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Ethyl 2-amino-4-(3-fluorophenyl)-4*H*-benzo[*h*]-chromene-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(Wae) = 0.009 Å
R factor = 0.084
wR factor = 0.289
Data-to-parameter ratio = 14.3

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

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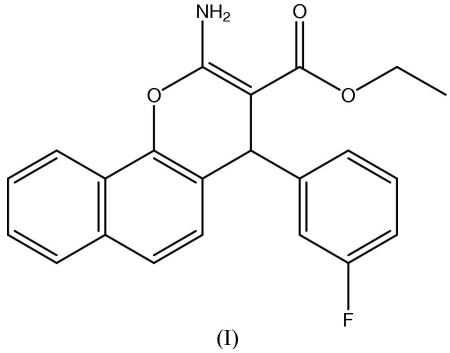
The title compound, $C_{22}H_{18}FNO_3$, was synthesized by the reaction of 1-naphthol with ethyl cyanocaetate and 3-fluorobenzaldehyde in ethanol under microwave irradiation. In the crystal structure, there are intramolecular N—H···O hydrogen bonds and intermolecular N—H···O and N—H···F hydrogen bonds. An intramolecular C—H···π interaction is also present.

Comment

Benzopyrans and their derivatives occupy an important place in the realm of natural and synthetic organic chemistry because of their biological and pharmacological properties (Morianka & Takahashi, 1977), such as antisterility (Brooks, 1998) 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, these are versatile synthons (Hatakeyama *et al.*, 1988).



(I)

We report here the crystal structure of the title compound, (I). The molecular structure of (I) is shown in Fig. 1, where the dashed line indicates the intramolecular N—H···O hydrogen bond (Table 2). The bond lengths and angles are unremarkable. In the crystal structure, molecules are linked by N—H···O and N—H···F intermolecular hydrogen bonds (Table 2 and Fig. 2). There is also an intramolecular contact which indicates a weak C—H···π interaction (Fig. 3 and Table 2), *viz*. C12—H12*A*···*Cg*1, where *Cg*1 is the centroid of the ring O3/C5/C4/C6/C13/C22. The combination of both types of rather weak interactions generates a three-dimensional network.

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (5 mmol) with ethyl cyanocaetate (5 mmol) and 3-fluorobenzaldehyde (5 mmol) in ethanol (2 ml) under microwave irradiation, using

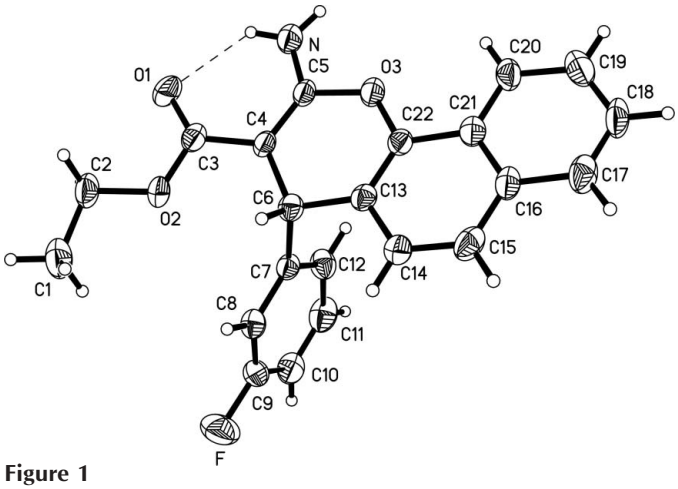


Figure 1
A view of the molecular structure of (I). The dashed line indicates the intramolecular N—H···O hydrogen bond.

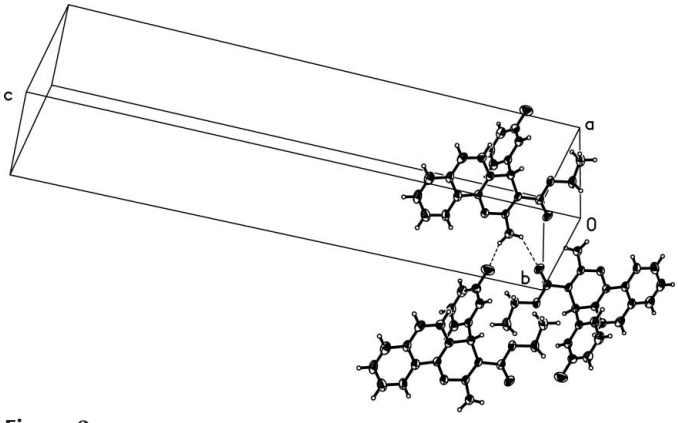


Figure 2
Part of the crystal structure of (I). Dashed lines indicate intermolecular N—H···O and N—H···F hydrogen bonds.

