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2-(4-Methylphenyl)-2-oxoethyl 3,4-dimethoxybenzoate

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Keywords: crystal structure; benzoate derivative; hydrogen bonding.

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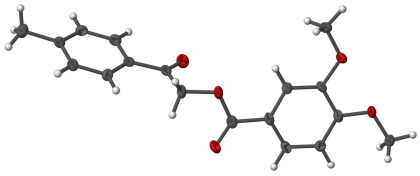
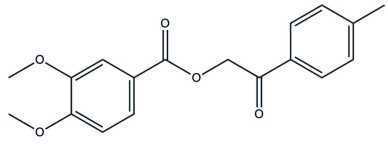
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{18}H_{18}O_5$, the dihedral angle between the mean planes of the two aromatic rings is 66.55 (8)°. The crystal packing features by intermolecular C—H···O contacts.

3D view



Chemical scheme



Structure description

Benzoate and its derivatives are well known heterocyclic compounds, which have a variety of biological activities. As a part of our ongoing research on benzoate derivatives (Kumar *et al.*, 2016), the title compound was prepared and characterized by single-crystal X-ray diffraction.

In the molecule (Fig. 1), the dihedral angle between the mean planes of the benzoate (C14–C19) and phenyl groups (C2–C7) is 66.55 (8)°. The crystal packing (Fig. 2) features C—H···O contacts (Table 1).

Synthesis and crystallization

Potassium carbonate was added to the solution of 3,4-dimethoxybenzoic acid (1) in water and the mixture was stirred for 30 min. Then, a solution of 2-bromo-1-(*p*-tolyl)ethanone (2) in ethanol was added and the reaction mixture was heated under reflux for 6 h. After completion of the reaction, ethanol was removed under reduced pressure. The obtained crystals were collected by filtration and recrystallized using ethanol.

Refinement

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

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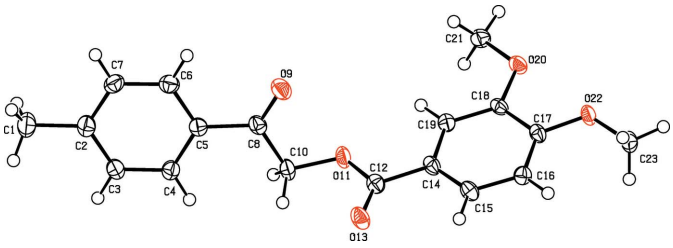

Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles of arbitrary radius.

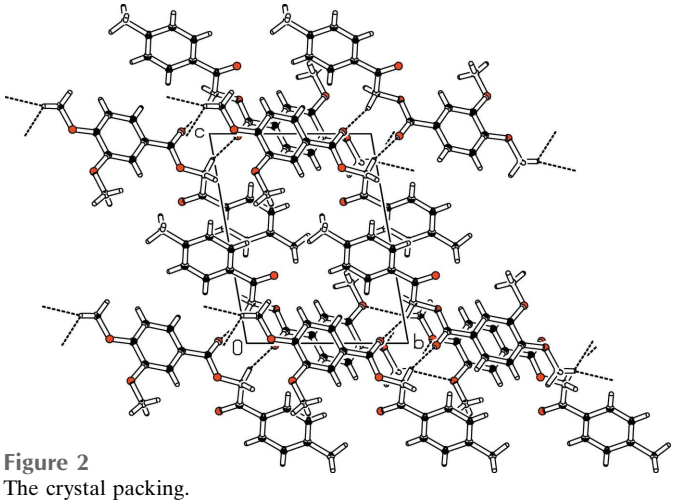

Figure 2
The crystal packing.

