where x, y, and z are fractional coordinates and the coefficients are in Å. This plane is nearly parallel to the $(0\bar{1}3)$ crystallographic plane. The spacing between the molecular planes related by the inversion at 0,0,0 is 3·176 Å and that related by the inversion at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$ is 3·684 Å. However, there are only a few intermolecular contacts less than 3·55 Å. These are listed in Table 3.

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References

BOWDEN, F. L. & LEVER, A. B. P. (1968). *Organomet. Chem. Rev.* **3**, 227–279.
CASH, G. G. & PETTERSEN, R. C. (1978). *Inorg. Chem.* **17**, 650–653.
HÜBEL, W. (1968). *Organic Synthesis via Metal Carbonyls*, edited by I. WENDER. New York: Interscience.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
MAITLIS, P. M. & MCVEY, S. (1965). *J. Organomet. Chem.* **4**, 254–255.
MARKBY, R., STERNBERG, H. W. & WENDER, I. (1959). *Chem. Ind. (London)*, pp. 1381–1382.
SHELDRICK, G. M. (1976). *SHELX 76: Program for Crystal Structure Determination*. Univ. of Cambridge, England.
STERNBERG, H. W., MARKBY, R. & WENDER, I. (1958). *J. Am. Chem. Soc.* **80**, 1009–1010.
VICTOR, R., BEN-SHOSHAN, R. & SAREL, S. (1973). *Tetrahedron Lett.* pp. 4211–4214.
VOLLHARDT, K. P. C. (1977). *Acc. Chem. Res.* **10**, 1–8.
YUR'EVA, L. P. (1974). *Usp. Khim.* **43**, 95–133.

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Structure of a Photo-addition Product of an α,β-Unsaturated Ketone and Allene, $C_{14}H_{22}O$†

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$C_{14}H_{22}O$ is triclinic, $P\bar{1}$, $a = 13{\cdot}173$ (5), $b = 13{\cdot}460$ (5), $c = 13{\cdot}038$ (6) Å, $\alpha = 104{\cdot}76$ (6), $\beta = 105{\cdot}33$ (4), $\gamma = 113{\cdot}59$ (5)°, $Z = 6$. The structure has been refined to $R = 0{\cdot}060$ and $R_w = 0{\cdot}061$ for the 4591 observed reflexions. The molecule contains a central six-membered ring which is *cis*-fused to a four-membered ring on one side and to a six-membered ring on the other. The central ring is a distorted chair, with substituents OH equatorial and CH_3 axial, and the other six-membered ring is a regular chair. The valency and torsion angles of the central ring appear to be susceptible to small but significant changes depending on the position of OH. The three independent molecules of the unit cell and their centrosymmetric equivalents are linked by O—H···O hydrogen bonds into hexamers. No links are present between the molecules of different hexamers.

Introduction

In order to study the geometry and conformational preferences of the $n.\pi^*$ excited states of α,β-unsaturated ketones, Wiesner (1977) and his collaborators prepared a number of photochemical addition compounds. Through addition of allene to the α,β-unsaturated ketone (I), the adduct (II) was produced, and this was subsequently reduced by sodium borohydride to compound (III) whose structure is reported here. The crystals were obtained from a solution of methanol and H_2O as colourless prisms which lose diffracting power fairly fast and turn opaque upon exposure to X-rays. This necessitated the use of four crystals for the collection of the full data set. A comparison of the intensities of all the reflexions within one octant of reciprocal space, measured with a fresh crystal and again with another after it had lost 30% of its diffracting power, showed that the intensity drop was systematic for all reflexions. The crystal system was determined from precession photographs as triclinic with $Z = 6$, and a careful examination of the interplanar spacings and intensity distribution of the reflexions within $2\theta = 20°$ did not reveal a higher symmetry. Also, the E distribution statistics were consistent with those of a centrosymmetric structure, and the analysis was therefore carried out in space group $P\bar{1}$ with three independent molecules in the asymmetric unit.

