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Methyl 2-amino-4-(2-fluorophenyl)-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) = 0.003 Å
R factor = 0.047
wR factor = 0.134
Data-to-parameter ratio = 14.4

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

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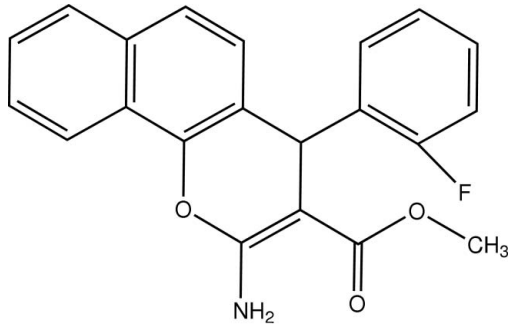
The title compound, $C_{21}H_{16}FNO_3$, was synthesized by the reaction of 1-naphthol with methyl cyanoacetate and 2-fluorobenzaldehyde in methanol under microwave irradiation. Intramolecular N—H···O and C—H···F hydrogen bonds interactions are present in the molecule.

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 anticancer activities (Hyana & Saimoto, 1987) and antisterility (Brooks, 1988). 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), all bond lengths and angles are normal. The pyran ring (Fig. 1) adopts a boat conformation, with atoms C15 and O1 displaced by 0.140 (5) and 0.049 (1) Å, respectively, from the mean plane through atoms C8, C9, C11 and C12. The dihedral angle between the C8/C9/C11/C12 and C16–C21 planes is 89.81 (4)°, similar to the corresponding angle of 82.30 (4)° in the structure that we reported recently (Tang *et al.*, 2006). Intramolecular N—H···O and C—H···F hydrogen bonds are present in the molecule (Table 1 and Fig.1). The former defines the orientation of the carboxylate group.

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (10 mmol) with methyl cyanoacetate (10 mmol) and 2-fluorobenzaldehyde (10 mmol) in methanol (3 ml), using piperidine (0.6 mmol) as catalyst under microwave irradiation for 6 min. Pure compound (I) was

obtained by recrystallization from methanol (m.p. 426–428 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{21}H_{16}FNO_3$
M_r = 349.35
Tetragonal, $P4_2/n$
a = 19.137 (3) Å
c = 9.4170 (19) Å
V = 3448.7 (10) Å^3
Z = 8
D_x = 1.346 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.966, T_{max} = 0.971
6972 measured reflections
3391 independent reflections
2082 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.134
S = 0.99
3391 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.12P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0206 (15)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0B···O2	0.86	2.08	2.694 (2)	128
C15—H15A···F	0.98	2.42	2.869 (2)	107

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_{iso}(H) = 1.2 or 1.5U_{eq}(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: *SHELXTL*.

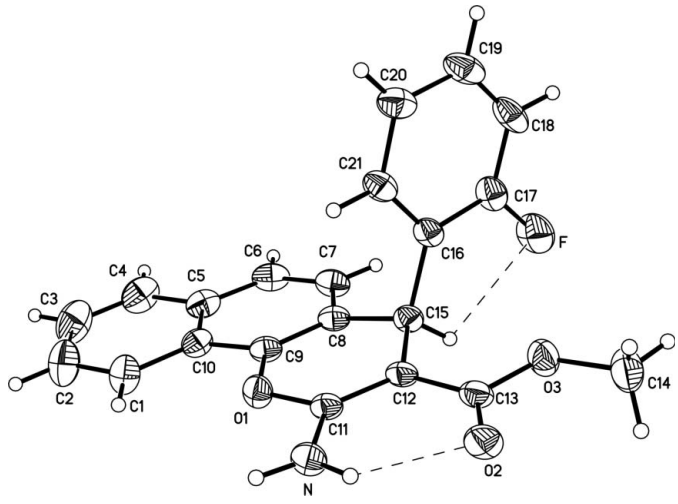


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Intramolecular hydrogen bonds are indicated by dashed lines.

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