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ISSN 2414-3146

Methyl 2-(benzoyloxy)benzoate

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Received 16 May 2016
Accepted 30 May 2016

Edited by H. Stoeckli-Evans, University of Neuchâtel, Switzerland

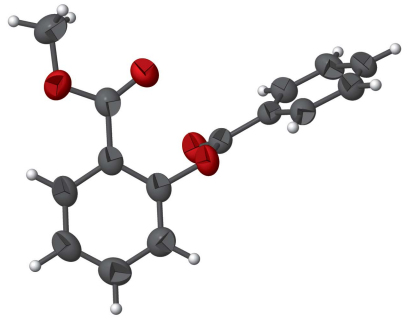
Keywords: crystal structure; benzoate derivative; medicinal importance; C—H$\cdots\pi$ interactions; offset π–π interactions.

CCDC reference: 1482493

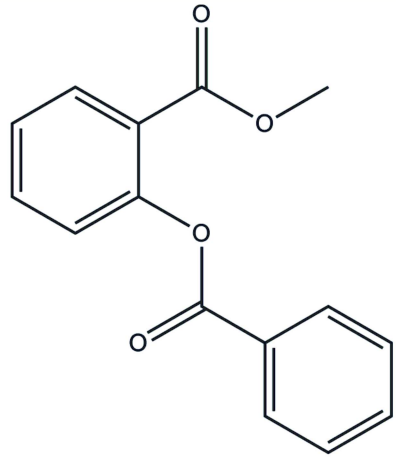
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{15}H_{12}O_4$, the dihedral angle between the two aryl rings is 68.19 (9)°. In the crystal, molecules are linked by C—H$\cdots\pi$ interactions forming chains along the *b*-axis direction. The chains are linked by offset π–π interactions [intercentroid distance = 3.6806 (14) Å], forming sheets lying parallel to (10$\bar{1}$).

3D view



Chemical scheme



Structure description

Benzyl benzoate and its derivatives represent an interesting class of compounds, as they improve the stability and odour characteristics of products in which they are used as the main ingredients. Benzoate derivatives have drawn much attention because of their medicinal activities, such as anti-microbial (Ankersen *et al.*, 1997) and anticancer (Revesz *et al.*, 2004). In addition, a series of benzyloxybenzaldehyde derivatives were prepared and tested against the HL-60 cell line for anticancer activity (Lin *et al.*, 2005). In view of the profound interest of these derivatives, we report herein on the synthesis and crystal structure of the title compound.

The molecular structure of the title compound is illustrated in Fig. 1. The dihedral angle between the benzene rings, C1–C6 and C10–C15, is 68.19 (9)°. The O9—C7═O8 group is almost coplanar with the aromatic ring C1–C6, with a dihedral angle of 8.11 (18)°, while it is inclined to the second benzene ring, C10–C15, by 74.8 (19) °. The O18—C16═O17 group is also almost coplanar with the benzene ring, C10–C15, to which it is attached with a dihedral angle of 5.1 (2)°.

There are no classical hydrogen bonds in the crystal structure. However, in the crystal, molecules are linked by C—H$\cdots\pi$ interactions, forming chains along the *b* axis direction (Table 1 and Fig. 2). The chains are linked by offset π–π interactions [$Cg1\cdots Cg1^{i}$ = 3.6806 (14) Å, where $Cg1$ is the centroid of ring C1–C6, interplanar distance =

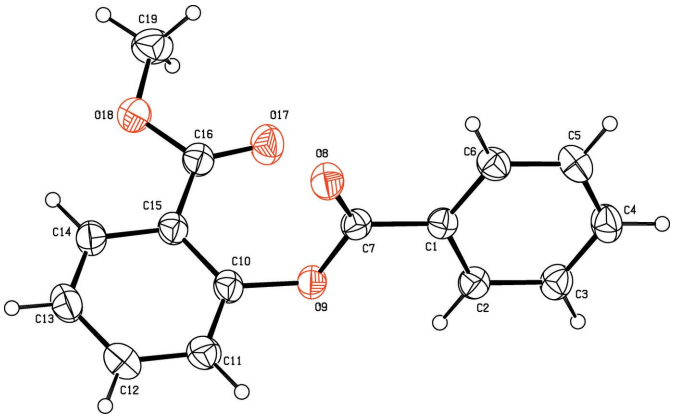


Figure 1
The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

3.4792 (7) Å, slippage = 1.201 Å; symmetry code: (i) $-x+1, -y, -z+2$], forming sheets lying parallel to $(10\bar{1})$; see Fig. 2.

