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Crystal structure of 3-[(2-acetylphenoxy)-carbonyl]benzoic acid

Mohammad Shoaib,[a] Ismail Shah,[a] Syed Wadood Ali Shah,[a] Muhammad Nawaz Tahir,[b]* Shafi Ullah[a] and Muhammad Ayaz[a]

[a]Department of Pharmacy, University of Malakand, Khyber Pakhtunkhwa, Pakistan, and [b]Department of Physics, University of Sargodha, Sargodha, Punjab, Pakistan. *Correspondence e-mail: dmntahir_uos@yahoo.com

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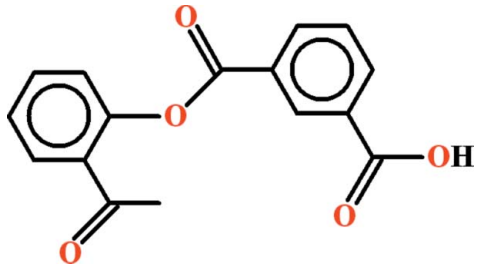
In the title compound, $C_{16}H_{12}O_5$, synthesized from isopthaloyl chloride and 2′-hydroxyacetophenone, the dihedral angle between the planes of the aromatic rings is 71.37 (9)°. In the crystal, carboxylic acid inversion dimers generate $R_2^2(8)$ loops. The dimers are linked by C—H···O interactions, generating (101) sheets.

Keywords: crystal structure; 3-[(2-acetylphenoxy)carbonyl]benzoic acid; hydrogen bonding; 2′-hydroxyacetophenone; isopthaloyl chloride.

CCDC reference: 1027627

1. Related literature

For related structures, see: Derissen (1974); Tanimoto *et al.* (1973).



2. Experimental

2.1. Crystal data

$C_{16}H_{12}O_5$ M_r = 284.26
Monoclinic, $P2_1/n$
a = 13.5081 (10) Å
b = 7.4743 (6) Å
c = 13.9421 (11) Å
β = 106.671 (3)°
V = 1348.48 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.38 × 0.28 × 0.25 mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.963, T_{max} = 0.977
10280 measured reflections
2655 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.115
S = 1.03
2655 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.84	2.6623 (18)	175
C4—H4···O5^{ii}	0.93	2.58	3.257 (3)	130

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON*.

Acknowledgements

The authors are grateful to the University of Malakand, Khyber Pakhtunkhwa, Pakistan, for provision of laboratory facilities for carrying out this research work.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7293).

References

Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Derissen, J. L. (1974). *Acta Cryst.* B**30**, 2764–2765.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Tanimoto, Y., Kobayashi, H., Nagakura, S. & Saito, Y. (1973). *Acta Cryst.* B**29**, 1822–1826.

supporting information

Acta Cryst. (2014). E**70**, o1153 [doi:10.1107/S1600536814021904]

Crystal structure of 3-[(2-acetylphenoxy)carbonyl]benzoic acid

Mohammad Shoaib, Ismail Shah, Syed Wadood Ali Shah, Muhammad Nawaz Tahir, Shafi Ullah and Muhammad Ayaz

S1. Comment

The title compound (I), (Fig. 1) has been synthesized for forming different metal complexes. The crystal structures of isophthalic acid and acetophenone have been published by (Derissen, 1974) and (Tanimoto, *et al.*, 1973) which are related to the title compound (I).

In (I) the group A (C1—C8/O1—O4) being like a part of isophthalic acid and benzene ring attached to it B (C9—C13) are almost planar with r. m. s. deviation of 0.0308 and 0.0034 Å, respectively. The dihedral angle between A/B is 71.98 (5)°. The acetaldehyde group C (O5/C15/C16) attached to ring B is of course planar. The dihedral angle between B/C is 9.56 (23)°. The molecules are dimerized due to coventional H-bondings of O—H···O type (Table 1, Fig. 2) forming $R_2^2(8)$ loop. The dimers are further interlinked due to C—H···O bondings where C—H is of benzene containing carboxylate and O is of acetaldehyde group.

