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(*E*)-3-(2,3-Dimethoxyphenyl)-1-(2-hydroxy-5-methoxyphenyl)prop-2-en-1-one

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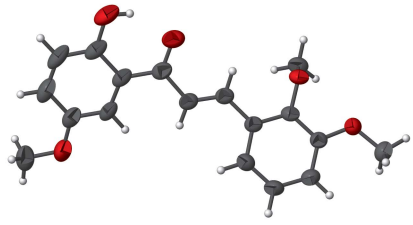
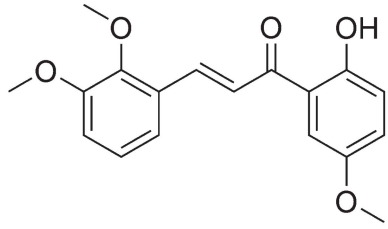
Structural data: full structural data are available from iucrdata.iucr.org

In the title chalcone derivative, $C_{18}H_{18}O_5$, the dihedral angle formed by the planes of the benzene rings is 29.6 (2)° and an intramolecular O—H···O hydrogen bond closes an *S*(6) ring. In the crystal, weak C—H···O hydrogen bonds link molecules into chains propagating along [001].

3D view



Chemical scheme



Structure description

Chalcones are α,β-unsaturated carbonyl (enone) compounds that connect two aromatic components. They are one of the secondary metabolites found in plants with a C6—C3—C6 skeleton. In this chalcone, the C3 skeleton is an open chain but in other metabolites such as flavones, the C3 skeleton is a closed chain. A variety of chalcones have been isolated from natural sources and synthesized due to their wide spectrum of biological activities (Singh *et al.*, 2014). In a continuation of research to develop new chalcones that show a broad range of biological activities (Jung *et al.*, 2015), the crystal structure of title compound (Fig. 1) has been determined. The structure of a related substituted chalcone compound was reported by Srividya *et al.* (2015).

The *trans* conformation of the C9═C10 double bond in the central enone group is confirmed by the C1—C9═C10—C11 torsion angle of 173.37 (17)°. The dihedral angle between the planes of the benzene rings is 29.6 (2)°. The C1═O1 double bond [1.239 (2) Å] is longer than the normal value as this group accepts an intramolecular hydrogen bond from the hydroxy group, thereby forming an *S*(6) ring (Table 1). Among the three methoxy groups attached to the benzene rings, the two groups at *meta* positions to the central enone group are tilted slightly [C5—C6—O3—C8 = −1.2 (3)° and C14—C13—O5—C18 = −7.5 (2)°] from their attached ring. However, the C atom of the group at the *ortho* position is orthogonal to the benzene ring [C13—C12—O4—C17 = 90.7 (2)°].

In the crystal, weak C—H···O hydrogen bonds link molecules into chains propagating along [001] (Fig. 2).

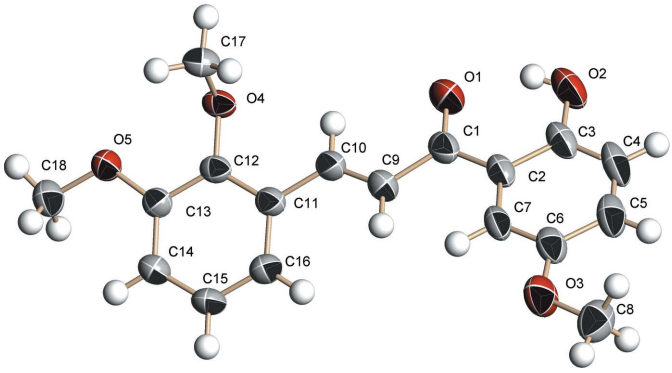


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

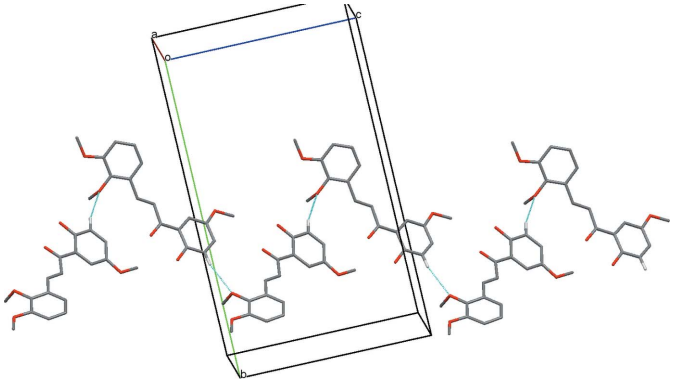


Figure 2
Part of the crystal structure of the title compound, showing the weak C—H···O hydrogen bonds as dashed lines. H atoms not involved in these interactions have been omitted for clarity.

