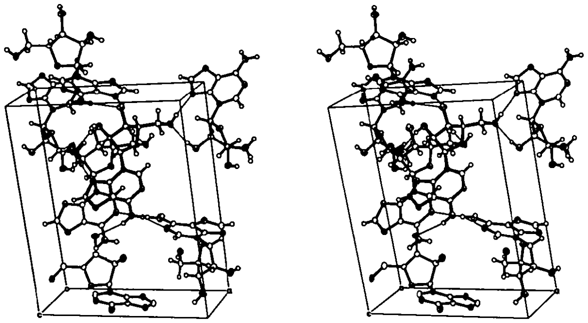

Fig. 2. Stereoscopic view of the unit cell with hydrogen bonding.

The adenine ring of the molecule is nearly planar (Fig. 1), but is slightly folded about the C(4)–C(5) vector with an interplanar angle of 2·5°. The torsion-angle values of N(1)–C(6)–N(6)–HN(6)*A* and N(1)–C(6)–N(6)–HN(6)*B*, 1 (3) and 171 (3)°, respectively, are indicative of the approximate coplanarity of the two amino H atoms at N(6) with the bicyclic ring. As in adenosine, the furanose ring is puckered with a C(3′) *endo* conformation.

A stereoview of the crystal packing arrangement is shown in Fig. 2. As with adenosine, all N and O atoms except N(3) and O(4′) [O(1′)] participate in a three-dimensional hydrogen-bonding scheme. In the crystal structure of adenosine the 6-amino group donates a hydrogen bond to both the N(7) and O(5′) atoms of a neighboring adenosine molecule. In tricanthine and 3-ethyladenine, this same amino group participates in a hydrogen-bonding scheme with two neighboring molecules. In tricanthine the N(1) atom of one neighbor and the N(7) atom of the other participate in this scheme, whereas in 3-ethyladenine it is the N(7) atom of one neighbor and the N(9) atom of the second neighbor that participate. 3-Isoadenosine represents yet another variation in the hydrogen-bonding pattern. The 6-amino-group H bonds with the N(1) atom of one neighbor and the O(3′) atom of the second neighboring molecule. The hydrogen-bond-related lengths and angles are provided in Table 2.

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Acta Cryst. (1988). C**44**, 510–512

Structure of (4*SR*,6a*RS*)-4-[2-(2-Methyl-1,3-dioxolan-2-yl)ethyl]-4,5,6,6a-tetrahydro-5,5,6a-trimethyl-2(1*H*)-pentalenone

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Abstract. $C_{17}H_{26}O_3$, $M_r = 278{\cdot}21$, triclinic, $P\bar{1}$, $a = 7{\cdot}545\,(1)$, $b = 10{\cdot}465\,(2)$, $c = 10{\cdot}629\,(1)$ Å, $\alpha = 95{\cdot}41\,(1)$, $\beta = 103{\cdot}32\,(1)$, $\gamma = 104{\cdot}30\,(1)°$, $V = 781{\cdot}1\,(2)$ Å^3, $Z = 2$, $F(000) = 304$, $D_x = 1{\cdot}18$ Mg m^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71073$ Å, $\mu(\text{Mo}\,K\alpha) = 0{\cdot}74$ cm^{-1}, $T = 133$ K, $R = 0{\cdot}046$, $wR = 0{\cdot}053$ with 1659 independent non-zero reflections. The stereochemistry is 4*SR*, 6a*RS*, the two fused rings form a boat-like arrangement and the crystal structure is stabilized by distinctive directionally-specific C—H···O hydrogen bonds.

Introduction. In the course of the stereoselective synthesis of (±)-silphenene from isophorone (Rao & Nagarajan, unpublished results) the pentalenone (1)

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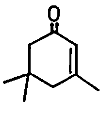
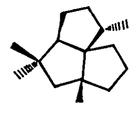
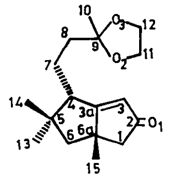
was synthesized. Its structure was established by NMR spectroscopy but the relative configuration at C(4) and C(6a) was impossible to deduce from spectroscopic data. Since enone (1) could be readily crystallized from hexane, its X-ray study was undertaken.



