

(E)-3-(3-Phenylpropoxy)but-2-enoic acid**Jing Li**Key Laboratory of Pesticides and Chemical
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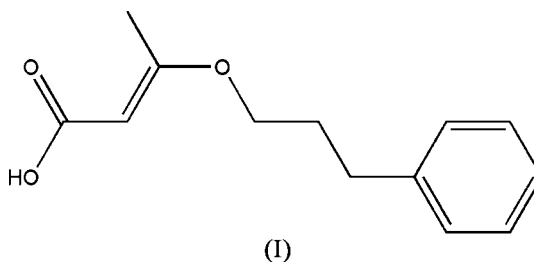
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Key indicatorsSingle-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.064
 wR factor = 0.179
Data-to-parameter ratio = 12.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $\text{C}_{13}\text{H}_{16}\text{O}_3$, molecules related by a centre of symmetry are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds between the carboxyl groups to form a dimer. The propoxy chain is disordered, with an occupancy ratio of 0.69 (1):0.31 (1) between two components with roughly enantiomorphic configurations.

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γ -Alkylation of 1,3-dicarbonyl compounds is a classic reaction in organic synthesis. Removal of a proton from the α -carbon using a base gives the corresponding enolate anion, and this active site can then form a C—C single bond (Carruthers, 1986). The title compound, (I), is obtained by hydrolyzing ethyl 3-(3-phenylpropoxy)but-2-enoate, which is a by-product of the γ -alkylation reaction (Niewöhner *et al.*, 2003).

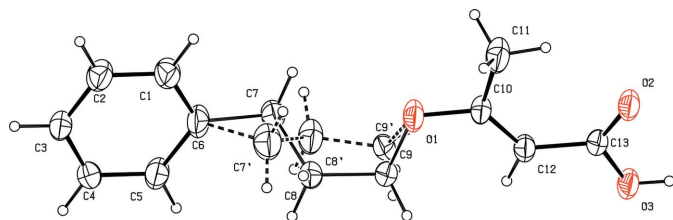


In the molecule of (I), the propoxy chain, O1—C9—C8—C7—C6, is disordered over two sites (Fig. 1), with group occupancies of 0.69 (1) and 0.31 (1) for the major and minor components, respectively. The configuration of the major and minor components are roughly enantiomorphic.

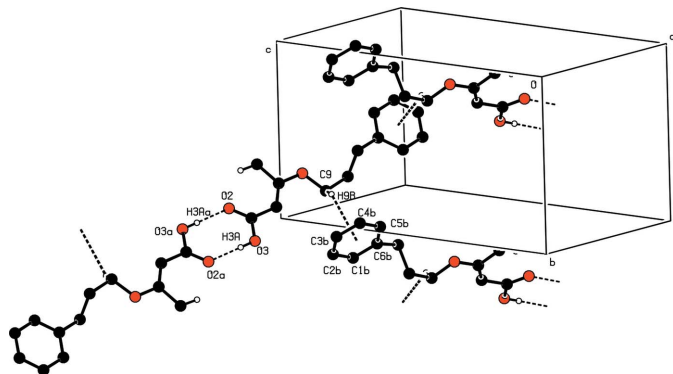
Intramolecular $\text{C}-\text{H}\cdots\text{O}$ and intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, and a $\text{C}-\text{H}\cdots\pi$ interaction, play a role in stabilizing the molecular and crystal structures of (I) (Table 1, Fig. 2).

Experimental

Ethyl 3-(3-phenylpropoxy)but-2-enoate (0.1 g, 0.4 mmol) was dissolved in dioxane (2 ml), and then 3.75 M sodium hydroxide (2 ml) was added. After stirring for 48 h at room temperature, the mixture was concentrated, treated with water (3 ml) and extracted with diethyl ether. The aqueous phase was cooled to 273 K and treated with 3 N hydrochloric acid until pH 1 was reached. It was then extracted with diethyl ether. The ether phase was dried and concentrated, yielding compound (I) (yield 18%). Single crystals of (I) suitable for X-ray diffraction were grown from a diethyl ether solution at 296 K.

**Figure 1**

A view of the molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. The minor disorder component is indicated by dashed lines.

**Figure 2**

A packing view of (I), showing hydrogen-bonds and C—H... π interactions (dashed lines). The suffixes a and b correspond to symmetry codes $(-x - 1, -y + 2, -z + 2)$ and $(\frac{1}{2} - x, 1/2 + y, 3/2 - z)$, respectively.

Crystal data

$C_{13}H_{16}O_3$	$Z = 4$
$M_r = 220.26$	$D_x = 1.183 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 8.8008 (9) \text{ \AA}$	$\mu = 0.08 \text{ mm}^{-1}$
$b = 9.0837 (9) \text{ \AA}$	$T = 292 (2) \text{ K}$
$c = 15.7827 (17) \text{ \AA}$	Block, colourless
$\beta = 101.470 (2)^\circ$	$0.30 \times 0.20 \times 0.10 \text{ mm}$
$V = 1236.5 (2) \text{ \AA}^3$	

Data collection

Bruker SMART 4K CCD area-detector diffractometer	2159 independent reflections
φ and ω scans	1586 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.026$
7552 measured reflections	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0769P)^2 + 0.4223P]$
$R[F^2 > 2\sigma(F^2)] = 0.064$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.179$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
2159 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
178 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$Cg1$ is the centroid of the phenyl ring.

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C11-H11A \cdots O2$	0.96	2.16	2.893 (4)	132
$O3-H3A \cdots O2^i$	0.82 (3)	1.83 (3)	2.651 (3)	178 (2)
$C9-H9B \cdots Cg1^{ii}$	0.97	2.93	3.659 (6)	133

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

All H atoms were initially located in a difference Fourier map. The coordinates of the O-bound H atom were refined freely, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but the methyl group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range $0.93\text{--}0.97 \text{ \AA}$ and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The atoms of the $O1-C9-C8-C7-C6$ linkage are disordered over two positions. The site-occupancy factors were refined to 0.69 (1) and 0.31 (1) and then fixed in the final refinement.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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