Synthesis and crystallization

To a solution of 2,3-dimethoxybenzaldehyde (392 mg, 2 mmol) in 20 ml of anhydrous ethanol was added 2-hydroxy-5-methoxyacetophenone (332 mg, 2 mmol) and the temperature was adjusted to around 276–277 K in an ice bath. To the cooled reaction mixture was added 2 ml of 50% aqueous KOH solution, and the reaction mixture was stirred at room temperature for 27 h. This mixture was poured into iced water (80 ml) and was acidified with 4 *N* HCl solution to give a precipitate. Filtration and washing with water afforded the crude solid of the title compound (yield 270 mg, 72%). Recrystallization of the solid from ethanol solution gave orange blocks (m.p. 370–371 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.83	1.81	2.543 (2)	146
C4—H4···O4^{i}	0.94	2.57	3.468 (2)	161

Symmetry code: (i) $x-1, -y+\frac{3}{2}, z+\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{18}O_5$
M_r	314.32
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	223
a, *b*, *c* (Å)	4.6056 (3), 24.7911 (13), 13.7523 (7)
β (°)	98.751 (3)
V (Å^3)	1551.93 (15)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.15 × 0.12 × 0.07
Data collection	
Diffractometer	Bruker PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2000)
T_{min}, T_{max}	0.985, 0.993
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	68518, 3870, 2340
R_{int}	0.100
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.050, 0.126, 1.02
No. of reflections	3870
No. of parameters	212
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.21

Computer programs: *APEX2* and *SAINT* (Bruker, 2000) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161492 [https://doi.org/10.1107/S2414314616014929]

(*E*)-3-(2,3-Dimethoxyphenyl)-1-(2-hydroxy-5-methoxyphenyl)prop-2-en-1-one

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(*E*)-3-(2,3-Dimethoxyphenyl)-1-(2-hydroxy-5-methoxyphenyl)prop-2-en-1-one

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.6056 (3) Å
b = 24.7911 (13) Å
c = 13.7523 (7) Å
β = 98.751 (3)°
V = 1551.93 (15) Å^3
Z = 4
$F(000)$ = 664
D_x = 1.345 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9647 reflections
θ = 2.2–24.1°
μ = 0.10 mm^{-1}
T = 223 K
Block, orange
0.15 × 0.12 × 0.07 mm

