Crystal and Molecular Structure of Tetra-acetyl Diborate

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The crystal structure of the title compound has been solved by direct methods from three-dimensional X-ray diffractometer data and refined by least-squares methods to R 0·050 for 1337 independent reflections. All the hydrogen atoms have been located. The crystals are monoclinic, space group $P2_1/c$, with a = 7·528, b = 8·231, c = 19·891 Å, β = 96·0°, Z = 4. Two distorted tetrahedra sharing a vertex are present in the molecule; two acetoxy-groups have only a B–O bond, whereas the remaining two are each linked to both boron atoms. The abnormally long and short B–O bond lengths (range 1·384—1·561 Å) are interpreted in terms of detailed valence neutralization. The molecules do not present strong intermolecular contacts, as shown by the considerable instability of the compound and the great thermal motion of the atoms.

THERE has been considerable discussion concerning the product obtained by reaction between orthoboric acid and acetic anhydride and, finally, it was identified and confirmed[1] as (I) tetra-acetyl diborate † $(AcO)_2$-$B{\cdot}O{\cdot}B(OAc)_2$ (TADB). Its derivatives with acetates of several uni- and divalent cations have been recently described.[2]

In order to find out more of the crystallo-chemical behaviour of boron,[3] we have carried out, and report here, the crystal and molecular structure of (I).

EXPERIMENTAL

Crystal Data.—$C_8H_{12}B_2O_9$, M = 273·6, Monoclinic, a = 7·528(3), b = 8·231(7), c = 19·891(5) Å, β = 96·0° (0·1), U = 1225·7 Å^3, Z = 4, D_c = 1·483 g cm^{-3}. Space group $P2_1/c$ from systematic absences: $0k0$ with k odd and $h0l$ with l odd. Cu-K_α radiation λ = 1·5418 Å; μ(Cu-K_α) = 11 cm^{-1}.

Preparation of the Crystals.—Acetic anhydride (20 ml) was added to a suspension of orthoboric acid (3 g) in acetic acid (10 ml) and the mixture heated on a vapour bath, under nitrogen, until all the solid had dissolved; (I) was then precipitated with dry ethyl ether and purified by successive precipitation from a solution in dry acetic acid by addition of ether. Acetic anhydride was then added to the microcrystalline powder; colourless prismatic crystals elongated along b and suitable for X-ray investigation were finally obtained after a few days, by keeping the mixture below 0 °C. The crystals (m.p. 147—148 °C) immediately decompose in the air; a prismatic crystal of cross-section *ca.* 0·65 mm was therefore mounted in a capillary which was sealed after saturation with acetic anhydride.

X-*Ray Data.*—The intensity data and unit cell parameters were measured with a Wooster four-circle diffractometer using Ni-filtered Cu-K_α radiation and setting the crystal with the b-axis parallel to the spindle axis. The cell parameters were obtained from the diffractometer setting for a number of reflections; intensity data were collected from 2234 independent reflections in the range $0 < 2\theta < 137°$ (*ca.* 79% of the limiting Cu-K_α sphere), using the ω scan mode, with a scan angle of 3° and a scan speed of 1° min^{-1}. Measurements of a reference reflection were repeated after every group of 30 reflections. The data were corrected for polarization and geometrical factors; no absorption correction was applied.

Determination and Refinement of the Structure.—The structure was solved by direct methods. The sign determination was undertaken by applying the Sayre relationship [4] to 222 reflections with normalized structure factors [5] $|E| > 1{\cdot}5$, by use of the program described in ref. 6. The positions of all the oxygen atoms were located from the E-map,[7] computed with the phases corresponding to the set with the highest consistency index (0·87). With these co-ordinates, successive three-dimensional Fourier syntheses revealed the positions of all the non-hydrogen atoms. The parameters derived from the last Fourier synthesis were refined, first isotropically and then anisotropically, by full-matrix least-squares calculations, performed with the Busing *et al.*[8] program ORFLS. In the refinement only 1337 non-equivalent reflections, having net intensity ranging from 5 to 1000 were used; reflections with lower intensity were coded as unobserved. On convergence a difference Fourier synthesis was calculated and the co-ordinates of all the hydrogen atoms were located: no spurious peaks appeared. Refinement was continued with anisotropic thermal parameters for all the non-hydrogen atoms and isotropic thermal parameters for the hydrogen atoms. A comparison between the observed and calculated structure factors suggested that many of the strongest reflections might be suffering from secondary extinction effects; the correction of Zachariasen [9] was applied (final value g 8·66 10^{-6}). The refinement results are summarized in Table 1.

