm$ would have required one unreasonably short tetrahedral cation–oxygen distance.) It was proposed (Geller, Grant & Colville, 1971) that the most probable magnetic space group for brownmillerite is $I_pbm'2'$.

A Mössbauer spectroscopic investigation (Geller, Grant & Fullmer, 1970; see also Geller, Grant & Gonser, 1971) of intermediate compositions (that is, $0 < x < 1$ in $\text{Ca}_2\text{Fe}_{2-x}\text{Al}_x\text{O}_5$) showed that there is a range of composition and of temperature in which two magnetic structures occur simultaneously. In the papers thus far published (Geller *et al.*, 1970; Geller *et al.*, 1971) it was thought that these corresponded to the $Pcm'n'$ of $\text{Ca}_2\text{Fe}_2\text{O}_5$ and (originally $Ic'm'm'$ but later) $I_pbm'2'$ of $\text{Ca}_2\text{FeAlO}_5$. Thus we decided to refine the structure of $\text{Ca}_2\text{Fe}_{1.28}\text{Al}_{0.72}\text{O}_5$, expecting, at room temperature, to find about 70% of the $I_pbm'2'$ structure and 30% of the $Pcm'n'$ structure (Geller, Grant & Fullmer, 1970). We did not know the scale of the domains and so we tested both coherent ($I \sim |F|$) and incoherent ($I \sim |F|^2$) scattering models. We found that the best fit of $|F_c|$ vs. $|F_o|$ was obtained when the structure was refined purely in $Ibm2$. This now presented difficulties in the interpretation of the results obtained by Mössbauer spectroscopy.

We next decided to refine the structure of the crystal with formula $\text{Ca}_2\text{Fe}_{1.40}\text{Al}_{0.60}\text{O}_5$. Past investigations appeared to fix the transition from the primitive ortho-

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