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1,2-Dibenzoyl-3,4-bis(4-methoxyphenyl)-cyclobutane

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The title compound, $C_{32}H_{28}O_4$, crystallizes with two conformers, α and β, in the asymmetric unit. In both conformers, the benzoyl and methoxyphenyl groups are in *cis* positions. The cyclobutane rings are puckered, with average absolute torsion angles of 10.8 (2) and 11.4 (1)° for conformers α and β, respectively. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds and benzene π–π interactions.

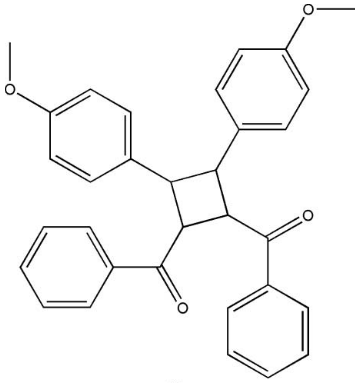
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.03 Å
R factor = 0.046
wR factor = 0.116
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.

Comment

Photodimerization of allyl compounds has been extensively studied in terms of reaction order in the solid state (Fernandes & Levendis, 2004), and compounds exhibiting this characteristic have been linked to biologically active systems (Dehmlow & Schmidt, 1990; Kort *et al.*, 2004). In our study of these aspects, we obtained the title compound, (I), a methoxyphenyl derivative.



(I)

The title compound crystallizes in a triclinic unit cell with two conformers, α and β, in the asymmetric unit (Fig. 1). A typical bonding pattern is observed for the cyclobutane ring system, with bond distances of around 1.57 Å (Table 1). Exceptions are the bonds C1*A*—C2*A* [1.536 (3) Å] and C3*B*—C4*B* [1.539 (3) Å]. In both conformers, the bond angles in the cyclobutane ring systems are nearly 90° (Table 1). An interesting observation is the out-of-plane deformation of the carbonyl group relative to the associated benzene ring, with the O—C—C—C torsion angle varying from −9.0 (3) to 22.5 (3)° (Table 1). Comparison of the torsional twist of the benzene ring with respect to the cyclobutane ring yields a significant difference between the two conformers, *viz*. C3*A*—C4*A*—C40*A*—C41*A* and C2*B*—C1*B*—C10*B*—C11*B* torsion angles of 68.8 (2) and 79.5 (2)° for conformers α and β, respectively.

The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2) and benzene

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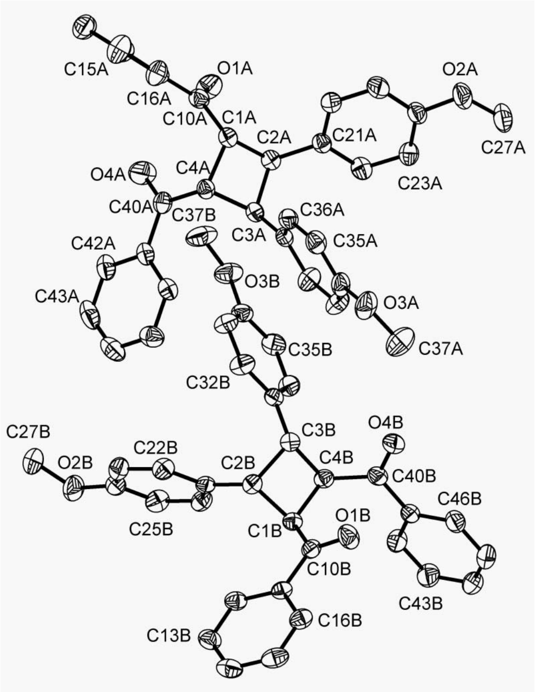

Figure 1

The two independent molecules of (I), viz. conformers α and β, shown with 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

