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2-[(4-Chlorophenyl)hydroxymethyl]cyclopent-2-en-1-one

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The crystal structure of the title compound, $C_{12}H_{11}ClO_2$, contains intermolecular O—H···O and C—H···O interactions, linking the molecules to form a three-dimensional framework. The dihedral angle between the cyclopentene plane and the benzene ring plane is 67.00 (7)°. C—H···π interactions are also present.

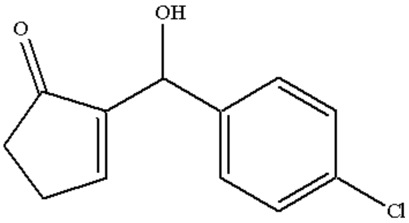
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.122
Data-to-parameter ratio = 13.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Baylis–Hillman adducts represent a series of useful intermediates in organic synthesis, leading to applications in biology (Kabat *et al.*, 1996; Kim *et al.*, 2002; Shi & Zhao, 2002). A few crystal structures of Baylis–Hillman products have been reported by us (Huo *et al.*, 2004*a*, *b*; Huo *et al.*, 2004). Here, we report another adduct, obtained from cyclopentenone and 4-chlorobenzaldehyde.



(I)

Selected geometric parameters of (I) are listed in Table 1 and the crystal structure is illustrated in Fig. 1. The value of the bond angle C3—C2—C6, 130.54 (17)°, is only slightly larger than the values, 128.3 and 129.1°, in the two crystallographically independent molecules of *syn*-2,5-bis(hydroxy(phenyl)methyl)cyclopent-2-enone butane-2,3-dione solvate (Shi *et al.*, 2002). The cyclopentene ring is planar with a

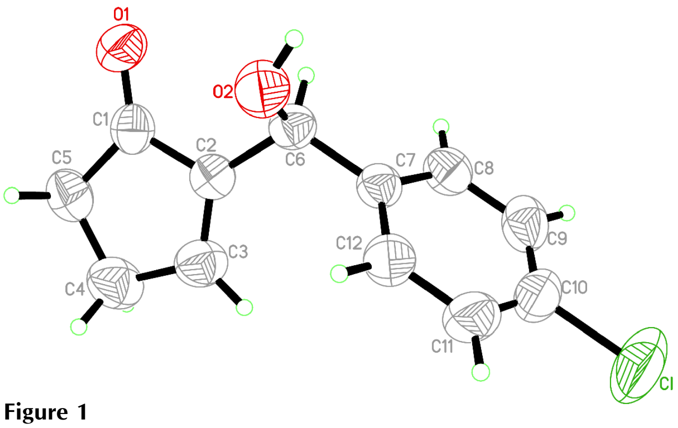


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

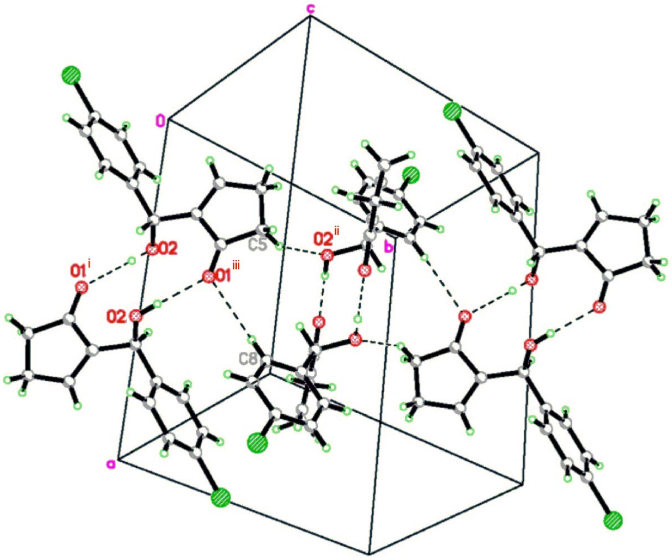


Figure 2
Crystal structure of (I), showing the intermolecular hydrogen bonding as dashed lines [symmetry code: (i) $1 - x, -y, 2 - z$; (ii) $x, -\frac{1}{2} - y, z - \frac{1}{2}$ (iii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$]. Selected atoms are labeled.

maximum deviation of 0.0199 (1) Å for atom C1. The dihedral angle between the cyclopentene plane and the benzene ring plane is 67.00 (7)°.

