

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

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Key indicators

Single-crystal X-ray study
 $T = 295$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.053
 wR factor = 0.119
 Data-to-parameter ratio = 15.3

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

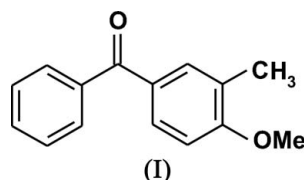
4-Methoxy-3-methylbenzophenone

The molecule of the title compound, $\text{C}_{15}\text{H}_{14}\text{O}_2$, is non-planar and the planes of the benzene rings intersect at an angle of $59.8(1)^\circ$. The crystal structure exhibits a significant short intermolecular π - π contact.

Received 15 March 2006
 Accepted 3 April 2006

Comment

The title compound, (I), a benzophenone derivative, is an intermediate for the preparation of antimicrobial β -apopicrodophyllin (Basavaraju & Anjanamurthy, 2003). Over 300 crystal structures of benzophenone derivatives in the Cambridge Structural Database (CSD, Version 5.26; Allen, 2002) highlight the importance of structural studies on such pharmaceutically useful compounds. The synthesis and structure of (I) are reported here.



A projection of (I) with the numbering scheme is shown in Fig. 1. Relevant geometric details are given in Table 1. The structure of (I), similar to other benzophenone analogues (Malathy Sony *et al.*, 2005; Mahendra *et al.*, 2005; Khanum *et al.*, 2005), is non-planar due to the strains associated with the overlapping of adjacent H atoms of aromatic rings. The planes through $\text{C}7$ - $\text{C}13$ and $\text{O}2/\text{C}1$ - $\text{C}7/\text{C}14/\text{C}15$ intersect at an angle of $59.1(1)^\circ$, whereas the planes of benzene rings ($\text{C}1$ - $\text{C}6$ and $\text{C}8$ - $\text{C}13$) intersect at $59.8(1)^\circ$. Atom $\text{O}1$ is displaced by $-0.226(2)$ and $0.960(2)$ Å with respect to aromatic rings $\text{C}1$ - $\text{C}6$ and $\text{C}8$ - $\text{C}13$. The dihedral angles between the planes associated with the carbonyl group $\text{O}1/\text{C}1/\text{C}7/\text{C}8$ and adjacent benzene rings $\text{C}1$ - $\text{C}6$ and $\text{C}8$ - $\text{C}13$ are $8.9(1)$ and $53.0(1)^\circ$, respectively. The conformation of the

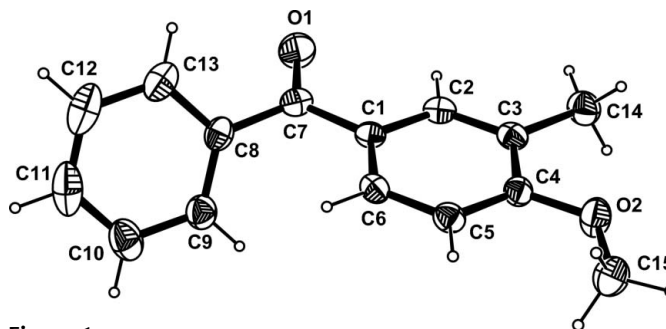


Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

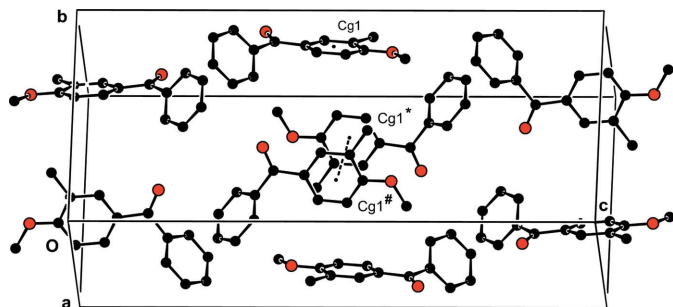


Figure 2

The crystal packing of (I). The short π - π contact is shown as a dashed line. Cg1 is the centroid of aromatic ring C1-C6. The asterisk (*) and hash (#) symbols indicate that the molecules are located at the symmetry positions $(\frac{1}{2} + x, \frac{3}{2} - y, 1 - z)$ and $(\frac{1}{2} - x, -\frac{1}{2} + y, z)$, respectively.