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† The compound is also called diboronoxide tetra-acetate, or pyroboron acetate.

[1] G. W. Gerrard and M. Wheelans, *Chem. and Ind.*, 1954, 758; R. G. Hayter, A. W. Laubengayer, and P. G. Thompson, *J. Amer. Chem. Soc.*, 1957, **79**, 4243; A. Perotti, M. Cola, and A. Parmigiani, *Gazzetta*, 1960, **90**, 1020; J. Goubeau and M. Lehman, *Z. anorg. Chem.*, 1963, **322**, 224.

[2] U. Kibbel, *Z. Chem.*, 1964, **4**, 104; 1965, **5**, 395; H. A. Lehmann, G. Kessler, P. Denecke, and G. Nickl, *Z. anorg. Chem.*, 1965, **340**, 16; A. Perotti and G. P. Caccini, 1965, Thesis (G.P.C.), University of Pavia, Italy; A. Perotti and P. Salvini, 1966, Thesis (P.S.), University of Pavia, Italy; U. Kibbel, *Z. anorg. Chem.*, 1968, **359**, 272.

[3] (*a*) A. Dal Negro, C. Sabelli, and L. Ungaretti, *Atti Accad. naz. Lincei, Rend. Classe Sci. fis. mat. nat.*, 1969, **47**, 353; (*b*) *Amer. Minerol.*, 1971, **56**, 1553; (*c*) E. Cannillo, A. Dal Negro, and L. Ungaretti, *ibid.*, in the press.

[4] D. Sayre, *Acta Cryst.*, 1952, **5**, 60.

[5] H. Hauptman and J. Karle, 1953, *Amer. Chem. Soc.*, Monograph N.3, Pittsburg, Polycrystal Book Service.

[6] R. E. Long, Ph.D. Thesis, 1965, University of California, Los Angeles.

[7] I. L. Karle, H. Hauptman, J. Karle, and A. B. Wing, *Acta Cryst.*, 1958, **11**, 257.

[8] W. R. Busing, K. O. Martin, and H. A. Levy, 1962, U.S. Clearing House Fed. Sci. Technol., *Inform. Report* ORNL TM 306.

[9] W. H. Zachariasen, *Acta Cryst.*, 1963, **A16**, 1139.

The atomic scattering factors given in ref. 10 were used for the structure factor calculations. The final parameter shifts were $<0{\cdot}16\,\sigma$. The observed and calculated structure factors are listed in Supplementary Publication No. 20404 (16 pp., 1 microfiche).* The final positions for the non-hydrogen atoms are listed in Table 2, the analysis of the anisotropic thermal parameters for the non-hydrogen atoms are listed in Table 3, and the positional and isotropic thermal parameters for the hydrogen atoms in Table 4.

TABLE 1

Summary of the refinement

Step	R (%)
(1) Structure factors, position of O atoms from E-map	60·0
(2) Structure factors, all non-hydrogen atoms	27·3
(3) 3 Cycles isotropic least-squares, 1337 independent reflections	16·5
(4) 3 Cycles anisotropic least-squares for all non-hydrogen atoms	7·5
(5) Hydrogen atoms from difference-Fourier included	6·2
(6) 2 Cycles with C, B, O anisotropic, H isotropic, and secondary extinction correction	5·0

TABLE 2

Final atomic co-ordinates with their standard deviations in parentheses

Atom	x/a	y/b	z/c
O(1)	0·8246(3)	0·1979(3)	0·3506(1)
O(2)	0·7778(3)	0·1170(3)	0·4546(1)
O(3)	0·8799(3)	0·4806(3)	0·3239(1)
O(4)	0·8171(2)	0·4045(2)	0·4366(1)
O(5)	0·7855(3)	0·3150(3)	0·5459(1)
O(6)	1·0936(3)	0·3384(3)	0·3929(1)
O(7)	1·0559(3)	0·2496(3)	0·4964(1)
O(8)	0·8704(4)	0·0844(3)	0·6007(1)
O(9)	0·5849(3)	0·4611(4)	0·3000(1)
B(1)	0·8908(4)	0·3678(4)	0·3776(2)
B(2)	0·8532(4)	0·2812(4)	0·4832(2)
C(1)	0·7692(4)	0·0955(4)	0·3913(2)
C(2) †	0·6864(6)	−0·0560(5)	0·3627(2)
C(3)	1·1534(4)	0·2900(4)	0·4508(2)
C(4) †	1·3514(5)	0·2778(6)	0·4672(2)
C(5)	0·8058(4)	0·2206(4)	0·6004(2)
C(6) †	0·7397(7)	0·2989(6)	0·6600(2)
C(7)	0·7245(5)	0·5224(4)	0·2891(2)
C(8) †	0·7504(7)	0·6446(6)	0·2353(2)