Dimers (Fig. 2), composed of enantiomers, are linked *via* strong intermolecular O2—H1···O1^{i} [symmetry code: (i) $1 - x, -y, 2 - z$] hydrogen bonds, forming $R_2^2(12)$ rings. Further examination of non-bonded contacts also revealed two intermolecular C—H···O bonds (Table 2; Fig. 2). These weak hydrogen bonds also help to stabilize the three-dimensional framework of the crystal structure. Additionally, a very weak intermolecular C—H···π contact (Table 2) exists, involving the π-system of the C7–C12 ring, whose centroid is *Cg* (Spek, 2003).

Experimental

The title compound was synthesized by the Baylis–Hillman reaction, as reported previously (Shi *et al.*, 2002; Luo *et al.*, 2002). In the former publication it was reported to be a colorless liquid, but we obtained single crystals by the following procedure. The colorless liquid was dissolved in anhydrous diethyl ether, and petroleum ether was added. The solution was allowed to evaporate at room temperature for 24–48 h. On completion of the evaporation, colorless crystals suitable for a single-crystal X-ray diffraction study were collected; m.p. 352 K. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26–7.35(*m*, 5H, ArH & CH), 5.53 (*m*, 1H, *CH), 3.58 (*br s*, 1H, OH), 2.45–2.64(*m*, 4H, CH_2).

Crystal data

$C_{12}H_{11}ClO_2$
$M_r = 222.66$
Monoclinic, $P2_1/c$
$a = 12.920$ (2) Å
$b = 11.411$ (2) Å
$c = 7.448$ (1) Å
$\beta = 99.329$ (2)°
$V = 1083.5$ (3) Å^3
$Z = 4$

$D_x = 1.365$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6533 reflections
$\theta = 2.4$–22.8°
$\mu = 0.33$ mm^{-1}
$T = 298$ (2) K
Rod, colorless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.908$, $T_{max} = 0.937$
4417 measured reflections

1903 independent reflections
1408 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 25.0°$
$h = -15 \rightarrow 15$
$k = -12 \rightarrow 13$
$l = -8 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.10$
1903 reflections
137 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.0122P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O2—C6	1.422 (2)	C2—C6	1.504 (3)
C2—C3	1.327 (3)	C6—C7	1.510 (2)
O1—C1—C2	126.16 (18)	C2—C3—C4	114.34 (19)
O1—C1—C5	125.16 (18)	C2—C6—C7	113.51 (14)
O2—C6—C2	106.53 (15)	C3—C2—C1	107.75 (17)
O2—C6—C7	112.12 (14)	C3—C2—C6	130.54 (17)
C1—C2—C6	121.71 (15)	C3—C4—C5	103.67 (17)
C1—C5—C4	105.47 (16)	C8—C7—C6	120.86 (16)
C2—C1—C5	108.65 (16)	C12—C7—C6	120.63 (17)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O1^{i}	0.82	1.96	2.778 (2)	178
C5—H5*B*···O2^{ii}	0.97	2.43	3.317 (3)	153
C8—H8···O1^{iii}	0.93	2.57	3.465 (2)	163
C9—H9···*Cg*^{iv}	0.93	3.36	3.497 (2)	91

Symmetry codes: (i) $1 - x, -y, 2 - z$; (ii) $x, -\frac{1}{2} - y, z - \frac{1}{2}$; (iii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iv) $x, \frac{1}{2} - y, z - \frac{1}{2}$. *Cg* is the centroid of the C7–C12 ring.

H atoms were placed in calculated positions and allowed to ride on their parent atoms, with U_{iso}(H) values set to $1.5U_{eq}$(parent atom) for the hydroxyl H atom and Csp^3-bound H atoms, and $1.2U_{eq}$(parent atom) for Csp^2-bound H atoms. The O—H distance was fixed at 0.82 Å and the C—H distances were fixed in the range 0.93–0.98 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2000); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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