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Key indicators

Single-crystal X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.049
wR factor = 0.126
Data-to-parameter ratio = 25.5

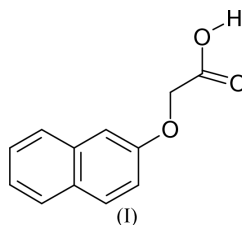
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Redetermination of 2-(naphthalen-2-yloxy)ethanoic acid

The redetermination of the title compound, $\text{C}_{12}\text{H}_{10}\text{O}_3$, agrees completely with the results previously reported by Pattabhi, Ragnathan & Chacko [*Acta Cryst.* (1973), **B34**, 3118–3120], but with improved precision. In addition, now reported are intermolecular $\pi-\pi$, $\text{C}-\text{H}\cdots\pi$ and aryl $\text{C}-\text{H}\cdots\text{O}$ interactions.

Comment

A search of the Cambridge Structural Database (Allen & Kennard, 1993) at the Chemical database service of the EPSRC (Fletcher *et al.*, 1996) reveals that the structure of the title compound, (I), was first reported (NAPXAC10; Pattabhi *et al.*, 1973) with $R = 0.083$ for 1623 reflections. The redetermination reported here with current refinement software and a more extensive set of intensity data yields a model of greater precision and improved R . Allowing for the different choice of unit cell ($P2_1/n$ for $P2_1/c$) and atom-labelling scheme (Fig. 1), the results of the redetermination (Table 1) agree in all respects with those of Pattabhi *et al.* (1973).



A feature unremarked on in the original determination but replicated here was the variation in $\text{C}-\text{C}$ bond lengths within the naphthalene ring system of the molecule. The bonds $\text{C}1-\text{C}2$, $\text{C}4-\text{C}5$, $\text{C}6-\text{C}7$ and $\text{C}9-\text{C}10$ at the corners of the ring system are in the range 1.3568 (19)–1.362 (2) Å (Table 1). The remainder are longer, ranging from 1.392 (2) to 1.4197 (18) Å . The difference between the bond lengths of the two groups is at least 0.03 Å and no less than 15 times the s.u.'s, and therefore significant and clearly of the type described elsewhere, *e.g.* for naphthalene (Brock & Dunitz, 1982).

The planarity of the molecule as a whole also merits comment. In addition to the undoubted planarity of the naphthalene group [maximum out of plane distance 0.0079 (13) Å for C7], the dihedral angles at the $\text{C}11-\text{C}12$ bond are clearly indicative of comparable planarity for the group comprising C11, C12 and O1 to O3 [maximum out of plane distance 0.0066 (9) for C11]. It is tempting then to estimate the angle between these planar entities by means of

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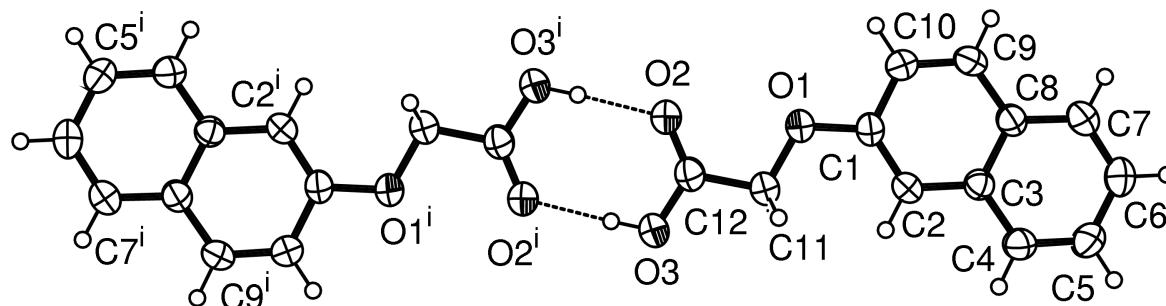


Figure 1

The centrosymmetric hydrogen-bonded dimer of (I). All non-H atoms of the asymmetric unit and selected non-H atoms of the symmetry related molecule [symmetry code: (i) $-x + 2, -y - 1, -z$] are labelled. Non-H atoms are shown as 50% probability ellipsoids and H atoms are shown as open circles. Dashed lines represent $\text{H} \cdots \text{O}$ contacts.