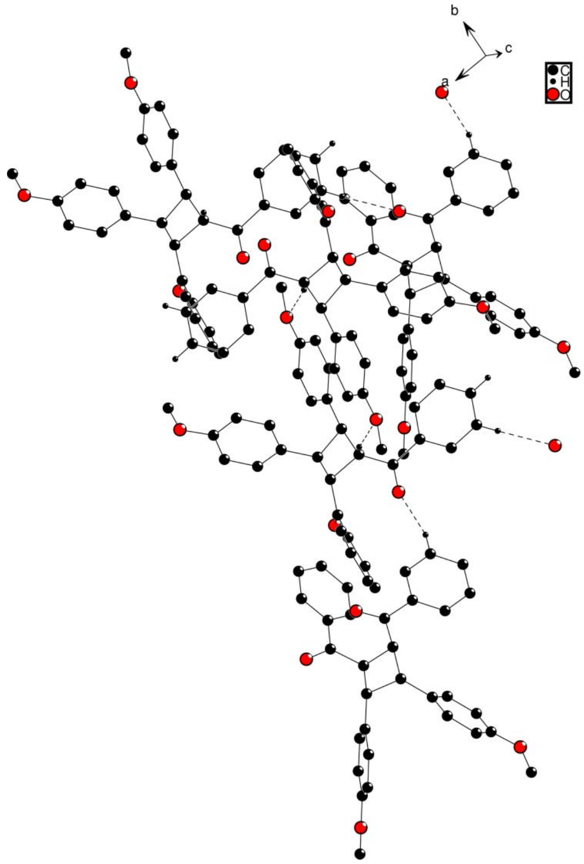

Figure 2

Crystal packing showing the intermolecular C—H$\cdots$O hydrogen bonding (dashed lines). H atoms not participating in hydrogen bonding have been omitted for clarity.

π–π interactions, evidenced by the short interplanar distance of 3.225 (4) Å between the C31*A*–C36*A* and C31*A*^{i}–C36*A*^{i} benzene rings [symmetry code: (i) $-x + 1, -y + 1, -z$].

A search of the Cambridge Structural Database (CSD, Version 5.26; Allen, 2002) was performed with a core cyclobutane ring as a target system (1952 entries and 2535 total observed parameters, excluding bicyclic butane structures). The average C—C bond distance in this ring ranged from 1.525 to 1.575 Å, with an average absolute torsion angle defined by the four C atoms of the cyclobutane ring of *ca* 9.6 °. The title compound shows an average bond distance of 1.562 (3) Å and average absolute torsion angles of 10.8 (2) and 11.4 (1)° for the α and β conformers, respectively.

Two sets of data are available for the unfunctionalized 1,2-dibenzoyl-3,4-diphenylcyclobutane (Caccamese *et al.*, 1978; Toda *et al.*, 1998), and both sets show a distortion in the cyclobutane ring (average torsion angles of *ca* 7.3°), comparable to the total average as mentioned above. A further functionalization of the two benzoyl groups, with both a methoxy and a hydroxy group (Katerere *et al.*, 2004), resulted in an additional increase in the average absolute torsion angle in the cyclobutane ring to *ca* 14°. Thus, additional functionalization on the benzene ring of the benzoyl group, presumably due to steric and carbonyl group effects, progressively affects the total distortion of the system as defined by the average absolute torsion angle in the cyclobutane ring (from 7 to 11 to 14°), for the non-functionalized, mono-functionalized [by a methoxy group, *viz*. (I)] and the bis-functionalized (methoxy and hydroxy) compounds, respectively.

Experimental

Anisylacetophenone (4-methoxychalcone, 1 g, 4.2 mmol) was dissolved with heating (*ca* 313 K) in ethanol (200 ml). The mixture was slowly evaporated in sunlight (*ca* 1 week) to yield light-yellow crystals. Repeating the procedure with a daylight lamp for 2 d gave the same result (yield: 0.7 g, 70%). ^{1}H NMR (CDCl$_3$, 300 MHz): 3.72 (*s*, 6H), 4.33 (*dd*, 2H), 4.68 (*dd*, 2H), 6.70 (*m*, 4H), 6.92 (*m*, 4H), 7.35 (*t*, 4H), 7.45 (*m*, 2H), 7.80 (*dd*, 4H). IR (KBr): 1659 (*s*), 1305 (*s*), 1263 (*s*), 1212 (*s*) cm^{-1}.

