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# Ethyl (*E*)-2-benzoyl-3-phenylpropenoate

**R. Hema,[a] V. Parthasarathi,[a]* K. Ravikumar,[b] B. Sridhar[b] and K. Pandiarajan[c]**

[a]School of Physics, Bharathidasan University, Tiruchirappalli 620 024, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Chemistry, Annamalai University, Annamalai Nagar 608 002, India

Correspondence e-mail: vpsarati@yahoo.com

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**Key indicators**

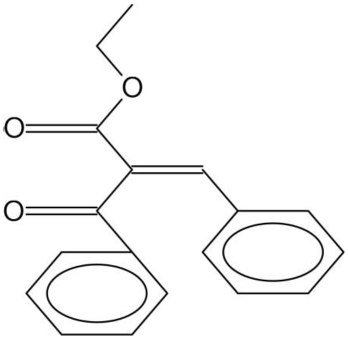
Single-crystal X-ray study
*T* = 273 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.035
*wR* factor = 0.102
Data-to-parameter ratio = 13.4

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

In the title molecule, $C_{18}H_{16}O_3$, the configuaration with respect to the C═C double bond is *E*. The benzoyl group is rotated out of the mean plane through the rest of the molecule by 88.31 (4)°. In the crystal structure, molecules are linked by weak intermolecular C—H···O interactions to form two-dimensional sheets.

## Comment

A novel derivative of ethyl 2-propenoate suppresses carcinogenesis and inducible nitric oxide synthase in rat tongue (Tanaka *et al.*, 2003). The potential of ethyl 2-methyl-2-propenoate derivatives to produce neurotoxicity has been investigated in adult male Spague–Dawley rats (Abou-Donia *et al.*, 2000). The X-ray crystal structure determination of the title compound, (I), has been undertaken to investigate the stereochemistry of the molecule and the conformational changes resulting from the presence of various substituents in the ethyl propenoate group, and the results are presented here.



(I)

In the title compound, (I), the C2═C3 bond length [1.340 (2) Å] is comparable with the unweighted mean double-bond distance [1.340 (13) Å] for C═C—C═O groups (Allen *et al.*, 1987). It also corresponds to the C2═C3 bond [1.347 (2) Å] in a similar related structure, methyl (*Z*)-3-[(4,6-dimethyl-2-pyrimidinyl)amino]-2-[(methoxy)(methylthio)-methyleneamino]propenoate, (II) (Sinur *et al.*, 1994). In two more related structures reported in the same paper, namely ethyl (*E*)-2-benzoyl-3-(dimethylamino)propenoate, (III), and ethyl (*Z*)-2-benzoyl-3-[(3-nitrophenyl)amino]propenoate, (IV) (Sinur *et al.*, 1994), the C2═C3 distances were reported as 1.385 (2) and 1.375 (2) Å, respectively.

The C1—C2 bond [1.482 (2) Å] in (I) is slightly longer than the conjugated C$sp^2$—C$sp^2$ bond length [1.464 (17) Å; Allen *et al.*, 1987], while in (II), (III) and (IV), the C1—C2 distances

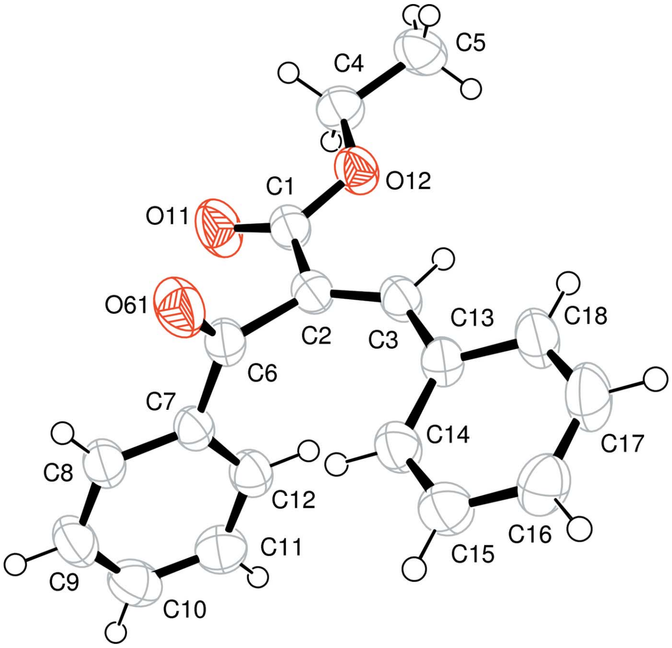


**Figure 1**
A view of (I), with displacement ellipsoids drawn at the 50% probability level and H atoms shown as small spheres of arbitrary radii.