† Carbon atoms belonging to methyl groups.

TABLE 3

Final anisotropic thermal parameters ($\times 10^4$) * and their standard deviations in parentheses

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
O(1)	179(5)	125(4)	22(1)	−14(3)	3(1)	−10(1)
O(2)	154(4)	98(4)	23(1)	−24(3)	3(1)	−10(1)
O(3)	153(5)	163(5)	22(1)	−5(4)	2(1)	16(1)
O(4)	133(4)	99(4)	20(1)	13(3)	3(1)	−4(1)
O(5)	152(4)	123(4)	19(1)	11(3)	12(1)	−7(1)
O(6)	119(4)	174(5)	22(1)	7(3)	4(1)	5(1)
O(7)	94(4)	133(4)	23(1)	9(3)	0(1)	−1(1)
O(8)	333(8)	166(5)	31(1)	59(5)	33(2)	14(2)
O(9)	166(5)	257(6)	36(1)	19(5)	−3(2)	8(2)
B(1)	128(7)	110(6)	22(1)	−20(5)	−1(2)	10(2)
B(2)	85(6)	112(6)	19(1)	13(5)	6(2)	−9(2)
C(1)	118(6)	106(6)	22(1)	−4(5)	−10(2)	−9(2)
C(2)	278(11)	117(7)	30(1)	−76(7)	−18(3)	−15(2)
C(3)	102(6)	105(6)	26(1)	−2(5)	3(2)	−7(2)
C(4)	103(7)	210(9)	36(1)	9(6)	−3(2)	4(3)
C(5)	145(7)	127(7)	22(1)	−23(5)	13(2)	−1(2)
C(6)	261(11)	157(8)	22(1)	−48(8)	39(3)	−12(3)
C(7)	180(8)	126(7)	19(1)	46(6)	3(2)	−6(2)
C(8)	324(13)	153(8)	22(1)	64(8)	12(3)	12(2)

* In 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

Hydrogen atom positional and thermal parameters with their estimated standard deviations in parentheses

Atom	x/a	y/b	z/c	$B/\text{Å}^2$
H(1)	0·739(6)	−0·159(6)	0·382(2)	3·8(1·0)
H(2)	0·661(5)	−0·057(5)	0·312(2)	4·3(1·0)
H(3)	0·585(8)	−0·099(8)	0·381(3)	9·3(2·0)
H(4)	1·376(6)	0·374(6)	0·511(3)	7·2(1·4)
H(5)	1·400(6)	0·356(6)	0·434(2)	5·7(1·3)
H(6)	1·373(6)	0·154(7)	0·477(2)	6·6(1·4)
H(7)	0·800(8)	0·385(8)	0·674(3)	7·9(1·8)
H(8)	0·722(7)	0·217(6)	0·694(3)	6·6(1·4)
H(9)	0·643(9)	0·381(9)	0·652(3)	10·8(2·3)
H(10)	0·794(5)	0·580(5)	0·200(2)	3·5(1·0)
H(11)	0·856(7)	0·749(7)	0·252(3)	8·6(1·7)
H(12)	0·651(7)	0·709(7)	0·224(3)	6·6(1·5)

DISCUSSION

The most important feature of the molecule is the presence of two boron–oxygen tetrahedra, sharing a vertex; all the other vertices are connected to carbon atoms belonging to the four acetoxy-groups (Figure 1). In this way two acetoxy-groups are linked *via* their oxygen atoms both to B(1) and B(2), whereas each of the other two acetoxy-groups has only one oxygen bonded to a boron atom. The two tetrahedra are arranged (Figure 1) so as to have two edges parallel

