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Weak hydrogen bonds in 5-(4-methoxyphenyl)-1-phenylpent-4-ene-1,3-dione

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Key indicators

Single-crystal X-ray study
T = 105 K
Mean σ(C–C) = 0.002 Å
R factor = 0.028
wR factor = 0.079
Data-to-parameter ratio = 13.6

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

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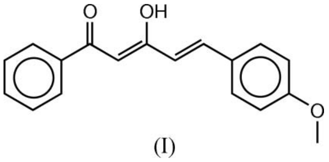
The title molecule, $C_{18}H_{16}O_3$, exists in the enolized form and displays a strong intramolecular hydrogen bond. Except for van der Waals interactions, the intermolecular contacts are dominated by weak hydrogen bonding of the type C—H···O and aromatic C—H···π.

Comment

In curcuminoid molecules, the enol H atoms always seem to be involved in a strong intramolecular hydrogen bond, and never engaged in intermolecular interactions. Therefore, when such molecules are without other classical hydrogen-bond donors, the packing of the molecules in the crystal structure is dominated by weak hydrogen bonding, as reported in earlier papers (Arrieta & Mostad 2000, 2003, 2004*a*,*b*). The molecule in the present study, (I), is depicted in Fig. 1.



(I)

The essentially planar molecule is slightly bent, and the phenyl group is rotated 8.6 (1)° about the C1—C13 bond, giving an angle of 9.65 (6)° between the aromatic rings. The molecules are stacked along the *a* axis in such a way that atoms C15 and C18 are positioned almost directly above the center of neighboring aromatic rings (Fig. 2). The distance from these atoms to the centers of the neighboring aromatic rings is 3.44 Å. The enol group is characterized by a strong intermolecular hydrogen bond; the distance between the two O atoms is very short. The rather long O1—H21 bond may indicate a broad energy minimum between O1 and O2. A similar enol-group geometry has been reported earlier (Arrieta & Mostad, 2003). In both structures, the displacement parameter for the enol H atom is large (almost five times that of the other H atoms).

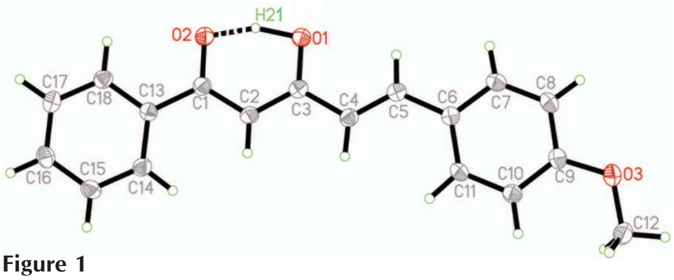


Figure 1
The numbering of the atoms in the title molecule, with displacement ellipsoids drawn at the 50% probability level.

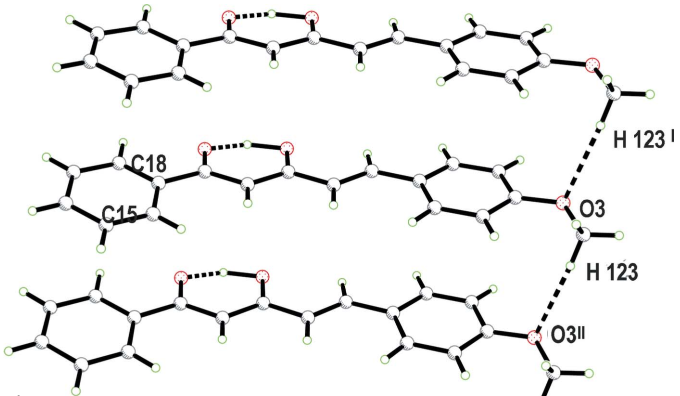


Figure 2
Interactions between molecules in the crystal structure. C—H⋯O bonds are in the direction of the *a* axis. [Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$.]

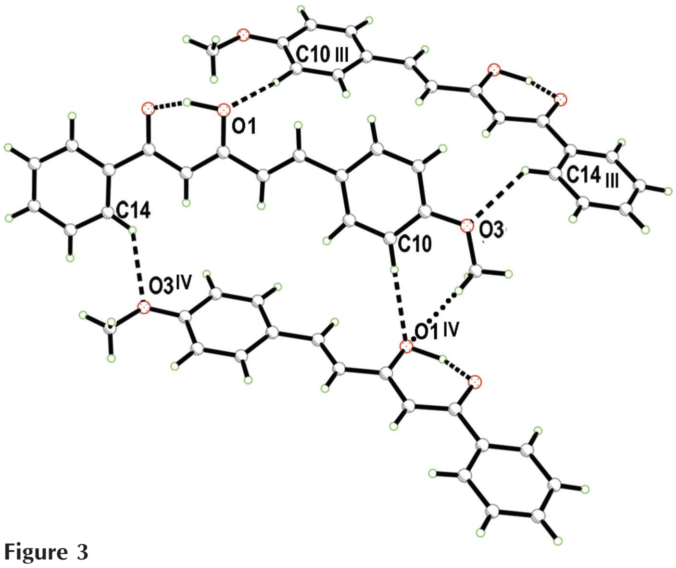


Figure 3
Interactions between molecules in the crystal structure. C—H⋯O bonds are in the direction of the *b* axis [Symmetry codes: (iii) $1 - x, -\frac{1}{2} + y, 1 - z$; (iv) $1 - x, \frac{1}{2} + y, 1 - z$.]

