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Methyl (*E*)-3-(2-formylphenoxy)acrylateS. Karthikeyan,^a K. Sethusankar,^{a*} R. Selvakumar^b and M. Bakthadoss^b^aDepartment of Physics, RKM Vivekananda College (Autonomous), Chennai 600 004, India, and ^bDepartment of Organic Chemistry, University of Madras, Maraimalai Campus, Chennai 600 025, India

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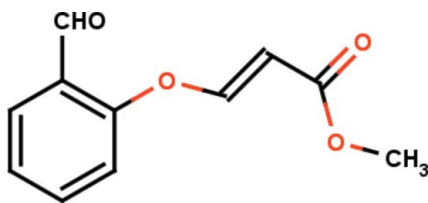
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.045; wR factor = 0.155; data-to-parameter ratio = 14.7.

In the title compound, $\text{C}_{11}\text{H}_{10}\text{O}_4$, the methyl acrylate substituent adopts an extended *E* conformation with all torsion angles close to 180° . The conformation of the keto group with respect to the olefinic double bond is typically *S-trans*. In the crystal, molecules are linked *via* pairs of $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming inversion dimers with an $R_2^2(8)$ graph-set motif. The dimers are further linked *via* $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming chains along $[001]$, which enclose $R_3^2(16)$ graph-set ring motifs. The keto group O atom accepts two $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For applications of acrylate derivatives, see: Xiao *et al.* (2008); De *et al.* (2011); Sharma (2011). For related crystal structures, see: Karthikeyan *et al.* (2012). For *E*-conformation aspects, see: Dunitz & Schweizer (1982). For resonance effects of acrylate, see: Merlino (1971); Varghese *et al.* (1986). For graph-set motif notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{10}\text{O}_4$
 $M_r = 206.19$
 Monoclinic, $P2_1/c$
 $a = 17.7458$ (8) Å

$b = 4.0629$ (2) Å
 $c = 14.5745$ (7) Å
 $\beta = 107.868$ (3)°
 $V = 1000.13$ (8) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹

$T = 293$ K
 $0.20 \times 0.15 \times 0.10$ mm

Data collection

Bruker SMART APEXII CCD
 diffractometer
 13052 measured reflections

2015 independent reflections
 1523 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.155$
 $S = 1.06$
 2015 reflections

137 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C9}-\text{H9}\cdots\text{O2}^{\text{i}}$	0.93	2.54	3.440 (2)	164
$\text{C8}-\text{H8}\cdots\text{O4}^{\text{ii}}$	0.93	2.61	3.529 (2)	171
$\text{C11}-\text{H11C}\cdots\text{O2}^{\text{iii}}$	0.96	2.63	3.578 (2)	168

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE*; 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 *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

SK and KS thank Dr D. Velmurugan, CAS in Crystallography and Biophysics, University of Madras, Maraimalai Campus, Chennai, India, for the X-ray intensity data collection.

Supporting information for this paper is available from the IUCr electronic archives (Reference: SU2732).

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supporting information

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Methyl (*E*)-3-(2-formylphenoxy)acrylate

S. Karthikeyan, K. Sethusankar, R. Selvakumar and M. Bakthadoss

S1. Comment

Cinnamic acid derivatives have received attention in medicinal research as traditional as well as recent synthetic antitumor agents (De *et al.*, 2011). They also possess significant antibacterial activities against *staphylococcus aureus* (Xiao *et al.*, 2008). Different substitutions on the basic moiety lead to various pharmacological activities, such as antioxidant, hepatoprotective, anxiolytic, insect repellent, antidiabetic, and anticholesterolemic (Sharma, 2011).

In the title molecule, Fig. 1, the methyl acrylate group is essentially planar, with a maximum deviation of 0.0264 (19) Å for atom C9. Its mean plane forms a dihedral angle of 31.74 (6)° with the benzene ring (C2—C7). The molecular dimensions are in excellent agreement with those reported for a closely related compound (Karthikeyan *et al.*, 2012).

The configuration of the keto group with respect to the olefinic double bond is typically *S-trans*, with the O2=C10—C9=C8 torsion angle = 178.78 (19)°. The methyl acrylate group adopts an extended *E* conformation with torsion angles C8=C9—C10=O2 = 178.78 (19)°, C8=C9—C10—O1 = -1.2 (3)°, C9—C10—O1—C11 = -178.82 (16)° and O2=C10—O1—C11 = 1.2 (3)°. The extended conformation is supported by the fact that the bond angles involving carbonyl O atoms are invariably enlarged (Dunitz & Schweizer, 1982).

