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3-[(4-Hydroxy-3-methoxyphenyl)methylene]-2,4-pentanedione

Li-Xia Pei,[a] Xian-Zhang Bu,[b] Lian-Quan Gu[b] and Seik Weng Ng[c]*

[a]School of Chemistry and Chemical Engineering, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, [b]School of Pharmaceutical Sciences, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

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Key indicators

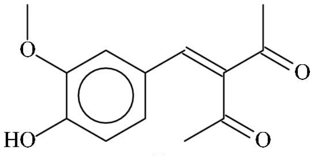
Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.041
wR factor = 0.137
Data-to-parameter ratio = 16.8

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

The molecules of the title compound, $C_{13}H_{14}O_4$, the condensation product of vanillin and acetylacetone, are hydrogen bonded into a linear chain arising from interaction between the hydroxy group of one molecule and the acetyl group of an adjacent molecule.

Comment

The synthesis of 3-[(4-hydroxy-3-methoxyphenyl)methylene]pentane-2,4-dione, (I), was undertaken as this compound is required for the synthesis of curcumin and other synthetic curcumins; curcumin is the principal component of turmeric that gives the spice its color and also its medicinal properties. Curcumin is prepared from the condensation of vanillin and acetylacetone (Pabon, 1964); other synthetic curcumins can be synthesized by using this procedure (Kim & Yang, 2004). Curcumin has been crystallographically characterized as the acetyl derivative (Lozada *et al.*, 2004).



(I)

The title compound can be considered as an alkene having two $CH_3C(O)-$ substituents on one side of the double bond and the $(CH_3O)(OH)C_6H_3-$ unit substituent on the other (Fig. 1). The acetyl group *trans* to the H substituent is not

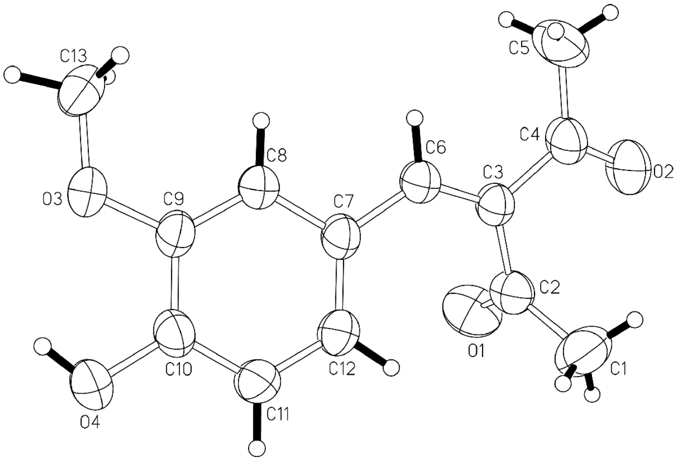


Figure 1
ORTEPII plot (Johnson, 1976) of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

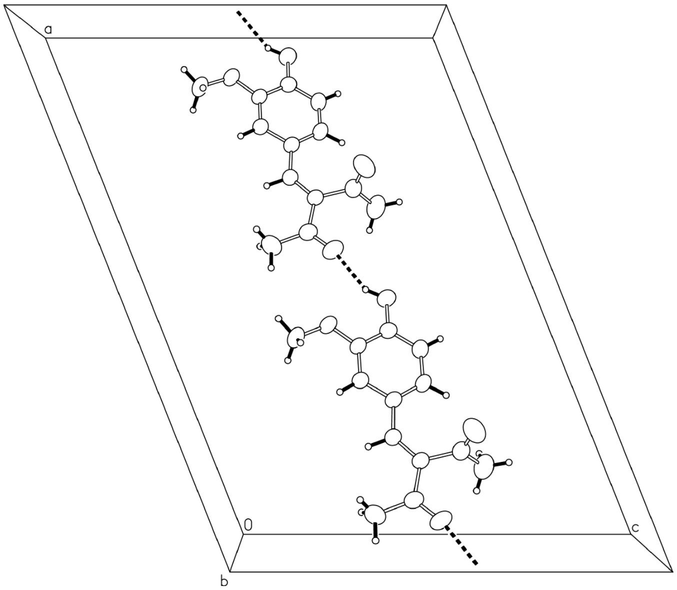


Figure 2
*ORTEP*II plot (Johnson, 1976) of the hydrogen-bonded chain structure in (I). [Symmetry code: (i) $\frac{1}{2}+x$, $y-\frac{1}{2}$, z]