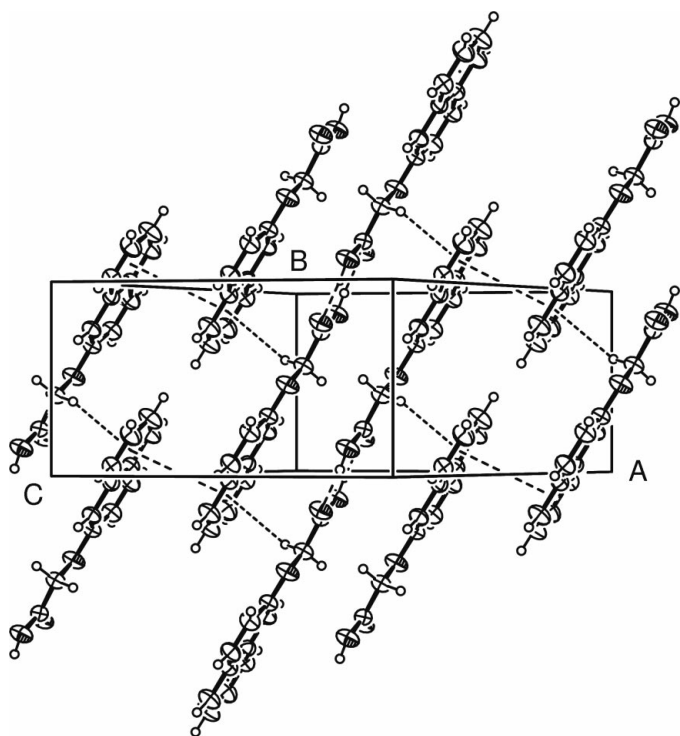


Figure 2

Part of a layer of molecules of (I) parallel to (001). The representation of the atoms is the same as in Fig. 1 and dashed lines represent intermolecular contacts (see text).

the dihedral angles at the C1–O1 bond which yield an average value of $5.88 (12)^\circ$. This, however, overestimates the value of $4.13 (5)^\circ$ from mean-plane calculation because it fails to take account of the effect of the C1–O1–C11–C12 torsion angle which accounts for the difference. The departure from planarity is not in any case large.

As shown in Fig. 2, in which the essentially planar molecules are seen edge-on, the molecules are packed face-to-face to form layers parallel to (001) at $z = 0$ and $\frac{1}{2}$. Three significant intermolecular contacts arise within the layers. The first of these is the previously reported centrosymmetrically hydrogen-bonded pairwise association of the molecules (Fig. 1 and Table 2). The others, elucidated by *PLATON* (Spek, 1990), both involve the π system of the C3–C8 ring (with centroid C_g) of the naphthalene group. In one of these, the rings are found in centrosymmetrically related pairs and are then by definition parallel to one another within the pair (Fig. 3). This interaction is characterized by the perpendicular distance between the parallel planes (3.604 \AA), the distance between their centroids [$C_g \cdots C_g^i$ 4.028 \AA ; symmetry code: (i) $1 - x, 1 - y, -z$] and the angle between these vectors (26.52°). Finally, there is an interaction of the form $\text{C11} - \text{H11B} \cdots C_g^{ii}$ [symmetry code: (ii) $x, y - 1, z$]. This is characterized by five parameters as: (i) the distance between H and the centroid of the π system ($\text{H} \cdots C_g^{ii}$ 2.792 \AA); (ii) the perpendicular distance from H to the plane of the aryl ring ($\text{H-perp} =$