piperidine as a catalyst. Pure compound (I) was obtained by recrystallization from ethanol (m.p. 406–409 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), 7.76 (*d*, 1H), 7.47–7.57 (*m*, 3H), 7.12–7.17 (*m*, 2H), 6.95–7.06 (*m*, 2H), 6.77–6.81 (*m*, 1H), 6.49 (*s*, 2H), 5.06 (*s*, 1H), 4.11 (*q*, 2H), 1.21 (*t*, 3H).

Crystal data

$C_{22}H_{18}FNO_3$
M_r = 363.37
Monoclinic, $P2_1/n$
a = 9.069 (2) Å
b = 6.136 (1) Å
c = 32.537 (7) Å
β = 96.87 (3)°
V = 1797.6 (6) Å^3
Z = 4
D_x = 1.343 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–13°
μ = 0.10 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
3741 measured reflections
3509 independent reflections
1679 reflections with $I > 2\sigma(I)$
R_{int} = 0.120
θ_{max} = 26.0°
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 7$
$l = -38 \rightarrow 38$
3 standard reflections
every 200 reflections
intensity decay: none

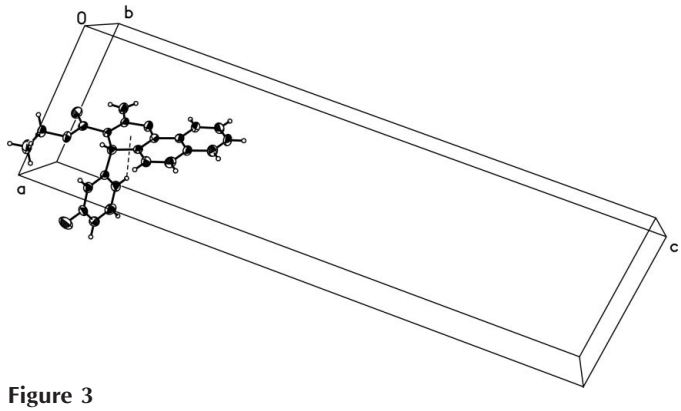


Figure 3
The C—H···π interaction in (I), shown as a dashed line.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.289
S = 1.00
3509 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 6.8P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0072 (17)

Table 1
Selected geometric parameters (Å, °).

F—C9	1.364 (7)	O3—C5	1.389 (6)
O1—C3	1.230 (7)	N—C5	1.322 (7)
O2—C3	1.343 (7)	C1—C2	1.490 (10)
O2—C2	1.444 (7)	C3—C4	1.447 (7)
O3—C22	1.385 (7)	C6—C7	1.533 (7)
C3—O2—C2	117.0 (5)	N—C5—C4	129.7 (5)
C22—O3—C5	118.1 (4)	N—C5—O3	108.0 (5)
O2—C2—C1	107.0 (6)	C4—C5—O3	122.3 (5)
O1—C3—O2	121.7 (5)	C8—C7—C6	120.6 (5)
O1—C3—C4	126.4 (6)	C12—C7—C6	120.5 (5)
O2—C3—C4	111.9 (5)	C13—C22—O3	122.8 (5)
C5—C4—C3	118.5 (5)	O3—C22—C21	113.4 (5)
C3—C4—C6	118.9 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0A···O1	0.86	2.15	2.732 (7)	125
N—H0A···O1^{i}	0.86	2.36	3.023 (7)	134
N—H0B···F^{ii}	0.86	2.46	3.166 (7)	140
C12—H12A···$Cg1$	0.93	2.72	3.069	103

Symmetry codes: (i) $-x+1, -y+2, -z$; (ii) $x-1, y+1, z$. Note: $Cg1$ is the centroid of the ring O3/C5/C4/C6/C13/C22.

All H atoms were positioned geometrically, with C—H = 0.93–0.98 Å and N—H = 0.86 Å; they were included in the refinement in the riding-model approximation, with U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.5 for methyl H atoms and 1.2 for all other H atoms.

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*.

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