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Ethyl 2-amino-4-phenyl-4*H*-benzo[*h*]-chromene-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.066
wR factor = 0.187
Data-to-parameter ratio = 14.9

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

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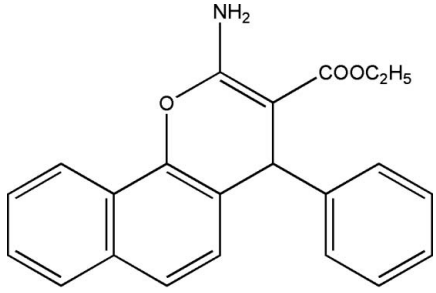
The title compound, $C_{22}H_{19}NO_3$, was synthesized by the reaction of 1-naphthol with ethyl cyanoacetate and benzaldehyde in ethanol under microwave irradiation. In the crystal structure, weak intermolecular N—H···O hydrogen bonds link the molecules into centrosymmetric dimers, which are held together by van der Waals forces.

Comment

Benzopyrans and their derivatives exhibit useful biological and pharmacological properties (Morianka & Takahashi, 1977), acting as antisterility (Brooks, 1988) and anticancer agents (Hyana & Saimoto, 1987). In addition, polyfunctionalized benzopyrans constitute the structural unit of a number of natural products and, because of the inherent reactivity of the inbuilt pyran ring, they may serve as versatile synthons (Hatakeyama *et al.*, 1988). We report here the crystal structure of the title compound, (I).



(I)

In the molecule of (I), (Fig. 1), all bond lengths and angles (Table 1) are normal. The intramolecular N—H···O hydrogen bond (Table 2) defines the orientation of carboxylate group. In the crystal structure, weak intermolecular N—H···O hydrogen bonds (Table 2) link the molecules into centrosymmetric dimers (Fig. 2). The crystal packing is further stabilized by van der Waals forces.

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (5 mmol) with ethyl cyanocaetate (5 mmol) and benzaldehyde (5 mmol) in ethanol (2 ml) by using piperidine (0.5 mmol) as catalyst under microwave irradiation. The pure compound (I) was obtained by recrystallization from ethanol (m.p. 418–419 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution. ^{1}H NMR ($CDCl_3$): δ 8.21 (*d*, 1H), 7.75 (*d*, 1H), 7.52–7.55 (*m*, 1H), 7.45–7.49 (*m*, 2H), 7.25–7.28 (*m*, 2H), 7.19–7.22 (*m*, 2H), 7.15 (*d*, 1H), 7.09–7.12 (*m*, 1H), 6.44 (*s*, 2H), 5.06 (*s*, 1H), 4.10 (*m*, 2H), 1.17 (*t*, 3H).

Crystal data

$C_{22}H_{19}NO_3$
M_r = 345.38
Monoclinic, $P2_1/c$
a = 12.178 (2) Å
b = 8.9950 (18) Å
c = 17.085 (3) Å
β = 110.03 (3)°
V = 1758.3 (6) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 9–12°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.4 × 0.3 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3595 measured reflections
3431 independent reflections
1642 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 26.0°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 10$
$l = -20 \rightarrow 19$
3 standard reflections every 200 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.187
S = 1.10
3431 reflections
231 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.9P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0076 (13)

Table 1
Selected geometric parameters (Å, °).

O1—C3	1.348 (5)	N1—C5	1.335 (4)
O1—C2	1.443 (4)	C1—C2	1.437 (5)
O2—C3	1.225 (4)	C3—C4	1.438 (5)
O3—C5	1.363 (4)	C6—C7	1.524 (5)
O3—C22	1.399 (4)		
C3—O1—C2	114.1 (3)	C4—C5—O3	123.5 (3)
C5—O3—C22	118.2 (3)	C13—C6—C7	109.4 (3)
C1—C2—O1	109.1 (4)	C4—C6—C7	112.6 (3)
O2—C3—O1	121.2 (4)	C8—C7—C6	120.0 (3)
O2—C3—C4	126.2 (4)	C12—C7—C6	122.7 (3)
O1—C3—C4	112.6 (3)	C14—C13—C6	120.4 (3)
C5—C4—C3	118.7 (3)	C20—C21—C22	124.0 (4)
C3—C4—C6	120.2 (3)	C13—C22—O3	122.4 (3)
N1—C5—C4	127.3 (3)	O3—C22—C21	113.5 (3)
N1—C5—O3	109.2 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H0A···O2	0.86	2.08	2.688 (4)	127
N1—H0A···O2^{i}	0.86	2.33	2.954 (4)	130

Symmetry code: (i) $-x+2, -y+1, -z$.

All H atoms were placed in calculated positions and refined as riding, with C—H = 0.93–0.98 Å, N—H = 0.86 Å and U_{iso}(H) = 1.2–1.3U_{eq} of the carrier atom.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

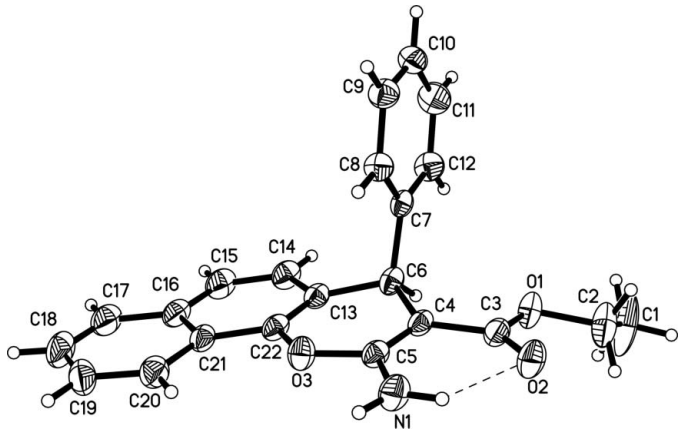


Figure 1
View of (I), showing the labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and the intramolecular hydrogen bond is indicated by a dashed line.

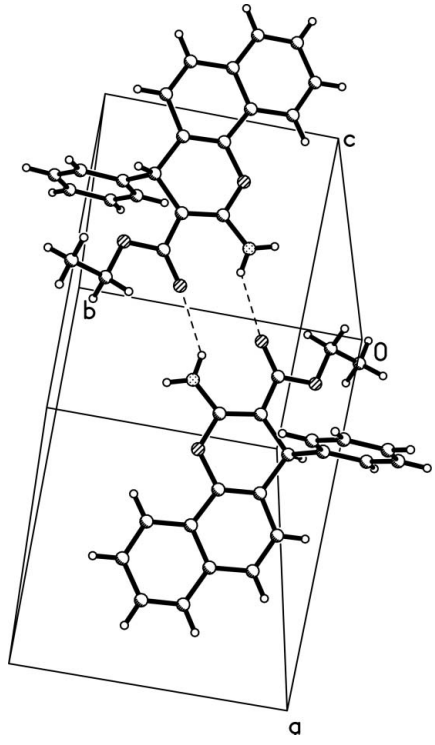


Figure 2
The hydrogen-bonded (dashed lines) centrosymmetric dimer in (I).

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