S2. Experimental

Isopthaloyl chloride (25 mmol) and 2′-hydroxyacetophenone (35 mmol) were refluxed in the aquauos solution of pyridine for 30 min. The mixture was cooled to room temperature and added to a beaker containing 2 N HCl. The crushed ice was added and stirred vigorously. The precipitate formed were obtained though filteration. The column chromatography was done ethyl acetate:n-hexane (4:6) to obtain the pure product. Light yellow prisms were obtained after two days.

S3. Refinement

All H atoms were geometrically placed [(O–H = 0.82 Å (hydroxyl), C–H = 0.93 Å (aromatic) and C–H = 0.96 Å (methyl) and refined as riding with with $U_{iso}(H) = xU_{eq}(C, O)$, where $x = 1.5$ for hydroxy & methyl and $x = 1.2$ for aromatic H-atoms.

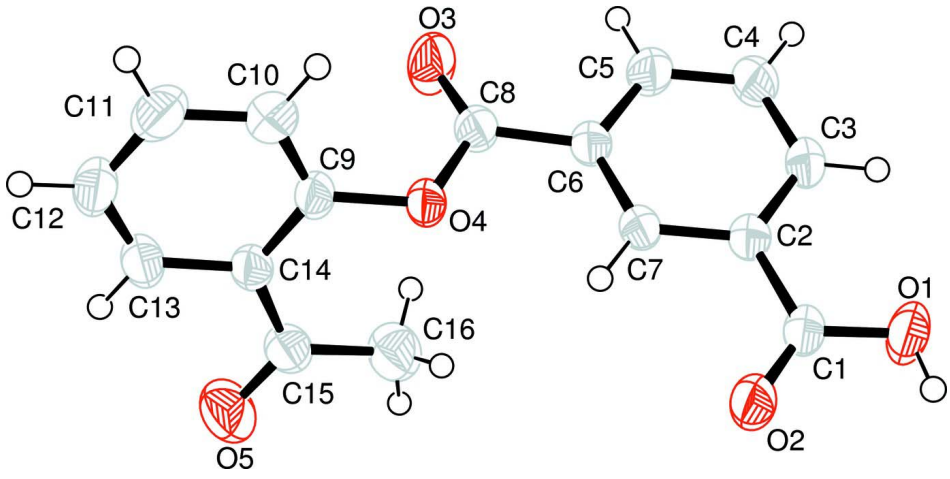


Figure 1

View of the title compound with displacement ellipsoids drawn at the 50% probability level.

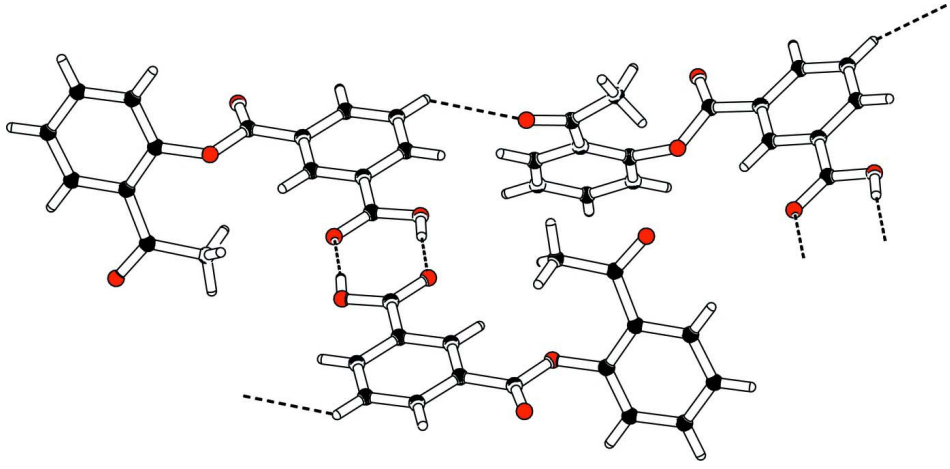


Figure 2

The partial packing (*PLATON*; Spek, 2009), which shows that molecules form dimers which are interlinked.

