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(*E*)-1-(2-Hydroxyphenyl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.040
wR factor = 0.035
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.

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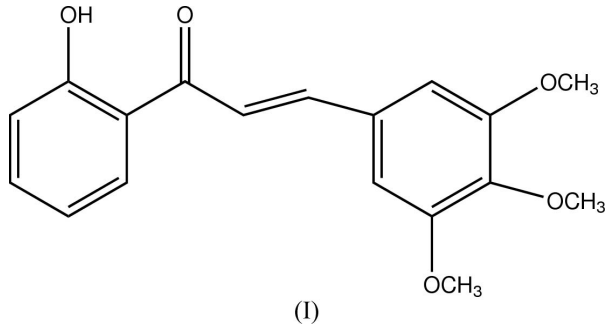
The title compound, $C_{18}H_{18}O_5$, was prepared by the condensation of 2-hydroxyacetophenone with 3,4,5-trimethoxybenzaldehyde. The trimethoxyphenyl and hydroxyphenyl rings of the chalcone system are approximately coplanar.

Comment

Chalcones, particularly those with hydroxy substituents, are important components of numerous natural products that show interesting biological and pharmacological activities (Kumar *et al.*, 2003; Liu *et al.*, 2001). They are also important intermediates in organic synthesis, such as in the use of 2-hydroxychalcones in the synthesis of flavanones (Chaturvedi *et al.*, 1992). We report here the structure of the title chalcone, (I).



(I)

The two aromatic rings are nearly coplanar [interplanar angle 15.33 (12)°]. Furthermore, the hydroxyphenyl ring subtends an angle of 5.39 (14)° at the central C—C═C—C section of the molecule; the corresponding angle for the methoxyphenyl ring is 9.95 (14)°, with the two benzene rings rotated in opposite directions. A classic intramolecular hydrogen-bonding interaction (Table 2) involves the hydroxy group and the adjacent ketone O atom to form a six-membered ring that promotes the planarity of the molecule.

Experimental

Compound (I) was prepared through condensation of 2-hydroxyacetophenone (5 mmol, 1.57 g) with 3,4,5-trimethoxybenzaldehyde (5 mmol, 0.68 g) in 20% NaOH solution (1 ml), using phase transfer TBAB (tetrabutylammonium bromide; 0.75 mmol, 0.25 g) under microwave irradiation for 5 min (yield 73%, m.p. 419–421 K). The reaction mixture was poured into water (100 ml) and filtered. After the usual work-up, the product was purified by chromatography on silica gel and crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a 95% ethanol solution.

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.32
Monoclinic, $P2_1/c$
a = 12.686 (2) Å
b = 8.588 (1) Å
c = 15.422 (3) Å
β = 108.00 (1)°
V = 1598.1 (5) Å^3
Z = 4
D_x = 1.307 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 33 reflections
θ = 3.0–14.5°
μ = 0.10 mm^{-1}
T = 296 (2) K
Block, yellow
0.35 × 0.30 × 0.16 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
3470 measured reflections
2989 independent reflections
984 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.5°
$h = 0 \rightarrow 15$
$k = 0 \rightarrow 10$
$l = -18 \rightarrow 17$
3 standard reflections
every 97 reflections
intensity decay: 1.9%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.035
S = 0.80
2989 reflections
216 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.001P)^2 + 0.075P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0061 (3)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.350 (3)	C8—C9	1.323 (2)
O1—H1*O*	0.832 (10)	C9—C10	1.466 (3)
O2—C7	1.239 (3)		
C12—O3—C16	117.7 (2)	O2—C7—C6	119.9 (3)
C13—O4—C17	113.1 (2)	C8—C9—C10	127.8 (3)
O2—C7—C8	120.4 (3)		
O1—C1—C2—C3	−179.7 (3)	O2—C7—C8—C9	8.2 (4)
C3—C4—C5—C6	0.0 (4)	C8—C9—C10—C15	1.1 (4)
O1—C1—C6—C7	1.6 (4)	C17—O4—C13—C14	77.0 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···O2	0.83 (1)	1.79 (2)	2.521 (3)	146 (3)

Crystals of (I) were weakly diffracting, with only 33% of the reflections considered to be observed. However, this fact did not adversely affect the solution and refinement processes. With the exception of H1*O*, which was located and freely refined, H atoms were positioned geometrically and allowed to ride on their parent atoms at C—H distances of 0.93 or 0.96 Å with $U_{iso(H)} = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997*b*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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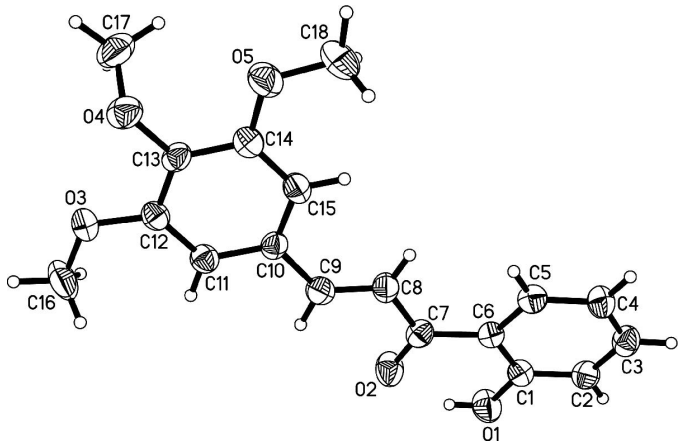

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

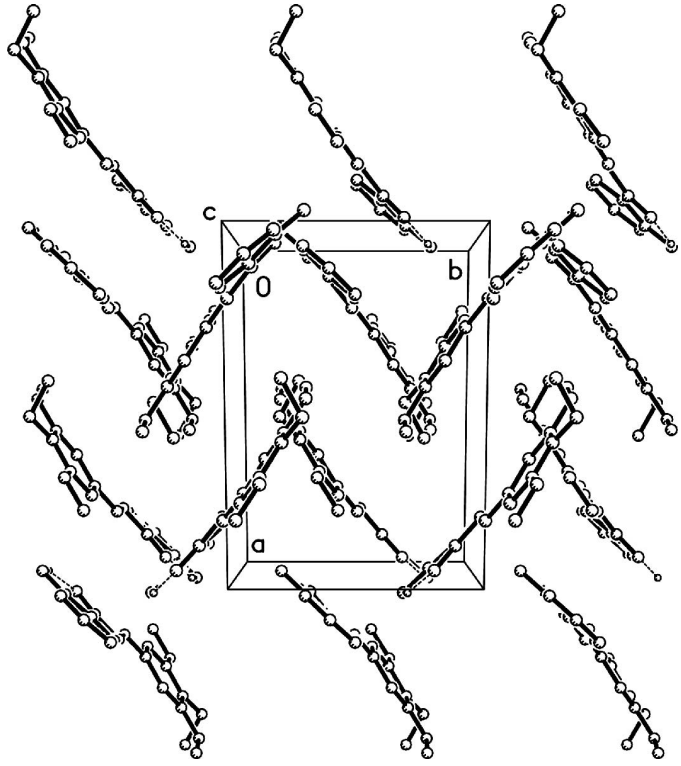

Figure 2
The molecular packing of (I). H atoms have been omitted.

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