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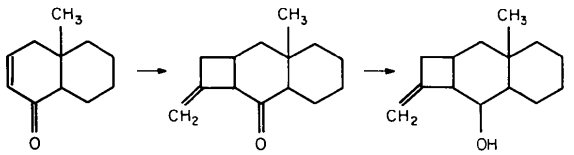

(I) (II) (III)

Experimental

Crystal data

$C_{14}H_{22}O$, $M_r = 206{\cdot}33$, m.p. 82–83 °C, triclinic, $P\bar{1}$, $a = 13{\cdot}173$ (5), $b = 13{\cdot}460$ (5), $c = 13{\cdot}038$ (6) Å, $\alpha = 104{\cdot}76$ (6), $\beta = 105{\cdot}33$ (4), $\gamma = 113{\cdot}59$ (5)°, $V = 1865{\cdot}4$ Å^3, $Z = 6$, $D_c = 1{\cdot}102$, $D_m = 1{\cdot}098$ g cm^{-3}, $F(000) = 684$, Cu $K\alpha$ [$\lambda(\alpha_1) = 1{\cdot}54050$, $\lambda(\alpha_2) = 1{\cdot}54434$ Å], μ(Cu $K\alpha$) = 4·78 cm^{-1}. The cell parameters were derived from the goniostat settings of six reflexions (2θ = 40–100°) and their equivalents, measured at 2θ and -2θ at a small take-off angle. The density was measured by flotation in aqueous KI solution at 22 °C.

Intensity data

Integrated intensities were collected on a Picker diffractometer equipped with a scintillation counter and a card reader/punch, by the θ–2θ scan method at a 4° take-off angle. The four crystals used for this purpose were cut into approximate spheres 0·2–0·3 mm in diameter. The 2θ scan range was 2·0° for $2\theta < 80°$, 2·4° for $80 \leq 2\theta < 100°$, and 3·0° for $2\theta \geq 100°$. In view of the fast crystal deterioration, the background of each reflexion was measured only once for 10 s at the high 2θ limit of each scan. Two reflexions of medium intensity were examined at regular intervals for scaling purposes. The net intensities, after scaling, were corrected for Lorentz and polarization effects, but not for absorption. Of the 6272 independent reflexions in the 2θ range 0–130°, 4591 (73%) had net counts significantly higher than background, and were considered observed.

Structure determination

The structure was determined by direct methods (Karle & Karle, 1963). The starting phases consisted of three for origin definition, two assumed with + and − signs and permuted manually, and two symbolic phases chosen by the program. These seven reflexions were among the 10 with the highest $|E|$'s so that the strongest $\sum_2$ triples were utilized in the very early stages of the procedure. These four trials were carried out on the 450 reflexions with $|E| \geq 1{\cdot}75$. The solution with all signs positive was rejected, and the next most consistent solution was extended to phase 902 of the 950 reflexions with $|E| \geq 1{\cdot}40$. The corresponding E map showed only 45 significant peaks corresponding to all C and O atoms of the three independent molecules. For this trial structure, assuming $B = 4{\cdot}0$ Å^2 for all atoms, R was 0·28. A difference map calculated after partial refinement, at $R = 0{\cdot}14$, produced the 66 H atoms.

Refinement was by the block-diagonal least-squares method, one block per atom, assuming anisotropic vibration for C and O and isotropic for H. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\{1 + [(|F_o| - 15)/30]^2\}$, and $|F_o| = 1{\cdot}7$–$171{\cdot}7$. The unobserved reflexions, as well as two very strong ($1\bar{2}0$ and 112) and one weak ($2\bar{4}2$) reflexions with high discrepancies were assigned zero weights. In the final cycle, $R = 0{\cdot}060$ and $R_w = 0{\cdot}061$ for the 4591 observed reflexions, $[\sum w\Delta^2/(m-n)]^{1/2} = 0{\cdot}72$, and the mean and maximum $\Delta(p)/\sigma(p) = 0{\cdot}16$ and 0·72 respectively. Only two of the unobserved reflexions were calculated higher than twice their threshold amplitudes. The residual electron density in the final difference map was within −0·20 and 0·23 e Å^{-3}. The atomic parameters are listed in Table 1.*

The scattering-factor curves were those of Hanson, Herman, Lea & Skillman (1964) for C and O, and of Stewart, Davidson & Simpson (1965) for H. All calculations were performed with the NRC system of crystallographic computer programs (Ahmed, Hall, Pippy & Huber, 1973).

