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Ethyl 4-acetoxy-6-(dimethylamino)-2-naphthoate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.135
Data-to-parameter ratio = 14.6

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

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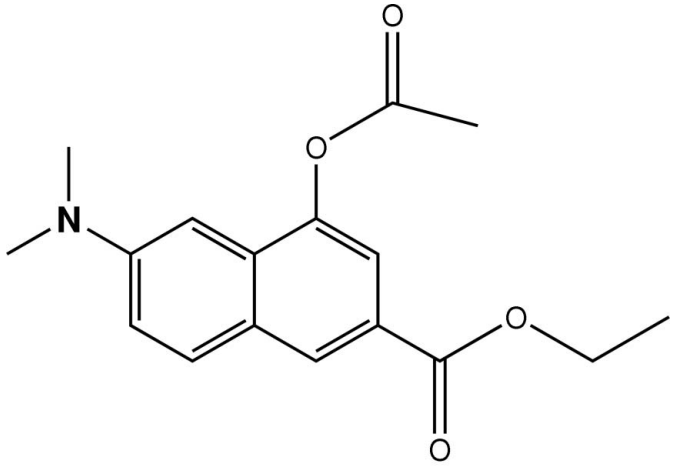
The title compound, $C_{17}H_{19}NO_4$, was synthesized by the Stobbe condensation reaction of *p*-dimethylaminobenzaldehyde and diethyl succinate, followed by cyclization of the Stobbe product. The crystal structure is stabilized by intermolecular C—H$\cdots\pi$ interactions.

Comment

The title compound, (I), is an intermediate in the preparation of photochromic compounds (Clarke *et al.*, 2001). We report here the molecular structure of (I), which is shown in Fig. 1.



(I)

The molecule is nearly planar except for the carboxylate groups and some of the hydrogen atoms. C—H$\cdots\pi$ interactions occur between C17—H18 and the C1–C4/C9/C10 ring, such that the distance between H18 and the ring centroid (*Cg*) is 2.97 Å, while the C—H$\cdots$*Cg*^{i} angle is 144° [symmetry code: (i) x, 1 + y, z].

Experimental

The title compound was prepared according to the procedures described by Tanaka *et al.* (2000) and Clarke *et al.* (2001), which involved the reaction of *p*-dimethylaminobenzaldehyde (4.47 g, 0.03 mol) and diethyl succinate (7.83 g, 0.045 mol) in the presence of ^{t}BuOK (6.72 g, 0.06 mol) at room temperature. The reaction mixture was neutralized with dilute HCl and extracted with ethyl acetate. Removal of the EtOAc afforded a red–brown solid. A solution of this solid with anhydrous sodium acetate (2.46 g, 0.03 mol) in acetic anhydride (100 ml) was refluxed for 6 h. The crystals formed were isolated by filtration. Yellow single crystals suitable for X-ray data collection were obtained by recrystallization from EtOAc–hexane (1:3 *v*/*v*).

Crystal data

$C_{17}H_{19}NO_4$
M_r = 301.33
Monoclinic, $P2_1/c$
a = 12.376 (3) Å
b = 7.0410 (16) Å
c = 17.945 (6) Å
β = 97.853 (12)°
V = 1549.1 (7) Å^3
Z = 4
D_x = 1.292 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
Prism, yellow
0.51 × 0.48 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
20499 measured reflections
2916 independent reflections
2326 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 25.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.135
S = 1.10
2916 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0701P)^2 + 0.2653P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.019 (3)

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.96 Å for aromatic and 0.97 Å for methylene H atoms, and with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC and Rigaku, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

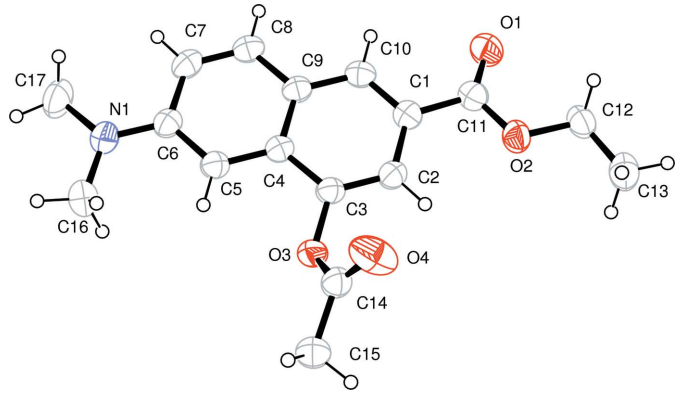


Figure 1
The molecular structure and atom-labelling scheme for (I), showing 40% probability displacement ellipsoids.

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