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(*E*)-1-(2-Hydroxy-3,4-dimethoxyphenyl)-3-(4-hydroxyphenyl)prop-2-en-1-one

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Key indicators

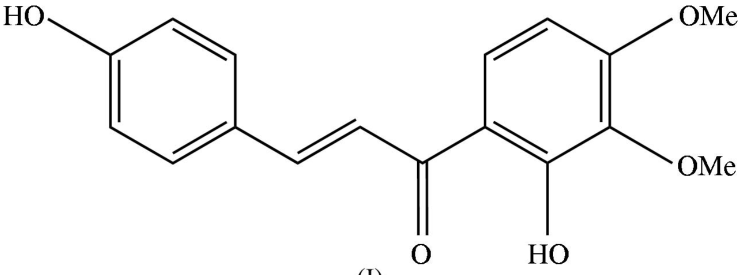
Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.118
Data-to-parameter ratio = 10.6

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

The title compound, $C_{17}H_{16}O_5$, crystallizes with two independent molecules in the asymmetric unit. These are oriented approximately orthogonal to one another. The keto group adopts an *s-cis* conformation; the six-membered ring formed by the O—H···O hydrogen bond and the adjacent benzene ring are approximately coplanar. In addition to an intramolecular O—H···O hydrogen bond, intermolecular O—H···O interactions link adjacent molecules into chains.

Comment

The title compound (I), also referred to as heliannone A (Macias *et al.*, 1997), is one of the bioactive flavonoids from *Helianthus annuus* cultivars which possibly play a role in the allelopathic activity of sunflowers. It was synthesized by Koteswara Rao *et al.* (2001) and we report the crystal structure here.



(I)

The two independent molecules, *A* and *B*, in the asymmetric unit of (I) are oriented approximately orthogonal to one another at an angle of 85.44 (5)°. Bond lengths and angles in the central enone unit agree with those found in chalcone derivatives with different *para* substituents (Jeyabharathi *et al.*, 2002; Rabinovich & Shakked, 1974). The keto group adopts an *s*-*cis* conformation, with C7—C8—C9—O5 torsion angles of 5.8 (2)° for molecule *A* and −0.4 (2)° for molecule *B*. The keto system is not strictly planar, as can be seen from the torsion angles in Table 1. The least-squares mean planes of the hydroxyphenyl and dimethoxyphenyl rings make dihedral angles with the keto group of 13.93 (5) and 7.32 (5)°, respectively, for molecule *A*, and 8.34 (5) and 8.42 (5)°, respectively, for molecule *B*. The dihedral angles between the rings themselves are 20.61 (5) for molecule *A* and 9.35 (5)° for molecule *B*. The enol rings formed by hydrogen bonding, comprising atoms O4*A*, O5*A*, C9*A*, C1′, C2′ and H30 in molecule *A*, and atoms O4*B*, O5*B*, C9*B*, C1″, C2″ and H30′ in molecule *B*, are

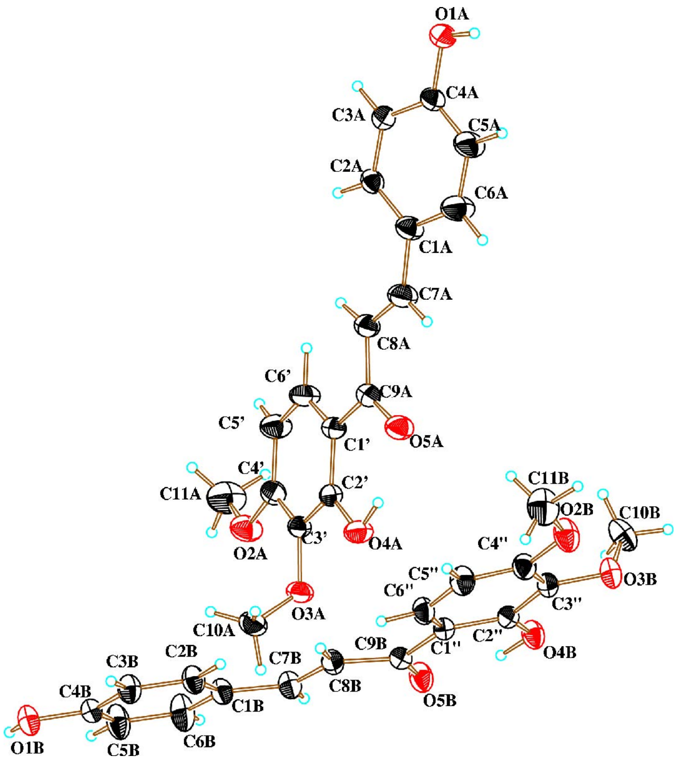


Figure 1
The structure of the asymmetric unit of (I), with 30% probability displacement ellipsoids.