Synthesis and crystallization

The solution of 2-hydroxybenzoic acid methyl ester (1.0 mmol) and benzoyl chloride (1.5 mmol) in pyridine was refluxed at 333 K. On completion of the reaction (monitored by TLC), the mixture was cooled to room temperature and pyridine was removed under reduced pressure. The residue obtained was dissolved in dichloromethane and washed with water. The separated organic layer was dried over anhydrous sodium sulfate and the solvent removed using a rotary evaporator. The crude product obtained was purified using silica gel column chromatography. Colourless block-like crystals were obtained by slow evaporation of a solution of the title compound in ethanol.

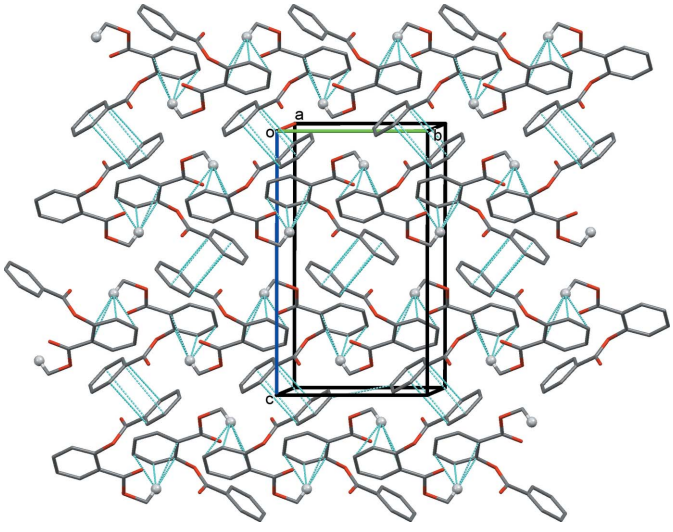


Figure 2
A view along the *a* axis of the crystal packing of the title compound. The C—H···π (see Table 1) and π–π interactions are represented by dashed lines, and for clarity only H atom H19*C* (grey ball) is included.

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

*Cg*2 is the centroid of the C10–C15 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19*C*···*Cg*2^{i}	0.96	2.83	3.625 (3)	140

Symmetry code: (i) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{12}O_4$
M_r	256.25
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	296
a, *b*, *c* (Å)	19.529 (5), 8.540 (2), 14.960 (4)
β (°)	93.334 (14)
V (Å^3)	2490.8 (11)
Z	8
Radiation type	Cu *K*α
μ (mm^{-1})	0.83
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6308, 2020, 1745
R_{int}	0.044
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.588
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.058, 0.156, 1.19
No. of reflections	2020
No. of parameters	174
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.28

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2008).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors would like to thank the SJB Institute of Technology, Kengeri, Bangalore, for their support. MM would like to thank the UGC, New Delhi, Government of India, for awarding project F.41–920/2012(SR).

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full crystallographic data

IUCrData (2016). **1**, x160867 [doi:10.1107/S2414314616008671]

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Methyl 2-(benzoyloxy)benzoate