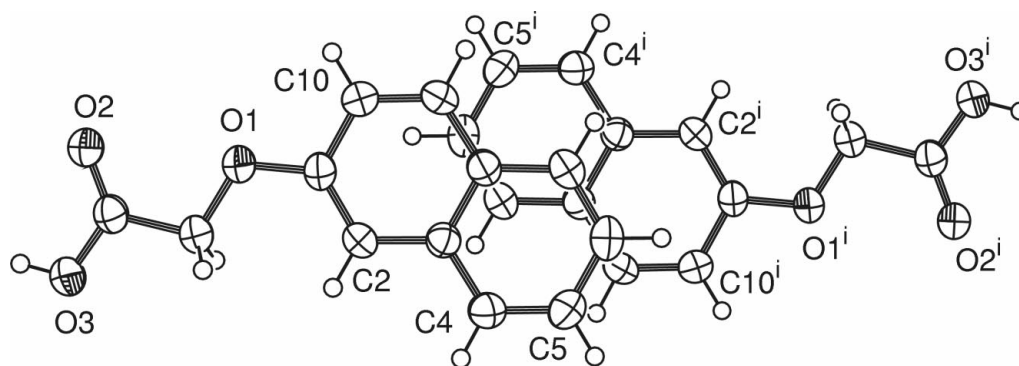


Figure 3

A π - π stacked pair of molecules of (I). The representation of the atoms is the same as in Figs. 1 and 2, and selected atoms are labelled [symmetry code: (i) $1 - x, 1 - y, -z$].

2.747 Å); (iii) the angle between these vectors ($\gamma = 10.28^\circ$); (iv) the angle C11–H11B \cdots Cgⁱⁱ (137.29°); and (v) the C11 \cdots Cgⁱⁱ distance (3.56 Å).

As noted above, the crystallographic centres of symmetry are involved in relationships between molecules within the layers. The layers themselves are related instead by the operation of the crystallographic *n*-glides, and equivalently by the crystallographic twofold screw axes. The tilt of the molecules relative to (010) therefore alternates from one layer to the next to yield a crisscross effect overall. The only interaction of any significance between the layers is the weak hydrogen-bond-type contact C5–H5 \cdots O2 (Table 2).

Experimental

As reported by Fries (1921), basic hydrolysis of methyl 2-naphthalenyloxyethanoate prepared by reaction of 2-naphthol and methyl bromoethanoate yielded (I), which was recrystallized from 1% aqueous hydrochloric acid solution [m.p. 432–433 K (literature value 428 K)].

Crystal data

C ₁₂ H ₁₀ O ₃	$D_x = 1.354 \text{ Mg m}^{-3}$
$M_r = 202.20$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1594 reflections
$a = 12.3112(10) \text{ \AA}$	$\theta = 3.0\text{--}25.5^\circ$
$b = 6.8401(5) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 13.1658(10) \text{ \AA}$	$T = 295(2) \text{ K}$
$\beta = 116.507(2)^\circ$	Needle, colourless
$V = 992.14(13) \text{ \AA}^3$	$0.60 \times 0.10 \times 0.03 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART 1000 CCD diffractometer	3577 independent reflections
φ and ω scans	1511 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 1999)	$R_{\text{int}} = 0.043$
$T_{\text{min}} = 0.782$, $T_{\text{max}} = 0.997$	$\theta_{\text{max}} = 32.5^\circ$
9994 measured reflections	$h = -18 \rightarrow 15$
	$k = -10 \rightarrow 9$
	$l = -19 \rightarrow 16$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0590P)^2]$
$wR(F^2) = 0.126$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.82$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3577 reflections	$\Delta\rho_{\text{max}} = 0.18 \text{ e \AA}^{-3}$
140 parameters	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

C1–C2	1.3580 (19)	C5–C6	1.392 (2)
C1–O1	1.3840 (16)	C6–C7	1.362 (2)
C1–C10	1.4091 (18)	C7–C8	1.4135 (19)
C2–C3	1.4197 (18)	C8–C9	1.4146 (19)
C3–C4	1.4125 (18)	C9–C10	1.3568 (19)
C3–C8	1.4167 (17)	O3–H3A	0.87 (2)
C4–C5	1.361 (2)		
C12–O3–H3A	110.8 (11)		
C2–C1–O1–C11	–6.1 (2)	O1–C11–C12–O2	0.55 (19)
C10–C1–O1–C11	174.35 (12)	O1–C11–C12–O3	–179.07 (11)
C1–O1–C11–C12	–177.02 (11)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
O3–H3A \cdots O2 ⁱ	0.87 (2)	1.79 (2)	2.6586 (14)	174.6 (18)
C5–H5 \cdots O2 ⁱⁱ	0.93	2.65	3.3220 (19)	129

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

Aryl and methylene H atoms were placed in calculated positions with C–H = 0.93 and 0.97 Å, respectively, and refined with a riding model with U_{iso} equal to $1.2U_{\text{eq}}$ of the C atom to which they are attached. The hydroxy H (H3A) atom was found in a difference map and refined isotropically in the usual manner.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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