Data collection

Bruker PHOTON 100 CMOS diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
T_{min} = 0.985, T_{max} = 0.993
68518 measured reflections
3870 independent reflections
2340 reflections with $I > 2\sigma(I)$
R_{int} = 0.100
θ_{max} = 28.4°, θ_{min} = 2.2°
h = −6→6
k = −33→33
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.126
S = 1.02
3870 reflections
212 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.4891P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7490 (4)	0.69540 (7)	0.89539 (13)	0.0489 (5)
O1	0.8227 (4)	0.72888 (5)	0.83684 (10)	0.0707 (4)
C2	0.5807 (4)	0.71236 (7)	0.97281 (13)	0.0463 (4)
C3	0.5374 (4)	0.76761 (7)	0.98920 (15)	0.0547 (5)
O2	0.6399 (4)	0.80615 (5)	0.93304 (12)	0.0727 (4)
H2	0.7084	0.7916	0.8871	0.109*
C4	0.3920 (5)	0.78349 (8)	1.06494 (18)	0.0655 (6)
H4	0.3710	0.8205	1.0772	0.079*
C5	0.2771 (4)	0.74642 (9)	1.12297 (16)	0.0628 (6)
H5	0.1766	0.7581	1.1737	0.075*
C6	0.3100 (4)	0.69169 (8)	1.10626 (15)	0.0538 (5)
C7	0.4625 (4)	0.67553 (7)	1.03285 (13)	0.0490 (5)
H7	0.4881	0.6384	1.0227	0.059*
O3	0.1997 (3)	0.65114 (6)	1.15815 (11)	0.0715 (4)
C8	0.0446 (5)	0.66650 (10)	1.23604 (16)	0.0731 (6)
H8A	0.1769	0.6853	1.2865	0.110*
H8B	−0.0316	0.6345	1.2641	0.110*
H8C	−0.1170	0.6901	1.2105	0.110*
C9	0.8415 (4)	0.63896 (7)	0.88882 (13)	0.0483 (4)
H9	0.7940	0.6139	0.9353	0.058*
C10	0.9897 (4)	0.62256 (7)	0.81934 (13)	0.0459 (4)
H10	1.0146	0.6478	0.7703	0.055*
C11	1.1186 (4)	0.56948 (6)	0.81078 (12)	0.0418 (4)
C12	1.2194 (4)	0.55511 (6)	0.72359 (12)	0.0390 (4)
C13	1.3518 (4)	0.50506 (6)	0.71390 (12)	0.0400 (4)
C14	1.3921 (4)	0.46977 (7)	0.79275 (13)	0.0493 (4)
H14	1.4852	0.4364	0.7876	0.059*
C15	1.2936 (5)	0.48418 (7)	0.87926 (14)	0.0572 (5)
H15	1.3200	0.4601	0.9327	0.069*
C16	1.1587 (5)	0.53265 (8)	0.88889 (14)	0.0541 (5)
H16	1.0925	0.5413	0.9484	0.065*
O4	1.1979 (3)	0.59122 (4)	0.64707 (8)	0.0437 (3)
C17	0.9323 (4)	0.58633 (8)	0.57896 (13)	0.0519 (5)
H17A	0.9226	0.5508	0.5493	0.078*
H17B	0.9287	0.6135	0.5281	0.078*
H17C	0.7653	0.5914	0.6133	0.078*
O5	1.4313 (3)	0.49495 (5)	0.62408 (9)	0.0483 (3)
C18	1.5974 (4)	0.44727 (7)	0.61420 (15)	0.0557 (5)
H18A	1.7771	0.4482	0.6612	0.084*
H18B	1.6454	0.4453	0.5480	0.084*

H18C	1.4826	0.4159	0.6266	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0567 (11)	0.0368 (9)	0.0496 (11)	−0.0016 (8)	−0.0031 (9)	−0.0031 (8)
O1	0.1060 (12)	0.0399 (7)	0.0672 (9)	0.0002 (7)	0.0168 (9)	0.0057 (7)
C2	0.0452 (10)	0.0341 (9)	0.0543 (11)	0.0057 (8)	−0.0088 (8)	−0.0076 (8)
C3	0.0497 (11)	0.0340 (9)	0.0739 (13)	0.0046 (8)	−0.0110 (10)	−0.0083 (9)
O2	0.0829 (11)	0.0346 (7)	0.0973 (12)	0.0037 (7)	0.0032 (9)	−0.0026 (7)
C4	0.0552 (12)	0.0403 (11)	0.0957 (16)	0.0130 (9)	−0.0056 (12)	−0.0230 (11)
C5	0.0513 (12)	0.0574 (12)	0.0766 (14)	0.0148 (10)	0.0001 (10)	−0.0231 (11)
C6	0.0468 (11)	0.0481 (10)	0.0635 (12)	0.0122 (9)	−0.0009 (9)	−0.0082 (9)
C7	0.0507 (11)	0.0362 (9)	0.0571 (11)	0.0109 (8)	−0.0009 (9)	−0.0098 (8)
O3	0.0770 (10)	0.0630 (9)	0.0802 (11)	0.0197 (8)	0.0303 (8)	−0.0026 (8)
C8	0.0602 (13)	0.0933 (17)	0.0670 (14)	0.0187 (12)	0.0140 (11)	−0.0105 (12)
C9	0.0577 (11)	0.0348 (9)	0.0524 (11)	−0.0001 (8)	0.0084 (9)	0.0014 (8)
C10	0.0587 (11)	0.0355 (9)	0.0427 (10)	−0.0030 (8)	0.0050 (8)	0.0007 (7)
C11	0.0469 (10)	0.0359 (8)	0.0419 (9)	−0.0021 (7)	0.0047 (7)	0.0024 (7)
C12	0.0413 (9)	0.0348 (8)	0.0401 (9)	−0.0066 (7)	0.0034 (7)	0.0050 (7)
C13	0.0420 (9)	0.0360 (9)	0.0422 (9)	−0.0046 (7)	0.0068 (7)	0.0007 (7)
C14	0.0580 (11)	0.0399 (9)	0.0498 (11)	0.0061 (8)	0.0070 (9)	0.0059 (8)
C15	0.0801 (14)	0.0472 (11)	0.0450 (11)	0.0117 (10)	0.0120 (10)	0.0145 (9)
C16	0.0717 (13)	0.0512 (11)	0.0414 (10)	0.0073 (10)	0.0146 (9)	0.0058 (8)
O4	0.0487 (7)	0.0368 (6)	0.0453 (7)	−0.0061 (5)	0.0065 (5)	0.0096 (5)
C17	0.0515 (11)	0.0540 (11)	0.0491 (11)	0.0026 (9)	0.0034 (9)	0.0118 (9)
O5	0.0586 (8)	0.0430 (7)	0.0455 (7)	0.0042 (6)	0.0150 (6)	0.0017 (5)
C18	0.0568 (12)	0.0503 (11)	0.0611 (12)	0.0078 (9)	0.0122 (10)	−0.0054 (9)