Acknowledgements

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References

Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···O13^{i}	0.97	2.48	3.399 (2)	157
C23—H25B···O20^{ii}	0.96	2.50	3.447 (2)	168
C23—H25B···O22^{ii}	0.96	2.57	3.281 (2)	131

Symmetry codes: (i) $-x, -y+2, -z+2$; (ii) $-x+1, -y, -z+2$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{18}O_5$
M_r	314.32
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	293
a, b, c (Å)	7.9805 (3), 8.6087 (3), 11.4674 (4)
α, β, γ (°)	99.678 (2), 99.173 (2), 91.495 (2)
V (Å^3)	765.55 (5)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.82
Crystal size (mm)	0.30 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker X8 Proteum
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10314, 2522, 2053
R_{int}	0.055
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.585
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.169, 1.06
No. of reflections	2522
No. of parameters	212
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.37

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

Kumar, S., Chandra, Dileep, C. S., Mahendra, M. & Doreswamy, B. H. (2016). *IUCrData*, **1**, x160867.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
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full crystallographic data

IUCrData (2017). **2**, x170183 [https://doi.org/10.1107/S2414314617001833]

2-(4-Methylphenyl)-2-oxoethyl 3,4-dimethoxybenzoate

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2-(4-Methylphenyl)-2-oxoethyl 3,4-dimethoxybenzoate

Crystal data

$C_{18}H_{18}O_5$
M_r = 314.32
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.9805 (3) Å
b = 8.6087 (3) Å
c = 11.4674 (4) Å
α = 99.678 (2)°
β = 99.173 (2)°
γ = 91.495 (2)°
V = 765.55 (5) Å^3
Z = 2
$F(000)$ = 332
D_x = 1.364 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 2522 reflections
θ = 5.2–64.5°
μ = 0.82 mm^{-1}
T = 293 K
Block, light yellow
0.30 × 0.25 × 0.20 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Rotating Anode
Graphite monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
10314 measured reflections
2522 independent reflections
2053 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 64.5°, θ_{min} = 5.2°
h = −9→9
k = −10→9
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.169
S = 1.06
2522 reflections
212 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
$w = 1/[\sigma^2(F_o^2) + (0.121P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: shelxl, $FC^*=KFC[1+0.001XFC^2\Lambda^3/SIN(2\Theta)]^{-1/4}$
Extinction coefficient: 0.013 (2)

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}
O9	−0.09184 (18)	0.75607 (14)	0.67623 (13)	0.0300 (5)
O11	0.19147 (17)	0.78179 (14)	0.83817 (13)	0.0261 (5)
O13	0.08365 (17)	0.82775 (14)	1.00977 (13)	0.0288 (5)
O20	0.42611 (16)	0.24229 (13)	0.82041 (12)	0.0234 (4)
O22	0.39935 (16)	0.15667 (14)	1.02101 (12)	0.0228 (4)
C1	−0.2712 (3)	1.3978 (3)	0.4619 (2)	0.0363 (7)
C2	−0.2107 (2)	1.2678 (2)	0.52842 (18)	0.0251 (6)
C3	−0.0852 (2)	1.2978 (2)	0.62938 (18)	0.0246 (6)
C4	−0.0257 (2)	1.1782 (2)	0.68962 (18)	0.0230 (6)
C5	−0.0911 (2)	1.0230 (2)	0.64858 (17)	0.0204 (6)
C6	−0.2189 (2)	0.9924 (2)	0.54882 (19)	0.0252 (6)
C7	−0.2772 (2)	1.1132 (2)	0.48971 (19)	0.0262 (6)
C8	−0.0261 (2)	0.8887 (2)	0.70705 (17)	0.0218 (6)
C10	0.1250 (2)	0.9248 (2)	0.80637 (19)	0.0246 (6)
C12	0.1576 (2)	0.7440 (2)	0.94225 (18)	0.0222 (6)
C14	0.2227 (2)	0.5891 (2)	0.96242 (18)	0.0210 (6)
C15	0.2037 (2)	0.5396 (2)	1.06763 (19)	0.0225 (6)
C16	0.2605 (2)	0.3952 (2)	1.09167 (18)	0.0211 (6)
C17	0.3360 (2)	0.29900 (19)	1.00736 (18)	0.0198 (6)
C18	0.3534 (2)	0.34750 (19)	0.89866 (18)	0.0189 (6)
C19	0.2971 (2)	0.4923 (2)	0.87634 (18)	0.0197 (6)
C21	0.4526 (3)	0.2905 (2)	0.71130 (18)	0.0257 (6)
C23	0.3883 (3)	0.1034 (2)	1.13133 (19)	0.0263 (6)
H3	−0.04010	1.40040	0.65730	0.0300*
H4	0.05780	1.20130	0.75740	0.0280*
H6	−0.26570	0.89020	0.52160	0.0300*
H7	−0.36240	1.09050	0.42290	0.0310*
H10A	0.21260	0.98460	0.78040	0.0300*
H10B	0.09130	0.98870	0.87600	0.0300*
H17	0.15210	0.60370	1.12370	0.0270*
H19	0.30870	0.52490	0.80460	0.0240*
H22	0.24800	0.36330	1.16350	0.0250*
H25A	0.44430	0.18050	1.19730	0.0390*
H25B	0.44210	0.00500	1.13200	0.0390*
H25C	0.27090	0.08890	1.13870	0.0390*
H28A	0.34490	0.30480	0.66470	0.0380*
H28B	0.51060	0.21090	0.66650	0.0380*
H28C	0.52010	0.38810	0.72930	0.0380*
H99A	−0.24360	1.49800	0.51370	0.0540*