Isophorone



(±)-Silphenene



(1)

Experimental. Colourless well-formed tablets from hexane, 0·80 × 0·30 × 0·16 mm with main faces (010) and (0$\bar{1}$0) and growth along [001], Nicolet *P*3/F diffractometer, lattice parameters from 25 high-angle reflections, no absorption or extinction corrections applied, minimum and maximum $(\sin\theta)/\lambda$ = 0·049, 0·538 Å^{-1}, ranges of *h*, *k* and *l* 0 to 9, −12 to 12, −12 to 12, standards (400, 050, 004) did not vary more than 5%, 2119 reflections measured, 1736 unique, 1659 non-zero at the 3σ level, $R_{\rm int}$ after merging 0·052, 77 unobserved reflections, structure solution with *SHELXS*86 (Sheldrick, 1985), least-squares refinement using *F*'s, hydrogens placed in calculated positions, all other atoms refined anisotropically, 211 parameters refined, $R = 0{\cdot}046$, $wR = 0{\cdot}053$, $S = 0{\cdot}065$, $w = 1{\cdot}00/\{[\sigma(F_o)]^2 + (0{\cdot}03F_o)^2\}$ where $\sigma(F_o)$ is the standard deviation based on counting statistics, maximum least-squares shift-to-e.s.d. ratio in penultimate refinement cycle 0·05, maximum and minimum peak heights in final difference Fourier synthesis 0·17 and −0·23 e Å^{-3}, $F(000) = 304$, scattering factors from *International Tables for X-ray Crystallography* (1974), refinements carried out with *SHELX*76 (Sheldrick, 1976).

Discussion. The final atomic parameters for the non-hydrogen atoms are given in Table 1.* Bond lengths and angles are listed in Table 2. The relative configuration is 4*SR*, 6a*RS*. The molecule has a bicyclo[3.3.0]octenone fused-ring structure. The two five-membered rings are bent together to form a boat-like arrangement.

The crystal structure as viewed down [001] is shown in Fig. 1, from which the relative stereochemistry at C(4) and C(6a) is revealed. Inversion-related molecules are linked by C—H···O hydrogen bonds which, in

Table 1. *Fractional coordinates* (× 10^5) *and equivalent isotropic thermal parameters for non-hydrogen atoms* (Å^2 × 10^4) *with e.s.d.'s in parentheses*

$$U_{\rm eq} = (U_{11}U_{22}U_{33})^{1/3}.$$

	x	*y*	*z*	$U_{\rm eq}$
C(1)	6531 (3)	13815 (2)	6293 (2)	278 (14)
C(2)	4627 (4)	12774 (2)	5692 (2)	295 (14)
C(3)	4674 (3)	11585 (3)	6330 (3)	272 (14)
C(3*A*)	6237 (3)	11871 (2)	7310 (2)	238 (13)
C(4)	7360 (3)	11082 (2)	8072 (2)	245 (13)
C(5)	9349 (3)	11675 (2)	7821 (2)	261 (13)
C(6)	9460 (3)	13187 (2)	7786 (3)	281 (14)
C(6*A*)	7400 (3)	13320 (2)	7541 (2)	247 (13)
C(7)	6499 (3)	9560 (2)	7731 (2)	257 (13)
C(8)	4843 (3)	9112 (2)	8348 (2)	272 (13)
C(9)	3562 (3)	7717 (2)	7789 (2)	257 (13)
C(10)	4573 (4)	6632 (2)	7940 (3)	343 (15)
C(11)	320 (4)	7173 (3)	7511 (3)	398 (17)
C(12)	747 (4)	6903 (3)	6234 (3)	407 (17)
C(13)	10951 (4)	11500 (3)	8897 (3)	372 (15)
C(14)	9416 (4)	11024 (2)	6486 (3)	335 (15)
C(15)	7122 (4)	14079 (3)	8755 (3)	343 (15)
O(1)	3354 (2)	12896 (2)	4816 (2)	401 (12)
O(2)	2072 (2)	7464 (2)	8451 (2)	302 (10)
O(3)	2657 (2)	7672 (2)	6454 (2)	286 (10)