Geometric parameters (Å, º)

C1—O1	1.239 (2)	C10—C11	1.456 (2)
C1—O1	1.239 (2)	C10—H10	0.9400
C1—C9	1.470 (2)	C11—C12	1.396 (2)
C1—C2	1.470 (3)	C11—C16	1.401 (2)
C2—C7	1.396 (3)	C12—O4	1.3735 (18)
C2—C3	1.407 (2)	C12—C13	1.398 (2)
C3—O2	1.358 (2)	C13—O5	1.3638 (19)
C3—C4	1.378 (3)	C13—C14	1.384 (2)
O2—H2	0.8300	C14—C15	1.383 (3)
C4—C5	1.375 (3)	C14—H14	0.9400
C4—H4	0.9400	C15—C16	1.368 (3)
C5—C6	1.388 (3)	C15—H15	0.9400
C5—H5	0.9400	C16—H16	0.9400
C6—O3	1.374 (2)	O4—C17	1.428 (2)
C6—C7	1.374 (3)	C17—H17A	0.9700
C7—H7	0.9400	C17—H17B	0.9700
O3—C8	1.427 (2)	C17—H17C	0.9700

C8—H8A	0.9700	O5—C18	1.426 (2)
C8—H8B	0.9700	C18—H18A	0.9700
C8—H8C	0.9700	C18—H18B	0.9700
C9—C10	1.320 (2)	C18—H18C	0.9700
C9—H9	0.9400		

