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2-Hydroxy-4-(methacryloyloxy)-acetophenone

G. Chakkaravarthi,[a] A. Anthonysamy,[b] S. Balasubramanian[b] and V. Manivannan[c]*

[a]Department of Physics, CPCL Polytechnic College, Chennai 600 068, India, [b]Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, and [c]Department of Physics, Presidency College, Chennai 600 005, India
Correspondence e-mail: manivan_1999@yahoo.com

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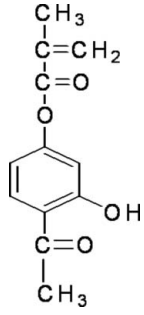
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.001 Å; disorder in main residue; R factor = 0.047; wR factor = 0.166; data-to-parameter ratio = 26.2.

In the title compound, $C_{12}H_{12}O_4$, the acetyl group is coplanar with the benzene ring, the dihedral angle being 1.00 (7)°; the methacryloyloxy group makes a dihedral angle of 34.67 (4)° with the benzene ring. The methyl and methylene groups in the terminal site are disordered equally over two positions. The molecular structure is stabilized by intramolecular O—H···O and C—H···O hydrogen bonds and the crystal packing is stabilized by intermolecular C—H···O and C—H···π interactions.

Related literature

For related literature, see: Gibson *et al.* (2006); Naka & Kubo (1999); Nicolaides *et al.* (1998); Parker & Braden (1989); Ren *et al.* (2006); Romero (2001). A similar acetophenone compound with a methyl group has been reported by Chakkaravarthi *et al.* (2007).



Experimental

Crystal data

$C_{12}H_{12}O_4$
M_r = 220.22
Triclinic, $P\overline{1}$
a = 6.6413 (2) Å
b = 7.2833 (3) Å
c = 12.4387 (4) Å
α = 84.020 (1)°
β = 87.447 (2)°
γ = 65.866 (1)°
V = 546.09 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
0.25 × 0.16 × 0.15 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.985
16320 measured reflections
4847 independent reflections
3225 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.166
S = 1.04
4847 reflections
185 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

Cg is the centroid of the C5–C10 ring.

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4	0.82	1.81	2.5334 (10)	147
C10—H10···O1	0.93	2.40	2.8163 (12)	107
C7—H7···O3^{i}	0.93	2.43	3.3372 (11)	164
C12—H12*C*···*Cg*^{ii}	0.96	2.81	3.6718 (12)	149

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2203).

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supplementary materials

Acta Cryst. (2007). E**63**, o4163 [**doi:10.1107/S1600536807046478**]

2-Hydroxy-4-(methacryloyloxy)acetophenone

G. Chakkaravarthi, A. Anthonysamy, S. Balasubramanian and V. Manivannan

Comment

Methacrylate derivatives have anti-inflammatory (Nicolaides *et al.*, 1998) and antipicornaviral (Romero, 2001) properties and are efficient as agonists for different receptors (Naka & Kubo, 1999). Methacrylate activated vinyl esters are readily polymerized by free-radical polymerization to form linear, branched or network polymers (Parker & Braden, 1989).

The geometric parameters in (I) (Fig. 1) are comparable with the reported values of similar compounds (Gibson *et al.*, 2006; Ren *et al.*, 2006). A similar acetophenone compound with methyl group has been reported (Chakkaravarthi *et al.*, 2007). The acetyl group is planar with the benzene ring [dihedral angle of 179.00 (7)°] and the methacryloyloxy group makes the dihedral angle of 34.67 (4)° with the benzene ring. The torsion angles O2—C4—C2—C3 and O2—C4—C2—C1 [−0.2 (6)° and 178.1 (8)°, respectively] indicate periplanar conformation of the respective moieties. The methyl and methylene groups in the terminal site are disordered over two positions with site occupancy factors of 0.50 (2). The molecular structure is stabilized by intramolecular O—H···O and C—H···O interactions and the crystal packing of (I) (Fig. 2) is stabilized by an intermolecular C—H···O hydrogen bond and a C—H···π interaction, involving the benzene C5—C10 ring (Table 1).

