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Ethyl 3-benzoyl-2-hydroxyprop-2-enoate

Jian Wang,[a] Wen Zhou[b] and Wen-Gui Xu[a]*

[a]Key Laboratory of Tumor Prevention and Therapy, Tianjin Medical University Cancer Institute and Hospital PET–CT, Tianjin 300071, People's Republic of China, and [b]School of Pharmaceutical Sciences, Tianjin Medical University, Tianjin 300071, People's Republic of China
Correspondence e-mail: wwbzzl@yahoo.com.cn

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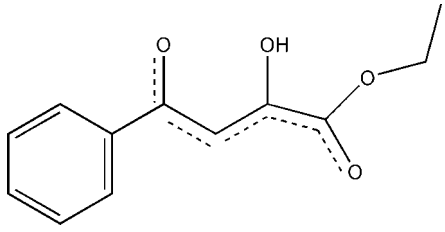
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.150; data-to-parameter ratio = 15.8.

In the title compound, $C_{12}H_{12}O_4$, the dihedral angle between the plane through the phenyl ring and the mean plane of the side chain is approximately 14°. The molecules, which contain an intramolecular O—H···O hydrogen bond, are linked end-to-end by weak C—H···O intermolecular hydrogen-bonding contacts, forming infinite one-dimensional chain systems in the crystal structure.

Related literature

For related literature, see: Davey & Ribbons (1975); Emerson *et al.* (1991); Aliev *et al.* (2000*a*,*b*); Bernstein *et al.* (1995); Desiraju & Steiner (2001).



Experimental

Crystal data

$C_{12}H_{12}O_4$
M_r = 220.22
Monoclinic, $P2_1/c$
a = 9.872 (4) Å
b = 13.498 (5) Å
c = 8.843 (3) Å
β = 105.464 (6)°
V = 1135.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 294 (2) K
0.26 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.981
6252 measured reflections
2316 independent reflections
1405 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.151
S = 1.02
2316 reflections
147 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O1	0.82	1.80	2.518 (2)	146
C5—H5···O3^{i}	0.93	2.67	3.405 (3)	137
C11—H11A···O1^{ii}	0.97	2.68	3.571 (4)	153

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2049).

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S1. Comment

1,3-Diketones are substrates for carbon-carbon bond hydrolysis by beta-ketolases. The 2,4-diketo acids examined for hydrolysis by acetopyruvate hydrolase from rat liver (EC 3.7.1.5) all have aliphatic side chains.(Davey & Ribbons, 1975) The acetopyruvate hydrolases cleave 2,4-diketopentanoic acid into pyruvate and acetate (Emerson *et al.*, 1991). Cleavage of analogues such as 2,4-diketo-4-phenylbutanoic acid was not reported. In order to discover the hydrolysis process of analogues, the title compound was synthesized.

In the title compound, the C—O and C—C bond lengths are in the normal range, and the dihedral angle between the plane of the phenyl ring and the mean plane of the side chain is approximately 14 °. The corresponding torsion angle C1—C6—C7—C8 is -14.0 (3) °. The molecule contains the typical O—H···O intra-molecular hydrogen bond graph set S(6) (Bernstein *et al.*, 1995). As shown in Figure 2, these monomers are associated end-to-end to form the $R^2_2(10)$ ring system, which is generated by different weak C—H···O hydrogen bonds (Table 1). These hydrogen bonds connect the molecules due to translational symmetry to assembly a chain system along the c direction. Similarly, the S(6) intra-molecular hydrogen bond type is also observed in 2-hydroxy-4-oxo-4-phenyl-3(*Z*)-butenic acid and 4-hydroxy-2-oxo-6-phenyl-3(*Z*),5(E)-hexadienic acid, but the monomer of the former is extended by the $R^2_2(8)$ (Bernstein *et al.* 1995) inter-molecular H-bonds, related by a centre of inversion to form a two-dimensional layer with head-to-tail packing architecture (Aliev *et al.*, 2000*a*,b). Head-to-tail packing is also observed in the structure of the title compound, and long H···O distances (Table 1) in weak intermolecular C—H···O contacts are extensively discussed in the literature (Desiraju & Steiner, 2001).