Discussion

The three independent molecules have the conformation shown in Fig. 1. The four-membered ring, A, is

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33862 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

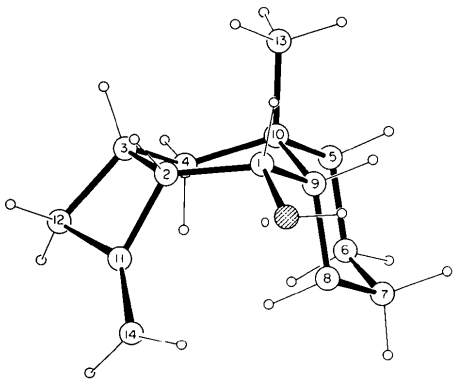

Fig. 1. A view of the molecular structure showing its conformation.

Table 1. *Positional parameters* ($\times 10^4$ *for* C *and* O; $\times 10^3$ *for* H) *of the three independent molecules*

	Molecule (I)			Molecule (II)			Molecule (III)		
	x	*y*	*z*	*x*	*y*	*z*	*x*	*y*	*z*
C(1)	994 (2)	3933 (2)	1489 (2)	1133 (2)	3766 (2)	8458 (2)	2239 (2)	8769 (2)	1551 (2)
C(2)	870 (2)	2714 (2)	1162 (2)	2411 (2)	4837 (2)	9031 (2)	2387 (2)	9492 (2)	2730 (2)
C(3)	1985 (3)	2601 (3)	1756 (3)	3460 (3)	4593 (3)	8968 (3)	3502 (3)	10781 (2)	3433 (3)
C(4)	2768 (3)	3532 (3)	3006 (3)	3076 (3)	3589 (3)	7845 (3)	4615 (2)	10851 (2)	3226 (2)
C(5)	3821 (3)	5664 (3)	4456 (3)	1529 (4)	1535 (3)	6291 (4)	5566 (3)	10434 (3)	1868 (3)
C(6)	3198 (3)	5467 (3)	5264 (3)	1257 (4)	1845 (3)	5269 (3)	6038 (3)	9716 (3)	2338 (3)
C(7)	2022 (3)	5483 (3)	4851 (3)	270 (4)	2167 (3)	5154 (3)	5076 (3)	8427 (3)	1772 (3)
C(8)	1192 (3)	4588 (3)	3595 (2)	591 (3)	3166 (3)	6277 (3)	3925 (3)	8248 (2)	1924 (3)
C(9)	1818 (2)	4791 (2)	2775 (2)	849 (2)	2835 (2)	7310 (2)	3417 (2)	8954 (2)	1437 (2)
C(10)	3029 (2)	4790 (2)	3162 (2)	1842 (3)	2478 (2)	7451 (3)	4371 (2)	10299 (2)	1960 (2)
C(11)	128 (3)	1760 (2)	1495 (3)	2924 (2)	5794 (2)	8595 (3)	2767 (2)	9278 (3)	3826 (2)
C(12)	1080 (3)	1382 (3)	1693 (3)	4129 (3)	5818 (3)	8935 (3)	3519 (3)	10601 (3)	4567 (3)
C(13)	3737 (3)	5220 (3)	2452 (3)	1910 (4)	1927 (3)	8346 (4)	3918 (3)	10930 (3)	1282 (3)
C(14)	−873 (3)	1443 (3)	1658 (3)	2502 (3)	6339 (3)	8070 (3)	2596 (3)	8338 (3)	4041 (3)
