

# 1-(2-Hydroxy-4-methoxyphenyl)- 3-(2,3,4-trimethoxyphenyl)prop- 2-en-1-one

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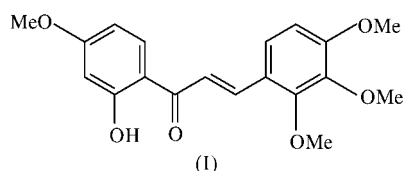
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The title compound, C<sub>19</sub>H<sub>20</sub>O<sub>6</sub>, crystallizes in the centrosymmetric space group *P2<sub>1</sub>/c* with one molecule in the asymmetric unit. The molecule is approximately planar and the dihedral angle between the phenyl rings is 11.0 (1)°. The H atoms of the central propenone group are *trans*. There is an intramolecular O—H···O hydrogen bond and the molecules are crosslinked by four intermolecular C—H···O hydrogen bonds, producing a three-dimensional network.

## Comment

Chalcone derivatives possess non-linear optical properties (Fichou *et al.*, 1988). 1,3-Diarylprop-2-enones (chalcones) are of great interest due to their biological activities, *viz.* antibacterial, antifungal and anti-inflammatory (Ahluwalia *et al.*, 1986; Bhat *et al.*, 1972; Mathew *et al.*, 1984; Oganessian *et al.*, 1986), and have been widely used as starting materials in many synthetic reactions (Awad *et al.*, 1960; Coudert *et al.*, 1988; Carrie & Rochard, 1963). The main feature of chalcone derivatives is the carbonyl functional group, which plays a significant role in the antibacterial activity of these compounds. Against this background and in order to obtain detailed information of the solid-state structures, an X-ray study of the title compound, (I), was carried out.



The molecular structure of (I) (Fig. 1) consists of two benzene rings attached to a propenone chain at the 1,3-posi-

tions. The molecule is approximately planar, with a dihedral angle between the two benzene rings of 11.0 (1)°. The two benzene rings make dihedral angles of 3.5 (1) and 8.6 (1)° with the O4=C7—C8=C9 group. An analysis of the weighted least-squares plane through the central C6—C7(=O4)—C8—C9—C10 chain shows that it is planar, with the largest displacement being 0.011 (2) Å for C8. The H atoms at C8 and C9 are *trans*.

The bond lengths O1—C13, O2—C14, O3—C15, O5—C1, O6—C3, C8=C9 and O4=C7 (Table 1) are comparable with the corresponding values in a similar structure (Sharma *et al.*, 1997). The O1 and O6 methoxy groups, which are both in *para* positions with respect to the point of attachment of the central chain to the benzene rings, show the tendency observed for anisoles to be coplanar with their attached benzene rings; these methoxy groups are oriented in opposite directions with respect to the central chain. The two methoxy groups (O2 and

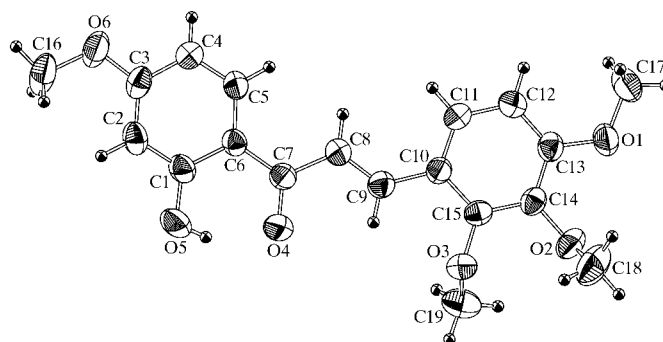


Figure 1

The molecular structure of the title compound (ZORTEP; Zsolnai, 1997), shown with 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

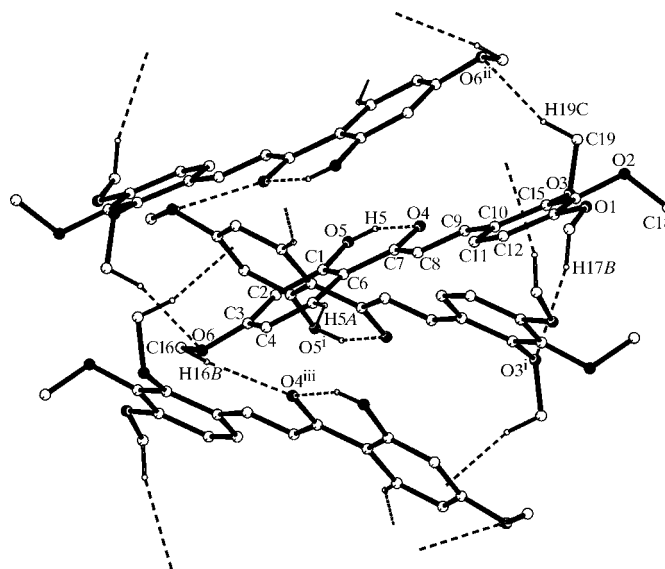


Figure 2

A plot showing a fragment of the hydrogen-bonded network. The hydrogen bonding is shown as dashed lines and the symmetry codes are as in Table 2. For the sake of clarity, H atoms not participating in hydrogen bonding have been omitted.