S2. Experimental

Sodium (2.3 g, 103 mmol) was added to absolute ethanol (133 ml). The mixture was cooled to -273 K and a mixture of diethyl oxalate (14.0 g, 96 mmol) and the ketone (96 mmol) was added slowly over a period of 20 min. A precipitate formed and stirring was continued for 4 h at room temperature. The precipitate was filtered, washed with absolute ethanol (20 ml) and dissolved in 2 N sulfuric acid (150 ml) and ether-extracted (3x150 ml), dried over Na_2SO_4 and ether removed. The residue was distilled under reduced pressure. The residue was recrystallized from ethanol and single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from acetone at room temperature. Elemental analysis (%) calcd for 1, $C_{18}H_{22}CuN_4O_8$: C 65.45, H 5.49; found: C 65.52, H 5.54.

S3. Refinement

H atoms were positioned geometrically and treated as riding with distances C–H = 0.93–0.97 Å, and O–H = 0.82 Å. The respective U_{iso}(H)=1.2Ueq(aromatic C and CH_2), U_{iso}(H)=1.5Ueq(CH_3), U_{iso}(*H*)=1.5(U_{eq})(hydroxyl O).

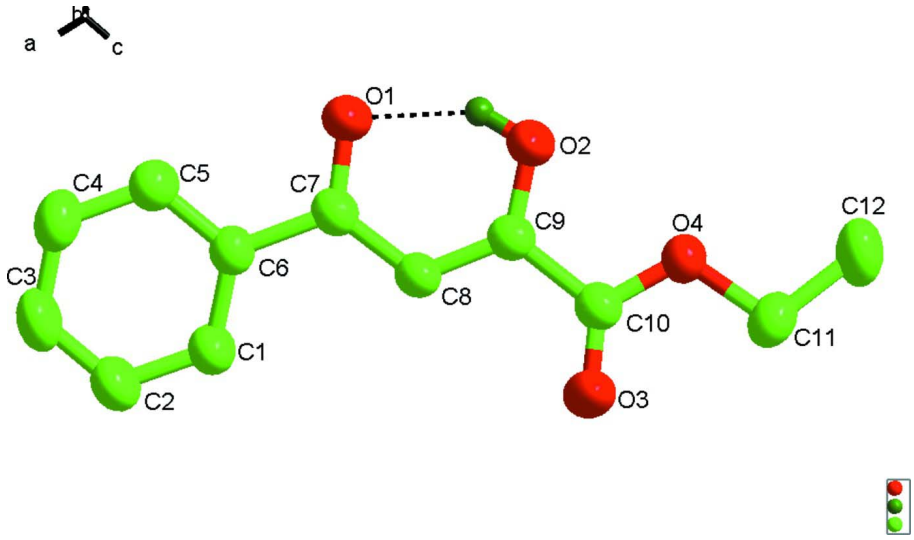

Figure 1

An *ORTEP* view of the labelled title molecule with 30% thermal ellipsoids. The intramolecular hydrogen bond is indicated by a dashed line.