Crystal data

$C_{15}H_{12}O_4$
M_r = 256.25
Monoclinic, $C2/c$
Hall symbol: -C 2yc
a = 19.529 (5) Å
b = 8.540 (2) Å
c = 14.960 (4) Å
β = 93.334 (14)°
V = 2490.8 (11) Å^3
Z = 8
$F(000)$ = 1072
D_x = 1.367 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2020 reflections
θ = 4.5–65.1°
μ = 0.83 mm^{-1}
T = 296 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
6308 measured reflections
2020 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.044
θ_{max} = 65.1°, θ_{min} = 4.5°
h = −22→22
k = −10→9
l = −11→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.156
S = 1.19
2020 reflections
174 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), FC*=KFC[1+0.001XFC$^2\Lambda^3$/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.0090 (9)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O8	0.33968 (6)	0.10878 (15)	0.89122 (8)	0.0565 (5)
O9	0.42601 (6)	0.15246 (15)	0.80123 (8)	0.0492 (4)
O17	0.33585 (7)	−0.02518 (16)	0.69827 (10)	0.0652 (5)
O18	0.26162 (7)	0.10223 (16)	0.60645 (8)	0.0610 (5)
C1	0.43639 (8)	−0.06021 (19)	0.90068 (10)	0.0422 (5)
C2	0.50358 (8)	−0.0827 (2)	0.87647 (11)	0.0466 (5)
C3	0.54205 (9)	−0.2026 (2)	0.91535 (12)	0.0536 (6)
C4	0.51445 (10)	−0.2986 (2)	0.97716 (13)	0.0574 (6)
C5	0.44773 (10)	−0.2784 (2)	1.00044 (13)	0.0586 (6)
C6	0.40884 (9)	−0.1591 (2)	0.96259 (12)	0.0512 (6)
C7	0.39454 (8)	0.0715 (2)	0.86554 (10)	0.0434 (5)
C10	0.38930 (8)	0.2783 (2)	0.76212 (11)	0.0457 (5)
C11	0.41229 (10)	0.4253 (2)	0.78495 (12)	0.0558 (6)
C12	0.37880 (11)	0.5548 (2)	0.74854 (14)	0.0631 (7)
C13	0.32272 (11)	0.5356 (2)	0.68963 (14)	0.0617 (7)
C14	0.30113 (10)	0.3877 (2)	0.66520 (13)	0.0543 (6)
C15	0.33425 (8)	0.2550 (2)	0.70022 (11)	0.0455 (5)
C16	0.31208 (9)	0.0961 (2)	0.67047 (11)	0.0473 (6)
C19	0.23598 (12)	−0.0457 (3)	0.57343 (14)	0.0693 (7)
H2	0.52230	−0.01750	0.83450	0.0560*
H3	0.58690	−0.21840	0.89960	0.0640*
H4	0.54100	−0.37830	1.00370	0.0690*
H5	0.42910	−0.34530	1.04170	0.0700*
H6	0.36390	−0.14480	0.97850	0.0610*
H11	0.45030	0.43780	0.82480	0.0670*
H12	0.39420	0.65490	0.76380	0.0760*
H13	0.29940	0.62280	0.66630	0.0740*
H14	0.26370	0.37600	0.62450	0.0650*
H19A	0.27090	−0.09800	0.54210	0.1040*
H19B	0.19650	−0.02870	0.53330	0.1040*
H19C	0.22340	−0.10930	0.62280	0.1040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O8	0.0495 (7)	0.0643 (9)	0.0571 (8)	0.0116 (5)	0.0150 (6)	0.0101 (6)
O9	0.0422 (7)	0.0571 (7)	0.0485 (7)	0.0056 (5)	0.0048 (5)	0.0130 (5)
O17	0.0709 (9)	0.0517 (8)	0.0717 (9)	0.0106 (6)	−0.0062 (6)	−0.0001 (6)
O18	0.0625 (8)	0.0631 (9)	0.0558 (8)	−0.0080 (6)	−0.0089 (6)	0.0073 (6)

C1	0.0447 (9)	0.0429 (9)	0.0387 (8)	−0.0001 (7)	0.0006 (6)	−0.0036 (6)
C2	0.0464 (9)	0.0495 (10)	0.0441 (9)	0.0008 (7)	0.0032 (7)	0.0002 (7)
C3	0.0509 (10)	0.0502 (10)	0.0594 (10)	0.0083 (7)	0.0008 (8)	−0.0031 (8)
C4	0.0689 (12)	0.0417 (10)	0.0608 (11)	0.0090 (8)	−0.0029 (8)	0.0010 (8)
C5	0.0727 (12)	0.0450 (10)	0.0589 (11)	0.0017 (8)	0.0118 (9)	0.0065 (8)
C6	0.0534 (9)	0.0490 (10)	0.0521 (10)	0.0007 (7)	0.0099 (7)	0.0012 (7)
C7	0.0434 (9)	0.0495 (9)	0.0372 (8)	−0.0008 (7)	0.0015 (6)	0.0002 (6)
C10	0.0445 (9)	0.0522 (10)	0.0410 (9)	0.0046 (7)	0.0088 (7)	0.0090 (7)
C11	0.0576 (10)	0.0602 (11)	0.0495 (10)	−0.0071 (8)	0.0027 (8)	0.0056 (8)
C12	0.0763 (13)	0.0502 (11)	0.0636 (12)	−0.0069 (9)	0.0108 (10)	0.0053 (9)
C13	0.0671 (12)	0.0515 (11)	0.0670 (12)	0.0088 (9)	0.0093 (9)	0.0148 (9)
C14	0.0505 (10)	0.0572 (11)	0.0552 (11)	0.0046 (7)	0.0029 (8)	0.0135 (8)
C15	0.0440 (9)	0.0517 (10)	0.0415 (9)	0.0036 (7)	0.0086 (7)	0.0080 (7)
C16	0.0457 (9)	0.0554 (11)	0.0416 (9)	0.0030 (7)	0.0085 (7)	0.0053 (7)
C19	0.0731 (13)	0.0728 (13)	0.0614 (12)	−0.0175 (10)	−0.0019 (9)	−0.0053 (10)

Geometric parameters (Å, º)

