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Diethyl naphthalene-1,4-dicarboxylate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.064
wR factor = 0.172
Data-to-parameter ratio = 14.0

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

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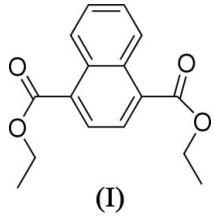
In the title compound, $C_{16}H_{16}O_4$, the two ester groups are not coplanar with the naphthyl ring system. The crystal packing is stabilized by C—H···O hydrogen bonds.

Comment

Naphthalene-1,4-dicarboxylic acid derivatives are a class of intermediates important for applications as monomers in the preparation of polymers (Fukuzumi *et al.*, 1994; Tsukada *et al.*, 1994). Previously, we have reported the crystal structures of dimethyl naphthalene-1,4-dicarboxylate (Jing *et al.*, 2005) and diphenyl naphthalene-1,4-dicarboxylate (Jing *et al.*, 2006). We now report the crystal structure of the title compound, (I).



(I)

The bond lengths and angles in (I) are normal (Table 1). The naphthalene ring system is slightly distorted from planarity, with dihedral angle of 5.9 (1)° between the two rings. As a result of steric effects, the groups at atoms C1 and C4 are

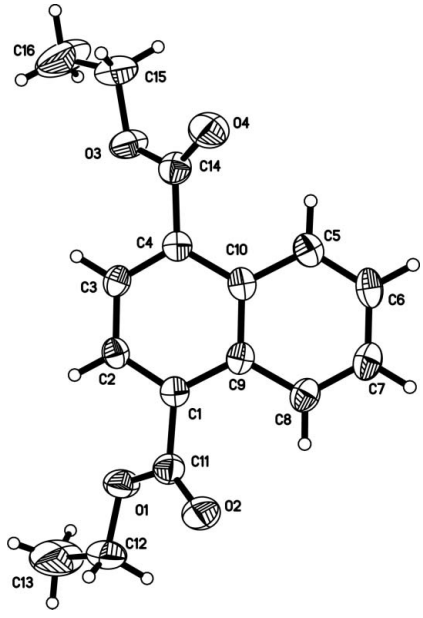


Figure 1
The structure of (I), showing 30% probability displacement ellipsoids and the atomic numbering.

twisted away from the plane of the naphthalene ring system (Fig. 1). The O1/O2/C1/C11 and O3/O4/C4/C14 planes form dihedral angles of 33.8 (1) and 35.3 (1)°, respectively, with the plane formed by atoms C1–C4/C9/C10. The crystal packing is stabilized by C—H···O hydrogen bonds (Table 2).

Experimental

Naphthalene-1,4-dicarboxylic acid (2 mmol) and a excess of thionyl chloride in dioxane (20 ml) were boiled under reflux for 6 h. The solution was distilled at reduced pressure. An excess of ethanol was added to the resulting yellow solid and reacted under reflux for one day. After the solution had cooled to ambient temperature, some water was added, affording a colourless solid. The solution was filtered to remove the ethanol and water. The filter cake was dissolved in ethanol and left to stand for one month at ambient temperature, after which colourless single crystals suitable for X-ray diffraction were obtained.

Crystal data

$C_{16}H_{16}O_4$
M_r = 272.29
Monoclinic, $C2/c$
a = 24.731 (7) Å
b = 7.2792 (16) Å
c = 16.151 (4) Å
β = 95.277 (19)°
V = 2895.2 (13) Å^3
Z = 8
D_x = 1.249 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colourless
0.58 × 0.48 × 0.36 mm

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction: none
2632 measured reflections
2569 independent reflections
1400 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 25.0°
3 standard reflections
every 97 reflections
intensity decay: 3.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.173
S = 1.01
2569 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.091P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0144 (15)

Table 1
Selected geometric parameters (Å, °).

O1—C11	1.328 (3)	O3—C14	1.336 (3)
O1—C12	1.438 (3)	O3—C15	1.441 (4)
O2—C11	1.194 (3)	O4—C14	1.194 (3)
C11—O1—C12	115.9 (2)	C14—O3—C15	116.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···O4	0.93	2.40	2.983 (3)	121
C8—H8···O2	0.93	2.37	2.982 (4)	123

H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C). The methyl groups were allowed to rotate but not to tip.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia,1997); software used to prepare material for publication: *SHELXL97*.

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