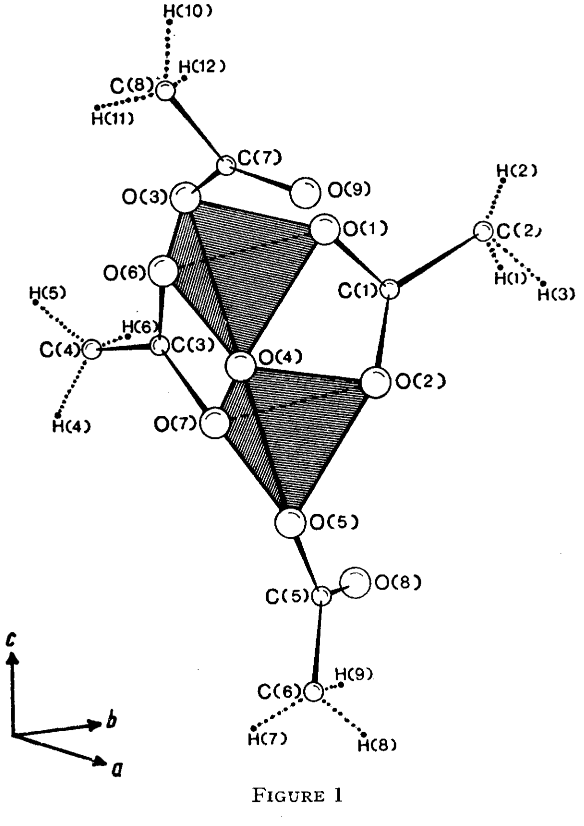

FIGURE 1

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* For details see Notice to Authors, No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20.

[10] H. P. Hanson, F. Herman, I. D. Lea, and S. Skillman, *Acta Cryst.*, 1964, **B17**, 1041.

[O(1)–O(6) and O(2)–O(7)] and two edges nearly aligned [O(3)–O(4) and O(4)–O(5)]; the angle formed by these last, O(3)–O(4)–O(5) (173·8°) is conditioned by the two bridging acetoxy-groups; in fact the O(1)–O(2) (2·235 Å) and O(6)–O(7) (2·231 Å) distances in the two bridging acetoxy-groups are arranged so that the two B–O tetrahedra can be approached with a B(1) · · · B(2) separation (2·265 Å) much shorter than those found in other borates. The abnormally short and long observed B–O bond lengths (Table 5) can be interpreted in terms of the principle of detailed valence neutralization, as suggested by Zachariasen,[11] who found in β-HBO_2 extreme values for tetrahedral B–O bond lengths of 1·433 and 1·553 Å, the mean value generally accepted being 1·47 Å.[3a,b,12,13]

TABLE 5

Bond lengths and angles concerning non-hydrogen atoms with estimated standard deviations in parentheses

(*a*) Distances (Å)

B(1)–O(1)	1·561(4)	C(1)–O(1)	1·269(4)
B(1)–O(3)	1·411(4)	C(1)–O(2)	1·267(4)
B(1)–O(4)	1·384(4)	C(1)–C(2)	1·480(5)
B(1)–O(6)	1·544(4)		
Mean	1·475	C(3)–O(6)	1·256(4)
		C(3)–O(7)	1·270(4)
B(2)–O(2)	1·551(4)	C(3)–C(4)	1·496(5)
B(2)–O(4)	1·384(4)		
B(2)–O(5)	1·422(4)	C(5)–O(5)	1·330(4)
B(2)–O(7)	1·543(4)	C(5)–O(8)	1·222(4)
		C(5)–C(6)	1·480(5)
Mean	1·475		
		C(7)–O(3)	1·340(4)
		C(7)–O(9)	1·206(4)
		C(7)–C(8)	1·496(5)

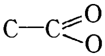
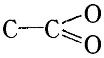
(*b*) Angles (°) *

