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3-(3,4-Dimethoxyphenyl)-1-(4-hydroxyphenyl)-prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.119
Data-to-parameter ratio = 13.8

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

The title molecule, $C_{17}H_{16}O_4$, is nearly planar, with a dihedral angle of 6.64 (6)° between the two aromatic rings. The configuration of the keto group with respect to the olefinic double bond is s-*cis*. O—H···O and C—H···π hydrogen bonds link the molecules to form layers parallel to the *ab* plane. The crystal structure is further stabilized by a number of C—H···O interactions, which form rings of $R_2^1(5)$, $R_2^1(6)$ and $R_1^2(5)$ graph set motifs.

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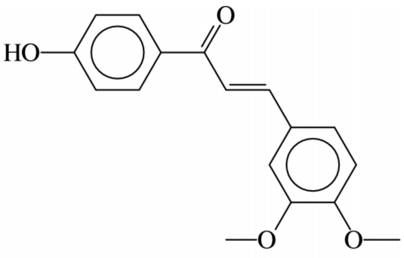
Comment

Chalcones and their heterocyclic analogues show various biological effects, *e.g.* anti-inflammatory, antitumour, antibacterial, antituberculous, antiviral, antiprotozoal, gastroprotective and others (Shibata, 1994; Gafner *et al.*, 1996; Opletalova & Sedivy, 1999; Xia *et al.*, 2000; Popova *et al.*, 2001). The cytotoxic, anticancer, chemopreventative, mutagenic, antiviral, antiprotozoal and insecticidal activities of a variety of chalcones have been reviewed by Dimmock *et al.* (1999). In addition, hydroxychalcones can be readily reacted with acryloyl and methacryloyl chloride to obtain acrylate and methacrylate monomers, which are useful for the preparation of photo-cross-linkable polymers (Subramanian *et al.*, 2001; Balaji & Nanjundan, 2002). The structure determination of the title compound, (I), was undertaken as part of our study of chalcones.



(I)

In the title molecule, the configuration of the keto group with respect to the olefinic double bond is s-*cis*, as seen from the C7—C8—C9—O1 torsion angle of −3.1 (2)° (Fig. 1). A similar conformation has been previously reported for related structures (Raj *et al.*, 1996, 1997; Jeyabharathi *et al.*, 2002; Ravishankar *et al.*, 2003). The two aromatic rings and the unsaturated ketone system are nearly coplanar, with a dihedral angle of 6.64 (6)° between the two rings. The two methoxy groups are almost coplanar with the attached ring, the C17—O3—C4—C5 and C16—O2—C3—C2 torsion angles being 11.5 (2) and −4.6 (2)°, respectively. The widening of the C5—C6—C7 [122.5 (1)°] and C6—C7—C8 [128.2 (1)°] angles from

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120° can be ascribed to the short interatomic contact between atoms H5 and H8 [2.24 (2) Å]. In addition, the strain induced by the short H8$\cdots$H11 [2.07 (2) Å] contact results in a slight opening of the C9—C10—C11 angle to 123.6 (1)°. Similar features have also been reported for a related structure (Ravishankar *et al.*, 2003). The close approach of methyl atoms H17*B* and H17*C* to atom H5 [H5$\cdots$H17*B* = 2.34 (2) Å and H5$\cdots$H17*C* = 2.34 (2) Å] and atom H16*C* to atom H2 [H2$\cdots$H16*C* = 2.28 (3) Å] results in the widening of the O3—C4—C5 [125.40 (11)°] and O2—C3—C2 [125.59 (11)°] angles, respectively. The narrowing of the O2—C3—C4 [114.65 (11)°] and O3—C4—C3 [114.71 (10)°] angles indicates that the lone pair-lone pair repulsion between the two methoxy O atoms is small compared with the H$\cdots$H repulsion involving the methyl H atoms.

