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2-(Methoxycarbonyl)-3-phenylprop-2-enyl-3-hydroxynapthalene-2-carboxylate

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Key indicators

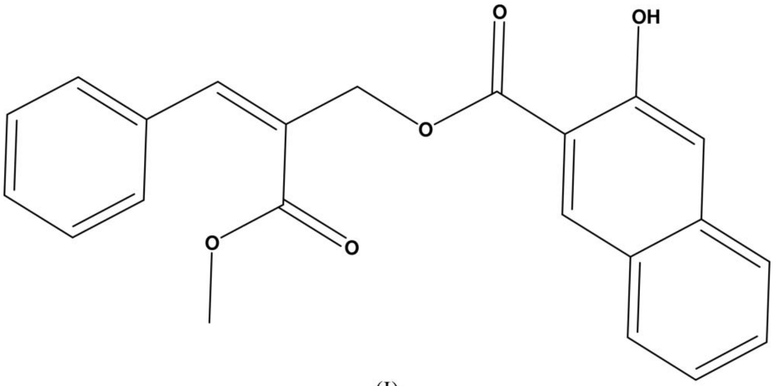
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.122
Data-to-parameter ratio = 16.7

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

In the title compound, $C_{22}H_{18}O_5$, the dihedral angle between the naphthalene plane and the phenyl ring is 81.25 (5)°. The crystal packing is stabilized by intermolecular O—H···O interactions, generating a centrosymmetric dimer with an $R_2^2(12)$ motif.

Comment

The Baylis–Hillman reaction (Basavaiah *et al.*, 2003) continues to attract the attention of various research groups as it leads to multifunctional derivatives, which have been successfully translated to a wide spectrum of synthetically important molecules. The study of nucleophilic substitution reactions of Baylis–Hillman adducts is an interesting endeavour, as it improves the functionality, thereby rendering the substitution products amenable to further chemical transformations. Towards this end we have carried out the nucleophilic substitution of a Baylis–Hillman bromide with 3-hydroxy-2-naphthoic acid. In view of the synthetic utility of nucleophilic substitution products of Baylis–Hillman bromides, we have undertaken the X-ray crystal structure determination of the title compound, (I).



(I)

Bond lengths and angles in (I) (Fig. 1) are within normal ranges (Allen *et al.*, 1987). The dihedral angle between the naphthalene plane and the C17–C22 phenyl ring is 81.25 (5)°. Atom O1 is displaced by 0.042 (1) Å from the plane of the naphthalene ring system.

The molecular structure is stabilized by O—H···O and weak C—H···O intramolecular interactions. The O1—H1···O2 intramolecular interaction generates an $S(6)$ ring. The crystal packing is stabilized by O1—H1···O2^{i} [symmetry code: (i) $1 - x$, $1 - y$ $1 - z$] intermolecular interactions, generating a centrosymmetric dimer with an $R_2^2(12)$ ring (Fig. 2). A short O2···O2^{i} contact of 2.7664 (14) Å is observed

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within a dimer. The dimers are interlinked by C—H⋯π interactions (Table 2) involving the C17–C22 phenyl ring.

Experimental

To a stirred solution of methyl 2-bromomethyl-3-phenyl prop-2-enoate (0.3 g, 1.18 mmol) in dimethylformamide (5 ml), 3-hydroxy-2-naphthoic acid (0.22 g, 1.18 mmol, 1 equivalent) and K_2CO_3 (0.16 g, 1.18 mmol, 1 equivalent) were added. The reaction mixture was stirred at room temperature until the disappearance of the starting materials, as monitored by thin-layer chromatography. The reaction mixture was then poured into water and extracted with ethyl acetate (3 × 30 ml). The organic layers were dried over Na_2SO_4 and evaporated under reduced pressure. The resulting crude product was purified by column chromatography (20–30% EtOAc–hexane mixtures, silica gel, 100–200 mesh) (yield 0.25 g, 60%). Crystals of (I) were grown by slow evaporation of an ethyl acetate solution.