O(1)–B(1)–O(3)	109·7	O(5)–C(5)–O(8)	123·5
O(1)–B(1)–O(4)	110·2	O(5)–C(5)–C(6)	112·3
O(1)–B(1)–O(6)	101·6	O(8)–C(5)–C(6)	124·2
O(3)–B(1)–O(4)	120·2	Mean	120·0
O(3)–B(1)–O(6)	103·4		
O(4)–B(1)–O(6)	110·0	O(3)–C(7)–O(9)	122·0
Mean	109·2	O(3)–C(7)–C(8)	111·7
		O(9)–C(7)–C(8)	126·3
O(2)–B(2)–O(4)	110·8	Mean	120·0
O(2)–B(2)–O(5)	110·0		
O(2)–B(2)–O(7)	103·5	B(1)–O(4)–B(2)	109·9
O(4)–B(2)–O(5)	112·3		
O(4)–B(2)–O(7)	111·1	C(1)–O(1)–B(1)	119·3
O(5)–B(2)–O(7)	108·8	C(1)–O(2)–B(2)	117·7
Mean	109·4	C(3)–O(6)–B(1)	118·8
		C(3)–O(7)–B(2)	118·0
O(1)–C(1)–O(2)	123·7	C(5)–O(5)–B(2)	125·4
O(1)–C(1)–C(2)	117·9	C(7)–O(3)–B(1)	122·6
O(2)–C(1)–C(2)	118·4		
Mean	120·0	O(1)–O(4)–O(2)	55·1
		O(6)–O(4)–O(7)	55·2
O(6)–C(3)–O(7)	124·1		
O(6)–C(3)–C(4)	118·3		
O(7)–C(3)–C(4)	117·6		
Mean	120·0		

* Estimated standard deviations are 0·3°.

Considering first the O(4) oxygen atom: its valence is neutralized by only two boron atoms and therefore the bond length is expected to be not too different from those occurring in B–O triangles. In fact the B(1)–O(4) and B(2)–O(4) distances (both 1·384 Å) agree with the B–O triangular distances (extreme values 1·280 and 1·433 Å,[14] mean 1·37 Å).

A little longer than this last value, but always shorter than the usual tetrahedral B–O bond lengths, are the B(1)–O(3) and B(2)–O(5) distances (1·411 and 1·422 Å respectively); the C(7)–O(3) and C(5)–O(5) bond lengths (1·340 and 1·330 Å respectively) are greater than the usual C–O single-bond distance in the ester grouping (1·315 Å),[15] which can be explained in terms of decreased π contribution and there being nearly full location of the double bond on the carbonyl groups. The O(3) and O(5) atoms therefore, having something less than one negative valence to be neutralized by boron atoms, will give a B–O bond length not very different from triangular B–O distances.

In the same way we can explain the long distances [B(1)–O(1) 1·561, B(1)–O(6) 1·544, B(2)–O(2) 1·551, and B(2)–O(7) 1·543 Å] concerning the oxygen atoms of the two acetoxy-groups, in which the two resonance forms  $\mathrm{C{-}C}\genfrac{}{}{0pt}{}{\mathrm{=O}}{\mathrm{-O}}$ and  $\mathrm{C{-}C}\genfrac{}{}{0pt}{}{\mathrm{-O}}{\mathrm{=O}}$ seem to have nearly equal contributions. In fact the four C–O bonds involved do not differ too much from each other [1·256(4)—1·270(4) Å]. According to this only about half a negative valence of each of these oxygen atoms remains to be neutralized by boron atoms.

In conclusion even if the mean B–O bond length for the two tetrahedra is 1·475 Å, the remarkable decrease or increase in this value may be very well explained by considering how much the negative valence of the oxygen atoms, which boron has to neutralize, differs from the expected value of 0·75. According to Zachariasen's[11] correlations between length and strength of the boron–oxygen bond the valence balance occurring for the two boron atoms is shown in Table 6 and seems to confirm the foregoing correlations.

TABLE 6

Bond	Distance/Å	Bond strength
B(1)–O(4)	1·384	0·957
B(1)–O(3)	1·411	0·895
B(1)–O(6)	1·544	0·593
B(1)–O(1)	1·561	0·555
Mean	1·475	Σ 3·000
Bond	Distance/Å	Bond strength
B(2)–O(4)	1·384	0·957
B(2)–O(5)	1·422	0·871
B(2)–O(7)	1·543	0·595
B(2)–O(2)	1·551	0·577
Mean	1·475	Σ 3·000

As far as the acetoxy-groups are concerned, the two carbonyl bonds C(7)–O(9) (1·206) and C(5)–O(8) (1·222 Å; 1·215 and 1·245 Å are the corresponding values generally

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[11] W. H. Zachariasen, *Acta Cryst.*, 1963, **A16**, 385.

[12] J. A. Konnert, J. R. Clark, and C. L. Christ, *Amer. Minerol.*, 1970, **55**, 1911.