In the crystal structure, screw-related molecules are linked by O4—H4$\cdots$O1^{i} hydrogen bonds to form chains along the *b* axis. The adjacent inversion-related chains are linked by centrosymmetric C17—H17*B*$\cdots$*Cg*^{x} interactions (*Cg* denotes the centroid of the aromatic C1–C6 ring) to form a molecular layer parallel to the *ab* plane (Fig. 2). The crystal structure is further stabilized by a number of C—H$\cdots$O interactions (Fig. 3 and Table 2), whose H$\cdots$O distances agree with the range reported for C—H$\cdots$O hydrogen bonds (Jeffrey, 1997; Desiraju & Steiner, 1999). The O4—H4$\cdots$O1^{i} and C14—H14$\cdots$O1^{i} interactions together form a pair of bifurcated acceptor bonds, which generate a ring with a graph set motif of $R_2^1(5)$ (Etter *et al.*, 1990). An $R_2^1(6)$ motif is formed by the C1—H1$\cdots$O2^{ii} and C7—H7$\cdots$O2^{ii} interactions. The C1—H1$\cdots$O2^{ii} and C1—H1$\cdots$O3^{ii} interactions constitute a pair of bifurcated donor bonds, generating a ring with a graph set motif of $R_1^2(5)$. All symmetry codes are given in Table 2.

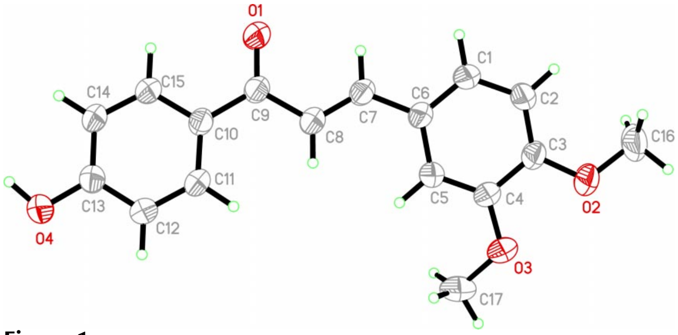

Figure 1
A view of the title molecule, with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

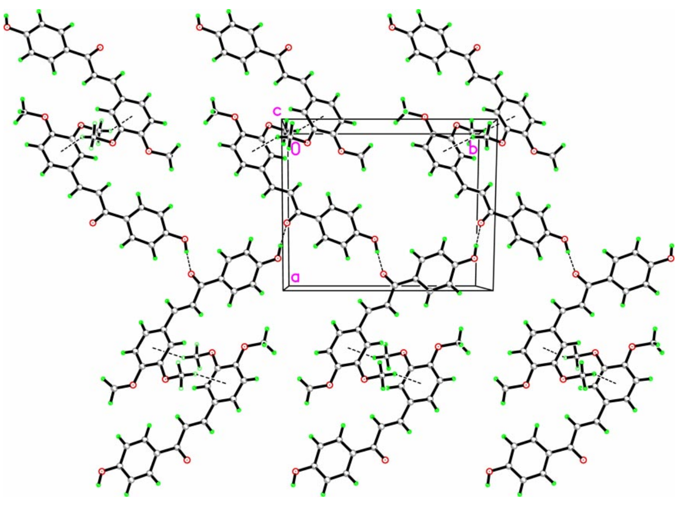

Figure 2
A view of the molecular layer, showing O—H$\cdots$O hydrogen bonds and C—H$\cdots\pi$ interactions.

Experimental

The title compound was prepared by stirring an alcoholic solution of 4-hydroxyacetophenone with 3,4-dimethoxybenzaldehyde in the presence of sodium hydroxide for 3 d at 273–283 K. The reaction mixture was then neutralized with dilute HCl and the precipitated crude product was filtered off, washed and recrystallized from ethanol.

Crystal data

$C_{17}H_{16}O_4$
M_r = 284.30
Monoclinic, $P2_1/n$
a = 10.5750 (6) Å
b = 12.8709 (7) Å
c = 11.0612 (6) Å
β = 103.071 (1)°
V = 1466.53 (14) Å^3
Z = 4

D_x = 1.288 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5614 reflections
θ = 2.4–28.2°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, yellow
0.60 × 0.56 × 0.52 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
9034 measured reflections
3530 independent reflections
2922 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.014
$\theta_{\rm max}$ = 28.3°
h = −14 → 13
k = −16 → 14
l = −13 → 14

