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Ethyl pamoate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.119
Data-to-parameter ratio = 13.8

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

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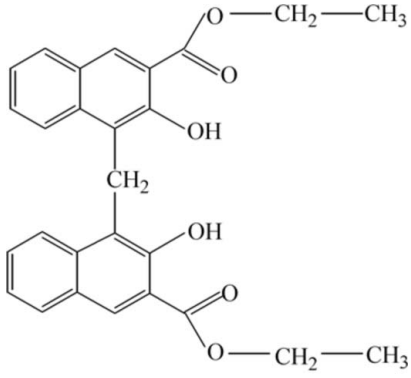
In the title compound, $C_{27}H_{24}O_6$, the dihedral angle between the two naphthalene ring systems is 84.79 (6)°. The unit-cell packing features weak π–π interactions along the *a* axis between adjacent molecules.

Comment

Organotin compounds have been widely used in homogeneous catalysis, and they have shown increased selectivity in a variety of chemical transformations (Edelman *et al.*, 1990; Orita *et al.*, 1999). In this work, the title compound, (I), was synthesized under the catalysis of an organotin compound. Although pamoic acid is a well known industrial precursor (Fei *et al.*, 2001), to the best of our knowledge, the crystal structure of its ethyl ester has never been reported.



(I)

The two naphthalene ring systems of (I) are almost perpendicular, with a dihedral angle of 84.79 (6)° (Fig. 1). The bond distances and bond angles are all normal. Two intramolecular O—H···O interactions (Table 1) are observed. The

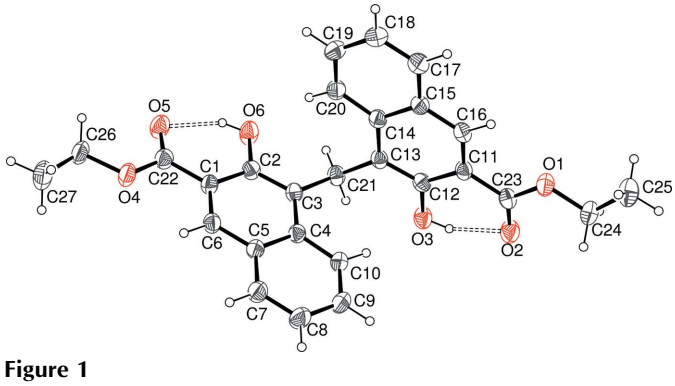


Figure 1
A view of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Hydrogen bonds are indicated by dashed lines.

naphthalene rings of adjacent molecules show centroid-to-centroid distances of 3.97 Å along the *a* axis (Fig. 2), indicating the presence of weak π–π interactions (Dong *et al.*, 2005).

Experimental

A mixture of pamoic acid (0.194 g, 0.5 mmol), NaOH (0.040 g, 1 mmol) and Bu_2SnCl_2 (0.152 g, 0.5 mmol) in ethanol (8 ml) was sealed in a 15 ml Teflon-lined stainless steel bomb and heated to 413 K for 3 days. Yellow crystals of (I) were obtained after slowly cooling to room temperature at the rate of 10 K h^{-1}.

Crystal data

$C_{27}H_{24}O_6$
M_r = 444.46
Monoclinic, $C2/c$
a = 14.556 (5) Å
b = 11.153 (5) Å
c = 27.617 (5) Å
β = 93.514 (5)°
V = 4475 (3) Å^3
Z = 8
D_x = 1.319 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, yellow
0.32 × 0.25 × 0.23 mm

Data collection

Bruker APEX CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.941, T_{max} = 0.965
10534 measured reflections
4127 independent reflections
2992 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.120
S = 1.01
4127 reflections
298 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.057P)^2 + 1.3961P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.82	1.86	2.589 (2)	148
O6—H4···O5	0.82	1.86	2.597 (2)	148

All C-bound H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.97 Å and U_{iso}(H)= $1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C). The hydroxyl H atoms were located in difference maps, repositioned in idealized locations and refined as riding, with U_{iso}(H) = $1.5U_{eq}$(O).

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-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

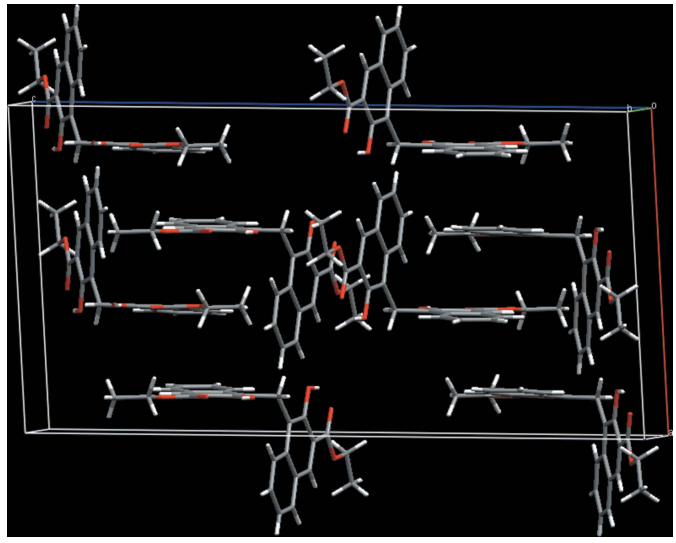


Figure 2
Packing diagram of (I).

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References

Bruker (1997). *SMART*. Version 5.622. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
Dong, X.-W., Wu, H. & Ma, J.-F. (2005). *Acta Cryst.* E**61**, m2400–m2401.
Edelman, M. A., Hitchock, P. B. & Lappert, M. F. (1990). *J. Chem. Soc. Chem. Commun.* pp. 1116–1117.
Fei, Z. F., Slawin, A. M. Z. & Woollins, J. D. (2001). *Polyhedron*, **20**, 3355–3360.
Orita, A., Sakamoto, K., Hamada, Y., Mitsutome, A. & Otera, J. (1999). *Tetrahedron*, **55**, 2899–2910.
Sheldrick, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.