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Methyl 2-amino-4-(3-nitrophenyl)-4*H*-benzo[*h*]chromene-3-carboxylate

Shi-Gui Tang,[a] Qing-Gang Tang,[b] Wen-Yuan Wu,[b] Cheng Guo[b]* and Chun-Xiang Ji[b]

[a]College of Life Sciences and Pharmaceutical Sciences, Nanjing University of Technolgy, Xinmofan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China, and [b]Department of Applied Chemistry, College of Science, Nanjing University of Technolgy, Xinmofan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China

Correspondence e-mail: guocheng@njut.edu.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.024
wR factor = 0.077
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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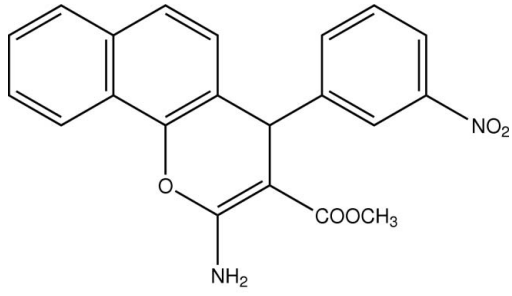
The title compound, $C_{21}H_{16}N_2O_5$, was synthesized by the reaction of 1-naphthol with methyl cyanoacetate and 3-nitrobenzaldehyde in methanol under microwave irradiation. Intermolecular C—H···O hydrogen bonds link the molecules into chains along the *b* axis and adjacent chains are connected *via* N—H···O hydrogen bonds.

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 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).



(I)

In the molecule of (I) (Fig. 1), all bond lengths and angles are normal. An intramolecular N—H···O hydrogen bond (Table 1) determines the orientation of the carboxylate group. The pyran ring adopts a flattened envelope conformation, with atom C11 displaced by 0.151 (2) Å from the mean plane through atoms C1, C10, O1, C18 and C19. The dihedral angle between the C1–C10/O1/C18/C19 and C12–C17 planes is 82.30 (4)°.

In the crystal structure, intermolecular C—H···O hydrogen bonds link the molecules into chains along the *b* axis. Adjacent chains are connected *via* N—H···O hydrogen bonds (Table 1). A C—H···π interaction involving the C12—C17 benzene ring is also observed.

Experimental

Compound (I) was prepared by the reaction of 1-naphthol (5 mmol) with methyl cyanoacetate (5 mmol) and 3-nitrobenzaldehyde

(5 mmol) in methanol (2 ml) by using piperidine (0.5 mmol) as catalyst under microwave irradiation for 8 min. The pure compound (I) was obtained by recrystallization from methanol (m.p. 416–418 K). Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{21}H_{16}N_2O_5$
M_r = 376.36
Triclinic, $P\overline{1}$
a = 8.2190 (16) Å
b = 9.2570 (19) Å
c = 13.203 (3) Å
α = 71.84 (3)°
β = 78.39 (3)°
γ = 82.73 (3)°
V = 932.8 (4) Å^3
Z = 2
D_x = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colourless
0.40 × 0.30 × 0.20 mm

Data collection

Enraf-Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.963, T_{max} = 0.984
3739 measured reflections
3658 independent reflections
2346 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 26.0°
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.077
S = 1.04
3658 reflections
254 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0249P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O5^{i}	0.86	2.22	3.0750 (18)	173
N1—H1B···O2	0.86	2.05	2.6616 (16)	127
C15—H15···O2^{ii}	0.93	2.48	3.373 (2)	161
C21—H21A···O5^{iii}	0.96	2.57	3.504 (2)	165
C21—H21C···$Cg1^{iv}$	0.96	2.83	3.5675 (19)	134

Symmetry codes: (i) $x-1, y+1, z$; (ii) $x, y-1, z$; (iii) $x, y+1, z$; (iv) $-x+1, -y+1, -z+1$. $Cg1$) 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_{iso}(H) = 1.2 or 1.5 times U_{eq}(carrier atom).

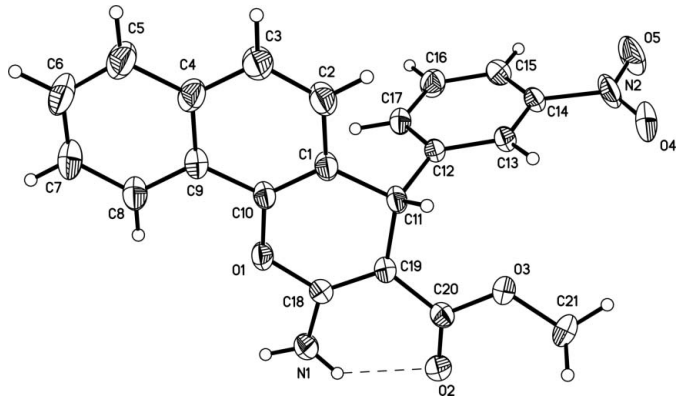


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and the intramolecular hydrogen bond is indicated by a dashed line.

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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