O(15)	−210 (1)	3756 (2)	1134 (1)	260 (2)	4149 (2)	8356 (2)	1408 (2)	7539 (2)	1225 (2)
H(1)	136 (2)	431 (2)	98 (2)	112 (2)	339 (2)	904 (2)	188 (2)	908 (2)	104 (2)
H(2)	61 (2)	231 (2)	28 (2)	261 (3)	528 (3)	990 (2)	158 (2)	952 (2)	263 (2)
H(3)	254 (2)	264 (2)	131 (2)	390 (3)	448 (3)	966 (3)	340 (2)	1146 (2)	335 (2)
H(4,1)	236 (2)	328 (2)	353 (2)	300 (3)	390 (3)	716 (2)	493 (2)	1042 (2)	365 (2)
H(4,2)	349 (2)	356 (2)	327 (2)	375 (2)	341 (2)	797 (2)	529 (2)	1175 (2)	361 (2)
H(5,1)	457 (3)	559 (3)	463 (3)	81 (3)	76 (3)	615 (3)	543 (3)	1023 (3)	102 (3)
H(5,2)	406 (3)	649 (3)	451 (3)	226 (3)	133 (3)	646 (3)	621 (3)	1130 (3)	228 (3)
H(6,1)	375 (3)	614 (3)	612 (3)	100 (3)	119 (3)	450 (3)	630 (3)	1000 (3)	323 (3)
H(6,2)	298 (3)	468 (3)	530 (3)	203 (3)	256 (3)	542 (3)	674 (3)	974 (3)	218 (3)
H(7,1)	160 (3)	529 (3)	536 (3)	12 (3)	236 (3)	453 (3)	483 (3)	802 (3)	95 (3)
H(7,2)	225 (3)	637 (3)	496 (3)	−53 (3)	144 (3)	495 (3)	533 (3)	788 (3)	208 (3)
H(8,1)	94 (2)	374 (2)	353 (2)	−5 (2)	345 (2)	627 (2)	412 (2)	851 (2)	282 (2)
H(8,2)	43 (3)	461 (3)	334 (3)	131 (2)	392 (2)	642 (2)	330 (2)	739 (2)	153 (2)
H(9)	198 (2)	559 (2)	279 (2)	11 (2)	212 (2)	714 (2)	328 (2)	863 (2)	62 (2)
H(12,1)	130 (3)	116 (3)	243 (3)	469 (3)	644 (3)	975 (3)	301 (3)	1089 (3)	484 (3)
H(12,2)	84 (3)	75 (3)	102 (3)	454 (3)	575 (3)	834 (3)	433 (3)	1097 (3)	527 (3)
H(13,1)	329 (3)	457 (3)	158 (3)	219 (3)	255 (3)	921 (3)	371 (3)	1050 (3)	41 (3)
H(13,2)	384 (3)	605 (3)	250 (3)	107 (4)	128 (4)	817 (3)	313 (3)	1085 (2)	129 (2)
H(13,3)	458 (3)	535 (3)	280 (3)	244 (3)	159 (3)	836 (3)	450 (3)	1171 (3)	151 (3)
H(14,1)	−109 (3)	79 (3)	190 (3)	307 (3)	701 (3)	799 (3)	206 (3)	752 (3)	337 (3)
H(14,2)	−137 (3)	188 (3)	152 (3)	165 (3)	619 (3)	789 (3)	296 (3)	845 (3)	487 (3)
H(15)	−21 (3)	444 (3)	127 (3)	−33 (3)	364 (3)	835 (3)	113 (3)	710 (3)	44 (3)