Experimental

2,4-Dihydroxyacetophenone (4.2 g, 27.60 mmol), triethylamine (3.85 ml, 27.67 mmol) and 150 ml of dry 2-butanone were taken in a 250 ml round bottom flask and the temperature was maintained at 273 K. Then the solution of methacryloylchloride (2.7 ml, 27.74 mmol) in 20 ml of 2-butanone was added dropwise to the mixture with constant stirring for 30 min. After the addition was over, the reaction mixture was stirred for another 6 h. The salt formed during the reaction was filtered and the filtrate was washed with water and dried over anhydrous $MgSO_4$. The filtrate was concentrated under reduced pressure and the crude product was purified by column chromatography (silica) using hexane and ethyl acetate mixture (9:1). Crystals suitable for X-ray analysis were grown by slow evaporation of an ethyl acetate solution.

Refinement

The site occupancy factors of the disordered methyl and methylene groups refined to 0.50 (2). H atoms for methylene C atoms were located in a difference map and refined isotropically. The remaining H atoms were positioned geometrically and refined as riding, with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl C, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic C—H, with O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(O) for OH. The distance restraints were applied to the disordered methylene C atoms.

Figures

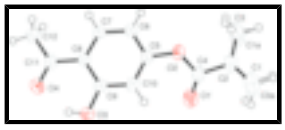

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

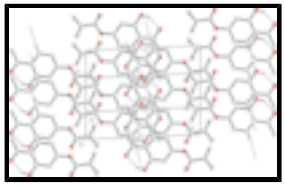

Fig. 2. The packing diagram of (I), viewed approximately down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

2-Hydroxy-4-(methacryloyloxy)acetophenone

Crystal data

$C_{12}H_{12}O_4$	$Z = 2$
$M_r = 220.22$	$F_{000} = 232$
Triclinic, $P\overline{1}$	$D_x = 1.339$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.6413$ (2) Å	Cell parameters from 6586 reflections
$b = 7.2833$ (3) Å	$\theta = 3.1$–35.1°
$c = 12.4387$ (4) Å	$\mu = 0.10$ mm^{-1}
$\alpha = 84.020$ (1)°	$T = 295$ (2) K
$\beta = 87.447$ (2)°	Needle, colourless
$\gamma = 65.866$ (1)°	0.25 × 0.16 × 0.15 mm
$V = 546.09$ (3) Å^3	

