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4′-Hydroxy-3′-methoxyacetophenone (acetovanillone)

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
H-atom completeness 91%
R factor = 0.041
wR factor = 0.122
Data-to-parameter ratio = 15.5

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

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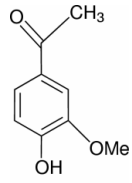
The crystal structure of acetovanillone, $C_9H_{10}O_3$, has been determined at 173 (1) K. All the C and O atoms are essentially coplanar, and the molecules pack in parallel planes as a result of intermolecular hydrogen bonds (C—H···O and O—H···O).

Comment

The title compound, (I), first synthesized in 1949 (Berlin *et al.*, 1949), is a popular model compound used to represent ketonic moieties in lignin; it has recently been used as a substrate to test the hydrogenation of several platinum metal-based systems (Hu & James, 2002). The molecular structure of (I) is shown in Fig. 1, and selected geometric parameter are listed in Table 1.



(I)

Four different C—O bond lengths are found in the molecule: C8—O3 1.230 (2) Å, C2—O2 1.372 (2) Å, C1—O1 1.353 (2) Å and C7—O2 1.431 (2) Å. The orientations of the carbonyl, methyl and methoxy groups with respect to the aromatic ring are defined by the torsion angles O1—C1—C2—C3 −179.3 (1)°, O2—C2—C1—C6 179.5 (1)°, O3—C8—C4—C3 −173.0 (1)°, C6—C5—C4—C8 −179.5 (1)° and C1—C2—O2—C7 179.5 (1)°. The groups attached to the aromatic ring deviate slightly from coplanarity with the ring. Two kinds of intermolecular hydrogen bonds exist in the crystal structure, O—H···O and C—H···O (Table 2), and these lead to parallel packing of the molecules (Fig. 2).

Experimental

The title compound was crystallized from a solution of 0.1 g of acetovanillone in 5 ml water, heated to totally dissolve the material. When the solution was cooled to room temperature, colorless crystals formed.

Crystal data

$C_9H_{10}O_3$
M_r = 166.18
Monoclinic, $P2_1/n$
a = 8.5144 (8) Å
b = 8.778 (1) Å
c = 10.712 (1) Å
β = 90.082 (5)°
V = 800.7 (1) Å^3
Z = 4
D_x = 1.378 Mg m^{-3}
Mo Kα radiation
Cell parameters from 4394 reflections
θ = 2.3–27.9°
μ = 0.10 mm^{-1}
T = 173.2 K
Prism, colorless
0.50 × 0.30 × 0.10 mm

Data collection

Rigaku/ADSC CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*d*TREK*; Molecular Structure Corporation, 2001)
T_{min} = 0.703, T_{max} = 1.000
6942 measured reflections
1860 independent reflections
1334 reflections with $I > 3\sigma(I)$
R_{int} = 0.039
θ_{max} = 27.9°
$h = -10 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2] > 2\sigma(F^2)$] = 0.041
$wR(F^2)$ = 0.122
S = 1.20
1748 reflections
113 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 0.00093|F_o|^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.353 (2)	C2—C3	1.381 (2)
O2—C2	1.372 (2)	C3—C4	1.404 (2)
O2—C7	1.431 (2)	C4—C5	1.387 (2)
O3—C8	1.230 (2)	C4—C8	1.486 (2)
C1—C2	1.404 (2)	C5—C6	1.385 (2)
C1—C6	1.388 (2)	C8—C9	1.495 (2)
C2—O2—C7	116.8 (1)	C3—C4—C5	119.3 (1)
O1—C1—C2	122.0 (1)	C3—C4—C8	121.7 (1)
O1—C1—C6	118.4 (1)	C5—C4—C8	119.0 (1)
C2—C1—C6	119.6 (1)	C4—C5—C6	120.6 (1)
O2—C2—C1	114.0 (1)	C1—C6—C5	120.2 (1)
O2—C2—C3	125.9 (1)	O3—C8—C4	119.9 (1)
C1—C2—C3	120.0 (1)	O3—C8—C9	120.1 (1)
C2—C3—C4	120.2 (1)	C4—C8—C9	120.1 (1)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82 (2)	2.29 (3)	2.682 (1)	110 (2)
O1—H1···O3^{i}	0.82 (2)	2.00 (3)	2.673 (1)	139 (2)
C9—H9B···O1^{ii}	0.98	2.67	3.497 (2)	143

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

The absorption correction is based on a three-dimensional analysis of symmetry-equivalent data and is performed along with batch scaling in a single step. The resulting transmission factors, therefore, include contributions from absorption, crystal decay, and detectable variations in beam intensity. Hydroxyl atom H1 was refined, while all other H atoms were placed in calculated positions and refined in riding mode.

Data collection: *d*TREK* (Molecular Structure Corporation, 2001); cell refinement: *d*TREK*; data reduction: *d*TREK*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1999); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1985–1992); molecular graphics: *TEXSAN*; software used to prepare material for publication: *TEXSAN*.

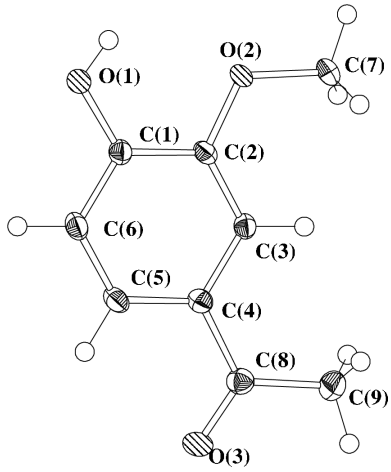


Figure 1
The molecular structure of acetovanillone, with the atom-numbering scheme and ellipsoids at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

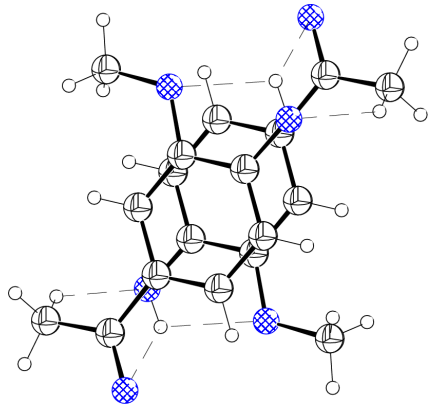


Figure 2
Packing diagram of acetovanillone, viewed down the *b* axis. O—H···O and C—H···O hydrogen bonds are shown as dashed lines, and all atoms are shown as spheres.

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