H99B	−0.39200	1.38420	0.43670	0.0540*
H99C	−0.21640	1.39410	0.39290	0.0540*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O9	0.0366 (9)	0.0196 (7)	0.0341 (9)	−0.0002 (5)	0.0066 (7)	0.0049 (6)
O11	0.0334 (8)	0.0210 (7)	0.0272 (9)	0.0128 (5)	0.0084 (6)	0.0090 (6)
O13	0.0348 (8)	0.0227 (7)	0.0329 (9)	0.0118 (6)	0.0152 (7)	0.0055 (6)
O20	0.0313 (8)	0.0185 (7)	0.0220 (8)	0.0066 (5)	0.0096 (6)	0.0021 (5)
O22	0.0286 (8)	0.0167 (6)	0.0258 (8)	0.0085 (5)	0.0077 (6)	0.0069 (5)
C1	0.0465 (14)	0.0344 (11)	0.0317 (13)	0.0144 (9)	0.0075 (11)	0.0134 (9)
C2	0.0264 (11)	0.0285 (10)	0.0241 (12)	0.0107 (8)	0.0109 (9)	0.0077 (8)
C3	0.0286 (11)	0.0205 (9)	0.0265 (12)	0.0043 (7)	0.0080 (9)	0.0057 (8)
C4	0.0225 (10)	0.0242 (9)	0.0228 (11)	0.0036 (7)	0.0032 (8)	0.0059 (8)
C5	0.0220 (10)	0.0218 (10)	0.0194 (11)	0.0042 (7)	0.0090 (8)	0.0039 (7)
C6	0.0261 (10)	0.0243 (10)	0.0239 (12)	0.0017 (7)	0.0040 (9)	0.0010 (8)
C7	0.0269 (11)	0.0318 (10)	0.0190 (11)	0.0072 (8)	0.0028 (8)	0.0024 (8)
C8	0.0259 (10)	0.0197 (9)	0.0224 (11)	0.0051 (7)	0.0106 (9)	0.0040 (8)
C10	0.0282 (11)	0.0178 (9)	0.0296 (12)	0.0070 (7)	0.0047 (9)	0.0082 (8)
C12	0.0182 (10)	0.0204 (9)	0.0276 (12)	0.0013 (7)	0.0027 (9)	0.0040 (8)
C14	0.0165 (10)	0.0171 (9)	0.0287 (12)	0.0033 (7)	0.0029 (8)	0.0029 (8)
C15	0.0204 (10)	0.0195 (9)	0.0288 (12)	0.0052 (7)	0.0089 (9)	0.0027 (8)
C16	0.0208 (10)	0.0216 (9)	0.0214 (11)	0.0017 (7)	0.0041 (8)	0.0047 (8)
C17	0.0163 (9)	0.0161 (9)	0.0266 (12)	0.0019 (7)	0.0023 (8)	0.0039 (8)
C18	0.0175 (9)	0.0158 (9)	0.0226 (11)	0.0033 (7)	0.0045 (8)	−0.0007 (7)
C19	0.0179 (9)	0.0187 (9)	0.0225 (11)	0.0006 (7)	0.0026 (8)	0.0042 (8)
C21	0.0327 (11)	0.0246 (9)	0.0208 (11)	0.0038 (8)	0.0088 (9)	0.0027 (8)
C23	0.0346 (11)	0.0220 (9)	0.0257 (12)	0.0083 (8)	0.0085 (9)	0.0093 (8)

