

Ethyl 3-hydroxy-2-naphthoate

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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.064
 wR factor = 0.147
Data-to-parameter ratio = 14.5For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the title compound, $\text{C}_{13}\text{H}_{12}\text{O}_3$, there are two independent molecules in the asymmetric unit. Both the molecules are nearly planar and an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond is observed between the hydroxy group and the O atom of the carbonyl group. The crystal structure is stabilized by $\pi-\pi$ stacking interactions.

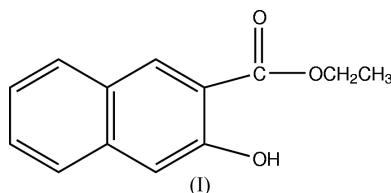
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Comment

The title compound, (I), is an important material for the preparation of many compounds (Gibney *et al.*, 1993; Franczyk *et al.*, 1992). Furthermore, 3-hydroxy-2-naphthoate and some of its derivatives are also interesting ligands because of the coordination ability of the ester group for metal ions. These properties stimulated the search for new methods to synthesize them and relate their activities to structures (Przhiyalgovskaya & Gaevskaya, 1970; Dogan *et al.*, 1998). In this paper, the X-ray crystal structure determination of (I) is reported.



The asymmetric unit contains two crystallographically independent molecules (Fig. 1). Bond lengths and angles in (I) are normal, and values for the two independent molecules agree with each other (Table 1). Both the independent molecules are almost planar and the dihedral angle formed by these planes is $67.82(5)^\circ$. In both molecules, an intramolecular hydrogen bond is observed between the hydroxy group and

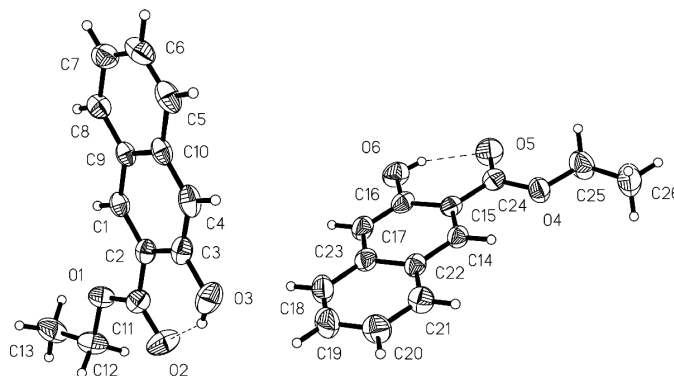


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids. $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds are shown as dashed lines.

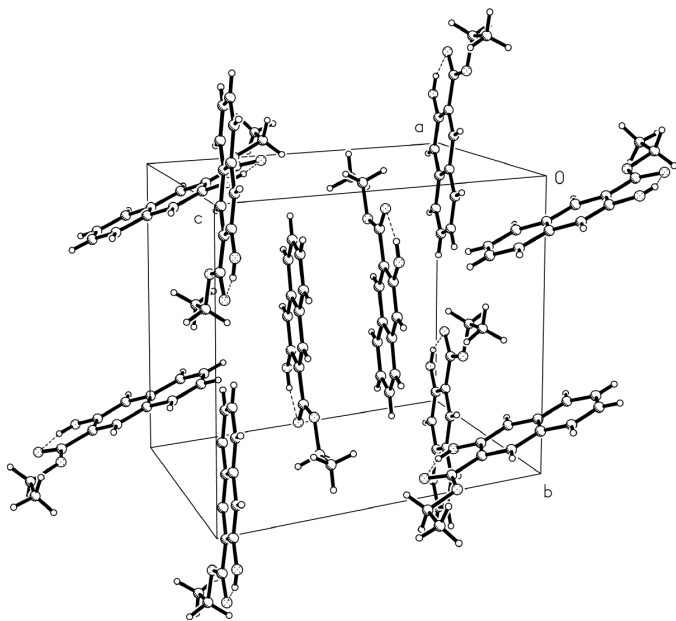


Figure 2
Packing diagram for (I). O—H...O hydrogen bonds are shown as dashed lines.

the O atom of the carbonyl group (Table 2). In the crystal structure, both independent molecules are involved in face-to-face π - π stacking interactions with their inversion-related partners (Fig. 2). The perpendicular distance between the benzene rings in the π - π stacking range from 3.408 to 3.529 Å, similar to those observed in related structures (Du *et al.*, 2003; Xiao *et al.*, 2000).

Experimental

Compound (I) was synthesized according a literature procedure (Gibney *et al.*, 1993). Single crystals suitable for X-ray analysis were grown from a saturated solution in methanol, by slow evaporation at room temperature.

Crystal data

$C_{13}H_{12}O_3$	$D_x = 1.304 \text{ Mg m}^{-3}$
$M_r = 216.23$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1884 reflections
$a = 13.8103 (9) \text{ \AA}$	$\theta = 2.3\text{--}20.9^\circ$
$b = 11.5082 (7) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 15.0809 (9) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 113.236 (1)^\circ$	Block, colourless
$V = 2202.4 (2) \text{ \AA}^3$	$0.30 \times 0.20 \times 0.10 \text{ mm}$
$Z = 8$	

Data collection

Bruker SMART CCD area-detector diffractometer	4236 independent reflections
φ and ω scans	2440 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.050$
$T_{\text{min}} = 0.973$, $T_{\text{max}} = 0.991$	$\theta_{\text{max}} = 26.0^\circ$
14 743 measured reflections	$h = -17 \rightarrow 17$
	$k = -14 \rightarrow 14$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.147$
 $S = 1.04$
 4236 reflections
 293 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2 + 0.255P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.0001$
 $\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C3—O3	1.361 (3)	C15—C24	1.465 (4)
C2—C11	1.468 (4)	C16—O6	1.361 (3)
C11—O1	1.325 (3)	C24—O4	1.322 (3)
C12—O1	1.453 (3)	C25—O4	1.453 (3)
O3—C3—C2	121.4 (3)	O6—C16—C15	121.4 (3)
O2—C11—O1	122.3 (3)	O5—C24—O4	121.8 (3)
O2—C11—C2	124.3 (3)	O5—C24—C15	124.2 (3)
C3—C2—C11—O2	−6.5 (4)	O2—C11—O1—C12	0.7 (4)
C1—C2—C11—O1	−6.7 (3)	C13—C12—O1—C11	177.8 (2)
C16—C15—C24—O5	5.9 (4)	O5—C24—O4—C25	0.4 (4)
C14—C15—C24—O4	5.7 (4)	C26—C25—O4—C24	178.5 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O3—H3...O2	0.82	1.91	2.632 (3)	146
O6—H6A...O5	0.82	1.90	2.627 (3)	146

H atoms were placed in calculated positions, with C—H distances of 0.93 (naphthalene nucleus), 0.96 (CH₃) and 0.97 Å (CH₂), and O—H distances of 0.82 Å. They were included in the refinement using a riding model with isotropic displacement parameters set equal to 1.2 U_{eq} of the carrier atom for the naphthalene nucleus and CH₂ H atoms, and 1.5 U_{eq} of the carrier for CH₃ and hydroxy H atoms.

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

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