3-[(2-Acetylphenoxy)carbonyl]benzoic acid

Crystal data

$C_{16}H_{12}O_5$
$M_r = 284.26$
Monoclinic, $P2_1/n$
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$b = 7.4743$ (6) Å
$c = 13.9421$ (11) Å
$\beta = 106.671$ (3)°
$V = 1348.48$ (18) Å^3
$Z = 4$

$F(000) = 592$
$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1971 reflections
$\theta = 1.9$–26.0°
$\mu = 0.11$ mm^{-1}
$T = 296$ K
Prism, light yellow
0.38 × 0.28 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.963, T_{max} = 0.977
10280 measured reflections
2655 independent reflections
1971 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 26.0°, θ_{min} = 1.9°
h = −15→16
k = −6→9
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.115
S = 1.03
2655 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0505P)^2 + 0.4011P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness 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 threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.36130 (10)	0.0311 (2)	0.47366 (9)	0.0704 (5)
H1	0.4166	0.0276	0.5177	0.106*
O2	0.46474 (9)	−0.0058 (2)	0.37694 (9)	0.0627 (4)
O3	0.14695 (9)	−0.0066 (2)	−0.05814 (9)	0.0669 (4)
O4	0.31564 (8)	−0.05274 (16)	0.01634 (7)	0.0411 (3)
O5	0.48190 (13)	0.2965 (2)	−0.11562 (12)	0.0767 (5)
C1	0.37753 (13)	0.0154 (2)	0.38694 (11)	0.0435 (4)
C2	0.28422 (12)	0.0221 (2)	0.29912 (11)	0.0370 (4)
C3	0.18668 (13)	0.0478 (2)	0.31086 (12)	0.0435 (4)
H3	0.1791	0.0628	0.3746	0.052*
C4	0.10086 (13)	0.0512 (3)	0.22840 (13)	0.0505 (5)
H4	0.0355	0.0673	0.2366	0.061*
C5	0.11211 (13)	0.0306 (3)	0.13359 (13)	0.0471 (4)
H5	0.0542	0.0336	0.0780	0.056*
C6	0.20966 (12)	0.0052 (2)	0.12066 (11)	0.0368 (4)
C7	0.29593 (12)	0.0005 (2)	0.20355 (11)	0.0370 (4)