Geometric parameters (Å, º)

O9—C8	1.216 (2)	C16—C17	1.388 (3)
O11—C10	1.433 (2)	C17—C18	1.405 (3)
O11—C12	1.354 (2)	C18—C19	1.386 (2)
O13—C12	1.203 (2)	C1—H99A	0.9600
O20—C18	1.372 (2)	C1—H99B	0.9600
O20—C21	1.427 (2)	C1—H99C	0.9600
O22—C17	1.361 (2)	C3—H3	0.9300
O22—C23	1.431 (2)	C4—H4	0.9300
C1—C2	1.505 (3)	C6—H6	0.9300
C2—C3	1.388 (3)	C7—H7	0.9300
C2—C7	1.393 (3)	C10—H10A	0.9700
C3—C4	1.387 (3)	C10—H10B	0.9700
C4—C5	1.398 (2)	C15—H17	0.9300
C5—C6	1.390 (3)	C16—H22	0.9300
C5—C8	1.496 (3)	C19—H19	0.9300
C6—C7	1.385 (3)	C21—H28A	0.9600

C8—C10	1.507 (3)	C21—H28B	0.9600
C12—C14	1.484 (2)	C21—H28C	0.9600
C14—C15	1.374 (3)	C23—H25A	0.9600
C14—C19	1.399 (3)	C23—H25B	0.9600
C15—C16	1.390 (2)	C23—H25C	0.9600
C10—O11—C12	116.65 (14)	H99A—C1—H99B	110.00
C18—O20—C21	116.91 (14)	H99A—C1—H99C	109.00
C17—O22—C23	117.24 (15)	H99B—C1—H99C	109.00
C1—C2—C3	121.05 (17)	C2—C3—H3	119.00
C1—C2—C7	121.07 (18)	C4—C3—H3	119.00
C3—C2—C7	117.87 (17)	C3—C4—H4	120.00
C2—C3—C4	121.50 (17)	C5—C4—H4	120.00
C3—C4—C5	120.11 (17)	C5—C6—H6	120.00
C4—C5—C6	118.75 (16)	C7—C6—H6	120.00
C4—C5—C8	122.23 (16)	C2—C7—H7	119.00
C6—C5—C8	119.01 (16)	C6—C7—H7	119.00
C5—C6—C7	120.44 (17)	O11—C10—H10A	110.00
C2—C7—C6	121.30 (18)	O11—C10—H10B	110.00
O9—C8—C5	121.72 (16)	C8—C10—H10A	110.00
O9—C8—C10	121.22 (16)	C8—C10—H10B	110.00
C5—C8—C10	117.06 (15)	H10A—C10—H10B	108.00
O11—C10—C8	110.52 (15)	C14—C15—H17	120.00
O11—C12—O13	123.23 (16)	C16—C15—H17	119.00
O11—C12—C14	112.10 (15)	C15—C16—H22	120.00
O13—C12—C14	124.67 (18)	C17—C16—H22	120.00
C12—C14—C15	118.14 (16)	C14—C19—H19	120.00
C12—C14—C19	121.80 (17)	C18—C19—H19	120.00
C15—C14—C19	120.04 (16)	O20—C21—H28A	109.00
C14—C15—C16	121.05 (17)	O20—C21—H28B	109.00
C15—C16—C17	119.34 (18)	O20—C21—H28C	109.00
