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Ethyl (*E*)-4-(2-formylphenoxy)but-2-enoate

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Key indicators

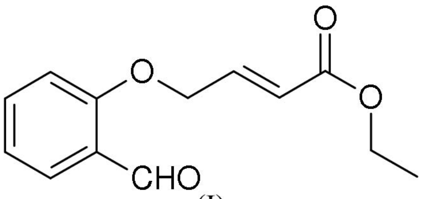
Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.048
wR factor = 0.126
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title compound, $C_{13}H_{14}O_4$, possesses normal geometric parameters. Its approximately planar conformation could be influenced by two intramolecular C—H···O interactions.

Comment

The title compound, (I), was prepared as a test substrate for an investigation into potential catalysts for the intramolecular Stetter reaction. The compound is well known and has been previously used in this context (Kerr *et al.*, 2002). In the present work, the synthesis used was that of Gong *et al.* (1998).



(I)

The molecule of compound (I) possesses normal geometric parameters (Table 1). The complete molecule is approximately planar (for the non-H atoms, the r.m.s deviation from the least-squares plane is 0.100 Å). This conformation might be stabilized by two intramolecular C—H···O interactions

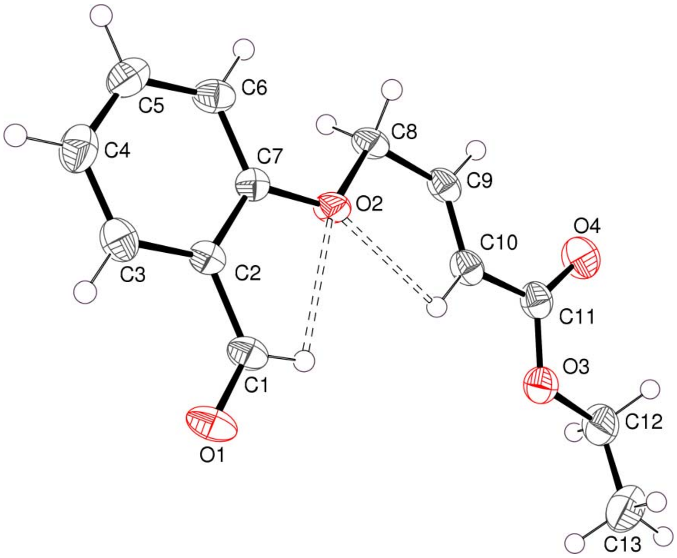


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as small spheres of arbitrary radi. The possible C—H···O interactions are indicated by dashed lines.

(Fig. 1, Table 2). The acute O—H···O bond angles are consistent with the intramolecular nature of these putative bonds. The r.m.s. deviation from the mean plane for atoms C1, C2, C7, C8, C9, C10 and O2 is 0.043 Å [maximum deviation 0.1005 (11) Å for O2].

There are no π–π stacking or other weak intermolecular interactions in (I) and the crystal packing (Fig. 2) is controlled by van der Waals forces.

