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

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Key indicators

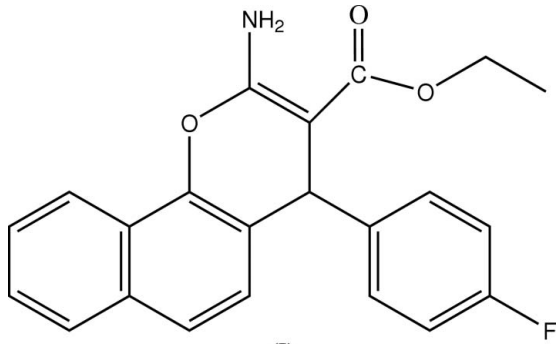
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.181
Data-to-parameter ratio = 14.2

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

The title compound, $C_{22}H_{18}FNO_3$, was synthesized by the reaction of 1-naphthol with ethyl cyanocaetate and 4-fluorobenzaldehyde in ethanol under microwave irradiation. The pyran ring adopts a flattened envelope conformation. The molecular conformation and the crystal structure are stabilized by N—H···O hydrogen bonds.

Comment

Benzopyrans and their derivatives occupy an important place in natural and synthetic organic chemistry because of their biological and pharmacological properties (Morianka & Takahashi, 1977) such as antisterility (Brooks, 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).



(I)

In the molecule of the title compound, (I) (Fig. 1), bond lengths and angles are normal. The pyran ring adopts a flattened envelope conformation, with atom C16 displaced by 0.304 (6) Å from the mean plane through atoms C4, C5, O3, C6 and C15. The dihedral angle between the C4–C5/O3/C6/C15 and C17–C22 planes is 88.02 (9)°. The molecular conformation and the crystal structure are stabilized by N—H···O hydrogen bonds (Table 1).

Experimental

The title compound was prepared by the reaction of 1-naphthol (10 mmol) with ethyl cyanoacetate (10 mmol) and 4-fluorobenzaldehyde (10 mmol) in ethanol (4 ml) using piperidine (0.5 mmol) as catalyst under microwave irradiation for 8 min. The pure compound was obtained by recrystallization from ethanol (m.p. 436–438 K). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{22}H_{18}FNO_3$
M_r = 363.37
Monoclinic, $P2_1/c$
a = 12.634 (3) Å
b = 16.037 (3) Å
c = 8.9890 (18) Å
β = 98.77 (3)°
V = 1800.0 (7) Å^3
Z = 4
D_x = 1.341 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, light brown
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} = 0.967, T_{max} = 0.972
3755 measured reflections
3519 independent reflections
2038 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
S = 0.99
3519 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 0.5068P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.013 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H0B···O2	0.86	2.09	2.700 (3)	127
N—H0A···O2^{i}	0.86	2.26	2.966 (3)	140

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

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 1.5 times U_{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*.

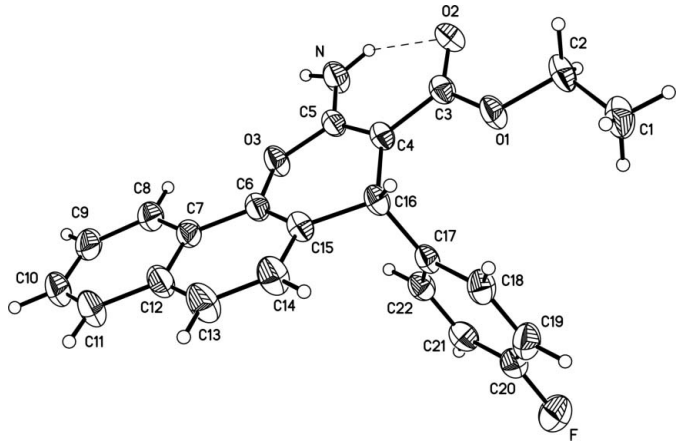


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and the intramolecular hydrogen bond is indicated by a dashed line.

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