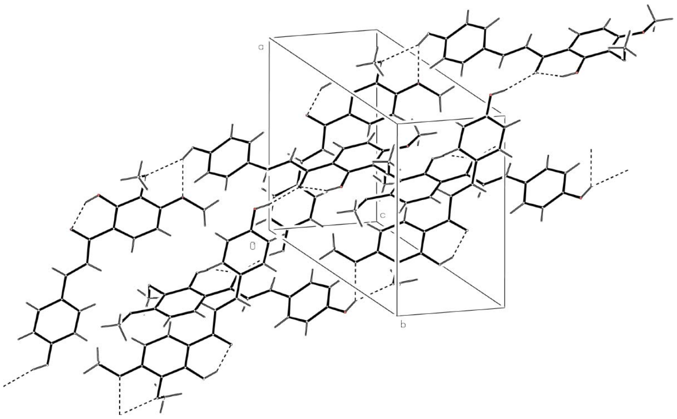


Figure 2
The molecular packing of (I), viewed down the b axis. H atoms bonded to C atoms have been omitted for clarity. Dashed lines indicate hydrogen bonds.

slightly distorted. The fused benzene and enol rings are approximately coplanar, with dihedral angles between the least-squares mean planes of the benzene and enol rings of 4.44 (5)° for molecule A and 2.36 (5)° for molecule B.

The structure of (I) is stabilized by intra- and intermolecular hydrogen bonds. The intramolecular interaction involves the hydroxyl group and the adjacent ketone O atom, with O4A—H30$\cdots$O5A 2.5534 (17) Å for molecule A and O4B–0H30′$\cdots$O5B 2.5784 (16) Å for molecule B. This classic interaction is the likely cause of the lengthening of the C2′—O4A [1.340 (16) Å] and C2″—O4B bonds [1.3391 (16) Å] and the shortening of the corresponding C9A═O5A [1.2538 (17) Å] and C9B═O5B bonds [1.2406 (17) Å] in comparison with normal C—O and C═O distances (Allen *et al.*, 1987). In addition, atom O5A forms an intermolecular hydrogen bond with a hydroxyl group from a neighbouring molecule B [O1B—H10′$\cdots$O5$A^{\rm ii}$ 2.8093 (16); symmetry code (ii): $x-1, y-1, z-1$], to generate an infinite chain parallel to the b axis (Fig. 2). Further stabilization derives from hydrogen bonds between the H atom of the hydroxyl group O1A and the methoxy atoms O2B and O3B. These form additional links between the primary chains [O1A—H10$\cdots$O3$B^{\rm i}$ 3.0616 (17) and O1A—H10$\cdots$O2B 2.8855 (19) Å; symmetry code (i): $-x+2, -y+2, -z+2$].

Experimental

The title compound was obtained from a solution of 2-hydroxy-3,4-dimethoxyacetophenone (98 mg, 0.5 mmol) and p-hydroxybenzaldehyde (61 mg, 0.5 mmol) in EtOH (10 ml). This was added dropwise to 50% aqueous KOH (10 ml). The resulting mixture was stirred at room temperature for 72 h. The reaction mixture was poured into ice-cold water, its pH was adjusted to 3–4 with 10% aqueous HCl and it was extracted with Et_2O (4 × 20 ml). The organic extract was washed with water and brine, dried over anhydrous $MgSO_4$ and evaporated under reduced pressure. The resulting residue was purified on a silica-gel column eluted with hexane–EtOAc (7:3) to give yellow crystals of the title chalcone, (I) (72 mg, 48%).