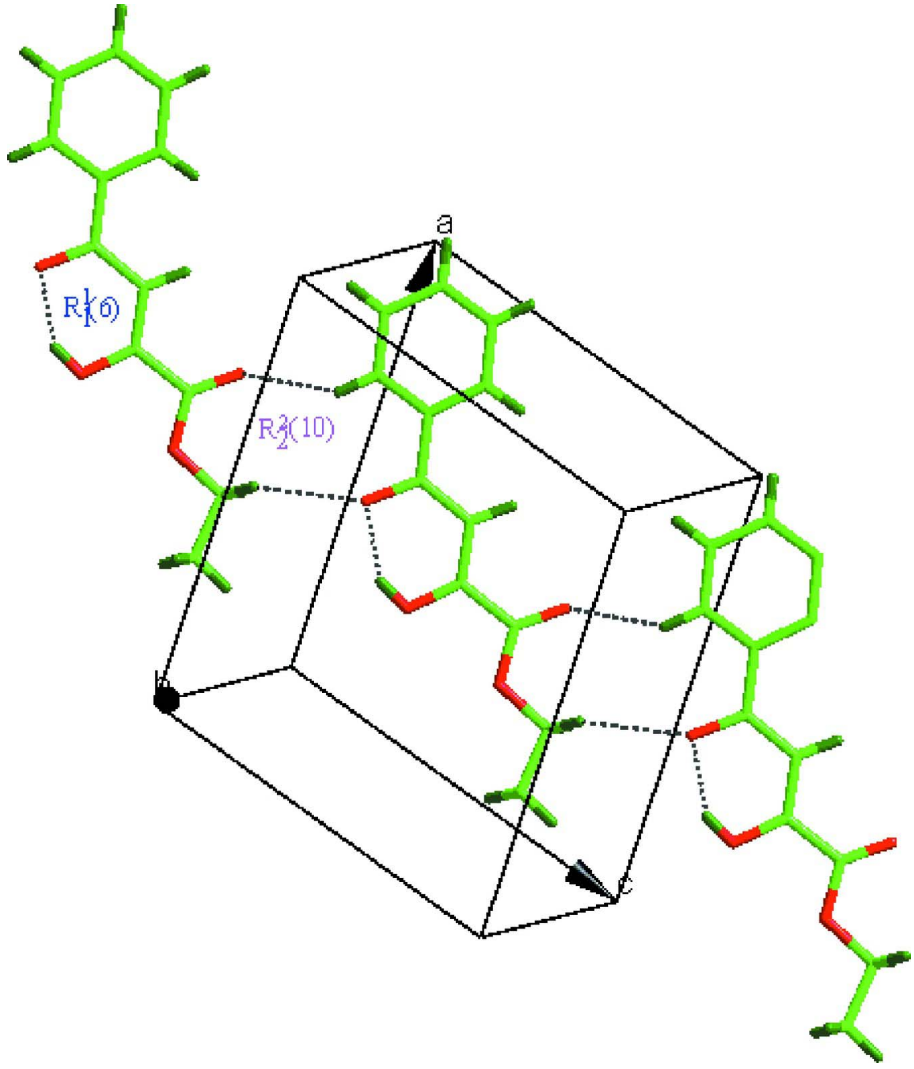

Figure 2

View of the one-dimensional weak hydrogen bonded chain structure.

Ethyl 3-benzoyl-2-hydroxyprop-2-enoate

Crystal data

$C_{12}H_{12}O_4$
$M_r = 220.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.872\ (4)$ Å
$b = 13.498\ (5)$ Å
$c = 8.843\ (3)$ Å
$\beta = 105.464\ (6)°$
$V = 1135.7\ (7)$ Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.288$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1800 reflections
$\theta = 2.6–26.2°$
$\mu = 0.10$ mm^{-1}
$T = 294$ K
Block, yellow
0.26 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.975, T_{max} = 0.981
6252 measured reflections
2316 independent reflections
1405 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 26.4°, θ_{min} = 2.1°
h = −12→11
k = −16→15
l = −9→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.151
S = 1.02
2316 reflections
147 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.0635P)^2 + 0.3304P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.58450 (15)	0.67082 (13)	0.25001 (17)	0.0695 (5)
O2	0.43270 (15)	0.65232 (14)	0.43639 (17)	0.0714 (5)
H2A	0.4506	0.6651	0.3531	0.107*
O3	0.62879 (18)	0.56869 (16)	0.80268 (18)	0.0871 (6)
O4	0.40548 (14)	0.61835 (11)	0.71546 (15)	0.0558 (4)
C1	0.9543 (2)	0.62744 (16)	0.4355 (3)	0.0589 (6)
H1	0.9547	0.6290	0.5407	0.071*
C2	1.0792 (2)	0.6156 (2)	0.3952 (3)	0.0708 (7)
H2	1.1633	0.6099	0.4732	0.085*
C3	1.0797 (3)	0.61223 (19)	0.2404 (3)	0.0726 (7)
H3	1.1636	0.6025	0.2136	0.087*
C4	0.9558 (3)	0.6232 (2)	0.1241 (3)	0.0721 (7)
H4	0.9566	0.6216	0.0192	0.087*
C5	0.8304 (2)	0.63658 (17)	0.1634 (3)	0.0603 (6)
H5	0.7473	0.6453	0.0849	0.072*
C6	0.8285 (2)	0.63697 (14)	0.3204 (2)	0.0479 (5)