O8—C7	1.202 (2)	C12—C13	1.375 (3)
O9—C7	1.360 (2)	C13—C14	1.374 (3)
O9—C10	1.401 (2)	C14—C15	1.392 (2)
O17—C16	1.200 (2)	C15—C16	1.485 (2)
O18—C16	1.335 (2)	C2—H2	0.9300
O18—C19	1.436 (3)	C3—H3	0.9300
C1—C2	1.395 (2)	C4—H4	0.9300
C1—C6	1.385 (2)	C5—H5	0.9300
C1—C7	1.470 (2)	C6—H6	0.9300
C2—C3	1.379 (2)	C11—H11	0.9300
C3—C4	1.370 (3)	C12—H12	0.9300
C4—C5	1.379 (3)	C13—H13	0.9300
C5—C6	1.373 (3)	C14—H14	0.9300
C10—C11	1.370 (2)	C19—H19A	0.9600
C10—C15	1.392 (2)	C19—H19B	0.9600
C11—C12	1.381 (3)	C19—H19C	0.9600
C7—O9—C10	116.36 (13)	O18—C16—C15	111.69 (14)
C16—O18—C19	116.14 (15)	C1—C2—H2	120.00
C2—C1—C6	119.84 (15)	C3—C2—H2	120.00
C2—C1—C7	121.64 (14)	C2—C3—H3	120.00
C6—C1—C7	118.45 (14)	C4—C3—H3	120.00
C1—C2—C3	119.38 (15)	C3—C4—H4	120.00
C2—C3—C4	120.22 (16)	C5—C4—H4	120.00
C3—C4—C5	120.69 (17)	C4—C5—H5	120.00
C4—C5—C6	119.79 (17)	C6—C5—H5	120.00
C1—C6—C5	120.07 (16)	C1—C6—H6	120.00
O8—C7—O9	122.63 (15)	C5—C6—H6	120.00
O8—C7—C1	125.17 (15)	C10—C11—H11	120.00
O9—C7—C1	112.17 (13)	C12—C11—H11	120.00

O9—C10—C11	116.52 (15)	C11—C12—H12	120.00
O9—C10—C15	121.67 (15)	C13—C12—H12	120.00
C11—C10—C15	121.75 (16)	C12—C13—H13	120.00
C10—C11—C12	119.66 (17)	C14—C13—H13	120.00
C11—C12—C13	119.92 (17)	C13—C14—H14	119.00
C12—C13—C14	120.04 (17)	C15—C14—H14	119.00
C13—C14—C15	121.30 (18)	O18—C19—H19A	110.00
C10—C15—C14	117.26 (16)	O18—C19—H19B	109.00
C10—C15—C16	122.05 (15)	O18—C19—H19C	109.00
C14—C15—C16	120.68 (15)	H19A—C19—H19B	109.00
O17—C16—O18	122.51 (16)	H19A—C19—H19C	109.00
O17—C16—C15	125.81 (16)	H19B—C19—H19C	110.00

C10—O9—C7—O8	−3.4 (2)	C4—C5—C6—C1	0.5 (3)
C10—O9—C7—C1	178.57 (13)	O9—C10—C11—C12	179.64 (16)
C7—O9—C10—C11	107.89 (17)	C15—C10—C11—C12	2.5 (3)
C7—O9—C10—C15	−74.9 (2)	O9—C10—C15—C14	−180.00 (16)
C19—O18—C16—O17	1.2 (3)	O9—C10—C15—C16	−1.3 (2)
C19—O18—C16—C15	−179.50 (15)	C11—C10—C15—C14	−2.9 (3)
C6—C1—C2—C3	−0.7 (2)	C11—C10—C15—C16	175.71 (16)
C7—C1—C2—C3	176.19 (15)	C10—C11—C12—C13	−0.1 (3)
C2—C1—C6—C5	0.4 (3)	C11—C12—C13—C14	−1.7 (3)
C7—C1—C6—C5	−176.55 (16)	C12—C13—C14—C15	1.2 (3)
C2—C1—C7—O8	−170.75 (16)	C13—C14—C15—C10	1.1 (3)
C2—C1—C7—O9	7.2 (2)	C13—C14—C15—C16	−177.55 (18)
C6—C1—C7—O8	6.1 (3)	C10—C15—C16—O17	3.7 (3)
C6—C1—C7—O9	−175.94 (14)	C10—C15—C16—O18	−175.58 (15)
C1—C2—C3—C4	0.0 (3)	C14—C15—C16—O17	−177.75 (18)
C2—C3—C4—C5	0.9 (3)	C14—C15—C16—O18	3.0 (2)
C3—C4—C5—C6	−1.2 (3)		

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C10–C15 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C19—H19*C*···*Cg*2^{i}	0.96	2.83	3.625 (3)	140

Symmetry code: (i) −*x*+1/2, *y*−1/2, −*z*+3/2.