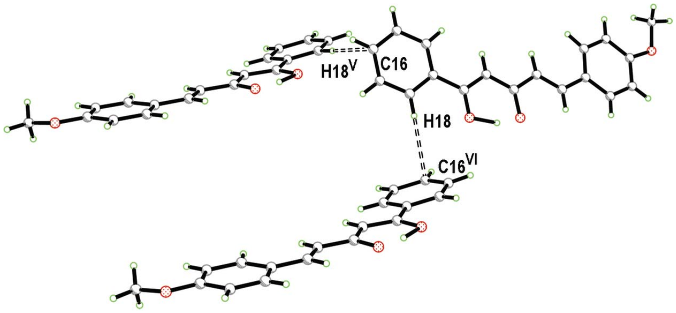


Figure 4
Interactions between molecules in the crystal structure. C—H⋯π bonds are in the direction of the *b* axis. [Symmetry codes: (v) $1 - x, \frac{1}{2} + y, -z$; (vi) $1 - x, -\frac{1}{2} + y, -z$.]

The intermolecular interactions display three types of weak hydrogen bonds: Csp^3—H⋯O, C_{ar}—H⋯O and C_{ar}—H⋯π. The first type connects the molecules along the *a* axis, as shown in Fig. 2, the second type connects the molecules in the *b*-axis direction, as shown in Fig. 3, while the tird type connects molecules in the direction of the *c* axis, as well as in the direction of the *b* axis, as shown in Fig. 4. The geometry of these intermolecular interactions is given in Table 2.

Experimental

The title compound was synthesized from benzoylacetone and 4-methoxybenzaldehyde (Arrieta *et al.*, 1992) and thin yellow needle-shaped crystals (m.p. 393–394 K) were obtained from an ethanol solution. Crystals suitable for the X-ray work were grown by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{18}H_{16}O_3$
$M_r = 280.31$
Monoclinic, $P2_1$
$a = 3.9754$ (8) Å
$b = 9.790$ (2) Å
$c = 17.958$ (4) Å
$\beta = 92.91$ (3)°
$V = 698.0$ (3) Å^3
$Z = 2$

$D_x = 1.334$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10099 reflections
$\theta = 2.1$–28.4°
$\mu = 0.09$ mm^{-1}
$T = 105$ (2) K
Plate, yellow
0.4 × 0.4 × 0.1 mm

Data collection

Bruker SMART diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{\min} = 0.965$, $T_{\max} = 0.991$
11132 measured reflections
1838 independent reflections

1794 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 28.3°$
$h = -5 \rightarrow 5$
$k = -13 \rightarrow 13$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.079$
$S = 1.12$
1838 reflections
254 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0881P)^2 + 0.4691P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 0.24$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.3110 (19)	C1—C13	1.485 (2)
O2—C1	1.2797 (19)	C2—C3	1.394 (2)
O3—C9	1.3657 (17)	C3—C4	1.4631 (19)
O3—C12	1.436 (2)	C4—C5	1.347 (2)
C1—C2	1.4203 (19)	C5—C6	1.4638 (19)
C9—O3—C12	117.43 (12)	C2—C3—C4	121.34 (14)
O2—C1—C2	120.09 (14)	C5—C4—C3	121.74 (14)
O2—C1—C13	117.65 (12)	C4—C5—C6	127.19 (14)
C2—C1—C13	122.25 (13)	C11—C6—C5	123.32 (13)
C3—C2—C1	119.89 (14)	C7—C6—C5	118.72 (13)
O1—C3—C2	121.03 (13)	C14—C13—C1	122.21 (13)
O1—C3—C4	117.63 (13)	C18—C13—C1	118.71 (13)
O2—C1—C2—C3	0.2 (2)	C12—O3—C9—C8	−174.56 (13)
C13—C1—C2—C3	−178.74 (13)	C12—O3—C9—C10	6.0 (2)
C1—C2—C3—O1	0.5 (2)	C7—C8—C9—O3	−179.07 (13)
C1—C2—C3—C4	−179.61 (13)	O3—C9—C10—C11	179.12 (13)
O1—C3—C4—C5	3.2 (2)	O2—C1—C13—C14	172.53 (14)
C2—C3—C4—C5	−176.65 (14)	C2—C1—C13—C14	−8.5 (2)
C3—C4—C5—C6	179.36 (14)	O2—C1—C13—C18	−8.7 (2)
C4—C5—C6—C11	−1.5 (2)	C2—C1—C13—C18	170.29 (13)
C4—C5—C6—C7	179.25 (14)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C18—H18···C16^{i}	0.96 (2)	2.75 (2)	3.708 (2)	178 (2)
C12—H121···O1^{ii}	0.95 (3)	2.68 (2)	3.556 (2)	154 (2)
C12—H123···O3^{iii}	0.94 (2)	2.65 (2)	3.545 (2)	158 (2)
C10—H10···O1^{iv}	0.99 (2)	2.55 (2)	3.525 (2)	172 (2)
C14—H14···O3^{iv}	0.97 (2)	2.57 (2)	3.3089 (19)	133 (2)
O1—H21···O2	1.19 (4)	1.31 (4)	2.4594 (16)	159 (4)

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

All H atoms were located from difference maps and refined in an isotropic approximation. As one would expect, due to the lack of anomalous scatterers the absolute structure determination (Flack, 1983) was inconclusive and Friedel pairs were merged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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