O1—C1—C9	119.15 (18)	C9—C10—H10	116.6
O1—C1—C9	119.15 (18)	C11—C10—H10	116.6
O1—C1—C2	120.38 (16)	C12—C11—C16	118.04 (16)
O1—C1—C2	120.38 (16)	C12—C11—C10	119.54 (15)
C9—C1—C2	120.43 (16)	C16—C11—C10	122.35 (16)
C7—C2—C3	117.66 (18)	O4—C12—C11	119.81 (14)
C7—C2—C1	122.50 (15)	O4—C12—C13	119.15 (14)
C3—C2—C1	119.83 (18)	C11—C12—C13	121.01 (15)
O2—C3—C4	118.67 (17)	O5—C13—C14	124.84 (15)
O2—C3—C2	121.58 (19)	O5—C13—C12	115.50 (14)
C4—C3—C2	119.7 (2)	C14—C13—C12	119.67 (16)
C3—O2—H2	109.5	C15—C14—C13	119.24 (16)
C5—C4—C3	121.45 (18)	C15—C14—H14	120.4
C5—C4—H4	119.3	C13—C14—H14	120.4
C3—C4—H4	119.3	C16—C15—C14	121.55 (17)
C4—C5—C6	119.7 (2)	C16—C15—H15	119.2
C4—C5—H5	120.1	C14—C15—H15	119.2
C6—C5—H5	120.1	C15—C16—C11	120.47 (17)
O3—C6—C7	116.04 (16)	C15—C16—H16	119.8
O3—C6—C5	124.80 (19)	C11—C16—H16	119.8
C7—C6—C5	119.2 (2)	C12—O4—C17	113.69 (13)
C6—C7—C2	122.20 (17)	O4—C17—H17A	109.5
C6—C7—H7	118.9	O4—C17—H17B	109.5
C2—C7—H7	118.9	H17A—C17—H17B	109.5
C6—O3—C8	117.49 (17)	O4—C17—H17C	109.5
O3—C8—H8A	109.5	H17A—C17—H17C	109.5
O3—C8—H8B	109.5	H17B—C17—H17C	109.5
H8A—C8—H8B	109.5	C13—O5—C18	117.41 (13)
O3—C8—H8C	109.5	O5—C18—H18A	109.5
H8A—C8—H8C	109.5	O5—C18—H18B	109.5
H8B—C8—H8C	109.5	H18A—C18—H18B	109.5
C10—C9—C1	121.63 (17)	O5—C18—H18C	109.5
C10—C9—H9	119.2	H18A—C18—H18C	109.5
C1—C9—H9	119.2	H18B—C18—H18C	109.5
C9—C10—C11	126.80 (17)		

C9—C1—O1—O1	0.0 (7)	O1—C1—C9—C10	−3.1 (3)
C2—C1—O1—O1	0.0 (7)	C2—C1—C9—C10	179.19 (17)
O1—C1—C2—C7	172.15 (17)	C1—C9—C10—C11	173.37 (17)
O1—C1—C2—C7	172.15 (17)	C9—C10—C11—C12	167.80 (18)
C9—C1—C2—C7	−10.2 (3)	C9—C10—C11—C16	−15.2 (3)
O1—C1—C2—C3	−8.7 (3)	C16—C11—C12—O4	−176.65 (16)

O1—C1—C2—C3	−8.7 (3)	C10—C11—C12—O4	0.4 (2)
C9—C1—C2—C3	168.97 (16)	C16—C11—C12—C13	1.1 (3)
C7—C2—C3—O2	−178.73 (16)	C10—C11—C12—C13	178.18 (16)
C1—C2—C3—O2	2.0 (3)	O4—C12—C13—O5	−4.5 (2)
C7—C2—C3—C4	2.4 (3)	C11—C12—C13—O5	177.70 (15)
C1—C2—C3—C4	−176.83 (17)	O4—C12—C13—C14	175.79 (15)
O2—C3—C4—C5	178.46 (19)	C11—C12—C13—C14	−2.0 (3)
C2—C3—C4—C5	−2.6 (3)	O5—C13—C14—C15	−178.06 (17)
C3—C4—C5—C6	0.8 (3)	C12—C13—C14—C15	1.6 (3)
C4—C5—C6—O3	−178.65 (19)	C13—C14—C15—C16	−0.3 (3)
C4—C5—C6—C7	1.2 (3)	C14—C15—C16—C11	−0.5 (3)
O3—C6—C7—C2	178.49 (16)	C12—C11—C16—C15	0.2 (3)
C5—C6—C7—C2	−1.4 (3)	C10—C11—C16—C15	−176.85 (19)
C3—C2—C7—C6	−0.4 (3)	C11—C12—O4—C17	−91.57 (18)
C1—C2—C7—C6	178.80 (17)	C13—C12—O4—C17	90.65 (19)
C7—C6—O3—C8	178.92 (17)	C14—C13—O5—C18	−7.5 (2)
C5—C6—O3—C8	−1.2 (3)	C12—C13—O5—C18	172.89 (15)
O1—C1—C9—C10	−3.1 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1	0.83	1.81	2.543 (2)	146
C4—H4···O4^{i}	0.94	2.57	3.468 (2)	161

Symmetry code: (i) $x-1, -y+3/2, z+1/2$.