molecule as a whole is essentially described by the torsion angles about the C1-C7 and C7-C8 bonds: C2-C1-C7-O1 = -7.0 (3) $^\circ$, O1-C7-C8-C13 = -50.7 (3) $^\circ$, C2-C1-C7-C8 = 173.61 (16) $^\circ$ and C1-C7-C8-C13 = 128.68 (19) $^\circ$. In contrast, the values of the corresponding torsion angles in an isomeric compound crystallizing in $P2_1/c$ (2-methoxy-5-methylphenyl phenyl ketone; Malathy Sony *et al.*, 2005) are: C6-C1-C7-O8 = 51.7 (3) $^\circ$, O8-C7-C9-C10 = 17.5 (4) $^\circ$, C6-C1-C7-C9 = -124.9 (3) $^\circ$ and C1-C7-C9-C10 = -166.0 (2) $^\circ$.

A view of the crystal packing is shown in Fig. 2. The crystal structure displays a significant short intermolecular π - π contact of face-to-face type, where the $Cg1 \cdots Cg1^i$ [symmetry code: (i) $-x, 2 - y, 1 - z$; Cg1 is the centroid of the ring C1-C6] separation and perpendicular distances are 3.55 and 3.48 Å, respectively. The rest of the short intermolecular contacts are in the range of van der Waals interactions.

Experimental

A solution of *o*-methylanisole (2 g, 0.0163 mol) in dry dichloromethane (15 ml) was treated with anhydrous aluminium chloride (2.183 g, 0.0163 mol). The reaction mixture was stirred continuously for 30 min. A solution of benzoyl chloride (2.3 g, 0.01637 mol) in dichloromethane (15 ml) was added dropwise over a period of 1 h to the reaction mixture at 298 K. After 24 h, 6 ml of concentrated HCl was added over 30 min. The reaction mixture was stirred for 24 h. Aqueous NaCl solution (10%) was added to break up the emulsion and the lower organic layer was separated and washed twice with 50 ml of 10% NaOH. The excess of dichloromethane was distilled off and the concentrated solution was left to stand overnight. Pale-yellow crystalline solids were separated and recrystallized from methanol (yield, 90%; m.p. 343 K).

Crystal data

C ₁₅ H ₁₄ O ₂	Z = 8
M _r = 226.26	D _x = 1.241 Mg m ⁻³
Orthorhombic, <i>Pbca</i>	Mo K α radiation
a = 8.582 (2) Å	μ = 0.08 mm ⁻¹
b = 11.314 (3) Å	T = 295 (2) K
c = 24.941 (7) Å	Block, colourless
V = 2421.5 (12) Å ³	0.34 × 0.31 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer	17509 measured reflections
φ and ω scans	2380 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1630 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.960, T_{\max} = 0.988$	$R_{\text{int}} = 0.048$
	$\theta_{\max} = 26.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.4398P]$
$R[F^2 > 2\sigma(F^2)] = 0.053$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.119$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.10$	$\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$
2380 reflections	$\Delta\rho_{\min} = -0.14 \text{ e \AA}^{-3}$
156 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, $^\circ$).

O1-C7	1.222 (2)	C1-C7	1.480 (3)
O2-C4	1.361 (2)	C3-C14	1.503 (3)
O2-C15	1.425 (2)	C7-C8	1.499 (3)
C4-O2-C15	119.04 (16)	O2-C4-C3	114.75 (16)
C6-C1-C7	122.92 (17)	O1-C7-C1	121.16 (18)
C2-C1-C7	119.31 (16)	C1-C7-C8	119.93 (16)
C2-C3-C14	121.87 (17)	C9-C8-C7	122.07 (17)
C4-C3-C14	120.42 (17)	C13-C8-C7	118.60 (19)
O2-C4-C5	124.29 (17)		

All H atoms were positioned geometrically and were refined as riding on their carrier atoms, with C_{ar}-H = 0.93 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}_{\text{ar}})$, and C_{methyl}-H = 0.96 Å with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PLATON.

The authors acknowledge the use of the CCD facility at the Indian Institute of Science, Bangalore, set up under the IRHPA-DST programme.

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