The significant difference in the bond lengths C10—O1 = 1.342 (2) Å and C11—O1 = 1.438 (2) Å is attributed to a partial contribution from the O=C=O⁺—C resonance structure of the O2=C10—O1—C11 group (Merlino, 1971). This feature, commonly observed for the carboxylic ester group of substituents in various compounds gives average values of 1.340 Å and 1.447 Å, respectively (Varghese *et al.*, 1986).

The crystal packing (Fig. 2 and Table 1) is stabilized by C—H···O intermolecular interactions. The molecules are linked into inversion dimers *via* C9—H9···O2 interactions resulting in an $R^2_2(8)$ graph-set motif (Bernstein *et al.*, 1995). The dimers are further consolidated by $R^2_3(16)$ graph-set ring motifs *via* C8—H8···O4 and C11—H11C···O2 interactions resulting in chains of molecules running parallel to the *c* axis; the keto group O atom (O2) is involved in bifurcated hydrogen bonding.

S2. Experimental

Salicylaldehyde (1 mmol) was dissolved in an aqueous solution of K₂CO₃ (1 mmol) and methyl propiolate (1 mmol) was added. The reaction mixture was stirred vigorously at room temperature. A turbid solution was formed by consumption of salicylaldehyde (monitored by TLC) in 5 min, the reaction mixture then became clear. The title compound was precipitated as a solid in water. The product was isolated by filtration without further purification [Yield 75%]. Block-like colourless crystals were obtained by slow evaporation of a solution in ethylacetate.

S3. Refinement

The H atoms could all be located in difference electron-density maps. In the final cycles of refinement they were treated as riding atoms: C—H = 0.93 and 0.96 Å for CH and CH₃ H atoms, respectively, with $U_{iso}(H) = 1.5 U_{eq}(C - \text{methyl})$ and =

1.2 $U_{eq}(C)$ for other H atoms.

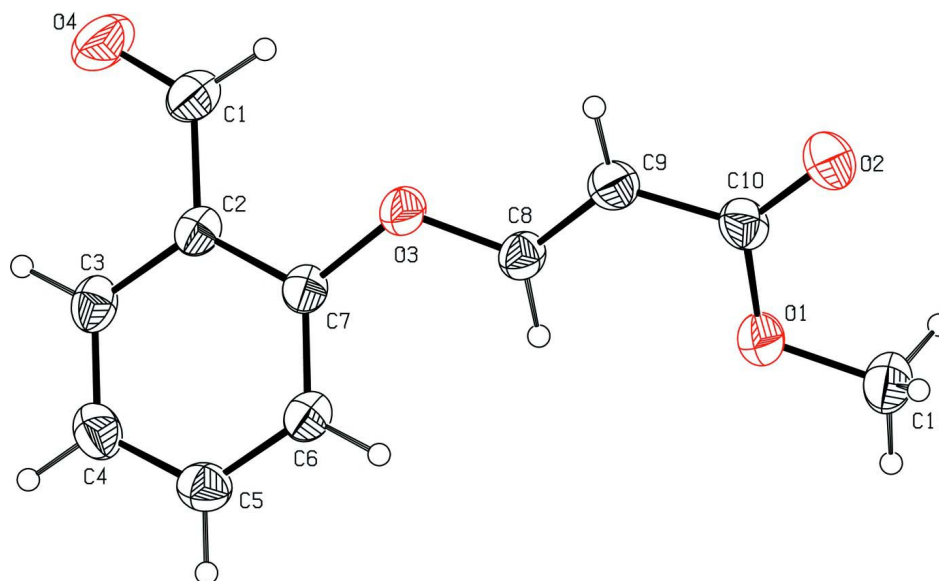


Figure 1

The molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

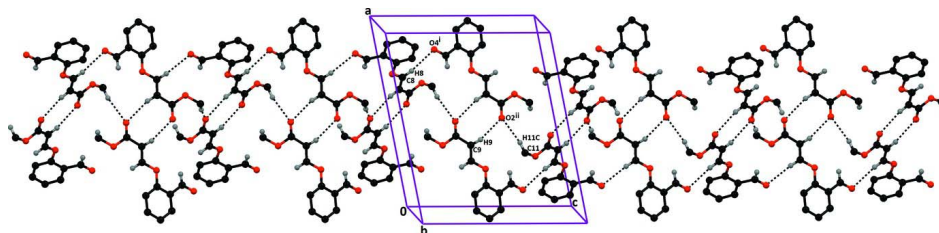


Figure 2

The crystal packing of the title compound viewed along the b axis, showing the formation of the $R^2_2(8)$ graph-set motif. The dimers are further consolidated by $R^2_3(16)$ graph-set ring motifs. Hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in these interactions have been omitted for clarity.)

