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Diisopropyl 2-(2-benzoyl-1-phenylethyl)malonate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.142
Data-to-parameter ratio = 16.0

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

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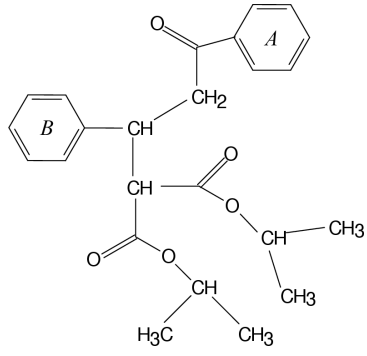
In the title compound, $C_{24}H_{28}O_5$, the keto group is planar. Weak C—H···O hydrogen bonds play a significant role in the crystal packing.

Comment

A number of compounds derived from chalcones possess multi-protecting biochemical activities, such as antifungal, antimalarial, antifertility *etc.* The biological activity of these compounds depends on the conformation of the keto group present in the molecule. Chalcones and their derivatives show inhibitory action against *S. aureus*, *F. graminearum* and *B. alli* (Marrian *et al.*, 1947). These compounds are also toxic to red spider mites (Eaton & Davis, 1950).



(I)

In the title molecule, (I), the bond distances are found to agree with the literature values (Allen *et al.*, 1987). The bond angles at phenyl rings *A* and *B* show no significant deviations from the standard value, except around atoms C14 and C19. The slight distortion in the exocyclic bond angles at C14 and C19 may be due to the steric interactions between the isopropyl groups and phenyl rings. The dihedral angle between phenyl rings *A* and *B* is 83.8 (2)° and they are oriented at angles of 8.7 (1) and 87.0 (1)°, respectively, to the keto group. The O5—C5—C4—C3 torsion angle of −17.7 (3)° shows that the molecule assumes an *s-cis* conformation for the keto system. Apart from van der Waals interactions, the packing of the molecules in the crystal is stabilized by intermolecular C—H···O interactions and one intramolecular interaction (Desiraju, 1991, 1996).

Experimental

To 1 mmol of $LiAlH_4$ in dry tetrahydrofuran (THF), 1 mmol of methyl 2-[*N*-(2-hydroxyphenylmethyl)amino]-3-methylbutanoate was added at room temperature in a dried side-arm flask and stirred

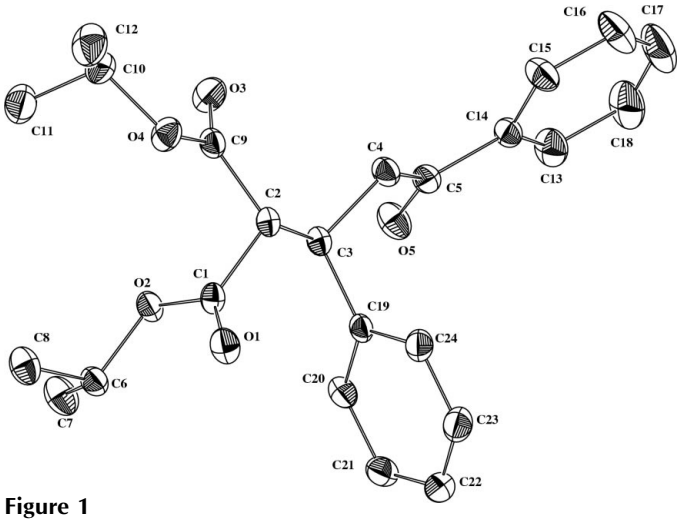


Figure 1
A view of the title compound, showing the atom-numbering scheme, with probability ellipsoids drawn at the 30% level.

for 4 h. To the stirred solution, 1,3-diphenyl-2-propen-1-one (5 mmol) and diisopropyl malonate (5 mmol) were added and stirring was continued for 7 h. The reaction was monitored throughout by thin-layer chromatography (TLC) until all the diisopropyl malonate had reacted. The reaction was quenched with 1 *N* HCl and the rpoduct was extracted with ethyl acetate. The combined organic layer was washed with $NaHCO_3$ and dried over anhydrous Na_2SO_4. Removal of the solvent under reduced pressure gave a syrupy mass which, on flash column chromatography, yielded a crystalline solid. Colourless needle-shaped crystals were obtained by slow evaporation at room temperature from a hexane–ethyl acetate (1:1) mixture.

Crystal data

$C_{24}H_{28}O_5$
M_r = 396.46
Triclinic, $P\overline{1}$
a = 6.031 (1) Å
b = 10.215 (1) Å
c = 19.069 (1) Å
α = 74.76 (1)°
β = 89.11 (1)°
γ = 76.87 (1)°
V = 1102.6 (2) Å^3
Z = 2
D_x = 1.194 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 14–25°
μ = 0.67 mm^{-1}
T = 293 (2) K
Needle, colourless
0.20 × 0.20 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
4453 measured reflections
4204 independent reflections
2392 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 71.9°
$h = -7 \rightarrow 6$
$k = 0 \rightarrow 12$
$l = -22 \rightarrow 23$
3 standard reflections
every 200 reflections
frequency: 120 min
intensity decay: <0.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.142
S = 1.01
4204 reflections
262 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.1575P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.199 (2)	C2—C3	1.543 (3)
O2—C1	1.333 (3)	C3—C19	1.517 (3)
O2—C6	1.465 (2)	C3—C4	1.535 (3)
O3—C9	1.199 (3)	C4—C5	1.508 (3)
O4—C9	1.329 (3)	C5—C14	1.492 (3)
O5—C5	1.212 (3)		
C14—C5—C4	118.8 (2)	C15—C14—C13	118.4 (2)
C7—C6—C8	113.1 (2)	C20—C19—C24	118.1 (2)
C11—C10—C12	112.6 (2)		
C9—C2—C3—C19	−170.52 (17)	C3—C4—C5—O5	17.7 (3)
C1—C2—C3—C19	−48.0 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O5^{i}	0.98	2.57	3.509 (3)	161
C4—H4A···O3	0.97	2.41	3.063 (3)	124
C7—H7A···O1^{ii}	0.96	2.54	3.437 (3)	156

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

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SDP* (Frenz, 1978); data reduction: *XCAD*-4-*PC* (Harms & Wocadlo, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997); software used to prepare material for publication: *PARST*97 (Nardelli, 1983, 1995).

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