H7	0.3613	−0.0169	0.1955	0.044*
C8	0.21703 (12)	−0.0183 (2)	0.01684 (12)	0.0404 (4)
C9	0.33430 (12)	−0.0900 (2)	−0.07617 (11)	0.0381 (4)
C10	0.30562 (14)	−0.2564 (3)	−0.11818 (13)	0.0485 (4)
H10	0.2692	−0.3350	−0.0892	0.058*
C11	0.33147 (15)	−0.3054 (3)	−0.20375 (13)	0.0560 (5)
H11	0.3132	−0.4178	−0.2319	0.067*
C12	0.38433 (14)	−0.1876 (3)	−0.24715 (13)	0.0552 (5)
H12	0.4012	−0.2199	−0.3049	0.066*
C13	0.41194 (13)	−0.0225 (3)	−0.20493 (12)	0.0476 (5)
H13	0.4474	0.0559	−0.2351	0.057*
C14	0.38845 (12)	0.0318 (2)	−0.11770 (11)	0.0375 (4)
C15	0.42394 (13)	0.2153 (2)	−0.07883 (12)	0.0456 (4)
C16	0.38994 (17)	0.3003 (3)	0.00334 (16)	0.0614 (5)
H16A	0.4174	0.4195	0.0148	0.092*
H16B	0.4148	0.2311	0.0635	0.092*
H16C	0.3158	0.3051	−0.0154	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0455 (8)	0.1379 (14)	0.0272 (6)	0.0069 (9)	0.0097 (5)	−0.0055 (7)
O2	0.0363 (7)	0.1189 (13)	0.0323 (6)	0.0023 (7)	0.0089 (5)	−0.0024 (7)
O3	0.0391 (7)	0.1245 (13)	0.0328 (7)	0.0139 (7)	0.0037 (5)	0.0015 (7)
O4	0.0325 (6)	0.0653 (8)	0.0264 (5)	−0.0005 (5)	0.0097 (4)	−0.0007 (5)
O5	0.0916 (11)	0.0748 (10)	0.0791 (10)	−0.0290 (9)	0.0495 (9)	−0.0073 (8)
C1	0.0401 (10)	0.0629 (12)	0.0286 (8)	−0.0013 (8)	0.0116 (7)	−0.0012 (7)
C2	0.0356 (9)	0.0454 (10)	0.0305 (8)	−0.0018 (7)	0.0104 (6)	0.0008 (6)
C3	0.0430 (10)	0.0594 (11)	0.0321 (8)	−0.0023 (8)	0.0169 (7)	−0.0031 (7)
C4	0.0338 (9)	0.0763 (13)	0.0449 (10)	0.0017 (9)	0.0171 (8)	−0.0054 (9)
C5	0.0329 (9)	0.0706 (13)	0.0360 (9)	0.0009 (8)	0.0071 (7)	−0.0027 (8)
C6	0.0324 (8)	0.0483 (10)	0.0300 (8)	−0.0011 (7)	0.0097 (6)	0.0008 (7)
C7	0.0313 (8)	0.0500 (10)	0.0312 (8)	−0.0018 (7)	0.0113 (6)	0.0011 (7)
C8	0.0317 (8)	0.0574 (11)	0.0311 (8)	0.0010 (7)	0.0074 (7)	0.0027 (7)
C9	0.0317 (8)	0.0567 (11)	0.0256 (7)	0.0028 (7)	0.0077 (6)	−0.0007 (7)
C10	0.0474 (10)	0.0557 (11)	0.0418 (9)	−0.0069 (9)	0.0118 (8)	−0.0027 (8)
C11	0.0544 (11)	0.0617 (13)	0.0480 (10)	−0.0025 (9)	0.0082 (9)	−0.0173 (9)
C12	0.0499 (11)	0.0804 (15)	0.0376 (9)	0.0033 (10)	0.0161 (8)	−0.0135 (9)
C13	0.0411 (9)	0.0692 (13)	0.0362 (9)	0.0021 (8)	0.0172 (7)	0.0005 (8)
C14	0.0296 (8)	0.0522 (10)	0.0307 (8)	0.0036 (7)	0.0087 (6)	0.0012 (7)
C15	0.0404 (9)	0.0555 (11)	0.0419 (9)	−0.0006 (8)	0.0135 (8)	0.0047 (8)
C16	0.0714 (14)	0.0531 (12)	0.0682 (13)	−0.0077 (10)	0.0335 (11)	−0.0133 (10)

Geometric parameters (Å, º)

O1—C1	1.2946 (19)	C6—C8	1.489 (2)
O1—H1	0.8200	C7—H7	0.9300
O2—C1	1.236 (2)	C9—C10	1.382 (2)

O3—C8	1.1953 (19)	C9—C14	1.394 (2)
O4—C8	1.3587 (19)	C10—C11	1.385 (2)
O4—C9	1.4111 (17)	C10—H10	0.9300
O5—C15	1.215 (2)	C11—C12	1.379 (3)
C1—C2	1.485 (2)	C11—H11	0.9300
C2—C3	1.387 (2)	C12—C13	1.372 (3)
C2—C7	1.395 (2)	C12—H12	0.9300
C3—C4	1.379 (2)	C13—C14	1.402 (2)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.382 (2)	C14—C15	1.502 (2)
C4—H4	0.9300	C15—C16	1.493 (2)
C5—C6	1.393 (2)	C16—H16A	0.9600
C5—H5	0.9300	C16—H16B	0.9600
C6—C7	1.386 (2)	C16—H16C	0.9600