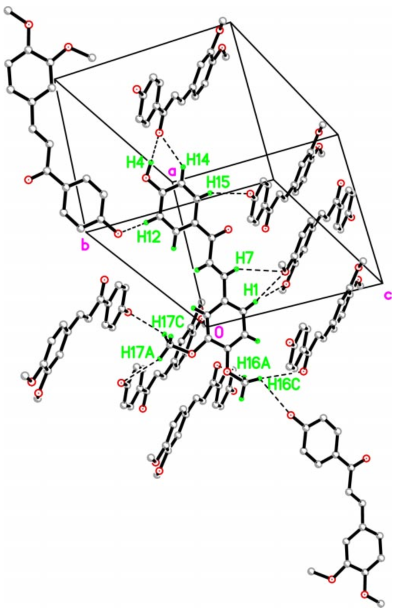

Figure 3
A view of the C—H$\cdots$O interactions. Only those H atoms involved in the interactions are labelled.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.119$
$S = 1.05$
3530 reflections
255 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.2916P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.018 (2)

Table 1
Selected geometric parameters (Å, °).

O1—C9	1.2359 (15)	O4—C13	1.3586 (15)
O2—C3	1.3607 (14)	C6—C7	1.4556 (15)
O2—C16	1.4146 (19)	C7—C8	1.3340 (17)
O3—C4	1.3643 (14)	C8—C9	1.4683 (16)
O3—C17	1.4188 (18)	C9—C10	1.4798 (16)
O2—C3—C2	125.59 (11)	C5—C6—C7	122.50 (10)
O2—C3—C4	114.65 (11)	C8—C7—C6	128.22 (11)
O3—C4—C3	114.71 (10)	C11—C10—C9	123.55 (10)
O3—C4—C5	125.40 (11)		
C16—O2—C3—C2	−4.6 (2)	C17—O3—C4—C3	−168.04 (13)
C16—O2—C3—C4	174.84 (15)	C7—C8—C9—O1	−3.1 (2)
C17—O3—C4—C5	11.5 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O1^{i}	0.93 (2)	1.76 (2)	2.686 (1)	176 (2)
C14—H14···O1^{i}	0.97 (2)	2.63 (2)	3.311 (2)	127 (1)
C7—H7···O2^{ii}	0.96 (2)	2.87 (2)	3.581 (2)	132 (1)
C1—H1···O3^{ii}	0.98 (2)	2.66 (2)	3.609 (2)	163 (1)
C1—H1···O2^{ii}	0.98 (2)	2.62 (2)	3.417 (2)	139 (1)
C12—H12···O4^{iii}	0.99 (2)	2.74 (2)	3.674 (2)	158 (1)
C15—H15···O4^{iv}	0.98 (2)	2.91 (2)	3.841 (2)	159 (1)
C16—H16*A*···O1^{v}	1.02 (2)	2.98 (2)	3.886 (3)	148 (2)
C16—H16*C*···O1^{vi}	0.97 (2)	2.66 (2)	3.528 (2)	149 (2)
C16—H16*C*···O4^{vii}	0.97 (2)	2.86 (2)	3.333 (2)	111 (1)
C17—H17*A*···O4^{viii}	1.01 (2)	2.95 (2)	3.808 (2)	144 (1)
C17—H17*C*···O4^{ix}	1.01 (2)	2.62 (2)	3.467 (2)	142 (1)
C17—H17*B*···*Cg*^{x}	0.99 (2)	2.89 (2)	3.803 (2)	154 (1)

Symmetry codes: (i) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (ii) $\frac{1}{2}+x, -\frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1-x, 1-y, -z$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (v) $x-\frac{1}{2}, -\frac{1}{2}-y, z-\frac{1}{2}$; (vi) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (vii) $x-1, y-1, z$; (viii) $\frac{1}{2}-x, y-\frac{1}{2}, -\frac{1}{2}-z$; (ix) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (x) $-x, -y, -z$.

All H atoms were located from a difference Fourier map and their positional and isotropic displacement parameters were refined. The range of C—H distances is 0.95 (2)–1.02 (2) Å and the O—H distance is 0.93 (2) Å. The $U_{\rm iso}$(H) values lie in the range 0.047 (4)–0.103 (7) Å^2. Owing to the large fraction of weak data at higher angles, the completeness of the data is low (0.97).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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