Methyl (*E*)-3-(2-formylphenoxy)acrylate

Crystal data

$C_{11}H_{10}O_4$

$M_r = 206.19$

Monoclinic, $P2_1/c$

Hall symbol: $-p\ 2ybc$

$a = 17.7458(8)\ \text{\AA}$

$b = 4.0629(2)\ \text{\AA}$

$c = 14.5745(7)\ \text{\AA}$

$\beta = 107.868(3)^\circ$

$V = 1000.13(8)\ \text{\AA}^3$

$Z = 4$

$F(000) = 432$

$D_x = 1.369\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2015 reflections

$\theta = 1.2\text{--}26.3^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, colorless

$0.20 \times 0.15 \times 0.10\ \text{mm}$

Data collection

Bruker SMART APEXII CCD diffractometer	1523 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.027$
Graphite monochromator	$\theta_{\text{max}} = 26.3^\circ$, $\theta_{\text{min}} = 1.2^\circ$
ω scans	$h = -22 \rightarrow 21$
13052 measured reflections	$k = -5 \rightarrow 5$
2015 independent reflections	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.155$	$w = 1/[\sigma^2(F_o^2) + (0.1001P)^2 + 0.0957P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2015 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
137 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.19462 (11)	0.6483 (5)	1.09554 (11)	0.0570 (5)
H1	0.2426	0.7588	1.1079	0.068*
C2	0.16126 (8)	0.4928 (4)	1.00055 (10)	0.0437 (4)
C3	0.08802 (9)	0.3378 (4)	0.97865 (12)	0.0518 (5)
H3	0.0621	0.3250	1.0252	0.062*
C4	0.05341 (10)	0.2034 (5)	0.88901 (13)	0.0574 (5)
H4	0.0040	0.1033	0.8746	0.069*
C5	0.09250 (10)	0.2182 (5)	0.82056 (12)	0.0545 (5)
H5	0.0692	0.1265	0.7600	0.065*
C6	0.16554 (9)	0.3671 (4)	0.84083 (11)	0.0484 (4)
H6	0.1918	0.3734	0.7946	0.058*
C7	0.19943 (8)	0.5068 (4)	0.93033 (10)	0.0425 (4)
C8	0.30241 (9)	0.7868 (4)	0.88985 (11)	0.0463 (4)
H8	0.2706	0.8014	0.8260	0.056*
C9	0.37579 (9)	0.8937 (5)	0.91462 (12)	0.0542 (5)
H9	0.4075	0.8649	0.9780	0.065*
C10	0.40974 (9)	1.0549 (5)	0.84737 (12)	0.0516 (4)

C11	0.39059 (12)	1.2299 (6)	0.68764 (14)	0.0655 (5)
H11A	0.4103	1.4452	0.7097	0.098*
H11B	0.3494	1.2484	0.6272	0.098*
H11C	0.4329	1.0976	0.6794	0.098*
O1	0.35946 (7)	1.0781 (3)	0.75754 (8)	0.0598 (4)
O2	0.47632 (7)	1.1596 (4)	0.86851 (10)	0.0723 (5)
O3	0.27287 (6)	0.6544 (3)	0.95771 (7)	0.0541 (4)
O4	0.16367 (9)	0.6413 (5)	1.15808 (9)	0.0821 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0536 (10)	0.0738 (13)	0.0448 (8)	0.0075 (8)	0.0171 (7)	0.0039 (8)
C2	0.0403 (8)	0.0498 (10)	0.0419 (8)	0.0095 (6)	0.0142 (6)	0.0084 (7)
C3	0.0432 (9)	0.0604 (11)	0.0562 (9)	0.0071 (7)	0.0220 (7)	0.0119 (8)
C4	0.0442 (9)	0.0590 (11)	0.0665 (11)	-0.0046 (8)	0.0134 (8)	0.0077 (8)
C5	0.0531 (10)	0.0542 (10)	0.0504 (9)	0.0009 (8)	0.0074 (7)	0.0007 (8)
C6	0.0485 (9)	0.0547 (10)	0.0443 (8)	0.0048 (7)	0.0175 (7)	0.0049 (7)
C7	0.0357 (7)	0.0485 (10)	0.0429 (7)	0.0054 (6)	0.0115 (6)	0.0082 (6)
C8	0.0411 (8)	0.0565 (10)	0.0435 (8)	0.0025 (7)	0.0162 (6)	0.0013 (7)
C9	0.0425 (8)	0.0710 (12)	0.0490 (9)	-0.0019 (8)	0.0137 (7)	-0.0027 (8)
C10	0.0383 (8)	0.0609 (11)	0.0580 (9)	-0.0018 (7)	0.0184 (7)	-0.0063 (8)
C11	0.0639 (11)	0.0721 (13)	0.0669 (11)	-0.0039 (10)	0.0297 (9)	0.0095 (10)
O1	0.0487 (7)	0.0767 (9)	0.0553 (7)	-0.0123 (6)	0.0181 (5)	0.0033 (6)
O2	0.0431 (7)	0.1021 (13)	0.0731 (8)	-0.0180 (7)	0.0197 (6)	-0.0050 (7)
O3	0.0403 (6)	0.0800 (9)	0.0427 (6)	-0.0071 (5)	0.0139 (5)	0.0042 (5)
O4	0.0787 (10)	0.1250 (15)	0.0509 (7)	-0.0008 (9)	0.0322 (7)	-0.0084 (7)