C1—O1—H1	109.5	C10—C9—O4	117.60 (14)
C8—O4—C9	118.33 (12)	C14—C9—O4	120.23 (15)
O2—C1—O1	122.65 (15)	C9—C10—C11	119.60 (17)
O2—C1—C2	121.50 (14)	C9—C10—H10	120.2
O1—C1—C2	115.84 (14)	C11—C10—H10	120.2
C3—C2—C7	120.03 (15)	C12—C11—C10	119.96 (18)
C3—C2—C1	121.19 (14)	C12—C11—H11	120.0
C7—C2—C1	118.78 (14)	C10—C11—H11	120.0
C4—C3—C2	120.27 (15)	C13—C12—C11	119.85 (16)
C4—C3—H3	119.9	C13—C12—H12	120.1
C2—C3—H3	119.9	C11—C12—H12	120.1
C3—C4—C5	119.91 (15)	C12—C13—C14	122.08 (17)
C3—C4—H4	120.0	C12—C13—H13	119.0
C5—C4—H4	120.0	C14—C13—H13	119.0
C4—C5—C6	120.44 (15)	C9—C14—C13	116.61 (16)
C4—C5—H5	119.8	C9—C14—C15	126.71 (14)
C6—C5—H5	119.8	C13—C14—C15	116.68 (15)
C7—C6—C5	119.71 (14)	O5—C15—C16	119.23 (18)
C7—C6—C8	122.18 (14)	O5—C15—C14	118.82 (16)
C5—C6—C8	118.11 (14)	C16—C15—C14	121.95 (15)
C6—C7—C2	119.65 (15)	C15—C16—H16A	109.5
C6—C7—H7	120.2	C15—C16—H16B	109.5
C2—C7—H7	120.2	H16A—C16—H16B	109.5
O3—C8—O4	122.75 (15)	C15—C16—H16C	109.5
O3—C8—C6	125.75 (15)	H16A—C16—H16C	109.5
O4—C8—C6	111.49 (13)	H16B—C16—H16C	109.5
C10—C9—C14	121.90 (14)		

O2—C1—C2—C3	−179.32 (18)	C5—C6—C8—O4	−176.17 (15)
O1—C1—C2—C3	1.4 (3)	C8—O4—C9—C10	−74.90 (19)
O2—C1—C2—C7	1.2 (3)	C8—O4—C9—C14	110.95 (17)
O1—C1—C2—C7	−178.08 (17)	C14—C9—C10—C11	0.3 (3)
C7—C2—C3—C4	0.4 (3)	O4—C9—C10—C11	−173.72 (15)

C1—C2—C3—C4	−179.14 (17)	C9—C10—C11—C12	−0.8 (3)
C2—C3—C4—C5	−0.6 (3)	C10—C11—C12—C13	0.5 (3)
C3—C4—C5—C6	0.4 (3)	C11—C12—C13—C14	0.2 (3)
C4—C5—C6—C7	0.0 (3)	C10—C9—C14—C13	0.4 (2)
C4—C5—C6—C8	179.31 (17)	O4—C9—C14—C13	174.31 (13)
C5—C6—C7—C2	−0.3 (3)	C10—C9—C14—C15	−179.79 (16)
C8—C6—C7—C2	−179.53 (15)	O4—C9—C14—C15	−5.9 (2)
C3—C2—C7—C6	0.1 (2)	C12—C13—C14—C9	−0.7 (2)
C1—C2—C7—C6	179.60 (15)	C12—C13—C14—C15	179.48 (16)
C9—O4—C8—O3	−5.1 (3)	C9—C14—C15—O5	170.50 (17)
C9—O4—C8—C6	175.91 (14)	C13—C14—C15—O5	−9.7 (2)
C7—C6—C8—O3	−175.87 (18)	C9—C14—C15—C16	−9.6 (3)
C5—C6—C8—O3	4.9 (3)	C13—C14—C15—C16	170.19 (17)
C7—C6—C8—O4	3.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.84	2.6623 (18)	175
C4—H4···O5^{ii}	0.93	2.58	3.257 (3)	130

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