Crystal data

$C_{22}H_{18}O_5$
$M_r = 362.36$
Monoclinic, $P2_1/c$
$a = 7.5053$ (7) Å
$b = 22.468$ (2) Å
$c = 10.7081$ (9) Å
$\beta = 106.101$ (1)°
$V = 1734.9$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
0.27 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: none
19634 measured reflections
4075 independent reflections
3620 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.122$
$S = 1.01$
4075 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.26$ e Å^{-3}

Table 1
Selected torsion angles (°).

C9—C10—C11—O2	−179.7 (1)	C12—C13—C14—O5	4.7 (2)
C1—C10—C11—O2	−2.0 (2)	C16—C13—C14—O4	1.5 (1)
C9—C10—C11—O3	−0.7 (2)	O5—C14—O4—C15	−2.3 (2)
C1—C10—C11—O3	177.0 (1)		

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

*Cg*1 is the centroid of the C17–C22 phenyl ring.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1⋯O2	0.82	1.93	2.644 (1)	145
O1—H1⋯O2$^{\rm i}$	0.82	2.45	3.106 (1)	137
C9—H9⋯O3	0.93	2.38	2.723 (2)	101
C16—H16⋯O4	0.93	2.27	2.687 (1)	107
C15—H15*B*⋯*Cg*1$^{\rm ii}$	0.96	2.67	3.446 (2)	138

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

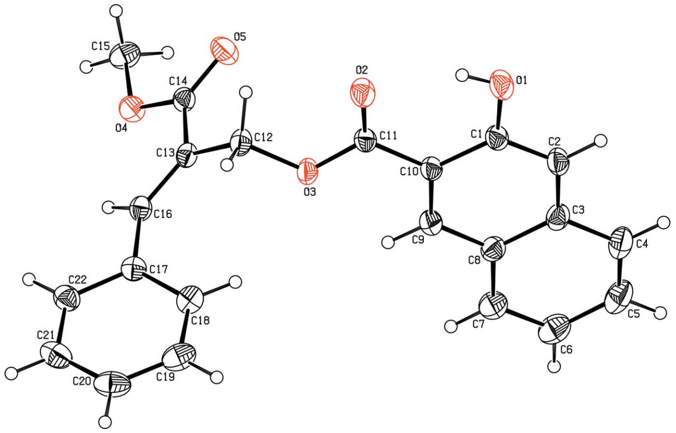

Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids.

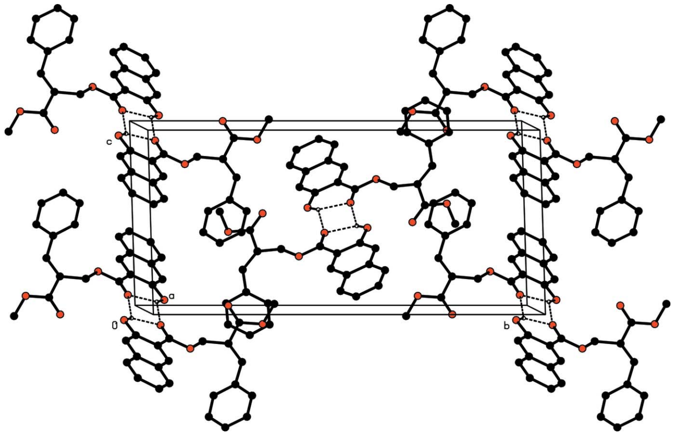

Figure 2
The molecular packing of (I), viewed approximately along the *a* axis. For clarity, H atoms not involved in the hydrogen bonds (dashed lines) have been omitted.

H atoms were positioned geometrically and allowed to ride on their parent atoms, with O—H = 0.82 Å, C—H = 0.93–0.97 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}$(methyl C,O) or $1.2U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; 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* and *PARST* (Nardelli, 1995).

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