Geometric parameters (Å, °)

C1—O4	1.200 (2)	C7—O3	1.3778 (18)
C1—C2	1.471 (2)	C8—C9	1.314 (2)
C1—H1	0.9300	C8—O3	1.3640 (18)
C2—C3	1.390 (2)	C8—H8	0.9300
C2—C7	1.3910 (19)	C9—C10	1.454 (2)
C3—C4	1.375 (3)	C9—H9	0.9300
C3—H3	0.9300	C10—O2	1.2035 (19)
C4—C5	1.380 (2)	C10—O1	1.342 (2)
C4—H4	0.9300	C11—O1	1.438 (2)
C5—C6	1.378 (2)	C11—H11A	0.9600
C5—H5	0.9300	C11—H11B	0.9600
C6—C7	1.380 (2)	C11—H11C	0.9600
C6—H6	0.9300		
O4—C1—C2	123.98 (17)	O3—C7—C2	115.83 (13)
O4—C1—H1	118.0	C6—C7—C2	120.61 (14)
C2—C1—H1	118.0	C9—C8—O3	120.00 (14)
C3—C2—C7	118.80 (14)	C9—C8—H8	120.0

C3—C2—C1	119.24 (14)	O3—C8—H8	120.0
C7—C2—C1	121.91 (15)	C8—C9—C10	122.98 (15)
C4—C3—C2	120.77 (15)	C8—C9—H9	118.5
C4—C3—H3	119.6	C10—C9—H9	118.5
C2—C3—H3	119.6	O2—C10—O1	122.10 (15)
C3—C4—C5	119.52 (16)	O2—C10—C9	124.32 (16)
C3—C4—H4	120.2	O1—C10—C9	113.58 (14)
C5—C4—H4	120.2	O1—C11—H11A	109.5
C6—C5—C4	120.83 (15)	O1—C11—H11B	109.5
C6—C5—H5	119.6	H11A—C11—H11B	109.5
C4—C5—H5	119.6	O1—C11—H11C	109.5
C5—C6—C7	119.46 (15)	H11A—C11—H11C	109.5
C5—C6—H6	120.3	H11B—C11—H11C	109.5
C7—C6—H6	120.3	C10—O1—C11	115.85 (13)
O3—C7—C6	123.51 (13)	C8—O3—C7	120.10 (12)
O4—C1—C2—C3	2.7 (3)	C3—C2—C7—C6	-0.5 (2)
O4—C1—C2—C7	-179.85 (17)	C1—C2—C7—C6	-178.03 (15)
C7—C2—C3—C4	-0.6 (2)	O3—C8—C9—C10	-176.28 (16)
C1—C2—C3—C4	176.94 (17)	C8—C9—C10—O2	178.78 (19)
C2—C3—C4—C5	1.0 (3)	C8—C9—C10—O1	-1.2 (3)
C3—C4—C5—C6	-0.3 (3)	O2—C10—O1—C11	1.2 (3)
C4—C5—C6—C7	-0.9 (3)	C9—C10—O1—C11	-178.82 (16)
C5—C6—C7—O3	178.55 (15)	C9—C8—O3—C7	-172.25 (16)
C5—C6—C7—C2	1.3 (2)	C6—C7—O3—C8	26.2 (2)
C3—C2—C7—O3	-178.02 (14)	C2—C7—O3—C8	-156.37 (14)
C1—C2—C7—O3	4.5 (2)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C9—H9...O2 ⁱ	0.93	2.54	3.440 (2)	164
C8—H8...O4 ⁱⁱ	0.93	2.61	3.529 (2)	171
C11—H11C...O2 ⁱⁱⁱ	0.96	2.63	3.578 (2)	168

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