Data collection

Bruker Kappa-APEXII diffractometer	4847 independent reflections
Radiation source: fine-focus sealed tube	3225 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 295$(2) K	$\theta_{max} = 35.9^\circ$
ω and φ scans	$\theta_{min} = 1.7^\circ$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.966$, $T_{max} = 0.985$	$k = -11 \rightarrow 11$
16320 measured reflections	$l = -19 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.047$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.166$	$w = 1/[\sigma^2(F_o^2) + (0.0909P)^2 + 0.037P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
4847 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
185 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C4	0.54938 (18)	0.71905 (16)	0.08087 (8)	0.0457 (2)	
C2	0.5104 (2)	0.75165 (17)	−0.03777 (8)	0.0480 (2)	
C1	0.688 (2)	0.690 (2)	−0.1025 (8)	0.086 (3)	0.50 (2)
H1A	0.681 (9)	0.652 (8)	−0.180 (5)	0.13 (2)*	0.50 (2)
H1B	0.835 (7)	0.591 (6)	−0.069 (3)	0.073 (12)*	0.50 (2)
C3	0.282 (2)	0.854 (2)	−0.0782 (10)	0.0607 (17)	0.50 (2)
H3A	0.2822	0.8570	−0.1556	0.091*	0.50 (2)
H3B	0.2173	0.9894	−0.0576	0.091*	0.50 (2)
H3C	0.1970	0.7813	−0.0477	0.091*	0.50 (2)
C1A	0.316 (3)	0.828 (3)	−0.0779 (13)	0.081 (4)	0.50 (2)
H1A1	0.279 (7)	0.864 (7)	−0.154 (4)	0.095 (12)*	0.50 (2)
H1A2	0.199 (5)	0.882 (6)	−0.042 (3)	0.052 (11)*	0.50 (2)
C3A	0.7115 (19)	0.6816 (18)	−0.1034 (8)	0.075 (3)	0.50 (2)
H3A1	0.6735	0.7183	−0.1785	0.112*	0.50 (2)
H3A2	0.7867	0.5373	−0.0907	0.112*	0.50 (2)
H3A3	0.8061	0.7432	−0.0840	0.112*	0.50 (2)
C5	0.34851 (15)	0.77482 (14)	0.24864 (7)	0.03768 (18)	
C6	0.16421 (15)	0.74879 (16)	0.29206 (7)	0.0410 (2)	
H6	0.0614	0.7390	0.2472	0.049*	
C7	0.13639 (14)	0.73772 (14)	0.40255 (7)	0.03719 (18)	
H7	0.0123	0.7224	0.4319	0.045*	
C8	0.29124 (13)	0.74898 (12)	0.47183 (6)	0.03183 (16)	
C9	0.47621 (13)	0.77413 (13)	0.42489 (7)	0.03407 (17)	
C10	0.50213 (14)	0.79139 (15)	0.31304 (7)	0.03920 (19)	
H10	0.6214	0.8138	0.2824	0.047*	
C11	0.26509 (14)	0.73650 (14)	0.58983 (7)	0.03631 (18)	
C12	0.07174 (17)	0.70661 (17)	0.64152 (7)	0.0448 (2)	
H12A	0.0908	0.6845	0.7185	0.067*	
H12B	0.0611	0.5915	0.6154	0.067*	
H12C	−0.0608	0.8247	0.6239	0.067*	
O1	0.72558 (15)	0.63635 (18)	0.12312 (7)	0.0732 (3)	
O2	0.35578 (12)	0.79405 (13)	0.13645 (5)	0.04900 (19)	
O3	0.63603 (12)	0.77904 (13)	0.48540 (6)	0.04826 (19)	
H3	0.6021	0.7732	0.5495	0.072*	

O4	0.40266 (14)	0.75142 (14)	0.64788 (6)	0.0531 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C4	0.0455 (5)	0.0536 (5)	0.0339 (4)	−0.0166 (4)	0.0071 (3)	−0.0048 (3)
C2	0.0570 (6)	0.0562 (6)	0.0314 (4)	−0.0238 (5)	0.0075 (4)	−0.0064 (4)
C1	0.099 (5)	0.120 (8)	0.040 (4)	−0.046 (5)	0.000 (3)	−0.008 (3)
C3	0.067 (4)	0.076 (3)	0.034 (3)	−0.024 (3)	−0.010 (3)	−0.0037 (18)
C1A	0.080 (6)	0.118 (8)	0.039 (4)	−0.033 (5)	0.007 (3)	−0.011 (4)
C3A	0.087 (4)	0.081 (4)	0.043 (4)	−0.021 (3)	0.035 (3)	−0.018 (3)
C5	0.0352 (4)	0.0469 (5)	0.0287 (3)	−0.0147 (3)	0.0028 (3)	−0.0036 (3)
C6	0.0343 (4)	0.0595 (5)	0.0323 (4)	−0.0215 (4)	−0.0005 (3)	−0.0067 (3)
C7	0.0298 (3)	0.0523 (5)	0.0337 (4)	−0.0209 (3)	0.0026 (3)	−0.0052 (3)
C8	0.0283 (3)	0.0383 (4)	0.0293 (3)	−0.0139 (3)	0.0014 (2)	−0.0044 (3)
C9	0.0279 (3)	0.0418 (4)	0.0342 (4)	−0.0154 (3)	0.0010 (3)	−0.0059 (3)
C10	0.0337 (4)	0.0527 (5)	0.0347 (4)	−0.0215 (4)	0.0057 (3)	−0.0051 (3)
C11	0.0348 (4)	0.0427 (4)	0.0305 (3)	−0.0148 (3)	0.0011 (3)	−0.0037 (3)
C12	0.0419 (5)	0.0586 (6)	0.0341 (4)	−0.0221 (4)	0.0059 (3)	−0.0015 (4)
O1	0.0458 (4)	0.1051 (8)	0.0423 (4)	−0.0040 (5)	0.0040 (3)	−0.0084 (4)
O2	0.0422 (4)	0.0737 (5)	0.0279 (3)	−0.0209 (3)	0.0037 (2)	−0.0038 (3)
O3	0.0353 (3)	0.0785 (5)	0.0406 (3)	−0.0318 (3)	0.0002 (3)	−0.0103 (3)
O4	0.0502 (4)	0.0843 (6)	0.0335 (3)	−0.0355 (4)	−0.0030 (3)	−0.0075 (3)