Experimental

A dry two-necked flask was charged with NaH (15 mmol, 360.4 mg). Dry dimethylformamide (40 ml) was added and the resulting suspension cooled to 273 K. Salicylaldehyde (10 mmol, 1.220 g, 1.06 ml) was added and the solution stirred for 20 min. Ethyl 4-bromocrotonate (11 mmol, 2.82 g, 2.01 ml) was added in one portion. The solution was then allowed to warm to room temperature and stirred for 1 h. Water (60 ml) was added, followed by extraction with Et_2O (3 × 50 ml). The combined organic phases were washed with saturated brine (75 ml), dried ($MgSO_4$) and the solvent removed. Chromatography of the resulting solid in 10% EtOAc in hexane allowed collection of the desired product (1.809 g, 77.2%), which was recrystallized from ethanol as colourless blocks or plates; m.p 342–344 K. Analysis, $C_{13}H_{14}O_4$ requires: C 66.66, H 6.02%; found: C 66.53, H 6.00%. Spectroscopic analysis: IR (KBr, ν_{max}, cm^{-1}): 2975.6 (Ar), 2902.4 (CH), 2859.5 (CHO), 1709.4 (CO_2Et), 1671 (CHO); ^{1}H NMR (250 MHz, $CDCl_3$, δ, p.p.m.); 10.5 (1H, *s*, CHO), 7.8 (1H, *d*, J = 8 Hz, Ph), 7.6 (1H, *t*, J = 8 Hz, Ph), 7.0 (3H, *m*), 6.2 (1H, *d*, J = 15 Hz, CH—CO_2Et), 4.8 (2H, *s*, CH_2), 4.2 (2H, *q*, J = 7 Hz, CH_2), 1.3 (3H, *t*, J = 8 Hz, Me); ^{13}C NMR (250 MHz, $CDCl_3$, δ, p.p.m.) 189.3 (CHO), 165.8 (CO_2Et), 160.2, 141.2, 135.9, 128.8, 125.1, 122.5, 121.4, 112.5, 66.8 (CH_2), 60.7 (CH_2), 14.2 (Me); MS (ESI$^+$): calculated: m/z 252.1230; found: 252.1232 [M+NH_4^+].

Crystal data

$C_{13}H_{14}O_4$
M_r = 234.24
Monoclinic, $P2_1/c$
a = 10.6759 (6) Å
b = 6.9487 (4) Å
c = 16.4346 (6) Å
β = 102.164 (3)°
V = 1191.81 (11) Å^3
Z = 4

D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2702 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 120 (2) K
Plate, colourless
0.46 × 0.27 × 0.09 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.957, T_{max} = 0.993
10 415 measured reflections

2712 independent reflections
1830 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.5°
h = −13 → 13
k = −8 → 8
l = −17 → 21

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.126
S = 1.02
2712 reflections
156 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.2428P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.036 (5)

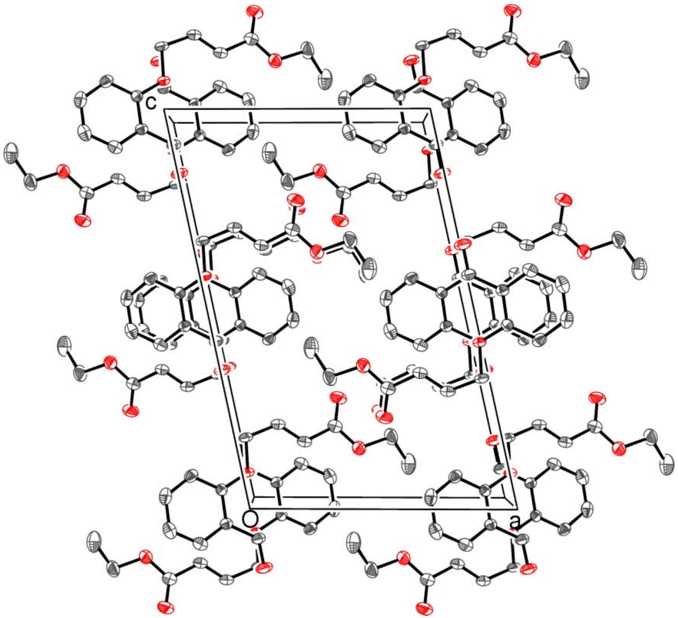


Figure 2
The unit-cell packing in (I), viewed down [010]. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

Table 1
Selected torsion angles (°).

O1—C1—C2—C7	179.50 (15)	O2—C8—C9—C10	5.2 (2)
C1—C2—C7—O2	−1.2 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2	0.95	2.39	2.7353 (17)	101
C10—H10···O2	0.95	2.38	2.7221 (19)	101

All H atoms were placed in calculated positions, with C—H distances in the range 0.95–0.99 Å, and refined as riding on their carrier atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor 1997); data reduction: *DENZO* (Otwinowski & Minor 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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