O3) adjacent to O1 are nearly perpendicular and directed on opposite sides of their attached benzene ring. This is due to steric hindrance, the O2...O3 contact distance being 2.780 (2) Å. The orientation of the O3 group is determined by the presence of the adjacent O2 group [O1...O2 = 2.661 (2) Å], while that of the O6 group involves an H16B...H2 contact of 2.29 Å.

The average O—C<sub>ar</sub> [1.373 (3) Å] and O—C<sub>sp</sub><sup>3</sup> [1.420 (3) Å] bond distances agree well with the corresponding literature values of 1.375 (3) and 1.421 (6) Å, respectively (Domiano *et al.*, 1979). The C—O distances of the two methoxy groups orthogonal to the benzene rings are systematically different; O2—C18 and O3—C19 [average 1.414 (4) Å] tend to be shorter than O1—C17 and O6—C16 [average 1.424 (4) Å], while O2—C14 and O3—C15 [average 1.378 (3) Å] tend to be longer than O1—C13 and O6—C3 [average 1.368 (3) Å]. These differences can be interpreted as being due to reduced phenyl—O-atom conjugation for the orthogonal O2 and O3 methoxy groups with respect to the nearly coplanar O1 and O6 groups.

Both pairs of exocyclic angles, *viz.* C1—C6—C7/C5—C6—C7 of 119.1 (2)/124.0 (2)° and C15—C10—C9/C11—C10—C9 of 119.2 (2)/123.2 (2)°, are asymmetric and this is due to the H5A...H8 (2.15 Å) and H11...H8 (2.33 Å) repulsive contacts for those larger than 120° and due to the O5—H5...O4 and C9—H9...O3 attractive contacts for those narrower than 120°. The C1—C6—C5 [116.9 (2)°] and C11—C10—C15 [117.6 (2)°] endocyclic angles at the points of attachment of the benzene rings to the central chain are less than 120° because of hybridization and VSEPR (valence-shell electron-pair repulsion) effects (Domenicano *et al.*, 1975*a,b*). The deformation of the endocyclic angles is quite similar in the two benzene rings. Interestingly, the endocyclic angles of the two benzene rings are practically equal according to the following values (the atom labels given are the central atoms of the endocyclic angles): C1/C15 = 120.9 (2)/120.9 (2)°, C2/C14 = 119.7 (2)/119.9 (2)°, C3/C13 = 120.8 (2)/119.9 (2)°, C4/C12 = 119.4 (2)/119.6 (2)°, C5/C11 = 122.1 (2)/122.1 (2)° and C6/C10 = 116.9 (2)/117.6 (2)°.

In the solid state, the title molecule is characterized by an intramolecular O5—H5...O4 hydrogen bond in which the hydroxy O atom acts as a donor to the adjacent keto O atom. This hydrogen bond is responsible for the coplanarity of the C1—C6 benzene ring with the central propenone chain. This hydrogen bond completes a six-membered ring with atoms O4, C7, C6, C1 and O5 [graph-set descriptor *S*(6); Bernstein *et al.*, 1995], which adopts a planar conformation.

In addition to normal van der Waals interactions, the crystal packing is stabilized by intermolecular C—H...O hydrogen bonds. Atoms C5 and C17 in the molecule at (*x*, *y*, *z*) act as hydrogen-bond donors to O5 and O3, respectively, in the molecule at (*x*,  $\frac{1}{2} - y$ ,  $\frac{1}{2} + z$ ). These two hydrogen bonds form an *R*<sub>2</sub><sup>2</sup>(22) ring. The molecules at (*x*, *y*, *z*) and (−*x*, 1 − *y*, 1 − *z*) are linked by C19—H19C...O6 hydrogen bonds into cyclic centrosymmetric *R*<sub>2</sub><sup>2</sup>(26) dimers. Atom C16 in the molecule at (*x*, *y*, *z*) donates one proton to atom O4 in the molecule at (−*x*,  $-\frac{1}{2} + y$ ,  $\frac{1}{2} - z$ ), forming a *C*(9) chain. Thus, the

symmetry-related molecules crosslinked by these hydrogen bonds generate a three-dimensional network (Fig. 2). The geometric details of the hydrogen bonds are given in Table 2.

## Experimental

2-Hydroxy-4-methoxyacetophenone was dissolved in ethanol and crushed KOH was added. The flask was immersed in a bath of crushed ice and a solution of 2,3,4-trimethoxybenzaldehyde in ethanol was added. The reaction mixture was stirred at 300 K and completion of the reaction was monitored by thin-layer chromatography. Ice-cold water was added to the reaction mixture after 48 h and the yellow solid that separated was filtered off, washed with water and cold ethanol, dried and purified by column chromatography on silica gel. Crystals of the title compound were obtained from a mixture of methanol and chloroform (9:1) by slow evaporation.

