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(*E*)-4-(4-Hydroxy-3-methoxyphenyl)but-3-en-2-one

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The title compound, $C_{11}H_{12}O_3$, was synthesized from 4-hydroxy-3-methoxybenzaldehyde and acetone. The molecule has a high degree of conjugation throughout the system and intermolecular hydrogen bonds connect adjacent molecules to form one-dimensional chains.

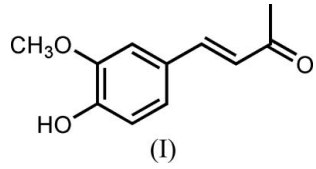
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.140
Data-to-parameter ratio = 18.0

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

Comment

α,β-Unsaturated ketones are an important class of pharmaceutical intermediates and have extensive application in the synthesis of natural products and drugs by 1,4 addition. As a precursor for further synthesis, we have synthesized the title compound, (I).



(I)

All the bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987) and the structural data confirm the *E* configuration about the C3═C4 double bond. Atoms C2, C3, C4 and O1 constitute a well defined plane and the benzene ring plane is inclined at 5.34 (1)° to this plane (Fig. 1). A weak intermolecular O2—H2···O1 hydrogen bond links adjacent molecules, forming one-dimensional chains (Fig. 2).

Experimental

4-Hydroxy-3-methoxybenzaldehyde (3.04 g, 20 mmol) was dissolved in 25 ml of acetone and 12 ml of dilute aqueous NaOH solution (10%) was added to the acetone solution with stirring. The mixture was allowed to stand overnight at room temperature and then the mixture was acidified with dilute aqueous HCl to give (*E*)-4-(4-hydroxy-3-methoxyphenyl)but-3-en-2-one as a yellow solid (yield 73%). The resultant precipitate was filtered off, washed with water and recrystallized from ethanol and dichloromethane (2:1), in air over a period of four days. After about three-quarters of the original solvent had evaporated, large colourless prisms of (I) were obtained (yield 61%).

Crystal data

$C_{11}H_{12}O_3$
M_r = 192.21
Monoclinic, $P2_1/c$
a = 9.602 (5) Å
b = 7.780 (4) Å
c = 13.478 (7) Å
β = 97.466 (8)°
V = 998.2 (9) Å^3
Z = 4
D_x = 1.279 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2995 reflections
θ = 2.6–27.9°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colourless
0.36 × 0.28 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer	1816 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.020$
Absorption correction: none	$\theta_{max} = 28.4^\circ$
8188 measured reflections	$h = -12 \rightarrow 12$
2355 independent reflections	$k = -10 \rightarrow 10$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.1629P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.046$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.140$	$\Delta\rho_{max} = 0.20$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.19$ e Å^{-3}
2355 reflections	Extinction correction: *SHELXL97*
131 parameters	Extinction coefficient: 0.024 (5)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O2—C8	1.3556 (18)	C10—C9	1.3785 (19)
C4—C3	1.336 (2)	C8—C7	1.380 (2)
C4—C5	1.459 (2)	C8—C9	1.394 (2)
C5—C6	1.388 (2)	C6—C7	1.379 (2)
C5—C10	1.400 (2)	O1—C2	1.2243 (18)
O3—C9	1.3686 (17)	C3—C2	1.454 (2)
O3—C11	1.413 (2)	C2—C1	1.490 (2)
C3—C4—C5	127.19 (15)	O3—C9—C10	125.48 (13)
C6—C5—C10	118.55 (12)	O3—C9—C8	114.63 (12)
C6—C5—C4	122.60 (13)	C10—C9—C8	119.89 (13)
C10—C5—C4	118.85 (13)	C7—C6—C5	120.63 (14)
C9—O3—C11	117.63 (12)	C4—C3—C2	124.57 (15)
C9—C10—C5	120.79 (13)	O1—C2—C3	119.85 (15)
O2—C8—C7	118.81 (13)	O1—C2—C1	119.74 (14)
O2—C8—C9	121.70 (13)	C3—C2—C1	120.40 (14)
C7—C8—C9	119.48 (13)	C6—C7—C8	120.64 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.82	2.08	2.772 (2)	141

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

H atoms were positioned geometrically [C—H distances of 0.93 (Csp^2—H) and 0.96 Å (methyl), and O—H = 0.82 Å]. U_{iso}(H) values were set equal to xU_{eq}(carrier atom), where $x = 1.2$ for CH and 1.5 for O and methyl C.

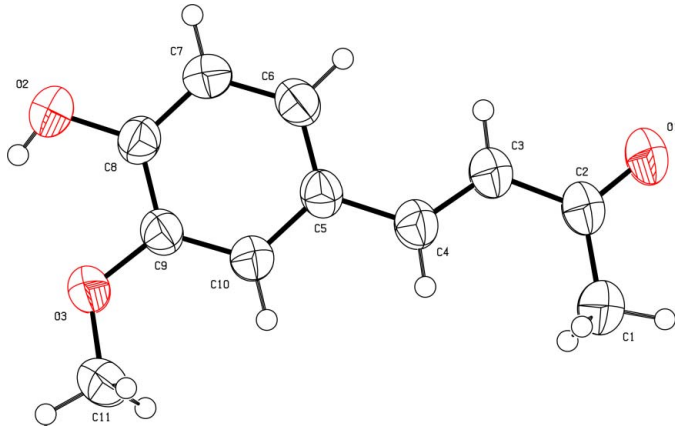


Figure 1
The structure of the title compound (1), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

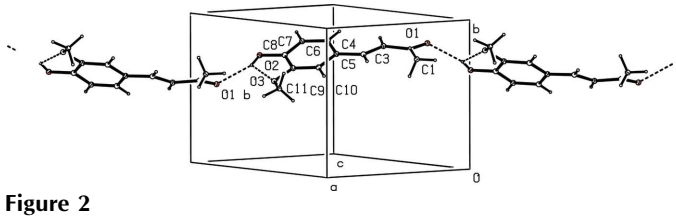


Figure 2
A view of part of the crystal packing. Dashed lines indicate hydrogen bonds.

Data collection: *SMART* (Bruker, 2002); 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: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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