coplanar with the double bond and the aromatic system as a twist is necessary to avoid crowding with the H atom of the aromatic ring. Such a severe twist was also noted in the aromatic compounds 3-[5-(3-nitrophenyl)furfurylidene]pentane-2,4-dione (Lokaj *et al.*, 1994), 3-[5-(2-nitrophenyl)furfurylidene]pentane-2,4-dione (Vrábel *et al.*, 1994) and 1-(2,2-diacetylvinyl)-3-phenylbenzo[*c*]thiophene (Hori *et al.*, 1989), as well as in the aliphatic compounds 3-acetyl-8-phenylocta-3,5,7-trien-2-one (Bai & Hu, 1983) and 3-acetyl-1-phenyl-2-pentene-1,4-dione (Usman *et al.*, 2002). In the title compound, the O atom of the *trans*-acetyl group interacts with the hydroxy group of an adjacent molecule to give rise to a chain structure (Fig. 2).

Experimental

The synthesis of the title compound from acetylacetone and vanillin was based on a report of almost half a century ago by Delest & Palland (1958). Piperidine (0.85 g, 10 mmol) was added to a dimethylformamide solution (30 ml) of acetylacetone (1 ml, 10 mmol) and vanillin (1.52 g, 10 mmol). The mixture was heated at 413 K for 6 h. The mixture was poured into water (300 ml) and the organic phase was extracted with ethyl acetate. The ethyl acetate extract was dried over sodium sulfate and the solvent removed under reduced pressure to yield the crude product, which was recrystallized from ethanol to afford yellow crystals in 50% yield. Analysis calculated for $C_{13}H_{14}O_4$: C 66.66, H 6.02%; found C 66.72, H 6.23%.

Crystal data

$C_{13}H_{14}O_4$
$M_r = 234.24$
Monoclinic, $C2/c$
$a = 20.792$ (3) Å
$b = 8.247$ (1) Å
$c = 15.097$ (2) Å
$\beta = 111.778$ (2)°
$V = 2403.7$ (5) Å^3
$Z = 8$
$D_x = 1.295$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1017 reflections
$\theta = 2.7$–27.0°
$\mu = 0.10$ mm^{-1}
$T = 295$ (2) K
Block, yellow
0.50 × 0.41 × 0.21 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
7252 measured reflections
2649 independent reflections
2139 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.013$
$\theta_{\rm max} = 27.1°$
$h = -23 \rightarrow 26$
$k = -10 \rightarrow 6$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.137$
$S = 1.01$
2649 reflections
158 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0809P)^2 + 0.8642P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.206 (2)	C2—C3	1.503 (2)
O2—C4	1.215 (2)	C3—C6	1.342 (2)
O3—C9	1.366 (2)	C3—C4	1.477 (2)
O3—C13	1.419 (2)	C4—C5	1.492 (2)
O4—C10	1.354 (2)	C6—C7	1.458 (2)
C1—C2	1.484 (2)		
C9—O3—C13	117.9 (1)	C3—C4—C5	121.5 (1)
O1—C2—C1	121.9 (1)	C3—C6—C7	131.3 (1)
O1—C2—C3	119.7 (1)	C6—C7—C12	125.2 (1)
C1—C2—C3	118.4 (1)	C8—C7—C12	118.2 (1)
C4—C3—C6	122.1 (1)	C6—C7—C8	116.5 (1)
C2—C3—C4	112.8 (1)	O3—C9—C8	126.0 (1)
C2—C3—C6	125.2 (1)	O3—C9—C10	114.1 (1)
O2—C4—C3	118.0 (1)	O4—C10—C11	119.1 (1)
O2—C4—C5	120.5 (1)	O4—C10—C9	121.4 (1)
O1—C2—C3—C6	71.9 (2)	O2—C4—C3—C6	178.3 (2)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O4—H4$\cdots$O2$^{\rm i}$	0.85	1.98	2.746 (2)	149

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

H atoms were placed in calculated positions (O—H = 0.85 Å, aromatic C—H = 0.93 Å and aliphatic C—H = 0.96 Å) and were included in the refinement in the riding-model approximation, with their displacement parameters tied to $U_{\rm eq}$ of the parent atoms by a ratio of 1.2 for the hydroxy and aromatic H atoms and 1.5 for the methyl H atoms. The hydroxy and methyl groups were rotated around the appropriate atom–atom axes to fit the electron density.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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