Table 2. *Intramolecular bond lengths* (Å) *and angles* (°) *for enone* (1) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·527 (4)	C(8)–C(9)	1·514 (3)
C(2)–C(3)	1·476 (4)	C(9)–C(10)	1·517 (3)
C(3)–C(3*A*)	1·330 (4)	C(11)–C(12)	1·483 (5)
C(3*A*)–C(4)	1·486 (3)	C(5)–C(13)	1·521 (4)
C(4)–C(5)	1·570 (3)	C(5)–C(14)	1·532 (4)
C(5)–C(6)	1·567 (3)	C(6*A*)–C(15)	1·534 (4)
C(1)–C(6*A*)	1·534 (3)	C(2)–O(1)	1·215 (3)
C(3*A*)–C(6*A*)	1·518 (3)	C(9)–O(2)	1·443 (3)
C(6)–C(6*A*)	1·559 (3)	C(11)–O(2)	1·405 (4)
C(4)–C(7)	1·537 (3)	C(9)–O(3)	1·417 (3)
C(7)–C(8)	1·535 (3)	C(12)–O(3)	1·420 (4)

C(2)–C(1)–C(6*A*)	104·6 (2)	C(1)–C(6*A*)–C(3*A*)	102·4 (2)
C(1)–C(2)–C(3)	107·1 (1)	C(1)–C(6*A*)–C(6)	119·4 (1)
C(1)–C(2)–O(1)	125·8 (1)	C(1)–C(6*A*)–C(15)	110·9 (2)
C(3)–C(2)–O(1)	127·0 (2)	C(3*A*)–C(6*A*)–C(6)	101·9 (2)
C(2)–C(3)–C(3*A*)	109·3 (3)	C(3*A*)–C(6*A*)–C(15)	109·9 (2)
C(3)–C(3*A*)–C(4)	135·5 (2)	C(6)–C(6*A*)–C(15)	111·2 (2)
C(3)–C(3*A*)–C(6*A*)	113·4 (2)	C(4)–C(7)–C(8)	110·6 (2)
C(4)–C(3*A*)–C(6*A*)	109·6 (2)	C(7)–C(8)–C(9)	114·9 (2)
C(3*A*)–C(4)–C(5)	100·5 (1)	C(8)–C(9)–C(10)	113·8 (1)
C(3*A*)–C(4)–C(7)	114·7 (2)	C(8)–C(9)–O(2)	107·9 (1)
C(5)–C(5)–C(7)	117·4 (2)	C(8)–C(9)–O(3)	109·2 (2)
C(4)–C(5)–C(6)	103·6 (2)	C(10)–C(9)–O(2)	109·0 (2)
C(4)–C(5)–C(13)	111·8 (2)	C(10)–C(9)–O(3)	110·5 (2)
C(4)–C(5)–C(14)	110·3 (2)	O(2)–C(9)–O(3)	106·1 (1)
C(6)–C(5)–C(13)	111·7 (2)	C(12)–C(11)–O(2)	105·1 (2)
C(6)–C(5)–C(14)	109·2 (2)	C(11)–C(12)–O(3)	103·7 (3)
C(13)–C(5)–C(14)	110·1 (2)	C(9)–O(2)–C(11)	108·5 (2)
C(5)–C(6)–C(6*A*)	107·5 (1)	C(9)–O(3)–C(12)	106·9 (2)

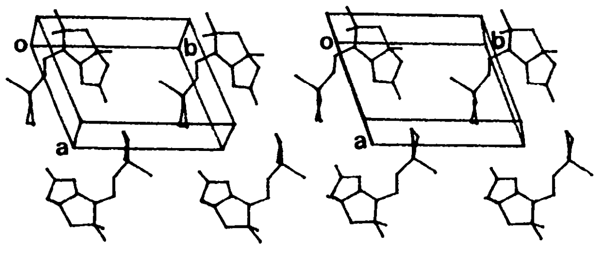


Fig. 1. Stereodrawing of the crystal structure of enone (1) looking down [001].

