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[3-Benzoyl-2,4-bis(3-bromophenyl)cyclobutyl]-(phenyl)methanone

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.026
wR factor = 0.062
Data-to-parameter ratio = 20.8

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

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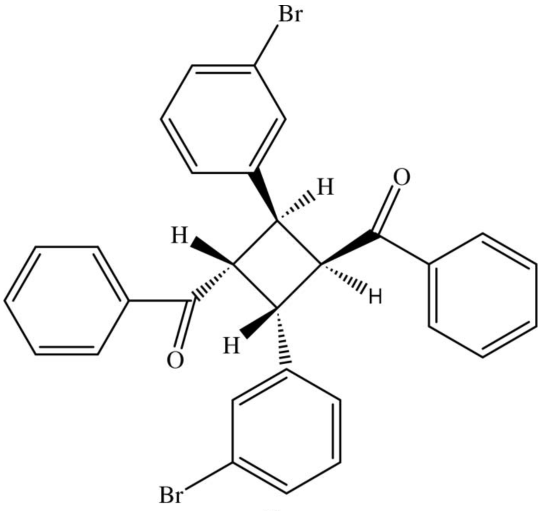
Molecules of the title compound, $C_{30}H_{22}Br_2O_2$, lie across crystallographic inversion centres. The dihedral angle between the phenyl and bromobenzene rings is 23.88 (8)°. Weak C—H···O interactions link the molecules into ribbons along the *a* axis.

Comment

Cycloaddition reactions can be used to produce rings containing different substituents as part of the total synthesis of complex natural products (Nicolaou & Sorensen, 1996). Cycloaddition reactions using microwave techniques are now well established (Kappe, 2004).



(I)

The title compound, (I), formed by [2 + 2] cylcloaddition using microwaves, lies across a crystallographic inversion centre; the asymmetric unit therefore contains one half-molecule (Fig. 1). The cyclobutane ring is planar. The dihedral angle between the phenyl and bromobenzene rings is 23.88 (8)°, both rings being approximately perpendicular to the cyclobutane ring with dihedral angles of 89.18 (11)° and 78.26 (10)° for the phenyl and bromobenzene rings, respectively. The value of the dihedral angle [30.44 (10)°] between the C7–C9/O1 unit and phenyl ring shows that the carbonyl group is twisted out of the plane of the phenyl ring. The C5—C6—C7—O1 torsion angle is −28.5 (2)°. All bond lengths and angles in (I) show normal values (Allen *et al.*, 1987).

The crystal structure of (I) (Fig. 2) shows that the molecules are arranged in a zigzag manner along the *b* axis, with weak C—H···O interactions (Table 2) linking the molecules into ribbons along the *a* axis.

Experimental

Compound (I) was isolated from a reaction containing 3-(3-bromophenyl)-1-phenylpropenone (290 mg, 1 mmol) and 2-amino-6-methylpyridine (108 mg, 1 mmol) during optimization of the process to synthesize substituted naphthyridine-type heterocyclic compounds (Goswami, Jana, Dey & Adak, 2007; Goswami, Jana, Hazra & Adak, 2007) under microwave irradiation (450 W, 20 min) using a domestic microwave oven (BPL 800 G). One of the components, namely 3-(3-bromophenyl)-1-phenylpropenone, dimerized under these conditions to afford compound (I) as a minor product; (I) (yield 10 mg, 7%) was purified by preparative thin-layer chromatography using petroleum ether–ethyl acetate (9:1 *v*/*v*) as eluent. Single crystals were grown by slow evaporation of a $CHCl_3$/EtOH solution (1:1 *v*/*v*) (m.p. 337–340 K).

Crystal data

$C_{30}H_{22}Br_2O_2$
$M_r = 574.28$
Monoclinic, $P2_1/c$
$a = 5.7813$ (1) Å
$b = 14.6794$ (2) Å
$c = 14.2828$ (2) Å
$\beta = 102.988$ (1)°
$V = 1181.11$ (3) Å^3
$Z = 2$
$D_x = 1.615$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 3.46$ mm^{-1}
$T = 100.0$ (1) K
Needle, colourless
0.46 × 0.14 × 0.09 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{\min} = 0.300$, $T_{\max} = 0.737$
18746 measured reflections
3429 independent reflections
2869 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 30.0$°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.062$
$S = 1.04$
3429 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 0.7106P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.55$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}

Table 1
Selected torsion angles (°).

C5—C6—C7—O1	−28.5 (2)	C8—C9—C10—C15	−30.8 (2)
C1—C6—C7—C8	−30.7 (2)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
C9—H9$\cdots$O1$^{\rm i}$	0.96	2.61	3.408 (2)	140

Symmetry code: (i) $-x+3, -y+1, -z+1$.

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for aromatic, and C—H = 0.97 Å and $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for CH H atoms.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

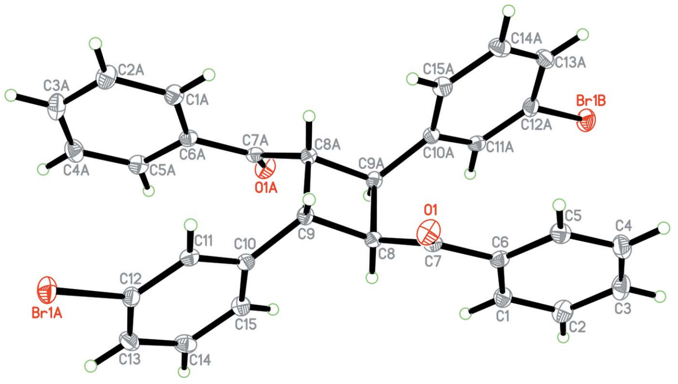


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. [Symmetry code: (A) $2-x, 1-y, 1-z$.]

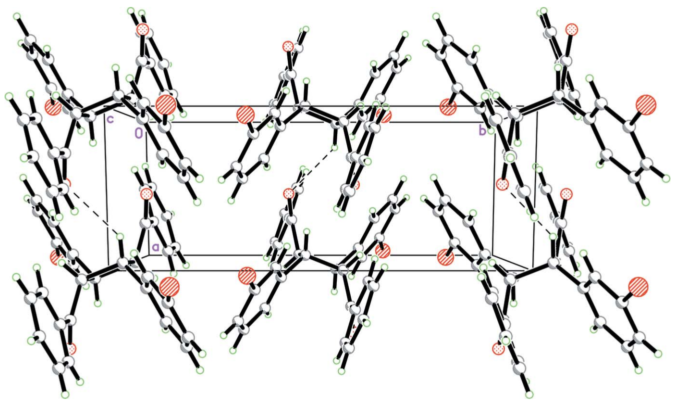


Figure 2
The crystal packing of (I), viewed down the *c* axis. C—H$\cdots$O interactions are shown as dashed line.

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