

6-(1-Hydroxy-2,2-diphenylethyl)-4,4-diphenyl-2-cyclohexen-1-one

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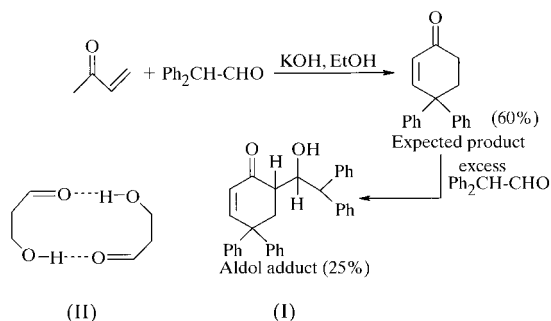
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The crystal structure of the title compound, $C_{32}H_{28}O_2$, (I), confirms the *erythro* stereochemistry of the aldol adduct. In the crystal, (I) forms centrosymmetric $O-H\cdots O=C$ hydrogen-bonded dimers which in turn are connected by $C-H\cdots O$ and $C-H\cdots \pi$ interactions.

Comment

During studies of the crystal structure of 4,4-diphenyl-2,5-cyclohexadien-1-one (Anthony *et al.*, 1998), the title compound, (I), was obtained as a by-product in the base-catalysed condensation of diphenylacetaldehyde with methyl vinyl ketone (Zimmerman *et al.*, 1968) (see scheme). The chemical structure is consistent with (I) based on its NMR and IR spectra. The stereochemistry of (I) was established by X-ray diffraction and confirmed to be *erythro* at C2 and C19 stereogenic centres. The ready reversibility of aldol condensation under the equilibrium conditions of alcoholic KOH medium provides the expected *erythro* diastereomer (House, 1972). The molecular geometry of (I) in the crystal is shown in Fig. 1 (Johnson, 1976).



In the crystal, inversion-related molecules of (I) form $O-H\cdots O=C$ dimers, (II), in the (110) layer between the hydroxy group and the carbonyl-O atom [$O2-H2A\cdots O1$ 1.90 (2) Å, 176.6 (15)°] (Fig. 2). Such dimers are in turn connected by $C-H\cdots O$ and $C-H\cdots \pi$ interactions (Desiraju & Steiner, 1999) along [100] from the phenyl- and olefin-H atoms to the carbonyl and phenyl (C21–C26) acceptor groups, respectively (C29–H29A $\cdots$ O1 2.59 Å, 161.4°; C5–H5A $\cdots \pi_{\text{centroid}}$

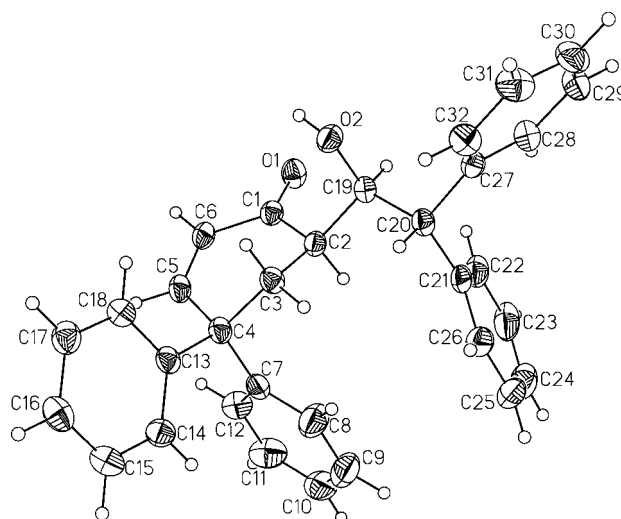


Figure 1
ORTEP (Johnson, 1976) diagram and atom-numbering scheme for (I); displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

2.73 Å, 170.0°). The chains are interlinked by $C_{\text{phenyl}}-H\cdots O_{\text{hydroxyl}}$ interactions along [010] (C16–H16A $\cdots$ O2 2.51 Å, 132.5°), not shown in Fig. 2.