Geometric parameters (Å, °)

C4—O1	1.1920 (14)	C5—C10	1.3751 (12)
C4—O2	1.3618 (12)	C5—O2	1.3886 (10)
C4—C2	1.4872 (14)	C5—C6	1.3912 (12)
C2—C1A	1.281 (14)	C6—C7	1.3770 (12)
C2—C1	1.339 (10)	C6—H6	0.9300
C2—C3A	1.464 (9)	C7—C8	1.4034 (11)
C2—C3	1.476 (14)	C7—H7	0.9300
C1—H1A	1.04 (6)	C8—C9	1.4076 (11)
C1—H1B	1.03 (4)	C8—C11	1.4680 (11)
C3—H3A	0.9600	C9—O3	1.3425 (10)
C3—H3B	0.9600	C9—C10	1.3932 (12)
C3—H3C	0.9600	C10—H10	0.9300
C1A—H1A1	0.97 (5)	C11—O4	1.2346 (11)
C1A—H1A2	0.85 (4)	C11—C12	1.4952 (13)
C3A—H1A	1.04 (6)	C12—H12A	0.9600
C3A—H1B	0.91 (4)	C12—H12B	0.9600
C3A—H3A1	0.9600	C12—H12C	0.9600
C3A—H3A2	0.9600	O3—H3	0.8200
C3A—H3A3	0.9600		
O1—C4—O2	123.66 (9)	C2—C3A—H3A3	109.5
O1—C4—C2	125.19 (10)	H3A1—C3A—H3A3	109.5
O2—C4—C2	111.14 (9)	H3A2—C3A—H3A3	109.5