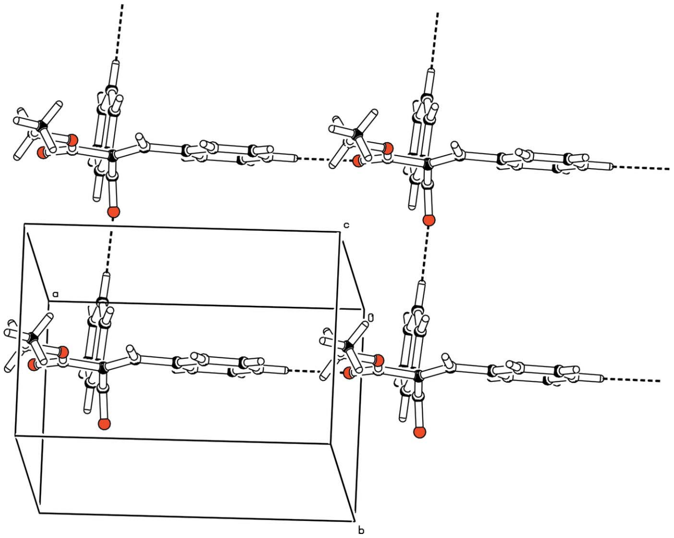


**Figure 2**
A partial packing plot of (I), showing hydrogen bonds as dashed lines.

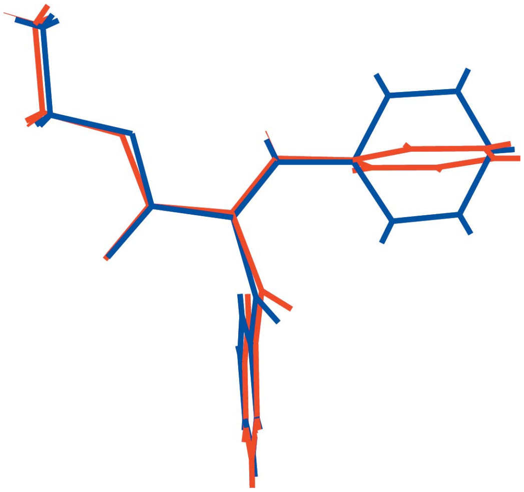


**Figure 3**
Superimposed fit of a molecule of (I) (blue) and its energy-minimized counterpart (red).

were reported as 1.474 (2), 1.457 (2) and 1.465 (2) Å, respectively. The C2—C6 bond [1.510 (2) Å] is longer than the above unconjugated bond length, but in both (III) and (IV) the C2—C6 bond distance is 1.481 (2) Å. The torsion angles C3—C2—C6—O61 [84.09 (16)°] and C1—C2—C6—O61 [−91.25 (13)°] show that the benzoyl group at C2 is oriented perpendicular to the mean plane through the rest of the molecule (C13–C18/C3/C2/C1/O11/O12/C4/C5). The dihedral angle between the benzoyl group and the mean plane is 88.31 (4)°.

In order to understand the effect of packing on the molecule, an energy minimization calculation was carried out on the isolated molecule using the *WINMOPAC* program (Shchepin & Litivinov, 1998). The superposition (Gans & Shalloway, 2001) of the fragment of (I) without the phenyl group (C13–C18) with the energy-minimized counterpart (without the phenyl group) gives an r.m.s. deviation of nearly zero, while the superposition of the structure of (I) with its energy-minimized counterpart with the phenyl group (Fig. 3) gives an r.m.s. deviation of 0.717 Å. The conformations of the molecules in the crystal structure and in the energy-minimized form are significantly different only in the orientation of the phenyl group. This is evident from the change in the torsion angle from −164.95 (13) to −81.36° for C2—C3—C13—C18 and 16.4 (2) to 101.85° for C2—C3—C13—C14. This change in torsion angle may be due to the possibility of free rotation of the phenyl group about the C3—C13 bond.

Superposition of the non-H atoms common to the structures of (I) and (II) and (I) and (III) give r.m.s. deviations of 1.811 and 1.743 Å, respectively. The crystal packing is stabilized by two weak intermolecular C—H···O interactions. Atom C11 acts as a donor for a weak intermolecular C—H···O interaction (Table 1) with carbonyl atom O61, to form a graph-set motif *C*(6) (Bernstein *et al.*, 1995). Atom C16 acts as a donor for a weak C—H···O intermolecular interaction with atom O11 of a symmetry-related molecule, which links the molecules into chains to form a graph-set motif *C*(9). The combination of both of these types of weak C—H···O interactions forms a two-dimensional sheet perpendicular to the *c* axis.

## Experimental

A mixture of benzoyl acetate (0.10 mol), benzaldehyde (0.10 mol), dry ammonium acetate (0.10 mol) and 95% ethanol (50 ml) was stirred with a magnetic stirrer for 5 min and warmed on a hot plate at 333 K for 25 min. The mixture was allowed to stand at 298 K for 2–3 d until no further crystals separated out. The crystals were filtered off and recrystallized from ethanol (m.p. 361 K, yield 85%).

*Acta Cryst.* (2006). E**62**, o708–o710 Hema *et al.* • $C_{18}H_{16}O_3$ **o709**

*Crystal data*

$C_{18}H_{16}O_3$
$M_r = 280.31$
Monoclinic, $P2_1/n$
$a = 10.8099\ (8)$ Å
$b = 7.2746\ (5)$ Å
$c = 18.5285\ (13)$ Å
$\beta = 94.3260\ (10)^\circ$
$V = 1452.89\ (18)$ Å$^3$
$Z = 4$
$D_x = 1.281$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 8602 reflections
$\theta = 2.3$–$27.9^\circ$
$\mu = 0.09$ mm$^{-1}$
$T = 273\ (2)$ K
Block, colourless
$0.18 \times 0.11 \times 0.09$ mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
13392 measured reflections
2557 independent reflections
2297 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 25.0^\circ$
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 8$
$l = -22 \rightarrow 22$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.102$
$S = 1.05$
2557 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.2059P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{\rm min} = -0.16$ e Å$^{-3}$

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C11—H11$\cdots$O61$^{\rm i}$ | 0.93 | 2.57 | 3.3911 (17) | 148 |
| C16—H16$\cdots$O11$^{\rm ii}$ | 0.93 | 2.57 | 3.3343 (16) | 140 |

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

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with $U_{\rm iso}({\rm H}) = 1.5U_{\rm iso}({\rm C})$, but were allowed to rotate freely about the C—C bond. All other H atoms in the structure were placed in geometrically idealized positions (C—H = 0.93–0.98 Å) and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm iso}({\rm C})$.

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: *WinGX* (Version 1.64.05; Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

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**o710** Hema *et al.* • $C_{18}H_{16}O_3$ *Acta Cryst.* (2006). E**62**, o708–o710