C7	0.6913 (2)	0.64484 (15)	0.3590 (2)	0.0490 (5)
C8	0.6759 (2)	0.62196 (16)	0.5096 (2)	0.0516 (5)
H8A	0.7552	0.6022	0.5926	0.062*
C9	0.5473 (2)	0.62671 (15)	0.5400 (2)	0.0497 (5)
C10	0.5333 (2)	0.60126 (17)	0.7012 (2)	0.0536 (5)
C11	0.3828 (2)	0.5971 (2)	0.8685 (2)	0.0641 (6)
H11A	0.4489	0.6341	0.9496	0.077*
H11B	0.3962	0.5270	0.8920	0.077*
C12	0.2353 (3)	0.6269 (2)	0.8617 (3)	0.0833 (8)
H12A	0.2239	0.6966	0.8405	0.125*
H12B	0.2164	0.6126	0.9604	0.125*
H12C	0.1710	0.5906	0.7799	0.125*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0472 (9)	0.1101 (13)	0.0500 (9)	0.0117 (8)	0.0109 (7)	0.0164 (9)
O2	0.0429 (9)	0.1209 (15)	0.0501 (9)	0.0102 (8)	0.0119 (7)	0.0166 (9)
O3	0.0623 (11)	0.1483 (18)	0.0514 (10)	0.0333 (11)	0.0161 (8)	0.0217 (10)
O4	0.0470 (8)	0.0774 (10)	0.0453 (8)	0.0045 (7)	0.0163 (6)	0.0043 (7)
C1	0.0463 (12)	0.0808 (16)	0.0507 (12)	0.0012 (11)	0.0151 (10)	0.0017 (11)
C2	0.0464 (13)	0.1006 (19)	0.0671 (15)	0.0019 (12)	0.0183 (11)	0.0075 (13)
C3	0.0546 (15)	0.0928 (19)	0.0803 (17)	0.0009 (13)	0.0353 (13)	0.0072 (14)
C4	0.0713 (17)	0.0946 (19)	0.0593 (14)	−0.0037 (13)	0.0326 (13)	0.0025 (13)
C5	0.0531 (13)	0.0767 (16)	0.0518 (13)	0.0000 (11)	0.0150 (10)	0.0068 (11)
C6	0.0455 (12)	0.0522 (12)	0.0480 (11)	−0.0005 (9)	0.0159 (9)	0.0019 (9)
C7	0.0419 (11)	0.0555 (12)	0.0492 (12)	0.0020 (9)	0.0117 (9)	−0.0019 (9)
C8	0.0420 (12)	0.0697 (14)	0.0424 (11)	0.0090 (9)	0.0101 (9)	0.0013 (10)
C9	0.0448 (11)	0.0597 (13)	0.0430 (11)	0.0042 (9)	0.0093 (9)	−0.0018 (9)
C10	0.0456 (12)	0.0693 (14)	0.0455 (12)	0.0050 (10)	0.0115 (10)	−0.0013 (10)
C11	0.0679 (15)	0.0823 (16)	0.0462 (12)	0.0021 (12)	0.0220 (11)	0.0012 (11)
C12	0.0776 (18)	0.104 (2)	0.0825 (18)	0.0151 (15)	0.0469 (15)	0.0085 (15)

Geometric parameters (Å, º)