Allen *et al.* (1999) have reported on the probabilities of formation of cyclic hydrogen-bond intermolecular motifs in organic crystal structures archived in the Cambridge Structural Database. Supramolecular synthon (II) occurs in 20 structures out of a possible 282 molecules that contain the β -hydroxy carbonyl functional group, giving it a probability of formation (P_s) of 0.07. While the P_s of motif (II) is low and its overall ranking 71/75 in the CSD analysis, it may be noted that the occurrence of this synthon is more frequent in crystal structures of aldol adducts (Fair *et al.*, 1985; Gleiter *et al.*, 1996; Gross & Finn, 1994; Lodge & Heathcock, 1987).

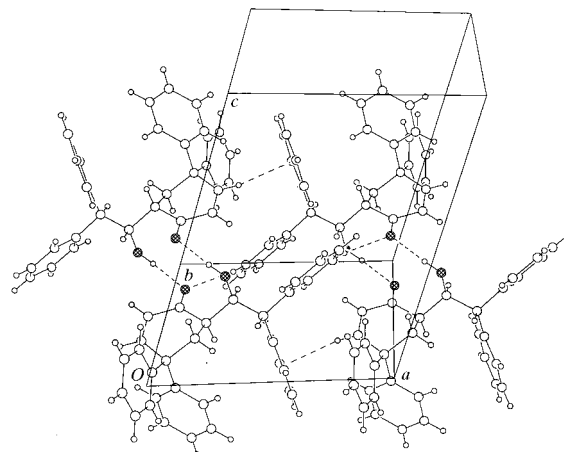


Figure 2
View of (I) in [101] showing the centrosymmetric $O-H\cdots O$ dimer, (II), and the intermolecular $C-H\cdots O$ and $C-H\cdots \pi$ interactions.

Experimental

Compound (I) was synthesized as shown in the scheme (Zimmerman *et al.*, 1968). Crystals were obtained upon recrystallization from 5% ethyl acetate/hexane (m.p. 457 K).

Crystal data

$C_{32}H_{28}O_2$	$Z = 2$
$M_r = 444.54$	$D_x = 1.228 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.6111 (9) \text{ \AA}$	Cell parameters from 1500 reflections
$b = 11.5199 (12) \text{ \AA}$	$\theta = 1.9\text{--}21.5^\circ$
$c = 11.5621 (12) \text{ \AA}$	$\mu = 0.075 \text{ mm}^{-1}$
$\alpha = 84.458 (1)^\circ$	$T = 168 (2) \text{ K}$
$\beta = 77.829 (1)^\circ$	Prism, colourless
$\gamma = 74.138 (1)^\circ$	$0.5 \times 0.31 \times 0.15 \text{ mm}$
$V = 1202.7 (2) \text{ \AA}^3$	

Data collection

CCD area-detector diffractometer	$\theta_{\max} = 26.37^\circ$
φ and ω scans	$h = -5 \rightarrow 11$
10624 measured reflections	$k = -14 \rightarrow 14$
4605 independent reflections	$l = -13 \rightarrow 14$
3460 reflections with $I > 2\sigma(I)$	Intensity decay: none
$R_{\text{int}} = 0.016$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2]$
$wR(F^2) = 0.093$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.051$	$(\Delta/\sigma)_{\max} = 0.001$
4605 reflections	$\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$
337 parameters	$\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bonding geometry ($\text{\AA}$, $^\circ$).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
$O2\cdots H2A\cdots O1^i$	0.914 (18)	1.903 (18)	2.8163 (14)	176.6 (15)
$C16\cdots H16A\cdots O2^{ii}$	0.95	2.51	3.2325 (19)	133
$C29\cdots H29A\cdots O1^{iii}$	0.95	2.59	3.512 (2)	161
$C5\cdots H5A\cdots \pi^{iv}$	0.95	2.73	3.67	170

Symmetry codes: (i) $-x, 2-y, -z$; (ii) $-x, 1-y, -z$; (iii) $1-x, 2-y, -z$; (iv) $x-1, y, z$.

The hydroxyl-H atom was identified from a Fourier synthesis and refined freely. H atoms bonded to carbon were generated at idealized geometries and refined isotropically using a riding model.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLUTON* (Spek, 1992); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1380). Services for accessing these data are described at the back of the journal.

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