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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.062
 wR factor = 0.184
Data-to-parameter ratio = 6.6

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

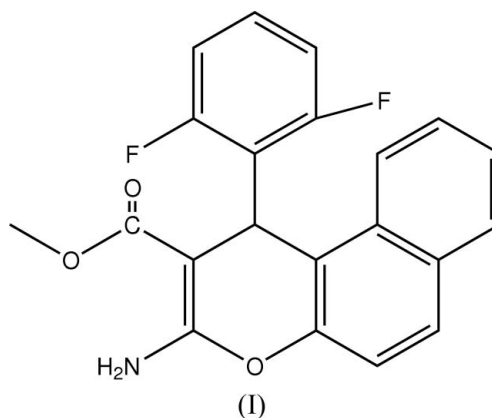
Methyl 2-amino-4-(2,6-difluorophenyl)-4*H*- naphtho[1,2-*b*]chromene-3-carboxylate

The title compound, $\text{C}_{21}\text{H}_{15}\text{F}_2\text{NO}_3$, was synthesized by the reaction of 2-naphthol with methyl cyanocetate and 2,6-difluorobenzaldehyde in methanol under microwave irradiation. The pyran ring adopts a boat conformation. In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$, $\text{C}-\text{H}\cdots\text{F}$ and $\text{C}-\text{H}\cdots\pi$ hydrogen bonds link the molecules into a three-dimensional framework.

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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 *et al.*, 1988) and anticancer activities (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).



In the molecule of (I), all bond lengths and angles are normal. The pyran ring adopts a boat conformation, with atoms O1 and C11 displaced by 0.234 (7) and 0.325 (7) Å, respectively, from the mean plane through atoms C1, C10, C18 and C19. The dihedral angle between the C1/C10/C18/C19 and C12–C17 planes is 88.9 (2)°. An intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond (Fig. 1) influences the orientation of the carboxylate group.

In the crystal structure, intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 1) link the molecules into chains along the [001] direction. The crystal packing is further stabilized by $\text{C}-\text{H}\cdots\text{F}$ hydrogen bonds and $\text{C}-\text{H}\cdots\pi$ interactions involving the C12–C17 benzene ring, which link the chains into a three-dimensional framework.

Experimental

Compound (I) was prepared by the reaction of 2-naphthol (10 mmol) with methyl cyanoacetate (10 mmol) and 2,6-difluorobenzaldehyde (10 mmol) in methanol (3 ml) using piperidine (0.6 mmol) as catalyst under microwave irradiation for 8 min. Pure compound (I) was obtained by recrystallization from methanol (m.p. 475–477 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Crystal data

$C_{21}H_{15}F_2NO_3$
 $M_r = 367.34$
 Monoclinic, Cc
 $a = 13.930$ (3) Å
 $b = 13.499$ (3) Å
 $c = 8.9040$ (18) Å
 $\beta = 96.95$ (3)°
 $V = 1662.0$ (6) Å³

$Z = 4$
 $D_x = 1.468$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 $0.40 \times 0.30 \times 0.30$ mm

Data collection

Enraf-Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.960$, $T_{\max} = 0.967$
 1687 measured reflections

1622 independent reflections
 1422 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\text{max}} = 26.0^\circ$
 3 standard reflections
 every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.184$
 $S = 1.06$
 1622 reflections
 245 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1421P)^2 + 0.538P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.32$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.47$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1A ⁱ ···O3	0.86	2.12	2.707 (6)	125
N1–H1B ⁱ ···O3 ⁱ	0.86	2.15	2.936 (5)	151
C5–H5 ⁱ ···F2 ⁱⁱ	0.93	2.48	3.297 (10)	146
C8–H8 ⁱ ···Cg1 ⁱⁱⁱ	0.93	2.74	3.578 (6)	150

Symmetry codes: (i) $x, -y + 1, z - \frac{1}{2}$; (ii) $x + \frac{1}{2}, y - \frac{1}{2}, z$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$. Cg1 is the centroid of the C12–C17 benzene ring.

All H atoms were placed in idealized positions and refined as riding, with C–H = 0.93–0.98 Å, N–H = 0.86 Å and $U_{\text{iso}}(\text{H}) = 1.2$ or 1.5 times $U_{\text{eq}}(\text{carrier atom})$. In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the final refinement.

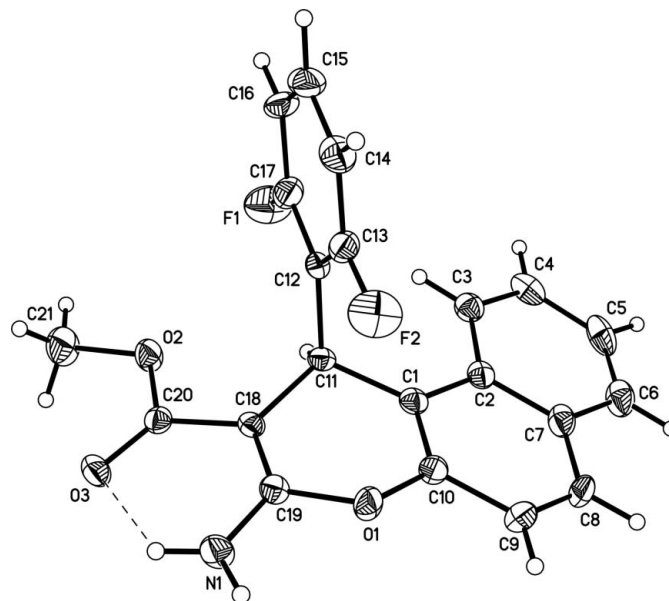


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and the intramolecular hydrogen bond is indicated by a dashed line.

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: *SHELXTL*.

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