non-planar, the central six-membered ring, *B*, is a distorted chair, and the terminal six-membered ring, *C*, is a regular chair, with each of the terminal rings *cis*-fused to the central ring. The two substituents on *B* are OH equatorial and CH_3 axial.

Bond lengths, valency and torsion angles of the three independent molecules, without thermal vibration correction and excluding those involving H, are listed in Tables 2, 3, and 4. The corresponding mean values are presented in Fig. 2. χ^2 tests on the equivalent quantities of the three molecules show that the discrepancies are not significant for 15 of the 17 bond lengths, for 21 of the 27 valency angles, nor for 12 of 16 torsion angles within the rings. The maximum deviations from the quoted mean values are 0·016 Å in the bond lengths and 1·7° in the valency angles. The corresponding maximum discrepancies in the torsion angles are 2·9° in ring *B*, 1·1° in rings *A* and *C*, and up to 4·6° in those involving the OH substituent. The quantities with high discrepancies, $\chi^2 > 9{\cdot}2$ and $P < 0{\cdot}01$, are

Table 2. *Bond lengths* (Å) *for the three independent molecules, and their* χ^2 *values*

	(I)	(II)	(III)	χ^2
C(1)–C(2)	1·514 (5)	1·521 (5)	1·508 (4)	4·2
C(1)–C(9)	1·540 (4)	1·525 (4)	1·524 (5)	9·3*
C(1)–O(15)	1·428 (4)	1·426 (4)	1·438 (4)	5·2
C(2)–C(3)	1·555 (5)	1·559 (5)	1·564 (5)	1·6
C(2)–C(11)	1·516 (5)	1·523 (5)	1·524 (4)	1·7
C(3)–C(4)	1·525 (5)	1·510 (5)	1·530 (5)	8·7
C(3)–C(12)	1·560 (5)	1·546 (5)	1·553 (5)	3·9
C(4)–C(10)	1·533 (5)	1·538 (5)	1·512 (4)	21·0*
C(5)–C(6)	1·509 (6)	1·492 (6)	1·510 (6)	5·7
C(5)–C(10)	1·548 (4)	1·530 (6)	1·550 (6)	7·6
C(6)–C(7)	1·511 (7)	1·511 (8)	1·501 (6)	1·7
C(7)–C(8)	1·524 (4)	1·531 (5)	1·513 (6)	5·4
C(8)–C(9)	1·527 (5)	1·520 (4)	1·531 (5)	3·3
C(9)–C(10)	1·542 (5)	1·549 (5)	1·556 (5)	3·9
C(10)–C(13)	1·536 (5)	1·536 (6)	1·540 (5)	0·4
C(11)–C(12)	1·519 (6)	1·516 (5)	1·513 (5)	0·6
C(11)–C(14)	1·308 (6)	1·303 (5)	1·310 (5)	1·0

* Significant discrepancies.

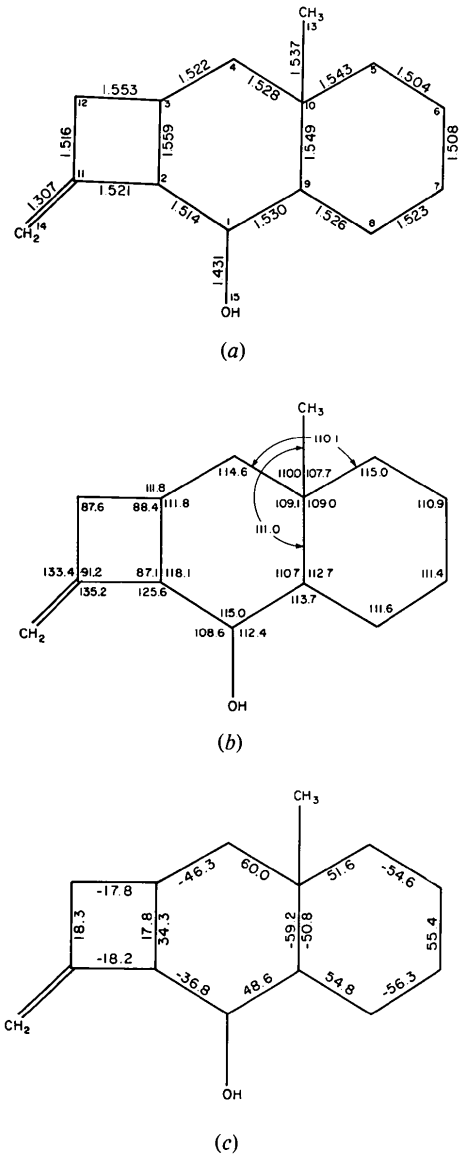

Fig. 2. Schematic drawings of the molecule showing the mean values of the bond lengths (Å), valency angles (°), and torsion angles (°) of the three independent molecules. Their e.s.d.'s are 0·003–0·004 Å for the bond lengths, 0·2° for the valency angles, and 0·4° for the torsion angles.

identified by an asterisk in Tables 2–4. These discrepancies appear to be attributable to slightly different positions of the OH groups as a result of their involvement in intermolecular hydrogen bonding, as will be discussed later. They also indicate that the central ring has a certain degree of flexibility shown by small variations in its valency and torsion angles.

The C—H lengths are in the range 0·91–1·08, mean = 1·014, and O—H range from 0·81 to 0·93, mean = 0·87 Å.