[13] J. A. Konnert, J. R. Clark, and C. L. Christ, *Amer. Minerol.*, 1971, in the press.

[14] H. Marezio, H. A. Plettinger, and W. H. Zachariasen, *Acta Cryst.*, 1963, **B16**, 594.

[15] N. C. Baird, M. J. S. Dewar, and R. Sustman, *J. Chem. Phys.*, 1969, **50**, 1275.

accepted for ketones[16] and esters[15] respectively) have to be considered as nearly pure $C{=}O$ double bonds and the decreased contribution to resonance has already been pointed out.

The difference between these two carbonyl bond lengths in (I) can be rationalized as being due to shorter intermolecular contacts concerning O(8) in respect of O(9). The C–C bond lengths in all the acetoxy-groups are in the range of the values found for compounds of the $C{-}C{=}O$ type. Where the C–C distances are shorter (depending on the resonance in the $C{-}CO_2$ group) the corresponding oxygen atoms involved have longer B–O bonds and *vice versa*.

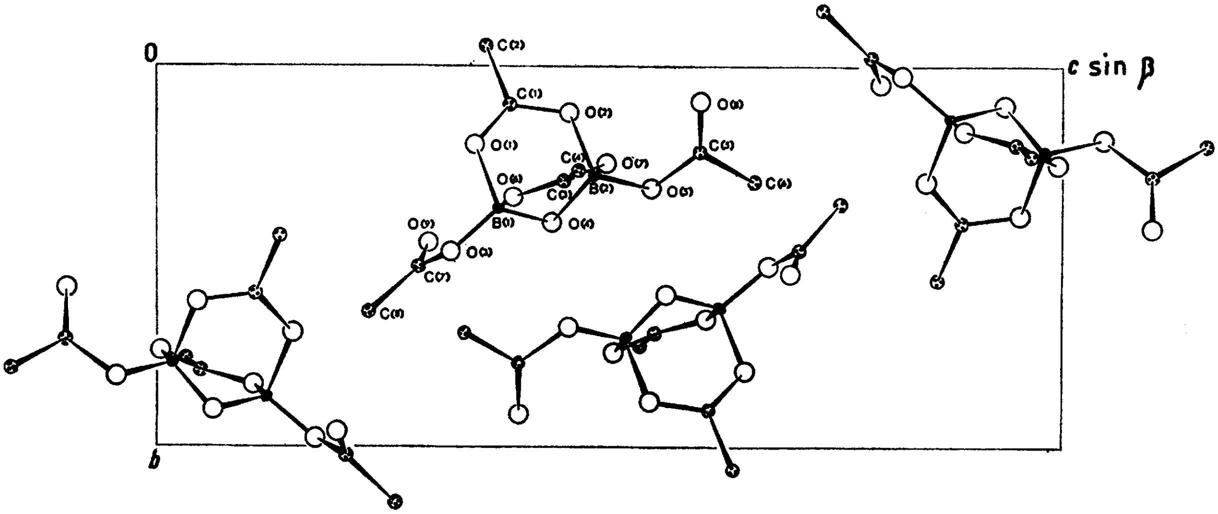

FIGURE 2

TABLE 7

Geometry of C–H bonds

(*a*) Distances (±0·06 Å)

C(2)–H(1)	0·99	C(6)–H(7)	0·87
C(2)–H(2)	1·02	C(6)–H(8)	0·98
C(2)–H(3)	0·95	C(6)–H(9)	1·00
C(4)–H(4)	1·18	C(8)–H(10)	0·96
C(4)–H(5)	1·02	C(8)–H(11)	1·19
C(4)–H(6)	1·04	C(8)–H(12)	0·92
		Mean C–H	1·01

(*b*) Angles (°)

H(1)–C(2)–H(2)	114(4)	H(7)–C(6)–H(8)	116(5)
H(1)–C(2)–H(3)	81(4)	H(7)–C(6)–H(9)	81(5)
C(1)–C(2)–H(1)	116(3)	H(8)–C(6)–H(9)	116(5)
C(1)–C(2)–H(2)	115(3)	C(5)–C(6)–H(7)	114(4)
C(1)–C(2)–H(3)	119(3)	C(5)–C(6)–H(8)	110(4)
Mean	109	C(5)–C(6)–H(9)	118(4)
		Mean	109
H(4)–C(4)–H(5)	91(4)		
H(4)–C(4)–H(6)	120(4)	H(10)–C(8)–H(11)	109(4)
H(5)–C(4)–H(6)	132(4)	H(10)–C(8)–H(12)	118(4)
C(3)–C(4)–H(4)	101(3)	H(11)–C(8)–H(12)	99(4)
C(3)–C(4)–H(5)	104(3)	C(7)–C(8)–H(10)	103(3)
C(3)–C(4)–H(6)	104(3)	C(7)–C(8)–H(11)	114(3)
Mean	109	C(7)–C(8)–H(12)	113(3)
		Mean	109

The four acetoxy-groups are planar, the mean angles around the central-carbon atoms being 120·0°.