Crystal data

$C_{17}H_{16}O_5$
$M_r = 300.30$
Triclinic, $P\bar{1}$
$a = 11.6831$ (13) Å
$b = 11.8477$ (13) Å
$c = 12.9212$ (14) Å
$\alpha = 111.911$ (2)°
$\beta = 111.296$ (2)°
$\gamma = 92.437$ (2)°
$V = 1512.9$ (3) Å^3

$Z = 4$
$D_x = 1.318$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8782 reflections
$\theta = 1.9$–25.0°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.25 × 0.25 × 0.13 mm

Data collection

Siemens SMART CCD area detector diffractometer
ω scans
Absorption correction: none
14 357 measured reflections
5298 independent reflections

4667 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 25.0°$
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.118$
$S = 1.06$
5298 reflections
502 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0699P)^2 + 0.2158P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.24$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.025 (3)

o1324 Radha Krishna *et al.* • $C_{17}H_{16}O_5$ *Acta Cryst.* (2005). E**61**, o1323–o1325

Table 1
Selected geometric parameters (Å, °).

O4*B*—C2″	1.3391 (16)	C8*A*—C9*A*	1.4650 (19)
O4*A*—C2′	1.3401 (16)	C1′—C9*A*	1.4644 (19)
O5*B*—C9*B*	1.2406 (17)	C8*B*—C7*B*	1.323 (2)
O5*A*—C9*A*	1.2538 (17)	C8*B*—C9*B*	1.4693 (18)
C1″—C9*B*	1.4711 (18)	C1*B*—C7*B*	1.4575 (19)
C8*A*—C7*A*	1.325 (2)	C1*A*—C7*A*	1.453 (2)
C6″—C1″—C9*B*	121.83 (12)	O5*B*—C9*B*—C8*B*	120.69 (12)
C2″—C1″—C9*B*	120.01 (11)	O5*B*—C9*B*—C1″	120.46 (11)
C7*A*—C8*A*—C9*A*	121.14 (14)	C8*B*—C9*B*—C1″	118.79 (12)
C6′—C1′—C9*A*	122.74 (12)	C2*B*—C1*B*—C7*B*	122.77 (12)
C2′—C1′—C9*A*	120.15 (12)	C6*B*—C1*B*—C7*B*	120.06 (13)
O5*A*—C9*A*—C1′	119.11 (12)	C8*B*—C7*B*—C1*B*	127.06 (14)
O5*A*—C9*A*—C8*A*	119.97 (12)	C2*A*—C1*A*—C7*A*	124.26 (13)
C1′—C9*A*—C8*A*	120.92 (12)	C6*A*—C1*A*—C7*A*	118.17 (14)
C7*B*—C8*B*—C9*B*	122.84 (14)	C8*A*—C7*A*—C1*A*	129.86 (15)
C2′—C1′—C9*A*—C8*A*	−174.55 (12)	C2″—C1″—C9*B*—C8*B*	−171.80 (12)
C7*A*—C8*A*—C9*A*—O5*A*	5.8 (2)	C9*B*—C8*B*—C7*B*—C1*B*	179.01 (13)
C7*A*—C8*A*—C9*A*—C1′	−173.69 (15)	C6*B*—C1*B*—C7*B*—C8*B*	175.54 (17)
C7*B*—C8*B*—C9*B*—O5*B*	−0.4 (2)	C9*A*—C8*A*—C7*A*—C1*A*	178.67 (16)
C7*B*—C8*B*—C9*B*—C1″	176.69 (13)	C6*A*—C1*A*—C7*A*—C8*A*	−170.5 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4*A*—H30···O5*A*	0.92 (2)	1.70 (3)	2.5534 (17)	153 (2)
O4*B*—H30′···O5*B*	0.94 (2)	1.74 (2)	2.5784 (16)	147 (2)
O1*A*—H10···O3*B*^{i}	0.83 (2)	2.27 (2)	3.0616 (17)	160 (2)
O1*A*—H10···O2*B*^{i}	0.83 (2)	2.30 (3)	2.8855 (19)	128 (2)
O1*B*—H10’···O5*A*^{ii}	0.90 (2)	1.92 (2)	2.8093 (16)	169 (2)

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

All H atoms, except those on the methyl groups of molecule *B*, were located in a difference Fourier map and only their positional parameters were refined. The remainder were included in calculated positions, with C—H distances constrained to 0.96 Å, and allowed to ride on their attached C atoms, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *enCIFer* (CCDC, 2003) and *PARST* (Nardelli, 1995).

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Acta Cryst. (2005). E**61**, o1323–o1325 Radha Krishna *et al.* • $C_{17}H_{16}O_5$ **o1325**