Table 3. *Valency angles (°) for the three independent molecules, and their χ^2 values*

The estimated standard deviations are 0·2–0·4°.

	(I)	(II)	(III)	χ^2
C(2)–C(1)–C(9)	114·8	114·6	115·5	5·0
C(2)–C(1)–O(15)	106·9	110·0	108·8	54·3*
C(9)–C(1)–O(15)	113·1	112·4	111·8	16·2*
C(1)–C(2)–C(3)	119·0	116·9	118·5	26·8*
C(1)–C(2)–C(11)	124·7	127·0	125·2	32·6*
C(3)–C(2)–C(11)	87·7	86·8	86·9	7·2
C(2)–C(3)–C(4)	111·9	113·0	110·6	32·1*
C(2)–C(3)–C(12)	88·2	88·8	88·1	3·2
C(4)–C(3)–C(12)	112·2	111·7	111·5	2·9
C(3)–C(4)–C(10)	114·4	115·0	114·5	2·3
C(6)–C(5)–C(10)	114·8	114·9	115·3	1·5
C(5)–C(6)–C(7)	110·9	111·2	110·5	2·3
C(6)–C(7)–C(8)	111·1	111·6	111·5	1·4
C(7)–C(8)–C(9)	112·0	110·9	111·9	8·2
C(1)–C(9)–C(8)	113·6	114·3	113·3	5·9
C(1)–C(9)–C(10)	110·6	110·6	110·9	0·8
C(8)–C(9)–C(10)	112·7	112·7	112·6	0·1
C(4)–C(10)–C(5)	110·4	110·5	109·4	8·2
C(4)–C(10)–C(9)	109·3	108·8	109·1	1·4
C(4)–C(10)–C(13)	110·0	109·8	110·3	1·4
C(5)–C(10)–C(9)	108·6	109·6	108·7	6·8
C(5)–C(10)–C(13)	107·7	106·9	108·4	12·5*
C(9)–C(10)–C(13)	110·9	111·3	110·9	1·2
C(2)–C(11)–C(12)	91·2	91·3	91·0	0·6
C(2)–C(11)–C(14)	135·3	135·6	134·8	3·7
C(12)–C(11)–C(14)	133·2	133·0	133·9	4·8
C(3)–C(12)–C(11)	87·5	87·5	87·7	0·3

* Significant discrepancies.

Table 4. *Torsion angles (°) for the three independent molecules, and their χ^2 values*

Assumed e.s.d. is 0·7°, based on the actual distribution of these angles in the fairly rigid four-membered ring.

	(I)	(II)	(III)	χ^2
Ring *B*				
C(1)–C(2)–C(3)–C(4)	32·9	34·8	−35·1	5·8
C(2)–C(3)–C(4)–C(10)	−45·5	−45·0	48·4	13·8*
C(3)–C(4)–C(10)–C(9)	60·3	58·1	−61·5	12·1*
C(4)–C(10)–C(9)–C(1)	−59·5	−59·6	58·4	1·8
C(10)–C(9)–C(1)–C(2)	47·8	51·5	−46·6	26·6*
C(9)–C(1)–C(2)–C(3)	−35·2	−39·1	36·1	17·0*
Ring *C*				
C(5)–C(6)–C(7)–C(8)	54·9	55·0	−56·2	2·1
C(6)–C(7)–C(8)–C(9)	−55·8	−55·9	57·3	2·9
C(7)–C(8)–C(9)–C(10)	55·2	54·6	−54·6	0·5
C(8)–C(9)–C(10)–C(5)	−51·5	−51·2	49·7	3·8
C(9)–C(10)–C(5)–C(6)	52·4	51·4	−50·9	2·4
C(10)–C(5)–C(6)–C(7)	−55·0	−54·1	54·8	0·9
Ring *A*				
C(2)–C(3)–C(12)–C(11)	−17·2	−17·7	18·5	1·8
C(3)–C(12)–C(11)–C(2)	17·7	18·1	−19·0	1·8
C(12)–C(11)–C(2)–C(3)	−17·7	−18·0	18·9	1·6
C(11)–C(2)–C(3)–C(12)	17·3	17·6	−18·4	1·3
OH substituent				
C(8)–C(9)–C(1)–O(15)	42·8	49·5	−44·2	51·0*
C(11)–C(2)–C(1)–O(15)	−52·3	−59·3	54·8	51·4*
C(10)–C(9)–C(1)–O(15)	170·7	178·1	−171·8	65·1*
C(3)–C(2)–C(1)–O(15)	−161·4	−166·9	162·8	33·3*

*Significant discrepancies.