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44490 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

general, have been increasingly recognized as being of importance in stabilizing organic crystal structures (Taylor & Kennard, 1984; Sarma & Desiraju, 1986). In the crystal structure of enone (1), these bonds are of two types: (*a*) to carbonyl oxygen, $O(1)(x,y,z)\cdots C(12)(-x,2-y,1-z)$ 3·10 Å; $O(1)(x,y,z)\cdots H(12a)(-x,2-y,1-z)$ 2·34 Å; $C(12)-H(12a)\cdots O(1)$ 127·4°; $H(12a)\cdots O(1)-C(2)$ 167·8°; (*b*) to ethereal oxygen $O(2)(x,y,z)\cdots C(15)(1-x,2-y,2-z)$ 3·52 Å; $O(2)(x,y,z)\cdots H(15b)(1-x,2-y,2-z)$ 2·45 Å; $C(15)-H(15b)\cdots O(2)$ 168·4°. The directional preferences of both these bonds seem to be pronounced, in keeping with trends earlier observed (Taylor & Kennard, 1984).

The X-ray data were collected by Dr Ward T. Robinson, Department of Chemistry, University of Canterbury, New Zealand, under the IUCr Commission on Small Molecules Programme for International Cooperation in Intensity Data Collection. We thank Dr Robinson for his kind assistance. One of us (VN) thanks the CSIR, New Delhi, for the award of a Junior Research Fellowship.

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Structure of WF-3681, 3-(2,5-Dihydro-4-hydroxy-5-oxo-3-phenyl-2-furyl)propionic Acid

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(*Received* 26 *August* 1986; *accepted* 12 *August* 1987)

Abstract. $C_{13}H_{12}O_5$, $M_r = 248·24$, monoclinic, $C2/c$, $a = 18·757$ (8), $b = 7·282$ (2), $c = 17·511$ (8) Å, $\beta = 91·20$ (3)°, $V = 2391$ (3) Å^3, $Z = 8$, $D_x = 1·379$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·54178$ Å, $\mu = 0·859$ mm^{-1}, $F(000) = 1040$, $T = 293$ K. Final $R = 0·054$ for 1409 unique observed reflections. The molecule contains two planar regions which differ in orientation by 5·7°. Distances from the carbonyl carbons to the center of the phenyl ring are not in the range found in the crystal structures of other potent aldose reductase inhibitor molecules.

Introduction. The crystal structure of the title compound, WF-3681 (Uchida, Itoh, Namiki, Nishikawa & Hashimoto, 1986; Okamoto, Uchida, Umehara, Kohsaka & Imanaka, 1984), has been determined as part of a study of structure–activity relationships of inhibitors of the enzyme aldose reductase. This enzyme, which catalyzes the reduction of glucose to sorbitol, has been implicated in the occurrence of numerous complications of diabetes including cataract formation (Kinoshita, 1974; Kinoshita, Kador & Datiles, 1981; Judzewitsch *et al.*, 1983). Effective inhibitors of aldose reductase are therefore of great pharmacological interest.

A structurally diverse group of compounds will inhibit aldose reductase, apparently at a common site (Kador & Sharpless, 1983). The mechanism of action and crucial structural features of these inhibitors are unknown. In a previous paper (Kissinger, Adman, Clark & Stenkamp, 1985), we have described the structure of the potent aldose reductase inhibitor, sorbinil. Here we describe the structure of WF–3681, an effective inhibitor of the enzyme which differs from other known inhibitors in containing a lactone ring.

Experimental. Sample of the compound provided by Fujisawa Pharmaceutical Co. Ltd as a gift. Crystallized by slow evaporation from ethanol and water solution. Crystal dimensions 0·50 × 0·25 × 0·15 mm. KRISEL control-updated Picker FACS-1 diffractometer. Nickel-filtered Cu $K\alpha$ radiation. Cell constants determined by least squares from angular settings of 20 reflections ($9 < 2\theta < 72°$). ω–2θ scans, 2° min^{-1}. Maximum $(\sin\theta)/\lambda = 0·5451$ Å^{-1} ($0 \le h \le 20$, $-7 \le k \le 0$, $0 \le l \le 18$). Data set of 1837 reflections, 1618 unique, 1409 with $F > 3\sigma_F$. 16 standard reflections collected every 8 h showed <2% variation in intensity. No deterioration correction. Empirical absorption correction (North, Phillips & Mathews, 1968) applied (correction factor 1·01–1·09).

Lorentz–polarization corrections applied. Structure solved by direct methods using *RANTAN*80 (Yao,

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