Interatomic distances and bond angles involving hydrogen atoms are reported in Table 7: the relatively poor values for C–H bond lengths and the high temperature factors for the hydrogen atoms indicate that the methyl groups are undergoing appreciable torsional oscillation in the crystal at room temperature.

Molecular Packing.—The crystal structure (Figure 2) is characterized by the presence of isolated molecules, between which there exist no strong intermolecular contacts, as shown by the considerable instability of the crystals and the great thermal motion of the atoms involved. Intermolecular contacts are listed in Table 8

TABLE 8

Van der Waals contacts (Å) with their standard deviations in parentheses

$O(9) \cdots H(9^{I})$	2·43(7)	$C(7) \cdots H(8^{IX})$	2·73(6)
$O(8) \cdots H(10^{II})$	2·52(5)	$C(6) \cdots H(3^{X})$	2·99(6)
$O(4) \cdots H(4^{III})$	2·61(5)	$C(8) \cdots H(2^{XI})$	3·00(4)
$O(3) \cdots H(7^{IV})$	2·65(6)	$C(8) \cdots H(8^{IX})$	3·09(5)
$O(9) \cdots H(2^{VI})$	2·74(4)	$C(2) \cdots H(11^{XII})$	3·11(6)
$O(9) \cdots H(12^{VII})$	2·74(5)	$C(5) \cdots H(3^{X})$	3·16(6)
$O(6) \cdots H(7^{IV})$	2·80(6)	$C(5) \cdots H(10^{XIII})$	3·18(4)
$O(8) \cdots C(1^{V})$	3·080(4)	$C(2) \cdots C(8^{XII})$	3·60(6)
$O(8) \cdots C(3^{XIV})$	3·247(4)		
$O(5) \cdots C(3^{III})$	3·283(5)	$H(8) \cdots H(10^{XIII})$	2·51(7)
$O(9) \cdots C(6^{I})$	3·304(5)	$H(2) \cdots H(11^{XII})$	2·54(7)
$O(6) \cdots C(8^{VIII})$	3·324(5)	$H(2) \cdots H(12^{XII})$	2·59(7)
$O(8) \cdots C(2^{V})$	3·348(6)		
$O(3) \cdots C(6^{IV})$	3·378(6)		

Roman numerals as superscripts refer to the following equivalent positions relative to the reference molecule at x, y, z:

I	$-x, 1-y, 1-z$	VIII	$1-x, -\frac{1}{2}+y, \frac{1}{2}-z$
II	$x, \frac{1}{2}-y, \frac{1}{2}+z$	IX	$x-1, \frac{1}{2}-y, -\frac{1}{2}+z$
III	$-x+1, 1-y, 1-z$	X	$-x, -y, 1-z$
IV	$1-x, 1-y, 1-z$	XI	$x-1, 1+y, z$
V	$1-x, -y, 1-z$	XII	$x-1, y-1, z$
VI	$-x, \frac{1}{2}+y, \frac{1}{2}-z$	XIII	$x-1, \frac{1}{2}-y, \frac{1}{2}+z$
VII	$-x, -\frac{1}{2}+y, \frac{1}{2}-z$	XIV	$-x+1, -y, 1-z$

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[16] L. E. Sutton, *Chem. Soc. Special Publ.*, No. 18, 1965.

within a limit of 0·2 Å greater than the sum of the relevant Van der Waals radii, for which the R_C 1·8, R_O 1·4, and R_H 1·2 Å values have been used:[17] only four of these are significantly shorter than the sum of the van der Waals radii of the atoms concerned.

[2/145 *Received, January 24th,* 1972]

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[17] A. I. Kitaigorodskii, ' Organic Chemical Crystallography,' (English translation), 1961, Consultants Bureau, New York, p. 7.