O1—C7	1.274 (2)	C4—H4	0.9300
O2—C9	1.299 (2)	C5—C6	1.394 (3)
O2—H2A	0.8200	C5—H5	0.9300
O3—C10	1.199 (2)	C6—C7	1.486 (3)
O4—C10	1.321 (2)	C7—C8	1.415 (3)
O4—C11	1.458 (2)	C8—C9	1.367 (3)
C1—C2	1.381 (3)	C8—H8A	0.9572
C1—C6	1.386 (3)	C9—C10	1.509 (3)
C1—H1	0.9300	C11—C12	1.496 (3)
C2—C3	1.371 (3)	C11—H11A	0.9700
C2—H2	0.9300	C11—H11B	0.9700
C3—C4	1.380 (3)	C12—H12A	0.9600
C3—H3	0.9300	C12—H12B	0.9600

C4—C5	1.383 (3)	C12—H12C	0.9600
C9—O2—H2A	109.5	C8—C7—C6	122.28 (18)
C10—O4—C11	116.17 (16)	C9—C8—C7	120.88 (19)
C2—C1—C6	120.5 (2)	C9—C8—H8A	118.4
C2—C1—H1	119.7	C7—C8—H8A	120.8
C6—C1—H1	119.7	O2—C9—C8	123.66 (19)
C3—C2—C1	120.2 (2)	O2—C9—C10	116.41 (18)
C3—C2—H2	119.9	C8—C9—C10	119.93 (18)
C1—C2—H2	119.9	O3—C10—O4	125.0 (2)
C2—C3—C4	120.2 (2)	O3—C10—C9	122.62 (19)
C2—C3—H3	119.9	O4—C10—C9	112.41 (17)
C4—C3—H3	119.9	O4—C11—C12	107.37 (18)
C3—C4—C5	120.1 (2)	O4—C11—H11A	110.2
C3—C4—H4	119.9	C12—C11—H11A	110.2
C5—C4—H4	119.9	O4—C11—H11B	110.2
C4—C5—C6	120.1 (2)	C12—C11—H11B	110.2
C4—C5—H5	120.0	H11A—C11—H11B	108.5
C6—C5—H5	120.0	C11—C12—H12A	109.5
C1—C6—C5	118.92 (19)	C11—C12—H12B	109.5
C1—C6—C7	122.10 (19)	H12A—C12—H12B	109.5
C5—C6—C7	118.96 (19)	C11—C12—H12C	109.5
O1—C7—C8	119.81 (18)	H12A—C12—H12C	109.5
O1—C7—C6	117.88 (18)	H12B—C12—H12C	109.5
C6—C1—C2—C3	−0.6 (4)	O1—C7—C8—C9	0.4 (3)
C1—C2—C3—C4	1.7 (4)	C6—C7—C8—C9	−177.77 (19)
C2—C3—C4—C5	−0.7 (4)	C7—C8—C9—O2	−0.4 (3)
C3—C4—C5—C6	−1.3 (4)	C7—C8—C9—C10	179.55 (19)
C2—C1—C6—C5	−1.4 (3)	C11—O4—C10—O3	1.2 (3)
C2—C1—C6—C7	176.9 (2)	C11—O4—C10—C9	−179.21 (18)
C4—C5—C6—C1	2.3 (3)	O2—C9—C10—O3	173.6 (2)
C4—C5—C6—C7	−176.0 (2)	C8—C9—C10—O3	−6.3 (3)
C1—C6—C7—O1	167.8 (2)	O2—C9—C10—O4	−6.0 (3)
C5—C6—C7—O1	−13.9 (3)	C8—C9—C10—O4	174.08 (19)
C1—C6—C7—C8	−14.0 (3)	C10—O4—C11—C12	176.8 (2)
C5—C6—C7—C8	164.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1	0.82	1.80	2.518 (2)	146
C5—H5···O3^{i}	0.93	2.67	3.405 (3)	137
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Symmetry codes: (i) *x*, *y*, *z*−1; (ii) *x*, *y*, *z*+1.