### Crystal data

C <sub>19</sub> H <sub>20</sub> O <sub>6</sub>	<i>D</i> <sub>x</sub> = 1.331 Mg m <sup>−3</sup>
<i>M</i> <sub>r</sub> = 344.35	Mo <i>K</i> α radiation
Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>	Cell parameters from 5955 reflections
<i>a</i> = 16.6025 (6) Å	<i>a</i> = 16.6025 (6) Å
<i>b</i> = 8.1908 (2) Å	<i>b</i> = 8.1908 (2) Å
<i>c</i> = 12.6432 (5) Å	<i>c</i> = 12.6432 (5) Å
$\beta$ = 92.3030 (10)°	$\beta$ = 92.3030 (10)°
<i>V</i> = 1717.93 (10) Å <sup>3</sup>	<i>V</i> = 1717.93 (10) Å <sup>3</sup>
<i>Z</i> = 4	<i>Z</i> = 4
	Rectangular slab, yellow
	0.32 × 0.28 × 0.20 mm

### Data collection

Siemens SMART CCD area-detector diffractometer	2285 reflections with <i>I</i> > 2σ( <i>I</i> )
$\omega$ scans	<i>R</i> <sub>int</sub> = 0.063
Absorption correction: empirical ( <i>SADABS</i> ; Sheldrick, 1996)	$\theta_{\max}$ = 28.3°
<i>T</i> <sub>min</sub> = 0.969, <i>T</i> <sub>max</sub> = 0.980	<i>h</i> = −22 → 22
11 685 measured reflections	<i>k</i> = −10 → 5
4236 independent reflections	<i>l</i> = −16 → 15
	Intensity decay: <1%

### Refinement

Refinement on <i>F</i> <sup>2</sup>	H-atom parameters constrained
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.058	<i>w</i> = 1/[σ <sup>2</sup> ( <i>F</i> <sub>o</sub> <sup>2</sup> ) + (0.06 <i>P</i> ) <sup>2</sup> ]
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.148	where <i>P</i> = ( <i>F</i> <sub>o</sub> <sup>2</sup> + 2 <i>F</i> <sub>c</sub> <sup>2</sup> )/3
<i>S</i> = 1.02	(Δ/σ) <sub>max</sub> < 0.001
4236 reflections	Δρ <sub>max</sub> = 0.20 e Å <sup>−3</sup>
226 parameters	Δρ <sub>min</sub> = −0.31 e Å <sup>−3</sup>

**Table 1**

Selected geometric parameters (Å, °).

O1—C13	1.370 (2)	O4—C7	1.246 (2)
O1—C17	1.423 (2)	O5—C1	1.337 (2)
O2—C14	1.376 (2)	O6—C3	1.367 (2)
O2—C18	1.411 (3)	O6—C16	1.425 (3)
O3—C15	1.380 (2)	C8—C9	1.323 (3)
O3—C19	1.416 (3)		
C13—O1—C17	117.6 (2)	C5—C6—C1	116.9 (2)
C14—O2—C18	115.4 (2)	C5—C6—C7	124.0 (2)
C15—O3—C19	116.8 (2)	C1—C6—C7	119.1 (2)
C3—O6—C16	117.8 (2)	C6—C7—C8	121.8 (2)
O6—C3—C4	115.0 (2)	C8—C9—C10	129.9 (2)
O6—C3—C2	124.2 (2)	C11—C10—C9	123.2 (2)
C16—O6—C3—C4	179.1 (2)	C7—C8—C9—C10	177.6 (2)
C16—O6—C3—C2	−2.1 (3)	C8—C9—C10—C15	175.0 (2)
C1—C6—C7—O4	−1.3 (3)	C17—O1—C13—C12	9.1 (3)
O4—C7—C8—C9	−0.6 (3)	C18—O2—C14—C13	97.3 (2)
C6—C7—C8—C9	178.5 (2)	C19—O3—C15—C14	−72.7 (2)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O5—H5...O4	0.82	1.78	2.503 (2)	147
C9—H9...O4	0.93	2.33	2.715 (2)	105
C9—H9...O3	0.93	2.40	2.789 (2)	105
C19—H19B...O2	0.96	2.46	3.051 (3)	120
C5—H5A...O5 <sup>i</sup>	0.93	2.45	3.315 (2)	154
C17—H17B...O3 <sup>i</sup>	0.96	2.56	3.449 (3)	154
C19—H19C...O6 <sup>ii</sup>	0.96	2.53	3.378 (3)	147
C16—H16B...O4 <sup>iii</sup>	0.96	2.41	3.301 (3)	155

 Symmetry codes: (i)  $x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (ii)  $-x, 1 - y, 1 - z$ ; (iii)  $-x, y - \frac{1}{2}, \frac{1}{2} - z$ .

All H atoms were fixed geometrically and allowed to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93 or 0.96 Å, and with  $U_{iso} = 1.5U_{eq}$ (methyl H) and  $1.2U_{eq}$ (other H).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1595). Services for accessing these data are described at the back of the journal.

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