Crystal data

$C_{32}H_{28}O_4$
$M_r = 476.54$
Triclinic, $P\overline{1}$
$a = 10.8522$ (12) Å
$b = 11.4576$ (2) Å
$c = 20.2987$ (2) Å
$\alpha = 93.580$ (1)°
$\beta = 95.311$ (1)°
$\gamma = 94.243$ (1)°
$V = 2500.1$ (3) Å^3

$Z = 4$
$D_x = 1.266$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1015 reflections
$\theta = 2.8$–24.4°
$\mu = 0.08$ mm^{-1}
$T = 295$ (2) K
Parallelepiped, light yellow
0.60 × 0.27 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
$T_{\min} = 0.975$, $T_{\max} = 0.981$
13113 measured reflections

8678 independent reflections
6125 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -24 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.116$
$S = 1.04$
8678 reflections
650 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0417P)^2 + 0.8936P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0083 (7)

Table 1
Selected geometric parameters (Å, °).

C1A—C2A	1.536 (3)	C1B—C4B	1.556 (3)
C1A—C4A	1.562 (3)	C1B—C2B	1.573 (2)
C2A—C3A	1.573 (3)	C2B—C3B	1.583 (3)
C3A—C4A	1.577 (3)	C3B—C4B	1.539 (3)
C2A—C1A—C4A	90.06 (14)	C4B—C1B—C2B	89.48 (13)
C1A—C2A—C3A	90.38 (14)	C1B—C2B—C3B	88.15 (13)
C2A—C3A—C4A	88.20 (13)	C4B—C3B—C2B	89.71 (13)
C1A—C4A—C3A	89.29 (13)	C3B—C4B—C1B	90.36 (14)
C1A—C2A—C3A—C4A	10.9 (2)	C1B—C2B—C3B—C4B	11.4 (2)
O1A—C10A—C11A—C12A	22.5 (3)	O1B—C10B—C11B—C16B	−9.0 (3)
O4A—C40A—C41A—C42A	1.3 (3)	O4B—C40B—C41B—C46B	21.0 (3)
C23A—C24A—O2A—C27A	9.5 (3)	C23B—C24B—O2B—C27B	16.1 (3)
C33A—C34A—O3A—C37A	15.3 (3)	C33B—C34B—O3B—C37B	9.8 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
C43A—H43A···O1B^{i}	0.93	2.46	3.382 (3)	174
C15B—H15B···O4A^{ii}	0.93	2.46	3.369 (3)	164
C44A—H44A···O1A^{iii}	0.93	2.47	3.234 (3)	139
C4A—H4A···O3A^{iv}	0.98	2.60	3.547 (3)	163

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}$ of the parent atom.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT* and *SHELXTL* (Siemens, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXL97*.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Brandenburg, K. (2000). *DIAMOND*. Release 2.1e. Crystal Impact GbR, Postfach 1251, D-53002 Bonn, Germany.
Bruker (1998). *SADABS* (Version 2004/1) and *SMART-NT* (Version 5.050). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT-Plus* (including *XPREP*). Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
Caccamese, S., McMillan, J. A. & Montaudo, G. (1978). *J. Org. Chem.* **43**, 2703–2704.
Dehmlow, E. V. & Schmidt, S. (1990). *Liebigs Ann. Chem.* pp. 411–413.
Fernandes, M. A. & Levendis, D. C. (2004). *Acta Cryst.* B**60**, 315–324.
Katerere, D. R., Gray, A. I., Kennedy, A. R., Nash, R. J. & Waigh, R. D. (2004). *Phytochemistry*, **65**, 433-438.
Kort, R., Komori, H., Adachi, S., Miki, K. & Eker, A. (2004). *Acta Cryst.* D**60**, 1205–1213.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1998). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Toda, F., Tanaka, K. & Kato, M. (1998). *J. Chem. Soc. Perkin Trans. 1*, 7, 1315–1318.