C1A—C2—C1	120.4 (9)	C10—C5—O2	123.66 (8)
C1A—C2—C3A	123.4 (9)	C10—C5—C6	121.75 (8)
C1—C2—C3	123.5 (8)	O2—C5—C6	114.48 (8)
C3A—C2—C3	126.5 (7)	C7—C6—C5	118.95 (8)
C1A—C2—C4	122.1 (8)	C7—C6—H6	120.5
C1—C2—C4	117.5 (5)	C5—C6—H6	120.5
C3A—C2—C4	114.4 (5)	C6—C7—C8	121.44 (7)
C3—C2—C4	119.0 (6)	C6—C7—H7	119.3
C2—C1—H1A	122 (3)	C8—C7—H7	119.3
C2—C1—H1B	118 (2)	C7—C8—C9	117.93 (7)
H1A—C1—H1B	108 (4)	C7—C8—C11	122.26 (7)
C2—C3—H3A	109.5	C9—C8—C11	119.81 (7)
C2—C3—H3B	109.5	O3—C9—C10	117.31 (7)
H3A—C3—H3B	109.5	O3—C9—C8	121.74 (7)
C2—C3—H3C	109.5	C10—C9—C8	120.93 (7)
H3A—C3—H3C	109.5	C5—C10—C9	118.93 (7)
H3B—C3—H3C	109.5	C5—C10—H10	120.5
C2—C3—H1A2	118 (3)	C9—C10—H10	120.5
H3A—C3—H1A2	132.3	O4—C11—C8	120.27 (8)
H1A1—C3—H1A2	131 (5)	O4—C11—C12	119.01 (8)
C2—C1A—H1A1	126 (3)	C8—C11—C12	120.72 (7)
C2—C1A—H1A2	124 (3)	C11—C12—H12A	109.5
H1A1—C1A—H1A2	108 (4)	C11—C12—H12B	109.5
C2—C3A—H1A	111 (4)	H12A—C12—H12B	109.5
C2—C3A—H1B	116 (3)	C11—C12—H12C	109.5
H1A—C3A—H1B	117 (4)	H12A—C12—H12C	109.5
C2—C3A—H3A1	109.5	H12B—C12—H12C	109.5
C2—C3A—H3A2	109.5	C4—O2—C5	121.97 (8)
H3A1—C3A—H3A2	109.5	C9—O3—H3	109.5
O1—C4—C2—C1A	174.4 (10)	C7—C8—C9—C10	1.28 (13)
O2—C4—C2—C1A	−4.8 (10)	C11—C8—C9—C10	−178.40 (8)
O1—C4—C2—C1	−2.7 (8)	O2—C5—C10—C9	178.52 (9)
O2—C4—C2—C1	178.1 (8)	C6—C5—C10—C9	2.40 (15)
O1—C4—C2—C3A	−2.9 (6)	O3—C9—C10—C5	176.14 (8)
O2—C4—C2—C3A	177.9 (6)	C8—C9—C10—C5	−2.76 (14)
O1—C4—C2—C3	178.9 (6)	C7—C8—C11—O4	−178.43 (9)
O2—C4—C2—C3	−0.2 (6)	C9—C8—C11—O4	1.23 (14)
C10—C5—C6—C7	−0.55 (15)	C7—C8—C11—C12	1.28 (14)
O2—C5—C6—C7	−177.00 (8)	C9—C8—C11—C12	−179.05 (8)
C5—C6—C7—C8	−1.00 (15)	O1—C4—O2—C5	−1.74 (18)
C6—C7—C8—C9	0.62 (14)	C2—C4—O2—C5	177.45 (9)
C6—C7—C8—C11	−179.70 (8)	C10—C5—O2—C4	38.32 (15)
C7—C8—C9—O3	−177.57 (8)	C6—C5—O2—C4	−145.30 (10)
C11—C8—C9—O3	2.75 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O4	0.82	1.81	2.5334 (10)	147

C10—H10···O1	0.93	2.40	2.8163 (12)	107
C7—H7···O3^{i}	0.93	2.43	3.3372 (11)	164
C12—H12C···Cg^{ii}	0.96	2.81	3.6718 (12)	149

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

Fig. 1

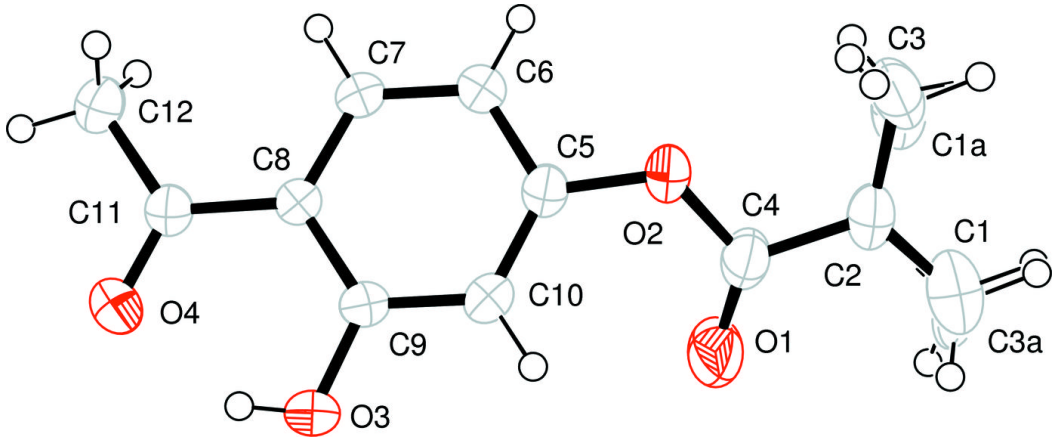

Fig. 2