O22—C17—C16	125.00 (17)	H28A—C21—H28B	109.00
O22—C17—C18	115.00 (16)	H28A—C21—H28C	109.00
C16—C17—C18	119.99 (16)	H28B—C21—H28C	110.00
O20—C18—C17	115.38 (15)	O22—C23—H25A	109.00
O20—C18—C19	124.65 (17)	O22—C23—H25B	109.00
C17—C18—C19	119.97 (17)	O22—C23—H25C	109.00
C14—C19—C18	119.59 (18)	H25A—C23—H25B	110.00
C2—C1—H99A	109.00	H25A—C23—H25C	109.00
C2—C1—H99B	109.00	H25B—C23—H25C	109.00
C2—C1—H99C	109.00		
C12—O11—C10—C8	−105.06 (18)	C5—C6—C7—C2	−0.3 (3)
C10—O11—C12—O13	−3.9 (3)	O9—C8—C10—O11	10.7 (2)
C10—O11—C12—C14	176.33 (14)	C5—C8—C10—O11	−169.40 (15)
C21—O20—C18—C17	−177.34 (16)	O11—C12—C14—C15	177.09 (16)
C21—O20—C18—C19	3.3 (2)	O11—C12—C14—C19	−4.8 (2)
C23—O22—C17—C16	−0.6 (3)	O13—C12—C14—C15	−2.7 (3)

C23—O22—C17—C18	178.63 (16)	O13—C12—C14—C19	175.41 (17)
C1—C2—C3—C4	−178.44 (18)	C12—C14—C15—C16	179.44 (16)
C7—C2—C3—C4	0.7 (3)	C19—C14—C15—C16	1.3 (3)
C1—C2—C7—C6	178.34 (18)	C12—C14—C19—C18	−178.94 (16)
C3—C2—C7—C6	−0.8 (3)	C15—C14—C19—C18	−0.9 (3)
C2—C3—C4—C5	0.5 (3)	C14—C15—C16—C17	−0.7 (3)
C3—C4—C5—C6	−1.6 (3)	C15—C16—C17—O22	178.78 (16)
C3—C4—C5—C8	177.30 (16)	C15—C16—C17—C18	−0.4 (3)
C4—C5—C6—C7	1.5 (3)	O22—C17—C18—O20	2.2 (2)
C8—C5—C6—C7	−177.43 (16)	O22—C17—C18—C19	−178.44 (15)
C4—C5—C8—O9	174.62 (17)	C16—C17—C18—O20	−178.56 (15)
C4—C5—C8—C10	−5.3 (2)	C16—C17—C18—C19	0.8 (3)
C6—C5—C8—O9	−6.5 (3)	O20—C18—C19—C14	179.13 (16)
C6—C5—C8—C10	173.57 (16)	C17—C18—C19—C14	−0.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O13	0.97	2.24	2.671 (2)	106
C10—H10*B*···O13^{i}	0.97	2.48	3.399 (2)	157
C19—H19···O11	0.93	2.42	2.733 (2)	100
C23—H25*B*···O20^{ii}	0.96	2.50	3.447 (2)	168
C23—H25*B*···O22^{ii}	0.96	2.57	3.281 (2)	131

Symmetry codes: (i) −*x*, −*y*+2, −*z*+2; (ii) −*x*+1, −*y*, −*z*+2.