Table 5. *Deviations* ($\times 10^3$ Å) *from the mean planes of the four atoms marked with* *, *for the three molecules*

	(I)	(II)	(II)
Ring *A*			
C(2)*	118	−119	−125
C(3)*	−114	117	122
C(12)*	117	−121	−127
C(11)*	−121	123	130
Ring *B*			
C(1)*	7	−21	3
C(2)	−400	443	−420
C(3)*	−8	21	3
C(4)*	8	−22	−4
C(10)	711	−709	710
C(9)*	−8	22	4
Ring *C*			
C(5)*	1	−5	−1
C(6)*	−1	5	1
C(7)	−656	657	661
C(8)*	1	−5	−1
C(9)*	−1	5	1
C(10)	649	−633	−636

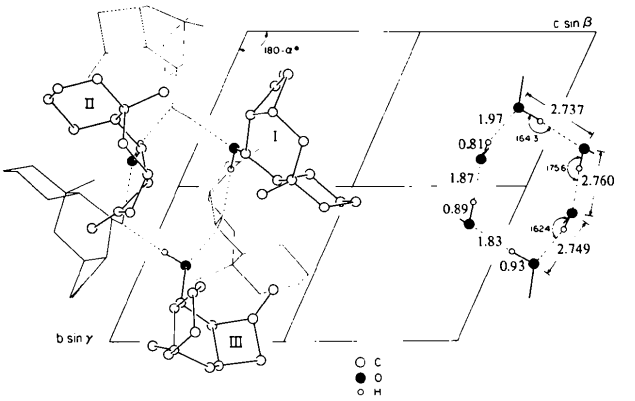

Fig. 3. A projection of the cell contents along the *a* axis, showing one of the hexamers on the left hand side, and the dimensions of the hydrogen bonds on the right. For clarity, only the O and H atoms of the hydrogen bonds are drawn on the right half of the unit cell.

Mean planes

For each of the three molecules, the four most coplanar atoms of ring *B* are C(1), C(3), C(4) and C(9), and those of ring *C* are C(5), C(6), C(8) and C(9). The deviations of these and the other atoms from the corresponding mean planes are listed for the three molecules in Table 5. In each case, the four atoms are coplanar except in ring *B* of molecule (II). While C(7) and C(10) are equidistant from the seat of ring *C*, there is a substantial difference between the deviations of C(2) and C(10) from the seat of ring *B*. In each molecule, C(2) is approximately 0·3 Å closer to the seat than C(10).

The dihedral angles $\angle AB$, $\angle BC$ and $\angle AC$ are 58·9, 62·2 and 74·8° in molecule (I), 59·4, 62·9 and 73·6° in (II), and 59·6, 61·3 and 74·6° in (III).

Hydrogen bonding

In this structure, the three independent molecules and their centrosymmetric equivalents are interlinked by O—H···O hydrogen bonds to form a hexamer, as shown on the left hand side of Fig. 3. Thus, each O acts both as donor and acceptor of a H atom. The dimensions of these bonds, presented on the right hand side of Fig. 3, are not identical for the three independent bonds. The hydrogen-bonding network is achieved by forcing the OH substituents into slightly different positions relative to their respective molecules, as indicated by the torsion angles involving OH. There are no other intermolecular distances shorter than van der Waals contacts between the molecules of adjacent hexamers.

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References

AHMED, F. R., HALL, S. R., PIPPY, M. E. & HUBER, C. P. (1973). NRC Crystallographic Programs for the IBM 360 System. Accession Nos. 133–147, *J. Appl. Cryst.* **6**, 309–346.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). *Acta Cryst.* **17**, 1040–1044.

KARLE, I. L. & KARLE, J. (1963). *Acta Cryst.* **16**, 969–975.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

WIESNER, K. (1977). Private communication.